# Mission Oriented R&D and the Advancement of Technology

The Impact of NASA Contributions

CASE FILE

VOLUME TWO

DENVER RESEARCH INSTITUTE UNIVERSITY OF DENVER

## MISSION-ORIENTED R & D AND THE ADVANCEMENT OF TECHNOLOGY: THE IMPACT OF NASA CONTRIBUTIONS

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#### INTRODUCTION

This Volume Contains Descriptions of NASA contributions to the advancement of major developments in twelve selected fields of technology. It is the second volume of a two-volume report presenting the findings of a NASA-sponsored study at the University of Denver Research Institute (NSR 06-004-063). Volume I dealt with an analysis of the nature, character and impact of the NASA contributions which are described in this volume.

The twelve fields of technology that are included in this study include: cryogenics, electrochemical energy conversion and storage, high-temperature ceramics, high-temperature metals, integrated circuits, internal gas dynamics, materials machining and forming, materials joining, microwave systems, nondestructive testing, simulation, and telemetry. These fields were selected on the basis of both NASA and nonserospace interest and activity.

Major developments in each field were identified through interviews with acknowledged leaders in the field. Identification of NASA contributions was limited to a few major developments in each field, selected from a more extensive list of developments in the field. Selection was on the basis of a general consensus among those interviewed, and after discussions with scientists, engineers and specialists in each of the fields.

NASA contributions were identified through interviews with senior NASA technical personnel and analysis of NASA and non-NASA published technical literature. The NASA contributions that were identified in the course of this study cannot be considered a complete listing, i.e., a census, nor representative (in the statistical sense) of what may actually exist. While an attempt was made to construct a reasonable sample of NASA contributions to permit generalizations of certain study results, such results have no statistical reliability. The contributions presented in this study represent only a fraction of the items identified in interviews with NASA personnel and through extensive literature searches. It was impossible in certain cases to obtain sufficient details to be reasonably certain the items qualified as contributions. In other cases, it was apparent that the items, while representing sound technological efforts designed to help achieve NASA's mission, did not serve to advance the development.

While this volume cannot be said to describe all of NASA's contributions to technological advancement and change, it does present a reasonable sample, and does allow qualitative inferences and assessments to be drawn.

Slightly over ten years ago, neither the means nor demand existed for volume liquefaction, transfer and storage of such cryogenic fluids as liquid helium or hydrogen. However, during the past decade a large number of significant technological developments and events occurred. These developments, in turn, advanced the entire field of cryogenic technology. A list of selected significant developments, as identified by technological leaders in the field of cryogenic technology is given in Table 1.

The role of the aerospace industry in helping to bring about these changes and advances in the state of cryogenics technology has been described by Flynn and Birmingham of the Bureau of Standards:

"In the process of meeting its own needs for large quantities of efficient rocket fuels (i.e., liquid hydrogen and oxygen), the aerospace programme has called into being large liquefier facilities (liquid oxygen, hydrogen, helium and flourine); engineering skills (roughly tenfold growth of the pool of trained specialists); development of components on a new scale (tanks, valves, pumps . . .) measurement techniques (for example, flowmeters rated for large rates of flow of cryogenic fluids) design data (a large body of accurate and reliable data on properties of materials and fluids at low temperatures having permanent archival value). The low temperature industry, so armed, now stands available to create new technologies for industry and commerce."\*

Much of the NASA program depends upon cryogenics,\*\* not only for cryopropellants but also for space chamber simulation, purging and pressurizing satellite sensors, and life support systems. In 1965, NASA's annual expenditures for cryogenic fluids alone exceeded \$50 million. At one time, NASA used nearly one-third of the national production of high-purity liquid hydrogen and, in 1968, one half of the helium production. It was the development of volume production tools, techniques, data and personnel for liquid hydrogen technology in the late 1950's and early 1960's which provided the resource base for a similar successful effort by NASA in the early and mid-1960's to develop liquid helium technology from laboratory to production levels.

<sup>\*</sup>Flynn and Birmingham, loc. cit.

<sup>\*\*</sup>Ibid, p. 1.

#### CRYOGENICS (01)

The field of cryogenic technology encompasses the production, maintenance, and application of very low temperatures. The major part of the field is concerned with the liquefaction of gases, and the storage, transport, and use of these liquefied gases. In practice, liquefied gases may be employed as cooling baths for research or industrial purposes as, for example, cooling the walls of a vacuum chamber or for cryoforming metals; or, alternatively, the thermophysical properties of the gases themselves may be used as in scarfing in steel mills, as rocket propellants, or as life support gases in hospitals, underwater, or space environment applications.

Cryogenic fluids are used in tonnage quantities for steel smelting: liquid fueled rocketry; purging and pressurizing a variety of industrial systems; cryopanel pumping and black body simulation for vacuum chambers; chemical plant processes; and in the case of liquefied natural gas, as a primary energy source. These fluids are also necessary in sensing and detection devices; superconductivity research for magnetic field studies, magnetohydrodynamics, fusion power, high-energy power transmission lines, motors and generators; cryosurgery, freeze preservation of food, tissue, blood and organs; cryoforming metals; and in basic physics research. Within the past decade cryogenics has evolved from an esoteric "science of the supercold" of limited interest to industry and a small number of basic researchers into a billion dollar annual market.\* Six cryogenic fluids of principal commercial and aerospace interest (oxygen, nitrogen, helium, hydrogen, argon and liquefied natural gas) had a combined shipment value in 1960 of \$166 million and in 1968 of \$532 million.\*\* Annual growth rate for production of these fluids is estimated at approximately 15 percent. By 1973 the market for these fluids should amount to annual value of \$1 billion, and by 1985 to about \$2.5 billion.\*\*\* These estimates are for the fluids alone and exclude hardware and plant construction.

<sup>\*</sup>T. M. Flynn, B. W. Birmingham, "Cryogenics and National Goals," NBS Institute for Basic Standards, Boulder, Colorado, in Advances in Cryogenic Engineering 14, 1968, p. 3.

<sup>\*\*</sup>Bureau of Census. <u>Current Industrial Reports</u>, Series M28H (60-70)-13, Supplement 2. Washington, D. C.: U. S. Department of Commerce.

<sup>\*\*\*</sup>T. M. Flynn and Collier N. Smith. "Trends in Cyrogenic Fluid Production in the United States." Boulder, Colorado: National Bureau of Standards, Institute for Basic Standards, p. 2. (Preprint)

#### TABLE 1

#### SIGNIFICANT DEVELOPMENTS IN CRYOGENIC TECHNOLOGY\*

- 1. Cryogenic components (01-01)
- 2. Cryoelectronics (01-02)
- 3. Cryometallurgy (01-03) 4. Cryoquenching (01-04)
- **\*\***5. Cryosurgery/cryobiology/cryomedicine (01-05)
- \*\*6. Cryolinsulation (01-06)
- **\*\***7. Josephson Junction (01-07)
- \*\*8. Large-scale cryogenic processing of food and steel (01-08)
- \*\*9. Liquefaction refrigeration, cryogenic components, storage and handling of cryogens (01-09)
- **\*\***10. Liquefied natural gas technology (01-10)
- Quantification/systematization of \*\*11. cryogenics (01-11)
  - 12. Semiconductor computer memory cores and logic circuits (01-12)
- \*\*13. Superconductivity technology (01-13)
  - 14. Tri-element detector (01-14)
  - 15. Waste disposal techniques (01-15)

<sup>\*</sup>Developments selected for identification of NASA contributions.

<sup>\*\*</sup>As identified by technological leaders in the field of cryogenics.

It was, then, necessary to create or further develop high volume hydrogen and helium separation, and liquefaction and pumping systems; cryogenic components, transfer lines; superinsulation; large cryogenic tank storage facilities and technology; thermodynamics, fluid dynamics and other fundamental data; and an extensive network of manufacturers, suppliers, engineers, professional communication channels, and technicians for liquid hydrogen rocketry to provide the resource base for many subsequent industrial technological and scientific cryogenic advances.\*

Significant technological and scientific developments in cryogenics, as identified in Table 1, which have been advanced by NASA contributions, include the following, among others.

#### Cryoinsulation (01-06)

The elaborate efficiency required of thermal insulations in the production, transport, storage, and use of cryogenic fluids is greater than for any other insulation application. It is insulation technology that underlies all of cryogenics.

In his original designs Sir James Dewar explored in the late 19th Century several of the major types of cryoinsulations still in use today: the reflecting-wall, high-vacuum thermos or dewar flask; multiple layer and evacuated powder insulations. Rigid foam and thin supports are two additional basic types or supplements in contemporary use.

The object in insulating cryogens is to reduce or effectively eliminate their intense evaporation by restricting heat paths from the atmosphere and conduction paths along container walls, supports, and the insulation itself. For low-temperature gases such as helium and hydrogen, the wide variety of evacuated multiple-layer reflective shield insulations most effectively perform these functions.

The transport of thermal energy between two regions at different temperatures may involve several different modes of transfer: (1) Energy may be transported by conduction by means of random kinetic collisions of gaseous molecules. If the intervening space between regions at different temperatures is evacuated the transfer rate is therefore reduced appreciably. (2) Conduction may also occur through nonmetallic solids by means of a similar mechanism involving atomic or molecular exchange from regions of high thermal energy to those at lower energy states. In metallic solids it is the free, mobile electrons which

<sup>\*</sup>K. D. Timmerhaus, R. F. Kamm, and J. D. Bays, "Wither Cryogenics," in International Advances in Cryogenic Engineering. New York: Plenum Press, 1965, pp. 1-3.

efficiently transport this thermal energy. (3) Finally, a third transport mechanism is thermal or infrared electromagnetic radiation. Since all bodies at temperatures above absolute zero radiate heat energy continuously, the insulation requirement here would be to provide infrared reflecting surfaces having low emissivities; which, it turns out, are those which are also good conductors such as copper, silver, and aluminum.

A superior very low-temperature insulation system, effectively eliminates gaseous conduction by the simple expedient of evacuating the space between regions of greater and less thermal energy; minimizes the through-paths aiding solid material conduction by means of minimum-contact area spacers; and provides multiple reflective surfaces to reduce radiation losses. Such as insulation is technically termed high performance (HP), multilayer, or superinsulation. Another material in increasing use is polyurethane foam, which is applied directly to the surface of interest, e.g., a cryogenic storage tank, or as a spacer in a laminate of aluminized mylar, aluminum foil or other materials.

The storage, transfer and use of very low-temperature cryogenic fluids would be impossible without today's sophisticated insulations and insulation systems. The large industrial gas manufacturers depend upon the performance of insulations for cost-effective operations. For example, over the road tanktrucks employing high-performance, multiple-layer, reflective-shielding insulation, are able to carry 40 percent more payload because of the reduced volume occupied by multilayer as against traditional powder insulation. In addition, the evaporation loss in such trucks is reduced four times. Modern cryoinsulation is the indispensable requirement for advanced cryogenic applications now and in the future.

In some areas of technology, existing knowledge was more or less adequate to meet the space program's needs. In other areas, it has been necessary for NASA to systematically advance the technology so that the needs could be met. In still other areas of technology, however, NASA requirements greatly exceeded available knowledge, data, equipment and capabilities. Cryoinsulation has been such an area. It has been necessary, for NASA to carry out a large cryoinsulation technology research and development program, covering such areas as: investigating the thermophysical properties of high-performance multilayer and conventional insulations; developing numerous new multilayer insulations; advancing insulation manufacturing fabrication and application technology; determining the thermophysical principles of thermal insulation systems; developing foam insulations, their application, equipment and techniques; developing sophisticated test methods; developing special cryogenic adhesives; and developing analytical models and data.

The cryoinsulation data, literature base and advanced equipment developed by NASA and NASA contractors\* will serve for many years as a major resource base which can be drawn upon by industry to meet advanced insulation requirements. NASA's contributions, then, are largely to advancing the state of technology, and not merely the development of discrete devices or techniques which may or may not be of value to the non-aerospace user, who ultimately will select from this technological mass the components of interest to him. It is not possible, therefore, to now determine the actual identity of potential applications, although the technologies applying advanced cryoinsulation technology will certainly include magnetohydrodynamics, superconducting power transmission and transportation systems, high-energy physics research, home appliances, food freezing and transportation, and basic cryogenic research, among others. Although the actual technological and economic impact of NASA's work in cryoinsulation is estimated as presently moderate, it is potentially high. Included among NASA's contributions to the technology of cryoinsulation are the following.

o Developed cryogenic insulating foams, application techniques and equipment (01-06-01).

For many insulation applications, low-density organic foams offer the advantages of ease of fabrication, relatively low cost, and a self-supporting structure. NASA has expended a considerable effort on analyzing the thermophysical properties of foams, systematically comparing commercial foams, developing new foams, comparing foam application equipment, and developing advanced application and inspection techniques. Foam insulations are applied extensively to the Centaur and Saturn space vehicle's cryogenic fuel tanks.

The Goodyear Aerospace Corporation examined polyurethane foams for thermal conductivity, response to vacuum conditioning, compression characteristics of open versus closed cell types, fabrication methods, permeability analyses of vapor barriers, and outgassing weight losses in a vacuum environment.

In another effort, A. D. Little developed a new plastic foam which adheres strongly to a

<sup>\*</sup>Some of the many state-of-the-art contractors which have participated in these efforts have been: A. D. Little, Inc., Linde, NRC, Lockheed, Boeing, General Dynamics, Martin, Goodyear, Aerojet-General, Bendix, G. E. and Northrup, among others.

metal surface with no cracking even at liquid hydrogen temperatures (-423 F.). The effect of cold shock is attenuated by small quantities of chopped glass fiber added to the urethane based mix combined with isocyanate.

A NASA project resulted in the development and testing of a lightweight polyurethane foam insulation that can be foamed in place on the outside of a cryogenic tank. This foam, which is used on the Saturn space vehicle, requires no strengthening or reinforcing to prevent cracking and splitting upon thermal cycling, and has low thermal conductivity.

Another A. D. Little project resulted in a "Thermal Insulation Systems" handbook which covers the technology of foams, applications, physical properties and thermal conductivity data. Because existing application equipment was not yet sufficiently reliable or versitle, NASA compared commercially available foam applications systems, modified and synthesized as advanced, relatively troublefree system, and developed a reliable preparation, application and surface finishing technique for spraying polyurethane foams.

In an attempt to increase insulation reliability, NASA contracted with North American Rockwell to develop a method for testing rigid, spray-applied, polyurethane foam cryogenic insulation under a realistic simulation of actual use conditions. The new technique employs the usual "dog-bone" tensile coupon on which is sprayed the candidate foam. The laminate is then carefully made defective in a representative manner and subjected to part or whole immersion in the cryogenic fluid of interest while in the novel strain compatibility tension test apparatus. Other reliability-directed efforts have resulted in NASA-developed nondestructive test techniques for analyzing and evaluating pore size, structural integrity, adhesion, cohesion, composition and void detection in cryogenic foams.

This body of advanced technological data and descriptive material is available for the use of manufacturers of refrigerators, cryogenic storage

tanks, natural gas tankers, railroad tank cars, and refrigerated trailer trucks, among others.

o Developed advanced multilayer insulation technology (01-06-02).

Multilayer insulation consists of many layers of alternate, radiation-reflecting shields, separated by low-conductivity spacers. This assembly is placed perpendicular to the flow of heat. Each layer contains a thin (usually 1/4mil.), low-emissivity radiation shield (usually aluminum foil, aluminized mylar, or goldized Kapton) enabling the layer to reflect a large percentage of the radiation it receives from a warmer surface. The radiation shields are separated from each other by nylon, dacron or silk netting, foam, or fiberglass to reduce the heat transferred from shield to shield by solid conduction. The gas in the space between the shields is evacuated to decrease conduction by molecules. Each component of multilayer insulation is therefore designed to perform a specific heat-transfer reducing function: radiation shields to attenuate radiation; spacers to decrease solid conduction; and evacuation to decrease gas conduction.

Research and development programs supported and stimulated by the space program have generated a considerable literature on advanced multilayer insulation designs; thermophysical properties; manufacturing, fabrication and attachment techniques; and test devices, methods and results. Although direct application of a complete space program insulation system to commercial needs is infrequent, components of these systems are both immediately and potentially useful to the non-aerospace community, including: manufacturing, fabrication and attachment techniques; test methods; data, charts, and analyses; etc.

Numerous NASA-supported investigations by A. D. Little have involved increasing the knowledge of the thermal behavior of multilayer insulations and obtaining data on the effects of relevant variables on their performance; developing new high-performance insulation systems; developing a flat-plate calorimeter (see contribution 01-06-08, below) and an emissiometer; and preparing a comprehensive survey on thermal insulation systems.

Under NASA sponsorship, Lockheed Missiles and Space Co. has developed lightweight, strong, low-conducting storage tank supports; carried out comparative analyses of alternate insulation designs and test methods; developed a glass-fiber calorimeter; developed computer programs for thermal analysis of venting cryogenic storage tanks, developed new lightweight superinsulations, one of which is assembled as buttoned, modular panels; and developed the charts, graphs, data, test results and handbooks attendant upon such investigations and designs.

The Boeing Company developed a mathematical model which yields more cost-effective designs. This model can be used to determine the optimum weighting of insulation materials for storage tanks, by treating such parameters as boil-off rate, insulation density, tank surface area, and performance level.

The Linde Division of Union Carbide has developed several high-performance insulations, under NASA sponsorship, and has been involved in investigating their thermophysical properties, and in manufacturing, fabricating and attachment techniques. One such major project resulted in a lightweight, self-evacuated multilayer insulation sealed-panel system. This is a new type of high-performance insulation which is strong, easily applied and removed, and highly efficient because of a new type of polyurethane foam developed especially for these panels. This foam has a uniform cell size and spacing, containing a cryopumping carbon dioxide gas, thus eliminating difficult and time consuming vacuum pumping, leak checking and sealing problems.

The Martin Marietta Corporation developed a technique to reduce boil-off losses in superinsulated cryogenic storage tanks by using the liquid hydrogen vent gas to cool piping penetrations to the tank, and investigated such alternative tank support materials as fiberglass and boron fiber epoxy composites.

The Goodyear Aerospace Corporation performed a comprehensive literature search of interstitial gas characteristics of multilayer insulation systems; and developed an advanced lightweight panel insulation system including fabrication techniques, and an analysis of structural and thermal characteristics.

Other NASA multilayer insulation research and development includes: development of dual seal insulation, a new type of insulation consisting of an inner layer of sealed cell Mylar honeycomb core with an outer helium purge channel of fiberglass reinforced phenolic honeycomb core; thermal conductivity measurements of insulation materials as a function of helium purge gas pressure; data on the effects of leaks and outgassing; comparative analysis of cryogenic valves; heat penetration data; determination of thermophysical properties of typical insulation materials and combinations; analytical investigations of thermal degradation of high-performance insulation in the vicinity of a penetration; investigation of alternative spacer materials; and development of battening and attachment methods.

These comprehensive technological contributions are of interest to manufacturers of industrial gases, cryogenic storage tanks, semi-trailer tank-trucks, railroad tankcars, cryogenic marine tankers, superinsulation and other cryogenic insulations; researchers in basic MHD and particle physics, and large consumers of cryogenic fluids such as steel mills and food processing facilities.

o Improved high-performance insulation manufacturing, fabrication, and application technology (01-06-03).

Although multilayer insulations are available at the inception of the space program in 1958, the two principal commercial types were either too fragile, too variable in performance, or too sensitive to application procedure for sophisticated requirements. Their thermophysical characteristics were incompletely understood, their variety still rudimentary and their manufacturing, fabrication and application techniques still underdeveloped. In addition to developing new insulations, NASA also advanced the processes and techniques necessary to fabricate and apply high-performance insulation systems to a wide variety of cryogenic storage tanks. Although most of the development work was performed in-house, many principal commercial insulation contractors contributed significantly, including Boeing, Linde, National Research Corporation (NRC), and Goodyear Aerospace Corporation.

Boeing developed manufacturing techniques for applying high-performance insulation, including

alternative insulation designs with such materials as silk or nylon netting, rigid polyurethane foam and polyethylene honeycomb spacers, and reflective shields of aluminized polyethylene.

Linde developed a manufacturing and application system for pre-evacuated superinsulation panels (see NASA contribution 01-06-02, above). The Linde effort resulted in the development of fabrication, test, and field repair techniques for these panels. The National Research Corporation contributed to the development of techniques and hardware for insulation wrapping of cryogenic containers, while the Goodyear Aerospace Corporation designed and fabricated the tooling for installation of high-performance insulation on a 105-inch diameter tank.

Other manufacturing technology advanced significantly by NASA include: developing a variety of insulation attachment designs and techniques; evaluating insulation materials and adhesives; developing advanced flexible vacuum jackets; developing techniques to insulate penetrations (e.g., fill and vent lines, instrumentation, tank supports); developing methods for improving insulation evacuation and leak detection; and developing comprehensive manufacturing plans, including battens, support pins, support and assembly fixtures, stepby-step procedures, handling techniques, cutting and patterning techniques, interweaving of the multilayers, etc.

Although much of this technology is immediately useful to manufacturers of multilayer insulation, cryogenic storage tanks, railroad tankcars, semitrailer tanktrucks, primary metals, hospital and food-processing systems, it will find increasing application as commercial, research and power generation needs become more sophisticated in the near and far future.

o Developed cryoinsulation handbooks (01-06-04).

Although numerous NASA contractor reports concerned with cryoinsulation are available to designers and manufacturers of commercial cryoinsulations, they are difficult to use since they are fragmented and directed at immediate space program needs. To overcome this, NASA developed several specialized publications for the non-

aerospace community. A. D. Little prepared a technological guidebook for the non-aerospace user of insulations. This publication (SP-5027) covered such topics as applications, principles, systems designs, materials, structures and other information relative to thermal protection systems at both high and low temperature. Another publication offered brief descriptions of thermal insulation developments, insulating flexible lines, manufacturing techniques for applying cryogenic insulation, multilayer insulation, etc. Lockheed Missiles and Space Company developed a comprehensive handbook, detailing high-performance thermal design criteria for such insulation materials as gases, metals, fiberglass laminates and multilayer insulations, their thermophysical properties, mathematical models, attachment and experimental methods. Information in this form and detail is particularly useful to insulation engineers and manufacturers in such industries as industrial gases, primary metals, petroleum, chemicals, and food.

o Developed advanced cryogenic adhesives (01-06-05).

In many cryogenic applications it is economically more effective to adhesively bond insulation to itself or to a storage tank wall, than to use pins, bands or other arrangements. However, the different thermal expansion coefficients of a tank-adhesive-insulation system create stress-induced problems such as disbonding, crazing, cracking, fatigue and failure at the joints.

NASA has therefore expended considerable effort on advancing, analyzing and developing practical adhesively bonded systems for cryogenic temperatures.

Development of filament-wound glass fiber storage tanks and their strut-supports have also received much NASA-supported research. Because of their high strength-to-weight ratio, these tanks and structural supports are of interest to both the space program and industry. Unfortunately, upon repeated thermal cycling they become porous. The attempted solution has been to adhesively bond a thin, flexible, non-permeable metal liner to the tank walls. Until recently, adhesives did not have sufficient shear and tensile strength to withstand the high axial and hoop stresses, did not have the toughness to resist transport failure, and did not have a contraction coefficient compatible with fiberglass, metal and insulation materials. Now, however, an adhesive developed under contract by the McDonnell

Douglas Corporation, consisting of urethane epoxy resins, meets these specifications. This adhesive has the high strength and modulus of the epoxy, and is not brittle. This adhesive is now a commercially available product.

o Developed the first accurate flat-plate calorimeter (01-06-06).

At the beginning of the space program, cryoinsulation technology was relatively primitive by today's standards. There were few requirements for advanced insulations, little government support, and virtually no commercial support for the expensive research necessary to develop the sophisticated understanding necessary for the development of sophisticated systems as well as the development of the physical insulation systems themselves. NASA contracted with A. D. Little (ADL) in the first of many such ADL-NASA programs to comprehensively investigate space program insulation requirements and the capability of insulation technology to meet those requirements. Since available test devices were often inadequate for precise analyses, ADL engineers developed the first accurate boil-off flat-plate calorimeter, the function of which was to measure the quantity of heat emitted or transferred through an insulation and thus yield a measure of its effectiveness. ADL has sold these commercially, and Lockheed Missiles and Space Company built one for in-house use. This device has played an important role in developing reliable and accurate basic performance data for many advanced insulation systems. It should find use with primary insulation manufacturers, and such advanced consumers as the chemical, petroleum and steel industries, government agencies, and other institutions involved in sophisticated cryogenics technology.

o Provided a major market for cryoinsulation materials (01-06-07).

Insulation is used extensively throughout the space program for both high and low-temperature applications. High-performance, multilayer insulation is used extensively in both ground and flight cryogenic storage tanks, railroad dewars, tanktrucks, transfer lines, in evacuated panels for space vehicles, satellites, command modules and interplanetary probes, in astronaut clothing, in many research

applications, and elsewhere. NASA has been one of the major consumers of high-performance multilayer insulation materials such as single and double aluminized Mylar, goldized Kapton, aluminum foil, Dexiglass, nylon, silk and dacron netting, various foams and other materials. These materials are used extensively in-house and by many NASA contractors such as McDonnell Douglas, Lockheed Missiles and Space Company, General Dynamics and others. Some principal suppliers have been Linde, National Research Corporation, Schjeldahl, Reynolds Aluminum, National Metallizing, and Standard Packaging, among others. The effect of NASA purchases has been to provide early market support for potentially commercial materials, long before the commercial purchasers could support such a market. The net effect has been to make these materials available in a quantity and at a price that would not have been otherwise possible at this early stage of development.

#### Josephson Junction (01-07)

In 1962, Brian D. Josephson, while still a graduate student at the University of Cambridge, predicted that a supercurrent consisting of correlated pairs of electrons could be made to flow across an insulating gap between two superconducting bodies, provided the gap was small enough. He further suggested that this tunneling of electron pairs through an insulator could take two forms, which have come to be known as the Josephson effects. Both forms have been observed in recent experiments.\* The Josephson effect occurs at the junction of a weak electrical contact between two pieces of superconducting metal. Devices based upon this principle are well adapted to handling very small signals with a very low noise level. Many of them have a very wide dynamic range. Often their calibration can be related directly to fundamental constants, making absolute measurements possible. Since the Josephson effect requires superconducting bodies, all devices using the principle must operate at a very low temperature, usually provided by a bath of liquid helium.

Josephson's theory provides an important explanation of the superconducting state; his phenomena in practice generated extremely short wave length electromagnetic radiation of much use to physicists; devices

<sup>\*</sup>Institute of Gas Technology. Proceedings of the First International Conference on LNG - Liquefied Natural Gas. Chicago: Illinois Institute of Technology, April 1968.

based on his discovery can measure very small magnetic fields, and provide very precise voltage references. Most importantly, the Josephson effects enable physicists to measure fundamental physical constants with an accuracy never before achieved.

NASA's role in advancing the use and understanding of Josephson effects has been primarily in funding development of basic Josephson Junction data and devices, such as a better understanding of the characteristics of Josephson oscillation and new methods for fabricating Josephson junctions. The significance of these contributions has been to reduce the time scale in which the advancement of the development of Josephson junction effects occurred, and the actual and potential scientific impact of these contributions are estimated to be high.

o Developing basic Josephson Junction data and devices (01-07-01).

The Josephson effect and its possible applications have been studied by the Institute for Basic Standards at the Boulder, Colorado facility of the National Bureau of Standards (NBS) under contract with the NASA/LeRC. The characteristics of Josephson oscillation were studied; methods for fabricating Josephson junctions devised; and a picovoltmeter and a noise thermometer for the millikelvin range were designed and tested.

Devices developed on the basis of this work performed on Josephson effect include the following, among others: very sensitive magnetometers/null detectors; very fast tunneling cryotron switches employed in computers; very slight potential difference voltmeters; nuclear particle magnetic reasonance detectors; and other very low threshold sensing devices, the most important of which may prove to be an absolute thermometer sensitive to a thousandth of a degree. These devices are of considerable importance to basic researchers in physics, cryogenics and electronics.

Interest at Lewis was in developing fundamental data useful for a cryogenic magnet-ometer for possible use in space shielding applications, and in developing a very small potential difference voltmeter; while the purpose of NBS which jointly funded the effort was to develop the information necessary for extremely sensitive instrumentation for making basic measurements.

### Liquefaction, Refrigeration, Cryogenic Components, Storage and Handling of Cryogens (01-09)

In practice, to liquefy a gas, it is first compressed, which causes its temperature to rise. While under compression this heat is dissipated through a heat exchanger, following which the gas is allowed to expand again but prevented from re-absorbing heat, which results in a cooler gas than it was prior to compression. Repeating the process eventually results in sufficient cooling to produce droplets, and thus, in time, a liquid. The liquefaction, transfer, handling and storage of such gases becomes progressively more elaborate as one descends the boiling point ranges from, say, carbon dioxide at -109 F.through oxygen (-297 F.), nitrogen (-320 F.), hydrogen (-423 F.) to, finally, helium (-452 F.).

Slightly over a decade ago only small-scale laboratory devices existed to liquefy, transfer and store cryogenic hydrogen and helium. The availability, sophistication and efficiency of pumps, valves, seals, control systems, transfer lines, heat exchangers, and technical data base were by today's standards either primitive or non-existent. In response to NASA, DOD, and AEC needs, contractors and suppliers developed the necessary base of cryogenic technology, and trained scientific and technical personnel, which led to a greatly expanded physical plant for the production, storage and handling of cryogenic fluids. Some of the major cryogenic fluids suppliers and systems contractors who were involved in this development are: Linde Division of Union Carbide; Air Products; Air Reduction; Cosmodyne; CVI; Pratt & Whitney; Cryogenic Engineering; and A. D. Little; among others. Some major component manufacturers are: Ingersol-Rand, Worthington & Borg-Warner for pumps; Annin & Hoke for valves; Linde & NRC for systems insulation; CVI & Gardner Cryogenics for transfer lines; and Linde, Beech Aircraft, Air Products, Air Reduction, Chicago Bridge & Iron, and Cosmodyne for large dewars, among other such firms.

Tonnage quantities of liquefied gases are required in steel mills (oxygen, fluorine); purging and pressurizing various research and industrial systems, such as cryopumping and space chamber black body simulation (nitrogen, helium); in the frozen food industry (nitrogen); and in lesser quantities for hospitals (oxygen, nitrogen, helium); welding (helium, oxygen, hydrogen); cryobiology and cryosurgery (nitrogen); cryoforming metals (nitrogen); basic superconductivity and particle research (helium, hydrogen); many laboratory sensors (helium); and on high-altitude commercial and military aircraft (oxygen).

The shipment value of the commonly used industrial gases (oxygen, hydrogen, helium, argon, and nitrogen) was \$344 million in 1960. By 1969 this had grown to \$705 million, and by 1973 it is projected to an annual value of \$1 billion. New capital expenditures for the production of these gases amounted to \$112 million in 1969.

To understand the nature of NASA's contributions to the advancement of the technology of cryogenic liquefaction, handling and storage, it is necessary to have a perspective on the state of cryogenic technology at the inception of the space program in 1958. At that time, large-scale, high-rate, liquid helium or liquid hydrogen liquefaction systems did not exist. Neither the liquefiers, storage facilities, transporters, transfer lines or components were in existence. Valves, seals, burst discs, flow-control devices, liquid-level sensors, adequately insulated pumps, and even simple fittings such as connections, tees, and unions for very low-temperature use, were also nonexistent.

NASA engineers were forced by these circumstances to use existing equipment from industrial gas manufacturers, and to adapt this equipment to their needs. These suppliers would characteristically examine NASA adapted and modified equipment, and then design and build a marketable version. One or more of the new designs would then be sold to NASA, following which the equipment would often be modified again to meet evolving NASA program requirements, which in turn again inspired a finished, marketable product. In this way, a continuous evolutionary development took place not only of cryogenic systems but of contractor personnel and plant capability.

NASA's evolutionary contributions to the technology of cryogenic liquefaction, handling and storage, have resulted in advancing the production levels of cryogenic fluids such as hydrogen, fluorine and helium from either very low or laboratory scale to tonnage volumes. These contributions include: (1) developing and improving technology for designing and constructing large-scale liquefiers, transporters, dewars, transfer lines and control systems; (2) developing and compiling data and specifications for safe handling and storage; (3) aiding in the development of an extensive network of trained personnel, contractors and suppliers; and (4) aiding in the growth and development of a wide-spread market for cryogenic fluids and components.\*

The collective significance of these contributions has been to greatly increase the sophistication, efficiency, and widespread availability of cryogenic technology, and to have brought about this advancement earlier than it would normally have occurred. The economic and technological impact of the collective contributions is estimated to be moderately high to high.

<sup>\*</sup>NASA supported the development of liquid helium technology from laboratory to commercial production and, as recently as 1968, used one-half of the national production of liquid helium. At present, it is estimated that the Government, directly or indirectly, accounts for over 90 percent of the total helium volume. NASA has also provided a significant, though not dominant, market for liquid oxygen (steel mills consume 70 percent of the national capacity) and other cryogenic fluids such as fluorine, neon and nitrogen.

o Development of specifications for triple-jacketed, no-loss, liquid-fluorine dewars (01-09-01).

As the most reactive element known, fluorine is both exceptionally useful and exceedingly difficult to store, transport and use. NASA uses considerable quantities of liquefied fluorine as a fuel oxidizer and therefore required a cryogenic dewar to contain the reactive liquid. Since no such suitable container existed, NASA personnel developed a design specification, for a triple-jacketed dewar containing no vent whatever. The inner jacket contains the liquid fluorine, the middle jacket contains liquefied nitrogen, and the outer jacket is evacuated. This no-loss design is now commonly employed by users of liquid fluorine such as Allied Chemical, Mathieson Chemical, Harshaw Chemical, Rocketdyne, McDonnell Douglas and General Dynamics. It would also be useful to and is probably already being applied in the steel industry.

o Development of specifications for first large-capacity, liquid-hydrogen tankcars (01-09-02).

Although liquid hydrogen is routinely shipped by tankcar throughout the United States today, large-capacity, liquid-hydrogen tankcars did not exist slightly over ten years ago. Since NASA had a requirement for such a tankcar, space agency personnel developed a specifications document for a 30,000-gallon, liquid-hydrogen tankcar with its own controlled venting system for the Linde Company. In order to respond to the growing NASA and Air Force markets, Linde, using their own funds, built six such cars in 1962. These cars are still in use, now serving commercial as well as space and military markets.

o Reduced fabrication costs of transportable liquid hydrogen dewars (01-09-03).

At the inception of the space program in 1958, the only moderately high-volume customers for liquid hydrogen were the Air Force and the AEC. NASA's needs for production, storage, transportation, and use, introduced a scale factor which could not be satisfied using existing technology. To meet immediate requirements NASA borrowed two 6,000-gallon, liquid hydrogen dewars (and other equipment) from the Air Force, each one costing \$90,000. In

practice these dewars proved to be inadequate due to high heat leaks (5-6 percent/day) and valve freezing. To overcome this, NASA, with the H. L. Johnson Company, drew up completely new designs and specifications. Changes involved: increasing inner-outer shell spacing; adding dried perlite purged with helium; redesigning supports and piping for maximum heat path; designing an evacuated man-hole lid system (a system used widely today); and replacing the high heat-leak, 6-inch valve system with smaller, bayonet-jointed, vacuum-jacketed piping with close male-female spacing containing a stationary gas film for insulation. The H. L. Johnson Company then built (1959-60) two such roadable (tanktruck) dewars for \$60,000 each. These dewars have very low heat-leaks (1-2 percent/day) and are still in use today. They were, in effect, pilot models for subsequent tanktruck designs. These design advances increased the efficiency and lowered the manufacturing cost of such dewars, now used throughout the United States to supply research facilities, steel mills, food hydrogenation facilities, and research facilities.

o Required industry to significantly improve transfer line technology (01-09-04).

Transferring cryogenic fluids from a source of supply (a dewar) to a point of use (a launch vehicle) is more of a problem than simply connecting the two with piping: (1) Transfer lines must be insulated for the particular liquefied gas they are to carry, and have no significant conduction points to the atmosphere. Otherwise, cold fluid pumped in at one end will come out as high-pressure gas at the other. (2) Materials of construction used in the transfer lines must be chemically and thermophysically compatible with the fluid. If not, contamination of the fluid may occur, or cracking of the material. (3) Welds must be of compatible materials so that expansion coefficients do not vary too much; welds must be of exceptionally high quality and flaw-free to withstand thermal cycling. (4) Cleanliness is necessary for all fluids and mandatory for explosive liquid oxygen. (5) Porosity control is crucial for helium, which can migrate through solid glass, and hydrogen, which burns with an intense, colorless and therefore hard to detect flame.

All of these problems plagued early space program personnel and transfer line contractors. The difficulty was that no customer had ever before had such requirements for high volume flow, with long distances between connections, and with high degrees of reliability, and cleanliness. NASA personnel therefore had to both demand and teach their suppliers (i.e., actually providing technical assistance in their plants) welding techniques, cleaning techniques, and quality control procedures none had experience before. For example, new assembly techniques had to be developed for transfer lines and x-ray inspections were made mandatory. These across-the-board requirements provided the base under which today's sophisticated transfer line technology was brought into existence.

o Development of competitive suppliers of transfer lines (01-09-05).

Cryogenic transfer line technology at the inception of the space program was rudimentary, with few manufacturing sources (see NASA contribution 01-09-05, above). NASA and the Air Force requirements for high flow-rates of extremely low-temperature fluids, under stringent reliability conditions, brought into being an entire new area of technological development in the field of cryogenics: the technology of transfer lines. With the advent of this new development, the need also developed for manufacturers, suppliers, designers and test personnel. As a major customer for these lines, NASA provided important, early support for this burgeoning market.

For example, Linde built a 20-inch diameter 8,400-foot transfer line at Edwards Air Force Base for NASA; Pratt & Whitney built a 3-inch diameter 3,000-foot line at West Palm Beach; every dewar manufactured for NASA also has at least one transfer line (see NASA contributions 01-09-01, 01-09-02, and 01-09-03, above, and 01-09-06, below); and all the rocket engine manufacturers supplying NASA have similar such facilities. NASA/LeRC alone has purchased in excess of \$250,000 worth of such lines.

Some of the suppliers to NASA have been Gardner Cryogenics, H. L. Johnson (later Cryovac), Process Engineering, Ross Construction, Vacuum Barrier and many other firms newly created or with added capabilities to design, test, manufacture and distribute transfer lines. Such lines are used by hospitals, steel mills, research facilities, food processors, chemical and petroleum manufacturers, and widely throughout industry and primary industrial gas manufacturers.

o Provided a major market for large cryogenic fluid dewars (01-09-06).

Every NASA test and launch facility requires high-volume use of such cryogenic fluids as liquid hydrogen, liquid oxygen, liquid helium, liquid nitrogen, and to a lesser extent, liquid neon and liquid fluorine. To handle these liquids, the various NASA installations required and purchased a wide variety of types and sizes of storage dewars, most of which, at the time of construction, were both qualitatively and quantitatively unique. Some of these major purchases include, among others: (1) four 34,000-gallon liquid hydrogen (LH2) railcars built by Linde which transport LH2 as well as act as storage dewars when parked on a siding, with one tankcar serving as a source, and the other as sink for recycled gas; (2) three 1,000-gallon liquid helium (LHe) dewars built by Cryogenics Engineering; (3) a 6,000-gallon, 6-inch valve, recirculating research dewar built by Beechcraft; (4) three 7,000-liter LHe dewars built by Linde; (5) a 200,000gallon LH2 dewar built by Chicago Bridge and Iron and Linde, which probably has the lowest heat leak rate in existence (i.e., less than one percent per year); (6) two 250,000-gallon LH2 dewars built at Aerojet General; (7) an 850,000-gallon LH2 dewars built at the LH2 supplier's facilities (Air Products, Linde, etc.). Many of these dewars required long and laborious reworking and adaptation, calling for continuous collaboration between NASA experts and their counterparts in industry. The results of these interactions were important advances in storage dewar technology. As the major market for many such special dewars, NASA both generated and helped stimulate this technology. Dewars of these capacities and types are used in steelmaking, basic research, hospitals, primary industrial gas manufacturing, and elsewhere.

o Accelerated development of large-scale helium liquefaction technology (01-09-07).

Prior to the space program (1958), the only significant use of large quantities of helium was the Navy requirement for filling dirigibles. To reduce shipment costs and increase stand-by supplies, the Navy constructed a 56 liter/hour helium liquefier. This liquefier was expected to reduce the volume of transported helium by 700 times in the liquefaction process, i.e., tanktrucks should then carry 700 times more helium as a liquid than they could as a gas. Unfortunately, the system was faulty and produced mostly a chilled gas with over 50 percent losses, which delayed its acceptance by the Navy. Since the space program badly needed liquid helium (LHe), NASA in collaboration with the National Bureau of Standards, reworked the system so that it could produce liquid helium.

The capacity of this system was soon inadequate for NASA's needs and Linde was contracted to develop, design, construct and deliver a turn-key system which would produce and store 100 liters/hour. At the time of construction, this was the world's largest helium liquefaction system. Shortly after this construction, Linde was again contracted to build a twin system. The numerous design innovations incorporated in this system advanced subsequent large-scale helium liquefaction technology.\* These contracts increased the scale of this technology by a factor of 15, since ordinary liquefiers were at that time producing only 8 liters/hour. Helium is now routinely separated from natural gas and liquefied by many American and European firms, some of which are Phillips Petroleum, Air Products, Kerr-McGee, Linde, Kansas Refined Helium, AIRCO, Gardner Cryogenics and others.

<sup>\*</sup>Since this was a costly R&D program leading to only a limited commercial market potential, probably no private organization would have undertaken it. Therefore, this large-scale system would probably not have been developed were it not for NASA's needs.

o Reduced cost of liquid helium (01-09-08).

Although liquid helium could be purchased at the beginning of the space program, it was only available in small quantities. Since dewar technology was not yet advanced enough to make shipments cost effective, it was also only available at the manufacturer's gate, i.e., the customer had to absorb the cost of the very high boil-off loss in transit between the time and place of purchase and his point of use. The cost for liquid helium in 1960 was about \$12/liter; today, liquid helium costs less than \$1/liter, delivered to the user. NASA's early, high-level efforts to develop practical production; storage and delivery techniques, advanced the technology to the point where unit costs were reduced substantially and led to subsequent efforts by industry which today permits routine manufacture and use of liquid helium at moderate cost. (See NASA contribution 01-09-07, above.)

o Aided in development of industrial capability for large-scale commercial production of liquid helium (01-09-09).

NASA's support of programs at Linde to develop two helium liquefaction systems, each 15 times larger than existing technology at the time permitted, resulted in the world's largest such liquefiers and storage dewars for helium. (See NASA contributions 01-09-07 and 01-09-08, above.) Since this involved considerable technological risk on the part of the contractor, NASA allowed a complete no-risk systems definition, and helped Linde develop the capability to manufacture such a system. Following successful completion of the contracted work for NASA, and based upon what had been learned, Linde built its own commercial facility for large-volume production of liquefied helium. The net result of NASA having reduced the risk was to aid Linde in developing its own capability for large-scale commercial production of helium.

o Developed first comprehensive liquid hydrogen safety manual (01-09-10).

The practical aspects of large-scale, low-temperature technology was at the inception of the space program rudimentary. Among the missing and

needed pieces for day-to-day engineering and operation was a hydrogen safety manual. Hydrogen gas is colorless and odorless, and therefore, impossible to detect by the senses; it can cause suffocation by displacing air; when mixed with air or oxygen it is explosively reactive with very little energy input (one-tenth that of a gasoline-air mixture); and burns with a colorless flame. Because of these problems, NASA convened an advisory panel on experimental fluids and gases to review the information required and prepare a comprehensive hydrogen safety manual. This document, the first of its kind, has received wide distribution throughout industry, government, and research facilities. It contains information on the hazards involved, design principles for gas detectors, buildings and chambers, dewars, materials compatibility, fittings and seals, fabrication methods for joints, contamination control, elimination of ignition sources, protection of personnel and equipment, blast effects, operating and emergency procedures, and much else. It answers a basic need for all users of hydrogen.

o Provided major market for cryogenic fluids (01-09-11).

The volumes of cryogenic fluids involved in space program operations are extremely large (see NASA contributions 01-09-01, 01-09-02, 01-09-03, and 01-09-06, above). As an example, each Atlas-Centaur satellite launch vehicle requires 2-1/2 tons of liquid hydrogen and 2,155 tons of liquid oxygen. Meeting the daily requirements to support development of the Saturn required 22 trailer loads of liquid hydrogen, 76 trailer loads of liquid oxygen and 26 trailer lcads of liquid nitrogen. Normal day-to-day research and maintenance operations also consume considerable quantities of these and other gases. As an example of industry response to space program needs, the following liquid hydrogen plants were constructed in addition to the plants at West Palm Beach, Florida which were originally built for the Air Force: a 6-ton/day plant in Torrance, Calif.; a 30-ton/day plant at Ontario, Calif.; a 30-ton/day plant at Long Beach, Calif.; a 60-ton/day plant at Sacramento, Calif.; and a 30-ton/day plant at New Orleans, La. With

decreased NASA requirements, these plants today produce liquefied gases for commercial customers, such as wire drawing operations in steel mills, hydrogenation of food, and basic research, among others. This cryogenic fluids capacity is directly attributable to space program needs and would not have otherwise been developed at the present scale and sophistication of operations. Among major suppliers to NASA have been AIRCO, Air Products, Linde and other such industrial gas producers.

o Developed systems technology and demonstrated feasibility of large-volume shipments of liquid helium by air (01-09-12).

Because the liquefaction of helium was still a laboratory process at the beginning of the space program, NASA's growing needs were met by Linde who designed, developed, and constructed the world's largest helium liquefaction system at the Lewis Research Center (see NASA contribution 01-09-07, above). Since large volumes of LHe were also needed as a purge and pre-chill gas for the Centaur missile at Cape Kennedy, it was necessary to develop air-transportable dewars. NASA borrowed a 5.000-gallon semi-trailer roadable dewar from the Air Force which was originally designed for liquid hydrogen (-423 F.). This dewar was completely redesigned, disassembled, and reworked for the lower temperature liquid helium (-452 F.). Representatives from the Linde Company, working with NASA cryogenics experts, drew up new specifications to satisfy the requirements of the Department of Transportation, Bureau of Explosives, Federal Aviation Agency, International Air Transport Association and the American Society of Mechanical Engineers, as well as NASA and Air Force constraints.

Leaks had to be located and repaired, walls reinforced, inner and outer jackets and supports redesigned, liquid-level gages redesigned, prechill cool down procedures developed, a document of operational procedures and safety regulations prepared, and a vent system with special pressure relief valves developed. NASA later contracted with Cryogenics Engineering Company to design, develop and build three additional 1,000-gallon air-shippable LHe dewars for the Centaur launch operations. The lightweight dewar patent and

design information developed on this project is now used by the contractor in his commercial sales. Liquid helium is now routinely shipped in 1,000-gallon dewars across the United States and overseas to Europe in CL-44's, DC-8's and Boeing 707's.

o Reduced cost of liquid hydrogen (01-09-13).

The components, devices, liquefaction, storage and transport systems that were developed in response to space program needs, and the economies of scale due to large-scale NASA purchases, had the effect of lowering the price of liquid hydrogen from about \$12/pound in 1956-1958 to \$0.25/pound in 1968. Today, because of the reduced NASA market, liquid hydrogen sells for slightly over \$1/pound. (See NASA contribution 01-09-11, above, for details on the massive use of cryogenic fluids by NASA, particularly liquid hydrogen.) Common availability, a network of suppliers and distributors and considerable price reduction of liquid hydrogen represent significant technological and economic consequences of space program requirements.

o Development of a boil-off reliquefier (01-09-14).

To maintain safe working pressures in the various liquid helium storage systems employed for research and space vehicle use, considerable quantities of boil-off gas must be vented to the atmosphere. To meet this requirement, A. D. Little developed a completely self-contained and portable boil-off gas recondensing system. The semi-trailer mounted system includes two high-pressure recovery vessels for the boil-off, pumped in with the two liquefier compressors, a make-up gasholder, liquefier, an intermediate 250 liter storage dewar and associated controls and instrumentation. savings at Cape Kennedy alone are estimated to amount to \$73,000 a year. This system would be valuable to other volume users of liquid helium. such as research facilities, in superconductivity applications, and large welding operations.

o Development of liquid oxygen - compatible laminated gasket composite (01-09-15).

Because liquid oxygen (LOX) cannot be permitted to contact most sealing materials, the seals most often used are variants on the chemically neutral fluorocarbons. Unfortunately, however, these materials cold flow when under compressive stress. which soon permits leakage of LOX with the attendant possibility of catastrophic explosion. Various fillers had been molded together with the fluorocarbons in an attempt to control cold flow, but without adequate success. To overcome this, the Whittaker Corporation, under a NASA contract, developed a LOX compatible seal of a glass fabric laminated structure in which the glass fiber bundle is not completely saturated with fluorocarbon polymer binder. This permits mechanical compressibility but has little cold flow at cryogenic temperatures. Various configurations using the flat seal concept were developed for O-rings, chevron seals, lip seals, ball seals, diaphragm and flexible tubing. This work would be of interest to producers and users of liquid oxygen, such as industrial gas manufacturers, steel mills and hospitals.

o Development of a liquid cryogenic lubricant (01-09-16).

Although many attempts have been made to develop reliable bearing lubricants for short-term use in cryogenic systems, none have really been adequate. The added requirement for long-term trouble-free bearing operation both in aerospace and commercial applications compounds the problem. NASA requirements called for reliable, trouble-free bearings in high-speed rocket turbopumps to run for several minutes, and cooling-system pumps to run with moderate speeds for several hours. To meet these requirements, a fluorinated polyether lubricant which remains fluid at cryogenic temperatures was developed at NASA/LeRC. Until this development, a dry lubricant film was employed which provided only boundary lubrication and thus failed to protect rolling elements or the bearing races, which required rapid replacement and often led to premature retainer failure and thus catastrophic bearing failure. Alternatively, the lubricant developed by space program personnel remains liquid at cryogenic temperatures, will not evaporate at operating temperatures, is chemically inert, will

not absorb water, has good heat transfer properties, and maintains a lubricating fluid film in the high-pressure region of a rolling element contact. This lubricant significantly extends bearing life and reliability, and could therefore be reliable in gas liquefaction plants and frozen food processing industries.

#### Quantification and Systematization of Cryogenics (01-11)

Prior to this nation's development of a major space effort, cryogenic activity was more an empirical art than a field of technology. Even today the field is unaffiliated with any major professional technical societies.

Quantification of cryogenic technology and systematizing it in data banks for widespread dissemination has been advanced by NASA and Air Force needs in missile and life support programs, by industry in response to growing cryogenics markets, and by the National Bureau of Standards seeking to understand very low-temperature physical phenomena.

Because of Air Force, NASA, industry, academic and National Bureau of Standards requirements, the annual Cryogenic Engineering Conferences now serve as the principal professional focus. These conferences are affiliated with the Division of Engineering, National Academy of Sciences, and are held annually on a different university campus. The proceedings of these conferences have been published with NASA and Air Force assistance. Besides these government and industry supported dissemination activities, fundamental research is carried out by NBS, private industry, and in government research laboratories, all directed at creating the broad data base required of a burgeoning and complex discipline, principally in low-temperature thermodynamics, particle mechanics, superconductivity, low-temperature liquefiers, refrigerators, and cryostats, as well as more mundane real-world hardware and systems problems in, for example, the rapidly growing liquefied natural gas industry.

A scientific data base and understanding of complex thermodynamic phenomena is an important ingredient in developing economically and socially important applications of cryogenics, such as massive power generation and transmission, medical cryogenics, high-energy physics, cryobiology, cryogenic electronics, magnetically suspended trains, and cryogenically propelled automobiles and aircraft, among others. As one expert expressed it: "I believe cryogenics will be to the second half of the 20th century what high-temperature processing was to the first half.\*

<sup>\*</sup> R. W. Temple, staff consultant, Arthur D. Little.

Since space program requirements in cryogenics far exceeded the available technology, NASA placed considerable effort into developing fundamental data, compiling and integrating existing but scattered information, and disseminating the resultant handbooks, manuals, charts, graphs, and bibliographies. These efforts included the support and development of: ortho-para hydrogen data, materials and fluids properties handbooks and manuals, considerable basic research by the National Bureau of Standards, dielectric properties of cryogenic materials, and the inauguration of an Aerospace Safety and Data Institute. These efforts have extended fundamental cryogenic knowledge and provided a data base from which present and potential research and technological development can proceed with confidence. The scientific, technological and economic impact of these collective contributions is estimated to be moderately high. NASA contributions include the following, among others.

o Development of fundamental ortho-para hydrogen data (01-11-01).

Ordinary liquid hydrogen exists in two molecular states, ortho and para, having opposed, anti-parellel electron spin. When liquid hydrogen converts naturally from the ortho state to the para state, heat is evolved. If left uncontrolled, this exothermic reaction creates boil-off rates sufficient to reduce volume by 70 percent. The ortho-para hydrogen conversion was a serious technical and economic problem to early industrial gas producers and their customers.

The National Bureau of Standards (NBS) subsequently developed a conversion catalyst yielding better than 95 percent parahydrogen, the properties of which were then investigated by NBS under NASA contract. The thermophysical properties data thus developed have been widely disseminated to industry, commerce, and research facilities. Some of the properties investigated include: equilibrium at normal boiling point; explosive limits in oxygen and air; heat or vaporization; heat of conversion ortho to para; and the velocity of sound in liquid hydrogen. These investigations represent an important addition to fundamental knowledge of cryogenic phenomena and is indispensable to users of liquid hydrogen, such as the food processing industry, steel mills, and in basic research.

o Development of the Aerospace Safety and Data Institute (01-11-02).

In the aftermath of the fatal January 27. 1967 Apollo fire, intensive investigations were initiated by NASA to review all safety programs. One important consequence of this effort was the development of the Aerospace Safety Research and Data Institute (ASRDI), the objectives of which are: to identify areas lacking appropriate technology to deal with safety problems and to initiate research to develop this technology; to author and compile state-of-the-art and summary publications; and to establish and operate a safety data bank covering all types of safety problems encountered in aerospace work such as: mechanics of structural failure, non-machine hazards, fire and explosion, propellants, fuels, lubricants, hydraulic fluids safety, cryogenic fluids safety, aircraft problems, structural materials limitations, industrial radiation, industrial safety manuals, codes and specifications, and more. Of particular interest are the ongoing research programs dealing with the interrelationships of systems handling, transportation and storage of cryogenic fluids and a major program to review oxygen safety technology and to develop new technology and data were necessary (equipment, standards, data, criteria). The data bank is available to industry as well as government agencies. ASRDI personnel are members of numerous safety committees and represent a variety of technical and scientific disciplines. The information compiled, developed and integrated is of considerable utility to all industrial gas manufacturers and users, research facilities, steel mills, hospitals, airlines, and throughout industry where safety is a problem.

o Preparation of aluminum alloy materials data handbooks (01-11-03).

Technical data on a group of aluminum alloys of interest as cryogenic materials of construction was developed by the Syracuse University Research Institute, under a NASA contract, and presented in a series of materials data handbooks. This comprehensive materials property information on aluminum alloys 2014, 2219, 5456, 6061 and 7075 was compiled from the Battelle Memorial Institute Defense Metals Information Center, and NASA documents and personnel, among many other sources.

This complete materials property information includes physical and mechanical property data at cryogenic, ambient and elevated temperatures, and is supplemented with useful information concerning materials procurement, metallurgy of the alloy, corrosion, environmental effects, fabrication and joining techniques, design data, and considerable reference information.

Handbooks of this thorough comprehensiveness are quite valuable to the engineer, and can represent considerable cost-savings for an industrial organization. They are, therefore, useful wherever aluminum alloys are employed.

o Preparation of a manual on the thermal expansion properties of common aerospace materials (01-11-04).

Because the relationship between thermal expansion and temperature of many common materials such as aluminum, copper, Teflon, silica, glass, and steel is nonlinear, design engineers cannot use known thermal expansion coefficients for large temperature changes. To provide useful data, experimentally derived thermal expansion curves for many common construction materials were developed by North American Aviation under a NASA contract and presented in a design manual. These data overcome the basic inaccuracy inherent in calculating coefficients to determine changes in length due to changes in temperature. This basic information is of use to engineers with design problems that must accommodate large temperature differentials, such as in automotive, ship and aircraft engine designs, and in power plants, fuel cells, steel mills, chemical plants, industrial gas plants, and cryogenic systems.

o Preparation of low-temperature mechanical properties manual (01-11-05).

The use of materials at low-temperatures depends upon the strength of the material and its ability to resist brittle failure caused by stress concentrations, multiaxial stresses, and impact. The ability of the material to resist brittle failure can be estimated from tests such as percent elongation, notch tensile ratio, and Charpy V-notch impact strength. To meet these data needs, a manual on the low-temperature mechanical properties of several important materials of construction was prepared by North American Rockwell Corporation under a NASA contract.

Included were data from the above-mentioned tests, for aluminum, magnesium, titanium, copper and various steel alloys. Data were developed from room temperature to liquid hydrogen temperatures. These data are useful in cryogenic engineering for selection and use of materials of construction and would be of interest to engineers in industrial gas manufacturing facilities, chemical and petroleum facilities, and to manufacturers of cryogenic components and systems.

o Determination of the fatigue properties of sheet, bar, and cast metallic materials for cryogenic applications (01-11-06).

The cryogenic fatigue and tensile properties of metallic materials of construction in sheet, wrought bar, and cast bar form were experimentally determined by the North American Rockwell, under contract to NASA, at room temperature, liquid nitrogen temperature (-320 F.), and at liquid hydrogen temperature (-423 F.). The results of these experiments were presented as stress versus number of cycles to failure and should find use in all cryogenic engineering design problems.

This effort is similar to NASA contributions 01-11-03, 01-11-04, and 01-11-05, described above, in that it is one part of an extensive series of NASA-sponsored projects to develop, compile and integrate basic cryogenic data useful to technologists and scientists concerned with low temperatures.

o Assistance in developing, compiling, integrating and disseminating basic cryogenics properties data (01-11-07).

The Cryogenic Division Laboratory and the Cryogenic Data Center of the National Bureau of Standards (NBS) are the nation's principal cryogenics research and information facilities. Since 1961, NASA has contributed support for both the Laboratory and the Center in developing, compiling, integrating and disseminating basic materials and cryogens properties data for use by academic institutions and researchers in industry and other government agencies. The Laboratory furnishes advisory, coordinating and consulting services on low-temperature phenomena

to persons involved in programs of materials research, measurement methodology and cryogenic engineering. The Data Center collects cryogenic literature and data worldwide, and evaluates, compiles, integrates and disseminates this information to users. Of the 69 projects currently underway at the Laboratory, 18 are funded by NASA. NASA support was also significant in the early development of the Laboratory in fundamental efforts to economically liquefy hydrogen, and in the determination and compilation of thermophysical properties data for oxygen, argon, helium, hydrogen, methane, air and other cryogens. This major scientific and technological information function has been supplemented by research on materials properties at cryogenic (and other) temperatures. For example, under NASA sponsorship NBS prepared data array and literature on the electrical resistivity behavior of 53 metallic elements in the cryogenic domain; a comprehensive document on the thermal conductivity of metals, alloys, and nonmetallic solids at cryogenic temperatures; a technique for predicting the thermal expansion coefficients of cryogenic metallic alloys; and cryogenic thermocouple calibration tables.

o Advanced understanding of cryogenic fluid behavior during transfer (01-11-08).

The transfer of cryogenic fluids from or to a dewar through transfer lines generates complex thermodynamic transients which often result in tank implosion, transfer line rupture or other catastrophic failures. A continuing series of research projects have therefore been supported by NASA directed at understanding and controlling the sophisticated fluid mechanics involved.

One such study by the National Bureau of Standards investigated the cool-down mechanics of transfer lines and resulted in an analytical relationship between fluid and line enthalpy, density, and velocity of sound in the warm gas which yields an estimate of the cool-down time to be expected.

A separate study by the Boeing Company involved a subscale experimental investigation of the fundamental mechanics or geysering,

which is the sudden expulsion of cryogenic fluid from a heated transfer line. The heat can derive from transfer through a wall or from the external environment. Geysering can cool and condense the source tank ullage\* gas, resulting in tank implosion, and severe impact loads resulting from the rapid refilling of the line. Boeing personnel derived a quantitative relationship between average heating rate, reservoir temperature and geysering period; documented their observations and analyses of the phenomenon; and identified areas requiring future study.

Another investigation by the Lockheed Missiles and Space Company involved an exhaustive study of cryogenic tank thermodynamics using laboratory model analyses derived from empirical data fed to a digital computer. Based on the computer printout, model systems were manipulated for both transfer line and receiver tank reactions to programmed parameters of flow, pressure, fluid phase, hardware configuration; and reaction of tank pressure in early transfer phase to the average size of liquid droplets in the liquid/vapor jet entering the tank (which was found to be the critical mechanism determining tank implosion).

These NASA-supported studies on fluid and materials behavior are particularly useful in large storage tank construction, tank farms where volume fluid handling is involved, fuel and supply tanks on ships and trains, oil and liquids long-line transmission, bulk handling facilities on barges, and fluid handling and treating in brewing, chemical and petroleum facilities.

o Determined feasibility of compact systems for a cryogenic capacitor (01-11-09).

Cryogenic capacitors represent a new development in cryoelectronics, holding considerable potential for applications in computers, masers, basic research and many other areas. These would be far smaller and more powerful than existing types, and would therefore be more cost-effective. To carry this further, the dielectric properties of three classes of promising

<sup>\*</sup>Ullage is the vapor space above the liquid cryogen in a storage tank, normally about 10 percent of its volume.

materials were investigated by General Electric under contract to NASA, for possible application as cryogenic dielectrics in capacitors: (1) inorganic bonded ferroelectric materials; (2) anodic coatings on metal foils; and (3) polar low temperature liquids. Study of the three principal parameters of permittivity of dielectric loss, voltage breakdown, and mechanical characteristics of the materials demonstrated the electrical objectives of such a capacitor were met, but that work remained in improving mechanical characteristics. General Electric personnel suggested approaches to the solution to this problem; made estimates of size, weight, and losses of potential cryogenic capacitor systems, and conclude that compact capacitor systems are feasible, especially since the peripheral components of such a system are well within existing technology.

o Preparation of a cryogenic data handbook (01-11-10).

A cryogenic reference work for design engineers, liquid gas suppliers, commercial development engineers, and research scientists, was prepared by Boeing and NASA personnel. This collection of cryogenic engineering information included extensive data on common cryogenic materials and fluids ordinarily scattered in various technical reports, journals, and textbooks with varying systems of units and outdated values. Where data was insufficient or incorrect, actual experimental and operational data were substituted. Data was also recalculated from metric to British units, where necessary, and available values extended to commonly required limits. Among the useful information are heat transfer rates for liquid nitrogen (LN2), temperature-enthalpy functions for LN2, saturated liquid/gas relations, linear contractions of Teflon, stainless steel and other ordinary cryogenic construction materials, gaseous air density, and compressibility factor versus pressure.

Engineers concerned with cryogenic technology have a continuing need for basic reference and design data. These data are ordinarily accessible only with difficulty, if at all. This Cryogenic Data Handbook is an attempt by NASA to overcome the problem by compiling, developing, integrating and arraying useful information in a handbook form which is useful to

in-house NASA operations to various NASA contractors and to the non-aerospace community.

o Preparation of a manual on special purpose thermocouples (01-11-11).

Special-purpose thermocouples are considered to be either: a new combination of thermoelectric materials applied to the solution of unique or conventional temperature-measuring problems; or the application of conventional thermoelectric materials in novel techniques of thermocouple fabrication, design, or instrumentation. Since the thermocouple plays such an important role both in the space program and industry, NASA contracted with the Midwest Research Institute to compile a comprehensive survey of NASA-developed thermocouple technology for reference and design use. The survey document is a complete text on thermocouple materials, principles, applications, designs and data. It covers thermocouple combinations for cryogenic temperatures, surface temperatures, gas temperatures, applications above 3000 F., and temperatures of solids; thermocouple use in energy-transfer gauges; the design, fabrication, and testing of thermocouples; and application techniques. The survey contains suggestions for applications in such industries as steel, food, natural gas, electronics, instrumentation, metals aerospace, petroleum, transportation, chemical, turbine engine and elsewhere.

# Superconductivity Technology (01-13)

In investigating the variation of electrical resistance with temperature, Heike Kammerlingh Onnes found that the electrical resistance in mercury becomes vanishingly small at a temperature just above the normal boiling point of helium (4.2 K). He called this point the transition temperature and the fundamental change in mercury its superconducting state, thus opening a whole new field of physics research.\* Over the past 55 years, another 900 superconducting materials had been discovered, including metals, semiconductors, alloys and compounds.

<sup>\*</sup>Onnes, H. Kammerlingh. "Disappearance of the Electrical Resistance of Mercury at Helium Temperatures." Proc. K. Akad. Wetenschappen, 19 (June 23, 1911), pp. 113-115.

Onnes also found that exceeding a certain critical current value abruptly re-established resistivity, and that if an external magnetic field were applied to the conductor at a certain temperature and strength it quenched the superconducting state. This critical current and critical magnetic field were further investigated by Silsbee, Meissner, Ochsenfeld and others, and the Meissner effect demonstrated that the magnetic field surrounding a normal conductor increased sharply in the superconducting transition.

The technological potential inherent in superconductivity provides the basis for enormously powerful magnets and motors for future commercial application in ships, atomic research, and electric power generation. For example, a 50-ton iron core magnet is required to produce the same magnetic field developed by a superconducting magnet weighing only a few pounds; the Meissner effect makes possible superconducting motors generating enormous torque values and run on friction-less magnetic bearings; and a 125.000 kVA superconducting transformer requires refrigeration of only about 150 kW, although combined copper and iron power losses in a similar conventional transformer would amount to 625kW.\*

The application of superconductivity to electric power engineering promises to increase capacity, reduce cost and improve reliability. For example, once a current is induced in a superconducting magnet, it will (theoretically) continue to produce an intense magnet field for over 100,000 years with no further power input, providing an energy source upon which one can draw.

Because of this potential, a wide variety of commercial applications of superconductivity technology were explored during the past decade, and are in various stages of advanced development, including: superconducting motors, high-capacity computer memory cores, superconducting power transformers and transmission lines, and superconducting magnets.

The General Electric Corporation and The Linde Division of Union Carbide Corporation, in cooperation with the Edison Electric Institute of New York and the Tennesee Valley Authority, Washington, D. C., are investigating various ways to use cryogenics and superconductivity in power transmission. Because electrical power requirements for our cities is expected to double over the next decade, yet public opinion in many areas rejects overhead power lines, the electric power industry is under some considerable pressure to develop adequate resources to answer the demand. Linde has begun an \$8 million, 12-year development

<sup>\*</sup>Timmerhaus, et al, loc. cit.

program to put an underground superconducting cable into operation by 1981, increasing efficiency 25-30 times that of conventional conductors.\*

One of the most important potential uses for superconducting magnets is as a critical component in developing electricity directly from a supersonic flow of hot ionized gases, a process known as magnetohydrodynamics (MHD). Approximately 75% of this country's power generation now requires an expensive and relatively inefficient three-step procedure of: first, heating large boilers; using the steam from the boilers to drive turbines; and then producing electricity from the turbine generators. MHD, alternatively, is an efficient, clean, one-step process. This method of power generation is far superior both in terms of increased efficiency (twice the efficiency of conventional power generation) and reduced pollutants (evolved gases are harmless carbon dioxide and nitrogen).

NASA contributions to the advancement of superconductivity have mainly dealt with the technological problems of moving the phenomenon of superconductivity out of the laboratory and into advanced development and early use. These contributions include the development of: (1) materials for superconducting devices that can be produced on a large scale at an economic price, such as niobium-titanium-copper compound superconductors and niobium nitride thin-film superconductors; (2) methods for fabricating large-scale superconducting devices, such as winding techniques to increase magnet efficiency; (3) techniques for stabilizing superconducting devices under large-scale operating conditions, such as external field stabilization methods to control flux jumping, and the use of superfluid helium as a coolant; and (4) a more advanced understanding of superconductivity theory, dealing with such areas as stability and flux jump, basic materials properties, and the effect of physical and chemical changes on high-field superconductors. The general significance of these contributions has been to reduce the time scale in which the advancement of the development of superconductivity has occurred, and the actual and potential impact (technological, scientific, economic, and social) of the contributions are estimated to be moderate to moderately high. Among the NASA contributions to the advancement of superconductivity technology are the following.

o Development of high field-strength, large-volume superconducting magnets (01-13-01).

The advent of superconductors with high-field (3-20 teslas) capability in the 1960's spurred

<sup>\*&</sup>quot;Superconducting Power Cables in 12 Years," <u>Industrial Research</u>, August 1969, pp. 19-20.

development by NASA/LeRC of large superconducting magnets. As a major customer requiring advanced materials and designs, NASA developed a continuing series of high field-strength, large-volume superconducting magnet technology. The Radio Corporation of America (RCA), for example, was contracted in 1963, to design and develop four 51-cm.-bore and one 15-cm.-bore superconducting magnets. The 15-cm.-bore magnet developed a field strength of 15 tesla, i.e., 150,000 gauss or the equivalent of a 14,000 pound per square inch bore pressure, and is still today the world's largest and strongest high field superconducting magnet.

o Fabrication and winding techniques to increase magnet efficiency (01-13-02).

- AP

During the course of RCA's development of five large superconducting magnets and associated systems for NASA/LeRC, fabrication and winding techniques had to be developed to control the flow of magnet current so that the current can be raised and lowered about the critical current in a reversible manner with no catastrophic change to the resistive state. Unfortunately, the penalties for this control can be high construction costs, low current density, and large magnet mass. Since overdesign is costly in magnet volume, materials, and performance, RCA developed and scaled up from laboratory to production levels a number of techniques to increase design efficiency. These techniques subsequently became part of the technological base used in their commercial sales.

One such technique involved scaling up to production levels a process for producing a practical composite Nb<sub>3</sub>Sn - a silver-stainless steel ribbon conductor of high strength, and thin cross-section. Hoop stress on the conductor and compressive loading of the magnet windings and internal structure are necessarily high because of the high current densities required. This stainless steel ribbon provided strength, the necessary alternative resistive current path, and minimum bulk.

Another technique invoves shorting strips, which is described in superconductivity contribution 01-13-06, below.

o Development of a composite conductor (Nb<sub>3</sub>Ti-Cu) (01-13-03).

Under contract to NASA/LeRC, AVCO-Everett Research Laboratory developed\* a niobium titanium wire-copper matrix (Nb<sub>3</sub>Ti-Cu) compound conductor that is today the standard conductor used in high field superconducting magnets.

Although current vs field characteristics are better for niobium stannide (Nb<sub>3</sub>Sn), it is an exceedingly brittle compound which cannot be drawn into wire or rolled into a tape, and requires highly specialized fabrication techniques. Alternatively, niobium titanium is a strong, ductile alloy which fabricates easily. Companies commercially supplying this composite are Airco Division of Air Reduction, Supercon Division of Norton, Cryomagnetics Corporation and Supertechnology, Inc.

o Reduced Cost of Superconducting Materials (01-13-04).

As a primary facility for the design, development, fabrication and testing of superconducting magnets, NASA/LeRC purchases significant quantities of superconducting materials. During 1970, for example, this facility purchased more composite conductor material from the Supercon Division of the Norton Company, a major supplier, than any other customer (13,000 feet of Nb3Ti-Cu composite conductor). Ordinarily, however, only the AEC is a larger market for superconductors. As an example of the effect of these large purchases, Nb3Ti-Cu cost \$1.25 per foot 5 years ago and today costs about \$0.60 per foot. Suppliers to NASA/LeRC have been RCA, AIRCO, Supercon, GE, Supertechnology, Inc., Cryomagnetics Corporation, Gardner Cryogenics, and AVCO-Everett Research Laboratories.

Since only NASA had the requirement for volume production quantities of high-field (3-20 tesla) superconductors, which are critical necessities for today's superconducting research, it was principally

<sup>\*</sup>Since there was a similar British effort underway at about the same time, it is not possible to pinpoint totally unique contributions.

due to responding to this market that the intermetallic compounds of niobium are widely available at the reduced cost at so early a date.

o External field stabilization (01-13-05).

Early experiments in making superconductive magnets showed that it was nearly impossible to achieve a current in the magnet windings equal to the current the wire could carry on a short sample test. This current degradation resulted in very poor performance of magnets made from the niobium stannide (Nb3Sn) or niobium zirconium (NbZr) materials, which were the first ones available for winding coils.

The poor performance of the materials when wound in coils was usually found to be in the low field portion of the windings. This generally required providing an external background field for the coil. Several magnets were wound by Lewis and RCA scientists using this external field stabilization. These magnets, in general, were made up of three physically independent coaxial coils. The outer sections were wound of Nb<sub>3</sub>Sn tapes. Each of the three coils was seriously degraded when operated independently but as the inner and center sections were immersed in higher background fields (from the outer coil) their critical currents increased until essentially the short sample performance was reached.

This now commonly employed external field stabilization technique to control flux jump was developed from theory to application in an early (1963) joint RCA-Lewis effort to control inner module instability, and without which superconducting solenoids would rapidly quench.

o Development of shorting strips as a stabilization device (01-13-06).

Superconducting current is difficult to maintain or vary in a magnet due to surges, current degradation, flux jump and other random or systematic instabilities which drive the magnet normal. Since superconductors carry very large amounts of energy, the release of this current from a superconductor to normally resistive materials will generate intense

heat, causing the liquid helium coolant necessary for the superconducting state to boil, which in turn heats adjacent superconductors and also drives them normal. The solenoid itself will, therefore, quench.

RCA developed, under contract to NASA/LeRC, a method of protection against collapsing fields consisting of interlayered shorted strips of copper foil laid across conductor layers at approximately 6 inch intervals around the circumference to provide alternate current paths for localized disturbances such as flux jumping. Evolutionary versions of this important technique are now found in commercial magnets as one of a family of stabilizing devices which also make a minimum contribution to bulk.

o Developing techniques for using superfluid helium as a stabilization device (01-13-07).

To prevent run-away current degradation and quenching, localized hot spots in superconducting magnets must be cooled immediately. The most commonly employed coolant, normal helium, has too high a viscosity and too low a flow rate to carry away all of the generated heat. Superfluid helium, on the other hand, has almost infinite heat conductivity and virtually no resistance to flow. It will, therefore, penetrate even the remote interstices of the magnet and instantaneously transport away critical heat.

The use of superfluid helium (2.17 K.) to achieve stabilization (see superconductivity contribution 01-13-05, above) in high-field superconducting magnets was developed from theory to practical application at NASA/LeRC. This technique is today a routinely applied part of the superconducting magnet state-of-the-art.

o Intermittent windings for stabilization (01-13-08).

A continuing problem in maintaining magnet superconductivity is to remove localized thermal surges at very high rates. Localized heat due to flux jumping if not closely controlled soon drives the magnet normal. The problem is to maintain coolant flow throughout the winding interstices at all times. Normal helium flow is insufficient and so superfluid

helium must be employed. But even this is not entirely adequate for stable magnetization.

ACVO-Everett Research Laboratory under contract to NASA/LeRC therefore developed a unique technique for providing helium flow channels by forming square rather than round compound conductors, which are then separated by intermittent spacers of sticky tape. This technique is presently reflected in commercial magnet designs.

o Development of high-volume liquid helium technology (01-13-09).

In order for superconductivity technology to advance, the widespread and economical availability of liquid helium was one indispensible prerequisite. Because liquid helium was required by NASA in volumes and at rates greatly in excess of the capabilities of commercial industrial gas producers for such applications as cryopumping and black body space simulation, and as a purge and precool gas in rocket systems, an entire technology had to be developed (see cryogenics development 01-09; liquefaction and refrigeration technology). Large-scale liquefiers, advanced insulation materials and techniques, tank cars, roadable and static dewars, and associated component hardware were developed to liquefy, transport, store and use high-volume liquid helium. Some of the many contractors involved were Linde Division of Union Carbide, Air Products, Air Reduction, Beech Aircraft, Goodyear, A. D. Little, Lockheed, Annin and Pratt & Whitney.

o Design and construction of a 'Magnetic Bottle' for fusion containment (01-13-10).

In order for fusion power to become a practical reality some means must be developed to confine the hot ionized plasmas from a thermonuclear reactor. Since these gases will be at temperatures in the range of 100 million C., no material substance can contain them. The most logical technique is to employ a suitably shaped magnetic field of sufficient intensity. The ionized particles are trapped and conveyed by magnetic lines of force which prevent them from actually contacting (and so disintegrating) the

walls of the chamber. Were an ordinary copper magnet employed, it would have to be several feet long, and would consume more power than the thermonuclear power generator could produce. The power required to start up and maintain an equivalent strength superconducting magnet is by comparison trivial. Such a fusion containment device is called a magnetic bottle.

o Advanced understanding of superconductivity theory (01-13-11).

In order to develop practical uses of superconductivity, NASA sponsored efforts which have led to a better understanding of superconductivity. These efforts include, among others: Theoretical studies of stability and flux jump as well as experimental studies of flux jumping in high field superconductors and the ac losses in superconduction coils (Atomics International); determination of ac losses in Type II superconductors (AVCO-Everett Research Laboratory); research on the effects of very strong magnetic fields and of magnetic field free environments on man and animals (Naval Aerospace Medical Institute); solution growth of large single crystals of Nb<sub>3</sub>Sn - Nb<sub>3</sub>Sb pseudo-binary system (RCA); analysis of the behavior of a square copperstabilized Nb<sub>2</sub>Ti conductor (AVCO-Everett Research Laboratory); study of the effect of tensile stress on current-carrying capacity of commercial superconductors such as niobium zirconium (NbZr) and niobium-tin (NbSn) at liquid helium temperatures (Lewis Research Center); and study of the properties of high-field superconductors at elevated temperatures (RCA).

o Analysis and development of niobium superconductors (01-13-12).

The intermetallic compounds of niobium are used to develop and sustain high-field super-conducting magnetization for work in thermonuclear power generation, plasma research, solid state physics, utility power generation and transmission, very high bit-density computers and other applications. NASA has performed significant in-house and contracted work to analyze the properties of various transition metal compounds of niobium and to

develop useful thermophysical data. Examples of this work include: preparation of superconducting thin films of transition metal interstitial compounds (Westinghouse); study of characteristics of NbZr superconducting strip (AVCO-Everett Research Laboratory); investigation of current degradation phenomena in NbTi (Atomics International); investigation of the superconducting properties of transition metal alloy systems (A. D. Little, Inc.); study of the nature of impurity resonances in electron energy bonds for both simple- and transition-metal impurities in both simple- and transition-metal matrices (Chicago University); study of the properties of high-field superconductors. ac field induced flux jumps (RCA); study and measurements of the magnetic anisotropy of critical current in wide sample superconductors (AVCO-Everett Research Laboratory); current distribution in a thin-film superconducting strip transmission line (Lewis Research Center); research on superconducting niobium-thorium eutectic alloy and superconducting composites (MIT); and high magnetic field superconducting properties of Nb3Sn films (RCA).

o Development of very-high-field niobium nitride superconductors (01-13-13).

Extremely pure niobium nitride (NbN) thin-film superconductors (10<sup>3</sup> Angstrom) capable of maintaining a magnetic field greater than 225 kilograms, were developed by the Westinghouse Corporation under NASA contract. Westinghouse now holds the patent on this development.

This development represents the first successful effort to produce ultrapure thin films of the NoN transition metal. Tests show thin film NoN supports magnetic fields equal to or better than Nb2Sn which is currently the highest field material available. Nb3Sn, however, is difficult to produce and is extremely brittle, which makes fabrication expensive and difficult. NoN is easier to manufacture, is less expensive, and is more stable than NbaSn. Another important characteristic is that its transition temperature of 14-16 K places its useful operation in the liquid hydrogen range, which is cheaper, although less safe than liquid helium. The NbN thin film samples are still superconducting at 180 Kilogauss at 4.2 K. This development could lead to far more widespread availability of low cost, low maintenance superconducting devices.

The field of electrochemical energy conversion and storage is concerned with direct conversion of the chemical energy of a fuel into electrical energy through electrochemical processes. The field also includes the storage of electrical energy as chemical energy and its eventual reconversion to electrical energy (the storage battery). Electrochemical energy conversion and storage devices generally can be grouped within three categories: fuel cells, primary batteries, and secondary (rechargeable) batteries.

In fuel cells, the fuel and an oxidizing agent are supplied separately and continuously to the cell electrodes, at which they undergo reaction; the reaction products are continuously removed. Theoretically, fuel cells can operate as long as they are supplied with the required active materials.

The primary battery is similar to the fuel cell (both may be classed as primary cells, i.e. the electrochemical reactions cannot be reversed) but the active materials (fuel and oxidizing agent) are stored within the cell electrodes. Therefore, cell life is limited by the amount of active materials initially stored in the cell.

Secondary batteries can be used and then recharged, the number of cycles varying from less than 10 to 1000 or more, depending on the type of battery. The common lead-acid storage battery used in automobiles is the best-known example of a secondary battery.

In both fuel cells and batteries, electric current is generated by electron transfer occurring in a chemical reaction. Electron transfer in the reaction processes is prohibited from occurring directly by the way in which the active materials are packaged: transfer is accomplished through an external circuit, resulting in the generation of electric current at a controlled rate dependent on the external circuit resistance. Unless the external circuit is closed, the reactions cannot proceed.

There has been a substantial amount of activity in this field during the last ten years. The activity includes research into the kinetics and catalysis of electrochemical reactions, the development of practical power systems for operation under the difficult environmental constraints of military and space missions, and development of reliable, relatively low-cost batteries for new commercial applications. Major impetus for fuel cell and battery activity in the early 1960's came from space flight and military mission requirements for highly reliable, light-weight, long-life power supplies. More recent activity has been spurred on by the search for new low-pollution energy sources to meet commercial, residential and transportation power demands. Electrochemical energy conversion and storage devices present an environmentally attractive alternative to fossil and nuclear fuel sources. A selected

list of significant developments in electrochemical energy conversion and storage, as identified by experts in the field, is given in Table 2.

The battery industry has grown from total sales of \$580 million in 1960 to just over \$1 billion in 1969. Except for military and space applications, fuel cell activity is primarily in the research and development stages, the largest current project being the natural gas industry's TARGET program at approximately \$7 million per year.

Many new electrochemical devices have shown promise in the laboratory and in military and space applications where high performance requirements had to be met, with cost a secondary factor. The basic problem in the field today is to develop commercially feasible, cost competitive systems. Activity in the field to meet this problem ranges from basic research to identify and develop non-noble metal (low-cost) catalysts to development of commercial manufacturing processes suitable for high-volume production.

Selected significant developments in the field of electrochemical energy conversion and storage (as listed in Table 2), and NASA contributions to the advancement of these developments, follow.

### Improvements in Alkaline Electrolyte Primary Batteries (02-01)

Alkaline electrolyte batteries are batteries which use alkalies such as potassium hydroxide and sodium hydroxide as electrolytes. They have several inherent advantages over the more common acid and salt electrolyte batteries: alkaline solutions are more stable and easier to transport than acid electrolytes, voltage-discharge characteristics are more uniform and relatively high current rates can be achieved at nearly constant voltage levels; and alkaline electrolytes are more compatible with the important zinc electrode.

Until recently, cost and technical difficulties associated with electrode and separator design, packaging, and storage life after activation, had slowed the development of alkaline electrolyte primary batteries. Incremental technological advances over the past few years have improved the performance of this class of batteries. These advances include use of Teflon, Kel-F and other proprietary plastics to solve the difficult problems of packaging and sealing the electrolyte; improved design and fabrication of electrodes; and long-lived separators.

Alkaline manganese dioxide-zinc dry cells such as Union Carbide's "Energizer," have found application in items such as tape recorders, hearing aids, portable radios and flashlights, and have captured an increasing share of the market over the past five years from the heretofore standard salt electrolyte Le Clanché cell. While these batteries cost three times as much as the Le Clanché cell, they can deliver about ten times as much power under heavy current drain conditions.

#### TABLE 2

# SIGNIFICANT DEVELOPMENTS IN THE FIELD OF ELECTROCHEMICAL ENERGY CONVERSION AND STORAGE (02)\*

- \*\*1. Improvements in alkaline electrolyte batteries (02-01)
- \*\*2. Biochemical fuel cell development (02-02)
  - 3. Fused salt systems (02-03)
- \*\*4. High-temperature secondary batteries (02-04)
  - 5. Development of hydrocarbon fuel cells (02-05)
- \*\*6. Development of hydrogen-oxygen fuel cells (02-06)
  - 7. Ion-exchange membranes (02-07)
  - 8. Lead-acid battery improvements (02-08)
- \*\*9. Metal-air batteries (02-09)
- 10. Metal-air and hydrogen-air fuel cells (02-10)
- \*\*11. Natural gas fuel cells (02-11)
- \*\*12. Nickel-cadmium battery improvements (02-12)
  - 13. Non-aqueous electrolytes for batteries (02-13)
  - 14. Non-noble metal catalysts (02-14)
  - 15. Oxygen gage and oxygen generation (02-15)
- \*\*16. Silver-zinc battery improvements (02-16)
- \*\*17. Solid electrolyte batteries (02-17)
  - 18. Solid electrolytes for fuel cells (02-18)
  - 19. Teflon applications (02-19)
  - 20. Thin-film metal and ceramic deposition (02-20)

<sup>\*</sup>As identified by technological leaders in the field of electrochemical energy conversion and storage.

<sup>\*\*</sup>Developments selected for identification of NASA contributions.

Other alkaline electrolyte primary batteries in production include the mercuric oxide-zinc battery, used in a number of industrial applications and for portable communications equipment; the copper oxide-zinc battery, used extensively by railroads for track circuits, crossing signals, etc.; and the silver-zinc battery, which can function as a primary or secondary battery.

NASA's major contributions have been directed primarily towards the improvement of one specific type of alkaline battery, which is more fully discussed under silver-zinc batteries (development 09-16). However, NASA's contributions to the advancement of silver-zinc batteries have also resulted in improvements in electrodes and separators for other alkaline batteries, and while minor, they should result in advancing the performance of these types of cells. These contributions include the following, among others.

o Development of improved zinc electrodes (02-01-01).

NASA efforts directed towards understanding the limiting processes of zinc electrodes and towards improving the electrode's performance and life resulted in increased power density and shelf life of batteries using zinc electrodes. While most of the effort dealt with zinc electrodes in silverzinc batteries, the results are applicable to the technology of zinc electrodes in other alkaline electrolyte batteries. (For a fuller description see NASA contribution 02-16-02.)

o Evaluation and characterization of separator materials and designs (02-01-02).

The performance of separators for use in alkaline electrolyte batteries was evaluated and characterized by NASA. Major performance measures used included chemical stability, structural reliability, mechanical diffusion properties and electrical characteristics. The evaluation was carried out by Electric Storage Battery Company (ESB), as well as by Borden, General Electric, Monsanto and TRW, under NASA contracts. This evaluation of separator materials advanced the technology of alkaline electrolyte batteries by the direction for further research on the development and improvement of separators.

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### Biochemical Fuel Cell Development (02-02)

A biochemical fuel cell uses biological catalysts (bacteria, enzymes, yeast) and organic matter (usually plant or animal waste products) as fuels to generate electricity. The concept of a biochemical fuel cell is not new, with the first demonstration in 1911 by M. C. Potter in England. The biochemical fuel cell usually contains an air or oxygen cathode and an anode which reacts with the biochemically produced fuel. Substances used as biochemical fuels include carbohydrates, urea, glucose and fatty acids.

Until the 1960's, the biochemical fuel cell was little more than a laboratory curiosity. The concept of a closed ecological cycle for space travel increased research efforts in this area.

There are several major potential applications of biochemical fuel cells. At present, most work on these cells is in the conceptual and exploratory research stages. Although the potential significance of biochemical fuel cells is major, the prospects for successful development of these cells is uncertain.

As noted, the cell can be used in closed cycle life support systems for space travel. Work in the early 1960's on this application aided in the development of a better basic understanding of biochemical fuel cell systems. The very low current density which is available from such a cell, now or in the foreseeable future, has resulted in virtual abandonment of research in biochemical fuel cells for space travel application. However, the work done has supported efforts concerned with other applications.

Fuel cells which use the metabolites in the body as fuel (e.g., glucose and oxygen in the blood) are being developed for use in pacemakers and implantable hearts. This line of research is being supported by the National Heart Institute of the National Institute of Health. For implantable hearts, several watts of power would be required, whereas for pacemakers only microwatts are required. Because of the apparently low inherent performance of the cells, the latter application appears more likely. Because these cells would be powered by natural fuels in the body, their installation could be permanent, in contrast to current pacemakers.

A low-power, long-life biochemical fuel cell could find application in areas of the world where fossil fuels are in short supply and in military uses. Many vegetables, fruits and grains have been investigated as possible fuel sources. However, for widespread commercial or government applications, an improvement of one or two orders of magnitude in current density is necessary, as in the development of a long life (about 10 years), cheap, reliable air electrode.\*

<sup>\*</sup>Austin, L. G. Fuel Cells: A Review of Government Sponsored Research, 1950-1964. Washington, D. C.: National Aeronautics and Space Administration, Office of Technology Utilization, Scientific and Technical Information Division, 1967.

NASA contributions to the advancement of biochemical fuel cells have been both direct and indirect. Their work in the early 1960's with contractors such as Marquardt, General Electric and Magna (also supported by the U. S. Army/USAERDC) developed basic knowledge of the biochemical and electrochemical reactions for a variety of biological fuels and catalysts. This work has had moderate but widespread impact on biochemical fuel cell development for applications other than space. NASA's development of an improved oxygen electrode for hydrogen-oxygen cells has influenced electrode design in biochemical fuel cells. The oxygen or air cathode is a critical element in the biochemical fuel cell system. NASA contributions include the following, among others.

o Development of Teflonated electrodes (02-02-01).

A biochemical fuel cell usually contains an air or oxygen cathode and an anode which reacts with a biochemically produced fuel. Teflonated electrodes, developed by NASA for use in hydrogen-oxygen fuel cells, are being used in modified form as cathodes in biochemical fuel cells.

The Teflonated electrode is made by embedding catalytic particles (e.g., platinum) or a grid (e.g., platinized nickel gauge) in Teflon. The electrode is relatively easy to fabricate, provides long-term stability in electrolytes, and has demonstrated reliable and predictable performance. For a full description of the development of this electrode see NASA contributions to hydrogen-oxygen fuel cells (contribution 02-06-02).

In the present early stage of biochemical fuel cell development, many types of electrodes are being tried. Although the Teflonated air or oxygen cathode is only one among many alternatives, it appears to provide satisfactory performance in several types of cells. Within the overall context of biochemical fuel cell development, the Teflonated air or oxygen cathode has been a valuable element in early development efforts.

o Provided the scientific and technological base for advanced development of biochemical fuel cells (02-02-02).

NASA's support of basic research in biochemical fuel cells during the early 1960's developed an extensive information base and stimulated government and industry interest in applications other than

space use. During the same period the U.S. Army/ USAERDC also actively supported basic research in biochemical fuel cells.

Fundamental studies of biochemical fuel cell reactions were funded by NASA at Philco-Ford (Aeronutronic) and the University of Pennsylvania from 1962 to 1964, and the reactions of several enzymes and microorganisms were characterized. Closed-cycle life support systems were studied by Marquardt, General Electric, and Magna Corporation at about the same time. Human wastes were the primary fuels. The NASA work, combined with parallel work supported by the U. S. Army/USAERDC, provided the scientific and technological base for more recent advanced developmental activities, such as those now being carried out at the National Heart Institute.

o Aided in setting the direction of current biochemical fuel cell research for biomedical applications (02-02-03).

The National Heart Institute is now supporting extensive research to develop biochemical fuel cells for use in pacemakers and implantable hearts. NASA/LeRC research personnel who were involved in NASA's biochemical fuel cell efforts have assisted the National Heart Institute in evaluating proposals in this area. Their familiarity with prior research, including the often undocumented "dead ends," has had an influence on the direction of current research.

## High-Temperature Secondary Batteries (02-04)

High-temperature secondary batteries are high energy-density systems generally operating in the temperature range 150 C. to 700 C., and capable of a number of charge cycles. Liquid electrolytes used in these cells are usually molten salts such as lithium chloride, or combinations such as lithium chloride/potassium chloride. (High-temperature solid electrolyte batteries are discussed in development 02-09.) Energy densities obtainable from these batteries are at least twice as high as lead-acid batteries and can run as high as seven to ten times greater than lead-acid energy densities. The higher figures provide densities in the order of 100-150 watt-hours per pound, a range often cited as the level needed for efficient use in automobiles to replace the internal combustion engine. While energy densities up to 1000 watt-hours per pound are theoretically possible, the estimates of 100-150 watt-hours are believed to be achievable in a practical system.

Because of their great potential, approximately 20 commercial firms are working on high-temperature secondary batteries, although less than half have publicly announced their activities. Major types of batteries under study are the sodium-sulphur battery (being developed by Ford Motor Company, Dow Chemical, Argonne National Laboratories and Atomics International), the lithium-chlorine battery (General Motors, Sohio), lithium-sulphur battery (General Motors, Atomics International), and lithium-selenium battery (Argonne National Laboratories).

Most of the work to date has been at the cell level, rather than at the battery system level. Major development difficulties at the present time are associated with materials due to the high temperatures and corrosiveness of the active substances in the battery.

The very high energy-density and power theoretically attainable from high-temperature secondary batteries could make them a significant alternative to the internal combustion engine as a power source in automobiles. The Army at Ft. Belvoir has sponsored work on the lithium-selenium battery for use in military vehicles. In another area of potential application, the National Heart Institute is investigating the use of lithium-selenium batteries in an implantable heart, with a battery output of 30 watts. The wide range of potential applications (automotive, military, biomedical) could have significant economic and technological impact, although the development of such batteries is presently in the laboratory stage.

NASA has had a limited role in the development of high-temperature secondary batteries. Fundamental research and cell development of two systems studied by NASA appear to contribute to the overall advancement of the technology of high-temperature secondary batteries: the potassium amalgam cell and the aluminum-chlorine cell.

NASA's contributions to high-temperature secondary batteries consist of the development of some promising cells, in parallel with the exploratory investigation by others of many cell types. The multiplicity of candidate systems characterizes the present stage of activity in this area. It is not possible at present to assess the potential significance of this work as no system as yet has progressed far enough for there to be a sorting out of candidate systems. Included among the NASA contributions are the following.

o Aluminum-chlorine battery development (02-04-01).

The development of an aluminum-chlorine cell with molten salt electrolytes was undertaken by NASA/ERC at Tyco Laboratories. The system shows promise as a rechargeable, high energy-density battery operating at the relatively low temperature

of 150 C. The low operating temperature should ease the materials and safety problems associated with high-temperature batteries. The solid aluminum in the system also is less hazardous than the molten lithium or sodium common to most other batteries in this category.

Much more research is required to assess the future applicability of this system. It is presently one promising cell among many in the high-temperature secondary battery field.

o Potassium amalgam regenerative cell (02-04-02).

Allison Division of General Motors, under contract to NASA/LeRC, developed and tested various electrolyte matrix combinations for a potassium amalgam regenerative cell. Preceding and during this work, Allison also was under contract to the Air Force for development of the potassium amalgam cell. The work for NASA and the Air Force was closely related and may have been a joint project.

The potassium amalgam cell promised high power, high energy-density and long life. An Air Force contract called for 5 kilowatts at 28 volts to last 1 year in space applications.

While the transition from laboratory cell to power system was never successfully achieved, the practical information developed on design of the high-temperature potassium system is of value in design of other high-temperature systems using lithium, sodium or potassium. No specific influence of this research on following work in high-temperature batteries has been identified but it is believed to have exerted some influence.

# Development of Hydrogen-Oxygen Fuel Cell Systems (02-06)

Electrochemical reaction of hydrogen and oxygen in a galvanic cell to produce electrical power was first demonstrated in the early 1800's. A number of laboratory cells were developed and tested during the first half of the 20th Century. Perhaps the most successful of these was the Bacon cell, an alkaline electrolyte, high temperature (approximately 250 C.) cell which was developed by Francis Bacon in England from approximately 1940 to 1960.

During the 1960's several types of hydrogen-oxygen fuel cells were developed into practical operating systems. Work was stimulated primarily by military and space mission requirements for high energydensity power systems that could operate for periods ranging from approximately one day to one month, at power levels from one to ten kilowatts. Within this operating region, fuel cells appeared to have performance advantages over alternative power sources such as battery, solar cells and nuclear power sources. The Bacon cell was improved and refined by Pratt and Whitney for the Apollo program. For the Gemini program, General Electric developed an acid electrolyte cell using an ion-exchange membrane to contain the electrolyte. Development of the ion exchange membrane cell also was directed towards military missions. Other major systems were developed by Allis-Chalmers (space and surface applications) and Union Carbide (surface applications). Much of the technology on the use of common hydro-carbon fuels in fuel cells was developed by the Army because of the logistics consideration in their missions.

Current work centers on development of systems using non-noble metal catalysts and common hydrocarbon fuels. Success in these areas would be required to achieve the major cost reduction and operating simplifications necessary for competitive commercial systems. One of the largest current efforts is to develop a fuel cell using natural gas. (For a complete discussion of natural gas fuel cells see development 02-11, below.)

Over the last decade hydrogen-oxygen fuel cells have developed from items of laboratory interest to a range of practical operating systems. The Gemini and Apollo fuel cell systems were key elements in the success of those space programs. If costs can be reduced, applications can be foreseen to uses such as emergency power, remote and portable power, automotive power, and even individual home power sources (see natural gas fuel cells, development 02-11).

The performance characteristics of hydrogen-oxygen fuel cells matched NASA's mission requirements for the Apollo and Gemini programs. Because the performance advantages overrode the relatively high cost per kilowatt-hour, NASA developed practical, high performance operating systems. During the same period, for similar reasons, the Army, Air Force and Navy actively supported hydrogen-oxygen fuel cell development. Without the impetus of military and space mission requirements, it is doubtful that such operating systems would now exist. Present work, limited in this country, but relatively active in Europe, is drawing upon the extensive base of technology developed by NASA and the military in the 1960's. NASA contributions to the advancement of the technology of hydrogen-oxygen fuel cells include the following, among others.

o Development of practical hydrogen-oxygen fuel cell operating systems (02-06-01).

For space application, performance factors such as low weight and volume, safety and reliability,

overcame the high cost of fuel cells and resulted in NASA's development of practical hydrogen-oxygen fuel cell operating systems. Major NASA contractors included Pratt and Whitney, GE, Electro-Optical Systems and Allis-Chalmers. In development of these systems a large number of incremental technological advances were made that have influenced overall fuel cell development. (Major technological advances are discussed below separately as individual NASA contributions.)

Among these incremental developments are improvements in temperature control, pressure regulation, electrode mounting for shock environments, and water removal subsystems. While these advances are generally system dependent (e.g., the temperature control subsystem of the Apollo cell is directly applicable to that cell), they have advanced the technology base for the development of other fuel cell systems. Much of this technology is critical to success in engineering design and production. However, little has been formally documented, with most of it found in internal memos, the skills of designers, and at times, implicit in design drawings. To a great degree such technology has been transferred through movement of people (company to company or project to project) and by informal personal contact.

o Development of the relatively long-lived matrix cell with Teflon-bonded electrodes (02-06-02).

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Starting in 1963, NASA/LeRC supported work at American Cyanimide to develop a high-performance, lightweight electrode system for hydrogen-oxygen fuel cells. Most of the effort was devoted to alkaline electrolyte cells, although electrodes for alkaline cells are made by imbedding platinized nickel gauze in Teflon. The electrodes are then pressed mechanically against the electrolyte matrix which is saturated with the electrolyte.

The American Cyanimide electrodes were originally used with an asbestos matrix. Subsequent NASA-supported work at TRW showed that asbestos was unstable in the electrolyte. Alternative matrix materials were identified in other NASA studies, and are now in experimental use: these include potassium titanate (Electro-Optical Systems) and zirconium oxide (Pratt and Whitney).

The matrix cell with Teflon-bonded electrodes had substantially increased life over electrodes of similar performance. Operating life of the Gemini ion exchange membrane cell was on the order of a few days; the matrix cell operating times are in excess of 1,000 hours.

o Development of an electrolytic regenerative hydrogenoxygen fuel cell (02-06-03).

A regenerative fuel cell is one in which the products of cell reaction are regenerated into reactants. Such a cell can function as a secondary (i.e., rechargeable) battery. The most probable application is to orbiting earth satellites. Regenerative cells consist of a fuel cell with a source of energy capable of dissociating the reaction product. Possible energy sources are heat, light, radiation, chemical (electrolytic) energy and electrical energy.\*

Since 1960, NASA has funded an extensive program at Electro-Optical Systems (EOS) to develop an electrolytically regenerative hydrogen-oxygen fuel cell. The wide ranging program has included development of matrix materials and designs, gas distribution methods, catalyst loadings, pressure and temperature control, and electrodes.

A patent was issued to NASA on the EOS regenerative fuel cell in April 1970. The cell is a candidate for synchronous satellite applications by COMSAT and the Air Force. It is believed that such a cell could provide twice the energy-density of nickel-cadmium batteries, and reduce the cost per channel of synchronous satellites. However, several fundamental problems first must be solved:

 Oxygen electrode lifetimes are less than a year; for communication satellites, 2-5 years life would be required. NASA has supported research efforts at Tyco Laboratories toward the solution of this problem.

<sup>\*</sup>Fuel Cells, Vielstich, p. 11.

- 2. A major technological advance in catalysis may be required to bring the costs to a point where the cell is suitable for commercial application.
- 3. Long-term stability of the cell needs improvement.

Another major factor affecting application is the rate of development of other types of high energy-density battery systems. At this point in time, prospects for commercial success of the hydrogen-oxygen regenerative cell are uncertain.

o Identification of the effects of small carbon dioxide concentrations on electrode life (02-06-04).

Recent research at NASA/LeRC has determined that very low concentrations of carbon dioxide (on the order of a few parts per million) can significantly affect the lifetime of electrodes in hydrogen-oxygen alkaline fuel cells. Limited electrode life-time has been a major problem in such cells. Although it is too early to estimate the importance of identifying low concentrations of carbon dioxide as adversely affecting electrode life, the direction of future electrode research and development could be significantly affected by this discovery.

# Metal-Air Batteries (02-09)

The metal-air battery consists of an oxygen or air cathode and a metal anode. Typical metal electrodes are zinc, aluminum, magnesium and cadmium. The zinc-air cell has been used for some time for remote power applications, such as railroad signalling installations and radio transmitters. Miniaturized metal-air dry cells have been used in hearing aids.

Recent improvements in air and oxygen electrodes have increased power and energy-densities significantly, sparking interest in these batteries for a wider range of application, such as in automotive and military power systems. For example, zinc-air batteries can provide an energy density of 80-100 watt-hours per pound. Because of their promise the Army has supported a great deal of research on these batteries in recent years for application as silent power sources.

There are two major obstacles which must be overcome if metal-air batteries are to find widespread application: rechargeability and shelf life. Although several companies are actively pursuing development

of rechargeable systems, efforts to make metal-air batteries rechargeable for a large number of cycles have had limited success. Another current problem with metal-air batteries is limited shelf life after activation.

The high energy- and power-densities of metal-air cells could lead to use of these batteries in automobiles. Dry cells could find application in a number of portable power uses, such as in portable tools. Success is contingent on solving the problems of shelf life after activation and rechargeability.

NASA's fuel cell efforts, have contributed to the improvement of the air cathodes in metal-air cells, resulting in increased power- and energy-density. Work on silver-zinc batteries should result in improved rechargeability of the zinc electrode, which could broaden the range of metal-air battery applications. Research on aluminum and cadmium electrodes, while not directed specifically towards metal-air battery application, has contributed to a better understanding of these electrodes, and probably will contribute to improvement of metal-air systems. In contrast to the Army's direct involvement in metal-air batteries, NASA's contributions have arisen primarily from work on related developments such as the hydrogen-oxygen fuel cell and silver-zinc battery and from basic research into electrode mechanisms. The present significance of these contributions range from moderate to minor, but the potential significance is somewhat greater. NASA contributions include, among others.

o Development of a rechargeable oxygen electrode (02-09-01).

NASA's work on the electrolytically regenerative hydrogen-oxygen fuel cell (contribution 02-06-03) resulted in development of a moderately successful rechargeable oxygen electrode. Development of such an electrode is necessary if metal-air batteries are to be successfully applied as secondary batteries. NASA has also sponsored recent research efforts at Tyco Laboratories on the development of rechargeable oxygen electrodes.

The NASA work has contributed to other industry efforts directed towards development of a successful secondary metal-air battery. At present, work on secondary metal-air batteries is still in the research and advanced development stages, and it is not possible to assess the relative importance of NASA's contributions. Potentially the contributions could be significant.

o Development of an inorganic separator (02-09-02).

Of specific importance for metal-air battery development is NASA's development of an inorganic separator, which could increase cycle capability of systems with zinc electrodes by a factor of four to ten. (The development of inorganic separators is more fully discussed under silverzinc battery improvements, development 02-16). Much of the work was done at McDonnel-Douglas Astropower. In recent NASA-supported efforts at Astropower, rechargeable zinc-oxygen cells with inorganic separators were tested and operated effectively for over 200 cycles. As in the oxygen electrode contribution cited above, the inorganic separator is potentially significant in advancing the development of a successful secondary metal-air battery. The inorganic separator effects are in parallel with other NASA and non-NASA projects, to improve zinc-air rechargeability. For a complete description of the development of inorganic separators, see silver-zinc battery improvements, contribution 02-16-01.

A NASA-supported project at Union Carbide tested several types of zinc anodes, and up to 175 cycles were obtained with one configuration. It is too early to determine what design or combination of designs will ultimately be commercially successful. (For a more complete description of NASA contributions to the advancement of zinc electrodes, see silverzinc batteries, contribution 02-16-02.)

o Development of aluminum electrodes (02-09-03).

Aluminum has long been considered as a replacement for zinc in alkaline metal-oxygen cells: it is superior to zinc in equivalent weight, negative electrode potential and electrochemical activity.\* Its principal drawbacks are excessive hydrogen gas and heat evolution and self-discharge characteristics in alkaline electrolytes. Non-alkaline electrolytes result in limited powerdensities.

<sup>\*</sup>Ibid.

NASA efforts to develop an aluminum electrode have focused on the electrochemical behavior of aluminum in molten salt electrolytes (see high-temperature secondary batteries, NASA contribution 02-04-01). While this work was aimed at developing a better understanding of the aluminum electrode in a high-temperature cell, the results, in part, are applicable to air cells, and demonstrate the technological interactions within the battery field.

o Determination of cadmium electrode failure mechanisms (02-09-04).

To determine why the capacity of cadmium battery plates decrease over time, and the dependency of such mechanisms on electrode preparation, a detailed study of the mechanisms affecting capacity changes and failure of the cadmium electrode was supported by NASA/LeRC. This research, carried out by General Electric, should have a significant effect on future cadmium electrode design.

# Natural Gas Fuel Cells (02-11)

Natural gas can be used in fuel cells in two basic ways: (1) the gas can be supplied directly to the anode and reacted electrochemically there; and (2) the natural gas can be reformed with steam at high temperature and pressure to yield hydrogen, carbon dioxide, methane and excess steam, with the hydrogen then supplied to the anode of a hydrogen-oxygen cell. Acid, alkaline and molten carbonate electrolyte cells have been studied for application with natural gas fuel.

The most significant effort in this area is the TARGET program funded by a consortium of natural gas companies. The original objective was to meet household energy requirements by fuel cell generated electricity. Recently, emphasis has switched to developing moderate size power plants (about 12 kilowatts) for application in places such as shopping centers and hospitals. Pratt and Whitney and the Institute of Gas Technology are the major contractors on this program. Cell lifetimes of 10,000 hours or more are being sought, but 2,000 hours is what is presently being achieved. Major emphasis is on the development of a cell with phosphoric acid electrolyte with a natural gas reformer.

Successful development of these cells would reduce the need for large central power generating stations with a resulting major impact on pollution and on the structure and growth of the electric power industry. Therefore, the potential economic and social impact of such a development is significant.

Most government activity in the area of hydrocarbon fuel cells has been through Army research activities at Ft. Belvoir and Ft. Monmouth. For logistics reasons, the Army has been interested in cells using common hydrocarbon fuels for silent electrical and automotive power sources. Due to its space mission, NASA has not had much interest in such cells. Not only do they not have the logistical need for using hydrocarbon cells, but the weight penalties inherent in using such fuels are contrary to space mission requirements. Therefore, NASA contributions to the development of natural gas fuel cells have been limited and indirect through work sponsored on hydrogen-oxygen fuel cells. These indirect contributions include, for example:

o Advancements in hydrogen-oxygen fuel cell technology (02-11-01).

NASA developed an extensive base of hydrogenoxygen fuel cell technology, including testing and analytical techniques, which are applicable to natural gas fuel cell work. The applicability is particularly direct in natural gas fuel cells which use gas reformers, because relatively standard hydrogen-oxygen fuel cells must be coupled with gas reformers in such a system. One of NASA's major fuel cell contractors was Pratt and Whitney, contractor on the TARGET program. The fuel cell engineering knowledge and testing and analytical skills developed at Pratt and Whitney, partially with NASA support, have undoubtedly been of value in the TARGET work. While indirect in nature, these NASA contributions served to advance the development of natural gas fuel cells.

# Nickel-Cadmium Battery Improvements (02-12)

The nickel-cadmium battery was first applied in the early 1900's. The active cathode material is nickel hydroxide; the active anode material is cadmium; and potassium hydroxide is the electrolyte. Low allowable depth of discharge and limited life hampered early development of the battery. From 1920 on, a series of developments, mostly in Europe, substantially improved battery performance. Nickel-cadmium battery production began in the U.S. shortly after World War II, using technology developed in Germany. The nickel-cadmium battery is generally used where requirements call for very high cycle-life and long lifetimes. Over the last ten years, reliability, power-density, shelf life and packaging density have increased, while battery cost has decreased. The system's high cycle-capability and long life has led to use in satellites. The closest competitor for space service, silver-zinc batteries, provide two to four times the energy density of nickel-cadmium

batteries but, for operational systems, have a cycle life an order of magnitude less than that of nickel-cadmium batteries. In addition, nickel-cadmium batteries are also unusually rugged and perform well at low temperatures.

Commercially, nickel-cadmium batteries are used in such applications as small, portable electric devices such as power tools, razors and electric toothbrushes, and for engine cranking and other uses. Improved nickel-cadmium battery performance has led to wide commercial application in recent years. Of special significance are improvements in quality control and ability to tolerate overcharge. For commercial application, batteries must be reliable and not overly susceptible to misuse. The most important improvements in the last ten years have been precisely in these areas, resulting in rapid growth of the nickel-cadmium market.

NASA has been actively involved in the improvement of nickel-cadmium batteries. Mission requirements imposed constraints of reliability, predictability and life, in excess of what was commonly available. Through extensive research, development and testing, failure modes were identified, quality control procedures specified and lifetimes of battery components improved. Resulting improvements in nickel-cadmium batteries have been applied selectively in commercial applications. The significance of NASA's role in nickel-cadmium battery development has been major and should continue to be so as expanding commercial application calls for higher performance and reliability. Among the NASA contributions to the advancement of nickel-cadmium battery development are the following.

o Development of improved ceramic seals (02-12-01).

Ceramic seals have been used in many battery systems. However, seals in common use are susceptible to chemical or electrolytic attack and are subject to high stresses from internal gas pressure.

Seal design is important to nickel-cadmium battery operation because of the tendency of cells to unbalance if oxygen generated during overcharge leaks out.

A seal developed by NASA incorporates a stress relief collar, allowing the battery terminals to move, and therefore enabling it to withstand more severe stress environments. Selection of the proper brazing alloy for the ceramic-to-metal seal was an important step in this process. Because of its high ductility, a silver brazing alloy was used. NASA contractors involved in the design of this improved ceramic seal were GE, Gulton, Sonotone and Eagle-Picher.

This ceramic seal has been important in batteries used in space applications, but has not yet found widespread commercial application. The lack of commercial acceptance is due to high fabrication costs and the belief that some leakage in commercial cells may be desirable from a safety standpoint in case of battery misuse.

o Improved battery separators (02-12-02).

Separators in nickel-cadmium cells have traditionally been made of cellulose fabric. The cellulose, however, had a relatively short service life because of instability in the alkaline electrolyte. NASA-sponsored work at Electric Storage Battery, Eagle-Picher and other contractors lead to the development of improved separators. Unwoven nylon and unwoven polypropylene provided the best performance. The nylon separators have a service life of 2 to 3 years. Polypropylene separators now under test may have a service life of up to ten years.

Separator development is still actively underway. The degree to which the improved nylon and polypropylene separators have found commercial application was not determined in the study, but they have had a significant impact on battery life in space applications and should find significant commercial application if they can be produced at reasonable cost.

o Development of auxiliary electrodes for charge control (02-12-03).

Overcharging of nickel-cadmium batteries was a frequent cause of cell failure. Overcharging can result in high gas pressures due to oxygen evolution and excessive heat generation which can cause the cell to rupture. Work had been done many years ago on auxiliary electrodes to control overcharging but development never proceeded very far. A control electrode can be used to signal the onset of oxygen overpressure by measurement of electrode voltage output; while a recombination electrode can be used to recombine excess oxygen generated during charging, thereby maintaining low oxygen pressures in the cell.

NASA/GSFC did significant work on auxiliary electrodes, both in-house and with contractors such as Gulton and General Electric. Development of both types of auxiliary electrodes was accomplished and

they have been used in space applications. (A related development at Goddard for charge control, the cadmium-cadmium coulometer, has also been used in space applications for charge control.) Battery manufacturers have been making commercial cells with auxiliary charge control electrodes, but the additional expense limits wide application. The National Institute of Health has been using auxiliary electrode technology, as developed on the GE contract, in implantable batteries for heart assist devices.

o Identification of battery failure modes (02-12-04).

Failure modes and variation in performance characteristics of nickel-cadmium batteries were not well understood until recently. NASA mission requirements of reliability and predictability led to development of an extensive testing program to identify failure modes and causes of performance variations. NASA/GSFC directed the program. Major contractors were Naval Ammunition Depot in Crane, Indiana and Inland Testing Laboratories. The program was successful and has had a major impact on nickelcadmium battery development. One example of failure modes which were identified deals with failures due to use of wetting agents on separators. The NASA testing program determined that these wetting agents. widely used in the industry, were a major cause of cell failure. Goddard alerted the industry to their findings. Based upon these findings, it appears that all nickel-cadmium specifications, aerospace as well as commercial, now state that no wetting agents shall be added.

o More uniform and predictable battery performance (02-12-05).

Nickel-cadmium cells supplied for space applications exhibited substantial performance variations (about 5-10 percent capacity variations) within a manufacturing lot and even wider variations on a lot by lot basis.\* After prolonged operation, supposedly identical cells exhibited differing performance characteristics. Coupled with the failure mode and performance tests cited above, NASA sponsored

<sup>\*</sup>Bauer, Paul. <u>Batteries for Space Power Systems</u>. National Aeronautics and Space Administration Publication, SP-172 (Washington, D. C.: Government Printing Office, 1968).

the development of more uniform and predictable materials and components for use in nickel-cadmium batteries. Contractors included GE, Gulton, Tyco and many others.

Knowledge developed in these investigations was summarized in a draft specification to be used for nickel-cadmium cells. Industry people were asked to study and suggest modifications to the specification. An interim meeting was held at Goddard in 1969 with industry representatives to discuss the specifications. After a period of draft to final version, the specification for nickel-cadmium batteries was issued in 1971. At least partly because of industry involvement in specification development, this specification has found wide use outside of aerospace applications. Its application to commercial batteries has been selective, with a balance struck between required performance characteristics and cost.

## Silver-Zinc Battery Improvements (02-16)

The silver-zinc battery is a high energy-density system (per unit weight and volume) which can deliver power at high current rates and a fairly constant voltage level. The silver oxide cathode and zinc anode are used in conjunction with a potassium hydroxide electrolyte. The silver-zinc battery has been known since the 1800's, but its cost and poor cycle life limited interest until the early 1940's. Interest was stimulated at that time because of potential military applications as a primary battery, and because of the development of an improved cellophane separator for secondary battery applications.\*

Cost of the silver-zinc battery is inherently high, primarily because of the silver-based cathode. Until recently, the application of this type of cell as a secondary battery has been limited due to low cycle life and shelf life (after activation). Therefore, major applications have been in military and space (planetary and satellite) missions, where high energy-densities (25-100 watt-hour/lb.), high current rates, and level voltage discharge characteristics, offset the system's disadvantages. The battery is often used as a primary battery to meet the high current-drain, short-time requirements of missile launching. The silver-zinc cell has also been tested in prototype electric cars by Yardney and General Motors.

<sup>\*</sup>The Primary Battery, Heise and Cahoon, pp. 225-226.

Shelf life, power-density, and utilization of the zinc active material of silver-zinc batteries, has improved considerably over the last few years. Major advances include improvement of organic separators and the development of a long life inorganic separator. Improvements in zinc electrode design have contributed to development work on related batteries, such as nickel-zinc and zinc-air batteries. There have also been a large number of relatively minor incremental improvements which have contributed to increasing the overall performance of this type of battery. These include advances in case design and methods of securing electrodes and separators to withstand severe aerospace environments.

Over the last ten years, much of the development work on silver-zinc batteries was supported by NASA, with primary involvement in this effort at JPL, Lewis and Goddard. The Air Force was also interested in silver-zinc battery development in the 1960's, but not as extensively as NASA. NASA's efforts range from basic electrochemical studies, to the detailed engineering of battery systems to meet aerospace environmental requirements (e.g., extremes of shock, vibration, temperature and acceleration). The result has been significant improvement in silver-zinc battery performance. Use of these batteries appears to be primarily restricted to military and space applications; however, some of the technology developed (e.g., improved zinc anodes, inorganic separators) does have application to other battery systems with more commercial potential.

o Development of high-cycle life inorganic separators (02-16-01).

Since the introduction of cellophane as a separator in silver-zinc batteries in 1941, organic separators, both cellulosics and non-cellulosic synthetics, have received the most research attention and have generally been the most widely used type. While these separators were successful by most performance standards (e.g., preventing migration of silver compounds between the positive and negative electrodes, and providing good resistance to zinc penetration), their cycle life was limited.

In a NASA program at Astropower Laboratory, McDonnell-Douglas, a proprietary inorganic separator was developed which should increase the cycle capability of the silver-zinc battery four to ten times over present capabilities. This increase makes the silver-zinc cell economically and technologically more competitive as a secondary battery system. For example, the silver-zinc battery, which has approximately twice the energy

density of a nickel-cadmium battery, can now be considered for some high-cycle applications where nickel-cadmium batteries are now used. (See metal-air batteries, contribution 02-09-02.)

o Zinc electrode improvements (02-16-02).

Zinc electrode performance has been one of the limiting factors in silver-zinc battery life. Problems include penetration of separators by zinc dendrites (a needlelike crystalline growth), structural weakness of the zinc electrode, poor self discharge characteristics, and high solubility of zinc oxidation products in the electrolyte.

Starting in the mid-1960's, the NASA program to improve zinc electrode performance ranged from basic studies of electrode reactions to fabrication and testing of a number of alternative electrode designs. Structural changes, modifications in charging procedures, use of limited electrolyte amounts, and electrode and electrolyte additives were among the improvements developed. Major NASA contractors included Yardney, Electric Storage Battery (ESB) and Leesona-Moos Corporation.

While individual contributions represent only small incremental advances in the technology of silver-zinc batteries, the aggregate effect of these contributions is a significant improvement in zinc electrode performance. Because the zinc electrode is used in so many other cells (e.g., zinc-air, nickel-zinc), these improvements do have an impact upon the development and advancement of other battery systems. (See metal-air batteries, contribution 02-09-02.)

o Improved organic separators (02-16-03).

Organic separators have been the type most commonly used in silver-zinc batteries, and it was only recently that an apparently successful inorganic separator has been developed (see contribution 02-16-01, above).

During the mid 1960's, however, NASA devoted some effort to improving organic separators (cellulosic and noncellulosic synthetics). At NASA/GSFC, modifications in cellulosic separators extended

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battery life by a factor of 5. Goddard specified the material properties (e.g., molecular weights) which must be controlled to maintain reliability. NASA contractors working on improving organic separators included Borden, Monsanto, Yardney, ESB and Westinghouse. Results of these contractor efforts included improvements in separator life, electrical properties and ability to withstand heat sterilization (a requirement for planetary missions).

o Improved silver-zinc battery system design (02-16-04).

Application of the silver-zinc battery system to space missions required engineering to meet difficult environmental conditions (e.g., shock and acceleration loads, temperatures, pressures). NASA/GSFC and JPL, successfully developed systems meeting these requirements. NASA contractors included ESB, Eagle-Picher, and Yardney, and extensive testing of the systems was performed at Naval Ammunition Depot-Crane. Improvements were made in case design, electrode fabrication, methods of securing electrodes and sealing of the battery. While these advances were important for space and military missions, they will apparently have only a minor impact on commercial applications.

## Solid Electrolyte Batteries (02-17)

Batteries using a solid electrolyte have been studied since 1935.\* In this type of battery the solid electrolyte conducts ionic species. not electrons. The solid electrolyte battery provides long shelf life, good charge stability, ruggedness, ease of miniaturization, and wide temperature operating range. Because the electrolyte is solid, the substantial problem of electrolyte control and containment in minaturized batteries is simplified. High energy-densities can be obtained at high temperatures, but, until recently, performance at low temperature was severely limited. Solid electrolyte batteries are now being considered for use in integrated microcircuit y. The low power and amperage requirements of such circuits is consistent with lowtemperature solid electrolyte battery performance. Costs at present have been said to be an order of magnitude too high for commercial application. The very long shelf life (often 10 years or more) of such batteries is of particular interest for military applications. In addition, hightemperature, high energy-density systems could find automotive and peak power applications.

<sup>\*</sup>Ibid, p. 385.

Research on solid electrolyte batteries and related applications (e.g., capacitors) is presently underway at a number of firms, including Atomics Internation, Gould Ionics, General Electric, Mallory and Union Carbide.

There are a number of solid electrolytes in use, mostly silverbased. Prior to 1960, the most common solid electrolyte was silver iodide. However, the low conductivity of solid electrolytes at ambient temperatures hindered the development of widespread interest and use of such batteries. Efforts in Germany and Japan in the early 1960's with the silver-sulphur-iodide electrolyte system (Ag3AI) resulted in an improvement of four orders of magnitude in conductivity at ambient temperatures over the silver-iodide electrolyte. Additional research identified complex double salts of a silver halide (e.g., KAg4I5) which provided further improvement in conductivity. However, energy-densities in these batteries are still lower than in more conventional battery systems.

The major improvements in solid electrolyte conductivities, coupled with the need for miniature power sources for integrated circuitry, point to a potentially major role for solid electrolyte batteries. The advantages of long shelf life, charge stability, miniaturization and low-temperature operation provides promise of significant military applications. Use of solid electrolytes as capacitors, timers, sensors (e.g., temperature, deformation) and coulometers, in addition to application in batteries, should result in broad and significant impact of research in this area.

The U. S. Army Electronics Command and the Signal Corps have been actively involved in solid electrolyte development. NASA's efforts to advance the technology of solid electrolyte batteries have been limited, due to the low energy-densities achieved with silver-based systems. However, research carried out by NASA on organic electrolytes has had some impact on solid electrolyte development. Basic research is now being conducted by NASA to look at non-silver based materials for high-energy density solid electrolytes but it is too early to estimate the impact of such work.

o Improved understanding of organic electrolyte systems (02-17-01).

Research on the physical and electrochemical properties of a large number of organic electrolyte materials was supported by NASA/LeRC in the 1960's. The encyclopedic nature of the work has had some influence and been of value to researchers in the related field of solid electrolytes. Major contractors were Mallory, Globe Union, Rocketdyne and Whittaker. An example of the interrelatedness of solid electrolyte battery development and the NASA efforts on organic

electrolytes is Mallory's work on a lithium-iodide battery for pacemakers. Their effort has drawn, to some degree, on NASA supported research findings, on organic electrolyte lithium batteries.

Another benefit from the work was development of analytical techniques to rapidly screen materials and electrode-electrolyte systems. Some of these techniques have been employed in solid electrolyte work.

o Improved basic understanding of nonsilver-based solid electrolyte materials (02-17-02).

Basic materials research is being conducted at NASA/LeRC directed towards identification of high energy-density (nonsilver-based) solid electrolytes. In a contract with Tyco Laboratories, NASA is trying to produce single crystal tubes of beta-alumina. They hope to obtain higher beta-alumina performance at temperatures lower than the 350 C. cited as being used by Ford Motor Company in their sodium-sulphur battery. Investigations of alkali metal ion conductors, fluoride ion conductors, and other materials are also being carried out at Lewis. The objective of these efforts is to "bridge the gap" between the materials field and the electrochemical field, which has proven to be a barrier in the further development of solid electrolytes. It is too early to estimate the potential impact of this NASA effort.

#### HIGH-TEMPERATURE CERAMICS (03)

High-temperature ceramic materials can be defined as those ceramics able to resist the hostile environment of high temperature, and the corrosive effects of a reactive system. In the context of this section, we have included those materials capable of withstanding temperatures greater than 2,750 F.

Literally hundreds of ceramic materials are available today. However, the number of materials that are actually useful in truly high-temperature applications (greater than 2,750 F.) is remarkably small. These generally include: oxides, such as alumina, beryllia, chromia, magnesia and zirconia; carbides, such as silicon carbide and boron carbide; nitrides, such as boron nitride and silicon nitride; borides, such as zirconium diboride and chromium boride; silicides, such as molybdenum disilicide; intermetallic compounds, such as nickel aluminide; and graphite.

The important properties of ceramics that fit them for use at high temperatures are that they have very high melting points, and potentially they retain a useful amount of strength at temperatures well above the capability of metals. As highly oxidized compounds, ceramics are strongly resistant to attack by nearly all chemicals. This accounts for many of their uses. They are indispensible where a structural material must withstand temperatures at which rapid oxidation usually destroys the strength of a metal. In spite of their well-known advantages, ceramic materials also have some severe weaknesses: lack of ductility creates an inability to withstand mechanical or thermal shock; they can be very costly or impossible to form into complex engineering shapes; and they generally have low absolute strength levels.

Because of their potential for high-temperature aerospace, electronic and industrial applications, ceramic materials have been subjected to intensive research and development efforts to overcome their weaknesses. In the last decade, a great deal of knowledge has been accumulated about ceramic materials, their microstructure, atomic character and the theoretical limits of their properties. This knowledge indicates that certain properties can be tailored to meet specific requirements, and that special properties or combinations of properties can be substantially improved over presently achievable levels. These research efforts have resulted in vastly improved materials, as well as in faster and improved ways to form and fabricate the materials. A list of significant advancements in high-temperature ceramics, as identified by technological leaders in the field, is given in Table 3.

Technological advances in the field of high-temperature ceramics have opened up a wide variety of new uses. In addition to applications in space systems and aircraft turbine engines, additional impetus has come from more severe operating conditions in industrial processes.

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# TABLE 3 SIGNIFICANT DEVELOPMENTS IN THE FIELD OF HIGH-TEMPERATURE CERAMICS\*

- \*\* 1. Advanced processing techniques (03-01)
  - 2. Alumina thin films (03-02)
- \*\* 3. Automotive exhaust catalysts (03-03)
  - 4. Brittle materials tensile test (03-03)
- \*\* 5. Ceramic automotive turbine materials (03-05)
  - 6. Ceramic turbine blades (03-06)
  - 7. Chemical strengthening (03-07)
  - 8. Chrome-magnesite brick (03-08)
  - 9. Complex diborides (03-09)
  - 10. Ferrous metal die casting (03-10)
  - 11. Fused silica refractories (03-11)
- \*\*12. Glassy Carbon (03-12)
- 13. High-alumina refractories (03-13)
- \*\*14. High-purity raw materials (03-14)
  - 15. High-temperature carbides (03-15)
  - 16. High-temperature shaft kilns (03-16)
- \*\*17. Hot extrusion (03-17)
- \*\*18. Hot forging (03-18)
  - 19. Melt-cast ceramics (03-19)
  - 20. Microstructure design (03-20)
  - 21. Pure magnesia bricks (03-21)
- 22. Pyrolytic graphite (03-22)
- \*\*23. Rare Earth magnets (03-23)
  - 24. Shaped sapphire parts (03-24)
  - 25. Silica insulat-on (03-25)
  - 26. Tonnage sintering (03-26)
- \*\*27. Transparent ceramics (03-27)
- \*\*28. Transparent Ferroelectrics (03-28)
- \*\*29. Understanding failure mechanisms (03-29)
  - 30. Yittrium alluminum garnate (03-30)
  - 31. Zinc oxide rectifier (03-31)

<sup>\*</sup> As identified by technological leaders in the field of high-temperature ceramics.

<sup>\*\*</sup> Developments selected for identification of NASA contributions.

The materials industries themselves have difficult refractory problems. In the primary metals industry there are needs for high-temperature ceramics in refining and melting, especially when the metals have high melting points and are easily contaminated by reactions with the crucible material; and the ceramics industry requires high-temperature materials for producing and firing new ceramic products. The electronics industry has many applications requiring ceramics with good electronic properties at elevated temperatures, as well as with adequate mechanical properties. And the atomic energy program has requirements for high-temperature ceramic materials with higher purities and special nuclear properties.

Total sales of refractory materials in the U.S. in 1968 amounted to more than \$535 million.\* These materials were consumed by a wide variety of industries including iron and steel, nonferrous metals, cement, glass, ceramics, chemical, petroleum and public utilities. However, sales of truly high-temperature ceramics represent only a small fraction of total refractory sales. In addition to traditional industry applications, there is growing consumption of high-temperature ceramic materials in the aerospace, electronics, and automotive industries.

In spite of present widespread application in the traditional industries, the engineering use of high-temperature ceramic materials is still limited. Specifically, the uniformity, reproduceability, reliability and mechanical performance of ceramic materials are presently below intrinsic capability. The restrictions on the wider use of ceramics has been largely attributed to inadequate knowledge and control of ceramic processing and finishing operations, reflecting the lack of a science of ceramic processing, and to the inherent brittle nature of ceramics.\*\*

Several advanced processing techniques are directed specifically to the problems cited above. Various controlled sintering and hot working methods are now being developed including rolling, forging extrusion and pressing, as well as cold forming followed by controlled crystallization. While these types of processes are well developed in metal systems, they are still in an experimental and advanced development stage in ceramics.

<sup>\*</sup>Kusler, D. J., <u>Impact of Changing Technology on Refractories</u>
Consumption, Washington: U. S. Department of Interior, Bureau of Mines,
1970 (IC 8494).

<sup>\*\*</sup>Committee on Ceramic Processing, Materials Advisory Board, Division of Engineering, National Research Council, Ceramic Processing, Washington: National Academy of Sciences, 1968 (Publication 1576).

NASA contributions to the advancement of the field of hightemperature ceramics have dealt primarily with the development of advanced processing techniques, such as hot extrusion and hot forging; with the development of a better understanding of ceramic failure mechanisms; and with the development of new applications for ceramics in automotive gas turbines and automotive exhaust afterburners.

### Advanced Ceramic Processing Techniques (03-01)

The bulk of ceramics produced today are densified by reasonably conventional sintering procedures. The rather large firing shrinkage experienced with conventional sintering (in the range of 15-16 percent by volume) produces difficulties in producing large shapes effectively and efficiently. Another difficulty is the lack of uniformity in shrinkage which is associated with dimensional variations and flaws in fired ceramics. Variations in porosity and pore anisotropy resulting from inadequate control in the forming process also account for many of the difficulties in using ceramic materials.

Various advanced forming processes have been successfully employed in the fabrication of ceramic materials for achievement of microstructure control, enhanced densification, composite fabrication and pressure bonding, which are not normally obtained by conventional sintering techniques. Developments in techniques such as hot forming, hot extrusion, hot swaging and hot forging, have brought about a fundamental change in the properties and types of ceramics that are available. Ceramic processing is moving away from the dry pressedsintered, or wet slipcast approaches to forming. These approaches have been historical in nature and a whole new range of processing techniques have been developed which relate directly to the deformation of the material and its final properties.

Several advanced processing techniques are directed specifically to the problems of preferred orientation in terms of phase, grain, and pore structure. The implications of preferred orientation in ceramic character have far-reaching significance in the general area of structural ceramics, as has already been accomplished in electronics and magnetics. Various methods are now available for accomplishing preferred orientation, e.g., controlled sintering and hot working processes such as rolling, forging, extrusion, and pressing. The full potential of many advanced processing techniques have not been exploited mainly because of the economic disadvantages relative to other forming processes. Primary commercial usage of these processes has been concerned with the preparation of refractory materials that are too difficult to densify by normal sintering procedures.

In recent years, those who work with ceramic materials have begun to conceptualize them in a way similar to plastics and metals. That is, ceramics have been integrated into the general field of materials

science. This type of thinking dates to the emergence of basic research on ceramic materials and changes in the educational process for ceramic scientists and engineers. This has brought about a change in thinking which has led to the development of a new set of advanced processing techniques.

These changes are significant because ceramic materials formed with processes that don't use traditional powder-sintering or wet slipcast approaches do not have shrinkages normally associated with ceramic materials. Components can be formed at close to desired sizes and close tolerance can be held. All the new processes are essentially deformation-type techniques, which do not make use of water and represent a fundamental change in ceramic processing. Based upon this, the potential for the use of ceramic materials in terms of property changes, such as microstructure control, is far greater than ever before.

NASA contributions to advancing the technology of new ceramic forming techniques are extensive and include major efforts to: (1) develop a better fundamental understanding of the relationship between processing and ceramic properties; (2) improve hot pressing techniques; (3) develop hot extrusion and hot forging into workable ceramic fabrication processes; and (4) develop fabrication and post-fabrication techniques for improving the mechanical behavior of ceramic materials. Among NASA's specific contributions to this development are the following.

o Development of hot-pressing techniques for very hightemperature ceramic materials (03-01-01).

> The melting point of hafnium carbide (HfC), 7030 F., is the highest reported for any metal or simple metal compound. Because of this HfC is potentially useful for structures to operate at very high temperatures. However, the best ceramic bodies produced out of HfC had on the order of 20 percent porosity, which indicated they were quite weak and not near the strength capabilities of the material. To fully investigate the potential of HfC as a high-temperature material, NASA/LeRC undertook a program to determine the effects of the hot-pressing variables of temperature, pressure and time, on the density and grain size of HfC. Result was HfC bodies of approximately 98 percent theoretical density, produced by hot pressing in a hydraulic press at pressures of 1,000 to 6.030 psi. and an inductively heated graphite die assembly at temperatures of 3,500 to 4,870 F.

This effort advanced the technology of hot pressing to a level it had not reached at that time (1960) and demonstrated that hot pressing could be used to form materials that could not be made any other way.

o Development of hot-pressing techniques for the fabrication of high-purity polycrystalline magnesia (03-01-02).

A fabrication technique for hot isostatic pressing of high-purity magnesia powders into 90 to 95 percent dense polycrystalline specimens was developed by NASA/JPL. The high-purity magnesia fabricated by this hot-pressing technique demonstrated grain growth approximately 100 times that found in magnesia of conventionally good purity. In addition, there was no evidence of reheat porosity in the pure magnesia at very high temperatures, as is found in less pure materials. The general technique appears to be useable for the fabrication of other finely divided powders.

o Increased understanding of effect of advanced processing on ceramic properties (03-01-03).

A series of NASA-supported studies on advanced processing techniques for ceramic materials has led to an increased understanding of the effect of these techniques on materials properties. These studies, carried out at the University of Washington, included correlations of characteristics of particles and agglomorates with ceramic forming processes and subsequent product properties, and the effects of processing variables on the micro and macrostructures of ceramic materials.

o Development of process for the hot extrusion of carbides (03-01-04).

The first process to successfully hot extrude very high-temperature ceramic materials, such as titanium carbide, was developed at NASA/LeRC in the early 1960's. Development of this process was based upon refractory metals extrusion technology developed at Lewis (see NASA contributions to the advancement of refractories metal technology in the field of high-temperature metals). In this process, titanium carbide powders are placed in

tungstum metal cans and compressed with a plunger to essentially 100 percent theoretical density before extrusion, so that extrusion step deals with a solid materials rather than voids.

Result of this process is a material with a cold-worked microstructure and preferred orientation (elongated grains). This NASA effort represents one of the first successful attempts to apply primary metal working techniques to ceramic materials and is the forerunner to subsequent work on the development of optimum parameters for hot extrusion of materials such as magnesia (see NASA contributions 03-01-05, below).

o Identification of optimum parameters for hot extruding ceramic oxides (03-01-05).

Under a NASA contract, Boeing Company identified the optimum parameters for hot extrusion of ceramic oxides and developed a process for hot extruding crack-free magnesia. A major part of this effort was determination of the effect of such advanced processing on material properties. The process entails extruding oxide materials in thick-walled cans using conventional extrusion techniques. A variety of shapes are possible, including square billets in a round can extruded through a round die; round billets in a round can extruded through a square die; billets extruded side-by-side and off-set from the can axis; and extrusion of an insulating can with a ceramic core (alternate metal and ceramic layers).

This effort developed basic information about the relationship of material strength to texturing, grain elongation, recrystallization and grain growth. Magnesia produced by this hot extrusion process exhibited 50 percent or more greater strength than hot-pressed magnesia of equivalent grain size. This increase in strength is due to decreased misorientation of grains by axial texturing. For a description of the effect of hot extrusion on fracture nucleation and propagation, see NASA contributions to better understanding of ceramic failure mechanisms, 03-29-01.

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o Development of a process for the high-temperature hydrostatic extrusion of ceramics (03-01-06).

A process for the high-temperature hydrostatic extrusion of ceramics, using solf solid or molten salts, was developed by Boeing and Nuclear Metals under a NASA contract. The process entails the use of a solid as the hydrostatic medium. The solid should be much softer than the ceramic material which is being extruded, such as calcium fluoride. The ceramic to be extruded is placed as the core in a doublewalled thin metal can. The interwall space is filled with a ceramic powder of the same composition as that in the core to provide insulation and service as an additional barrier against a leak in the outer wall of the can. The can is then placed in an extrusion press liner filled with the soft solid and extruded using conventional techniques. This process allows the extrusion of ceramic materials, such as calcia, at temperatures from 500 to 800 F. lower than that needed for extrusion in a thick-walled can.

o Development of forging processes for refractory ceramic oxides (03-01-07).

Forging processes for refractory metal oxides are now being developed at Avco under NASA sponsorship. This effort, to date, indicates that the deformation involved in the forging process strain-hardens the ceramic materials. The fracture process seems highly dependent on attainment of a critical stress, though prior strain at lower stresses may also have an effect. There is also evidence of strain-enhanced grain growth which may be responsible for the strain hardening. The most hopeful result is extensive evidence of grain boundary sliding. The compressure surfaces revealed more deformation features than tensile surfaces, which suggests dislocation motion.

The results of this effort support the view that there is sufficient ductility in fine-grained alumina to allow forging at moderate temperatures. o Development of a technique for increasing mechanical strength of brittle materials by inducing surface compressure stresses (03-01-08).

Various forming and post-forming techniques are used to develop specific mechanical properties in ceramic materials. Since fracture in ceramic materials is often initiated by tensile stresses on the surface, the introduction of compressure stresses in a fabrication technique can be used to inhibit crack initiation and propagation. This technique of inhibiting crack initiation is now used with alumina by controlled heating and cooling. Under a NASA contract, Ceramic Finishing Company is taking a different approach to developing surface compressive stresses. Their approach is to induce the stresses by capitalizing on the differences in thermal expansion coefficients in two-phase structures (see NASA contributions to the application of ceramics in automobiles, 03-05-02).

### Automotive Applications for High-Temperature Ceramics (03-05)

Increasing concern about the adverse environmental effects of the internal combustion engine has made present automobile design the subject of intense research and development efforts. These efforts have taken two main thrusts: (1) replacement of the internal combustion engine with some lesser polluting type of engine, such as the gas turbine; and (2) reducing the amount of pollutants in the exhaust of the internal combustion engine by some sort of automotive exhaust reactor (afterburners).

Both of these approaches are close to commercial reality. For example, Ford Motor Company recently announced they will offer turbines for trucks and buses in the 1970's, and several nonexperimental turbine-powered vehicles produced by other companies are already on the road. In terms of internal combustion engines, the U. S. Environmental Protection Agency has already set emission standards on future automobiles that will almost certainly require afterburners. Both the turbine and the afterburner approach require high-temperature materials capable of withstanding severe thermal shock.

While ceramics offer unique high-temperature properties they have not found large-scale application in aircraft turbine engines due to their inherent brittleness and relatively poor thermal shock resistance. Reliability levels for modern commercial and military aircraft are too high to allow use of presently available ceramic materials in such critical parts as turbine blades. Because of this, operating temperatures of gas turbines are limited by the properties of available

high-temperature metals. Since turbine efficiency is directly related to operating temperature, engine designers have tried a variety of cooling techniques to raise allowable use temperatures of metals and coatings. These techniques often entail expensive fabrication steps and tend to reduce overall engine efficiency. (For a full description of this problem and the various metals which are finding application in jet engines, see the fields of high-temperature metals and internal gas dynamics.)

Since turbine engines in buses, trucks and automobiles do not need to meet the same stringent reliability standards as found in aircraft, ceramic materials offer a promising alternative to high-temperature alloys. By using ceramic materials for parts such as turbine blades, the operating temperatures, and thereby operating efficiencies, can be increased to the point where automotive turbines can be made economically very attractive.

In addition to the use of ceramics in the turbine engine itself, there are good prospects for the use of ceramics in turbine-engine regenerators. Regenerators are used to take heat from exhaust gases and pass it to incoming air, thereby increasing the turbine's efficiency and fuel economy. The heat exchanger's temperature is too high for most ordinary iron-base metals (not superalloys) but is safe for ceramic regenerators which can operate to 2,000 F. and higher. Corning Glass Works is already manufacturing a glass-ceramic regenerator for production model gas turbine engines and the Ford gas turbine will use two glass-ceramic rotary regenerators each.

In the improvement of internal combustion engines, ceramics may be the most likely materials for use in automotive afterburners to prevent air pollution. Ceramics offer automotive exhaust reactors the advantages of low cost, excellent oxidation resistance and high-temperature capabilities. As in turbine use, the brittle nature of most ceramics will require the development of either new or modified ceramic materials, or special designs to compensate for brittleness.

NASA's contribution to advancing the application of ceramic materials in automobiles is manyfold: NASA has been involved in surveying the materials problems associated with turbine or afterburner use; they have screened and critically evaluated existing materials; they are involved in improving existing materials; and they are developing special designs to meet turbine and afterburner needs. Specific NASA contributions follow.

o Evaluation of ceramic materials for use in automotive turbine engines (03-05-01).

While numerous ceramic materials appear to be candidates for use in automotive gas turbine engines, they have never been critically evaluated

and compared in consistent and realistic tests for this type of application. NASA/LeRC carried out a major screening program on 18 to 20 different ceramic materials, including oxides, nitrides, borides and carbides. Testing was done in a simulated turbine environment with the materials exposed to high gas velocity, high temperature and severe thermal shock. Based upon these tests, silicon carbide and silicon nitride appear to be the most promising ceramic materials for automotive turbine applications. They exhibit excellent thermal shock and oxidation resistance but have a continuing problem with brittleness. Several research programs have been planned and are now being sponsored by NASA to reduce or circumvent the brittleness problem by -both-material-and-physical-changes-(see-NASAcontribution 03-05-02, below).

o Development of improved ceramic materials for automotive turbine applications (03-05-02).

Based upon information developed in an initial materials screening process (NASA contribution 03-05-01, above) NASA/LeRC is now attempting to improve silicon carbide and silicon nitride materials for automotive turbine applications. Two general techniques are being developed by industrial firms under NASA contract: (1) by making composites; and (2) by developing surface compressure stresses.

In the first approach, Avco is attempting to improve strength, and impact energy absorption, and to inhibit crack propagation in both silicon carbide and silicon nitride by developing a limited second phase in monolithic pieces of the main materials.

In the second approach, Ceramic Finishing Company of State College, Pennsylvania is attempting to develop compressure surface stresses by capitalizing on the differences in thermal expansion coefficients in two-phase structures. Since fracture in ceramic materials is often initiated by tensile stresses on the surface, the introduction of compressive stresses can be used to inhibit crack initiation and propagation. This technique of inhibiting crack initiation is now used with alumina, a single-phase material, by controlled heating and cooling of the surface and the core.

o Determination of automotive exhaust reactor material needs (03-05-03).

Because of NASA's extensive work with alloys and ceramics for applications in the 1,800 to 1,900 F. range, the National Air Pollution and Control Administration (now the Environmental Protection Agency) asked NASA/LeRC to analyze the materials problems associated with automotive exhaust reactors. Lewis determined that ceramics may be the most likely material because of the advantages of low cost, excellent oxidation resistance and high-temperature capability. Based upon this effort, Lewis is now involved in designing and testing a workable reactor (see NASA contribution 03-05-04, below).

o Design of an automotive thermal reactor using ceramic materials (03-05-04).

NASA/LeRC has designed and built a prototype automotive thermal reactor for pollution control which comes very close to meeting EPA 1975 standards for carbon monoxide and hydrocarbons. Several ceramic materials have the necessary thermal shock and oxidation resistance, but mechanical shock is still a problem. Major candidates for use in the reactor are glass-ceramics, and modifications of silicon nitride and silicon carbide (see NASA contribution 03-05-02, above). The design is unique in that it places all of the ceramic parts compression. Several ceramic companies are now making the hardware to meet Lewis specifications and drawings.

The Lewis reactor has been extensively tested on an engine test stand and has more than 1,200 miles of actual road testing in an automobile. The design is presently being modified by developing a honeycomb ceramic material for future reactor designs.

#### Glassy Carbon (03-12)

Glassy carbon is a material that has the same chemical and physical properties as glassy materials, such as chemical inertness and zero porosity, but is made of carbon. It is manufactured by decomposing polymeric materials under controlled conditions. One recent advance in

the use of glassy carbon has been to manufacture it as a glaze which is then applied to graphite or an oxide material with which it is compatible. As a glaze it can be used to coat a reinforced graphite material, which therefore extends the usage of glassy carbon far beyond its original strength-limited applications.

Glassy carbon is innocuous in the body and therefore can be used as a possible replacement for bones. The present glassy carbons are still only used in laboratory experimentation. As a glaze to coat reinforced graphite or oxide ceramics, it has the potential for use in pipes and related types of processing equipment because of its unique insulating properties and chemical inertness.

The original invention of glassy carbon occurred in Japan in the late 1950's. It was first sold by the Japanese as a glassy carbon crucible. A glassy carbon glaze can find widespread use in medical areas to coat bone replacements. In addition, it has potential as an impervious coating on graphite which, for the first time, would take advantage of the high-temperature properties of graphite without the disadvantages that graphite has in terms of porosity, and corrosion resistance.

NASA has contributed to the advancement of this development by developing and compiling basic data and by actively working with medical researchers in developing new medical applications. Specific contributions follow.

o Developed engineering and design data on glassy carbon (03-12-01).

In a series of research programs at NASA/LeRC and NASA/JPL selected engineering and design data on glassy carbon was developed, evaluated and compiled. Data included: ablation performance in different environments; magnetic susceptibility as a function of magnetic field strength, heat treatment and high-temperature tensile deformation; and tensile creep behavior. This data should assist others in developing applications for glassy carbon.

o Development of biocompatible glassy carbons (03-12-02).

High-strength glassy carbon forms, developed by North American Rockwell under a NASA contract, have been found to be chemically, biologically and physically compatible with fluids and tissues in the human body. In addition to their high strength and long-term biocompatibility, glassy carbon can be fabricated easily into complex shapes and may be sterilized by several of the presently used techniques. Because of these characteristics, these materials are now being evaluated as surgical impants, as implantable splints, epithelial bone extensions and replacement heart valves. Substitution for previously used metals may allow implants to be used much more extensively and safely.

#### Availability and Control of High-Purity Ceramic Raw Materials (03-14)

Impurities in ceramic materials have a profound affect on final characteristics, especially in very-high-purity and ultra-high-purity compositions. Impurities control and influence grain boundaries, phase boundaries and nonstoichoimetry. The mutual dependence of both processing and properties on the chemical and particulate character of the raw materials used, has been well understood for some time. Over the past decade, there has been increased awareness of the need to control the character of the ceramic raw materials with the over-all objective of determining the limits to which final ceramic materials properties can be controlled using purity materials. In the past, buyers tended to purchase what the producer was selling. Ceramic users have become much more concerned about raw materials, particularly such items as chemical composition and grain size distribution. This emphasis on control of purity has brought about an attendant increase in the ceramic material properties, and consequently, has led to the much more widespread availability of high-purity raw materials. In reality, the emphasis is more on controlled purity than on higher purity.

The change that occurred in the ceramic field, when it started to move from an art into a science, placed a greater demand upon control of purity of raw materials. Research investigators needed high-purity materials to explore the relationship of the various ingredients to the properties and structure. Use of increasing amounts of high-purity raw materials by researchers made such materials more available on a larger scale, and these materials have eventually found their way into the fabrication of many commercial items. Demand generated by various electronics and aerospace companies and the type of research carried out, led to an increased emphasis on the raw materials. For example, the only way to develop certain specific electronic properties in a ceramic material is through the use of controlled high-purity ingredients, such as in the ruby laser which is achieved through the use of high-purity raw materials.

High purity of raw materials allow desired properties and structure to be specified and developed. While many other technologies are necessary for structure and property development it still remains that such developments could not be done without the availability of high-

purity raw materials. The control and use of high-purity materials should be instrumental in reducing the variability often found in the strength properties of ceramics. This variability often forces ceramic users to overdesign since they cannot be sure of strength minimums. Emphasis on control has had an impact upon the supplier and a potentially greater impact on all users of ceramic materials. A large increase in ceramic material properties has been brought about with little change in the materials themselves.

While this development is of profound importance to the entire field of high-temperature ceramics, it is difficult if not impossible to identify specific contributions to advancing the technology and availability of high-purity raw materials. Demand for pure materials, created by ceramics research supported by NASA, DOD and the AEC, has probably been the most important ingredient in advancing the technology to its present state. This advance has been evolutionary and cumulative, with no single input from any one agency readily recognizable or separatable from the inputs of the others.

#### Hot Extrusion (03-17)

The technology for extruding metals and polymers has been well developed over the past 25 years. Until recently, ceramic materials could only be extruded at room temperature as a preconsolidation process, and then diversified by hot pressing or firing at high temperatures. Recent advances in the technology of the process, however, have led to the hot extrusion of several ceramic materials.

Hot extrusion of ceramics is limited to elongated shapes, usually metal clad. In nearly all cases, the ceramic is encased in a metal can prior to extrusion. The entire assembly is heated and extruded. Very dense ceramics with elongated grains result.

Usually, ceramics must be heated above their conventional fabricating temperatures for extrusion. However, if the ceramic has some ductility, processing temperatures may be lower. Ceramics that have been extruded include, among others, titanium carbide, uranium dioxide, alumina, magnesia, and beryllia.

Hot extrusion has posed serious problems on extrusion die materials. High pressures required usually necessitate the material to be extruded to be preheated and rapidly placed in a relatively cold extrusion chamber and extruded through relatively cold dies. What effect this has on material response is generally unknown and may be a limiting factor in character uniformity. Major problems with hot extrusion are associated with the lack of high temperature extrusion dies and the inability to characterize the material response.

Much of the present technology in the hot extrusion of ceramics is based upon experience gained with metals and ceramic hot pressing, and basic research into sintering and grain growth.

One of the largest elements of cost associated with ceramic materials is the difficulty in shaping and forming finished parts. These costs, in many cases, may be excessive. Development of a successful hot extrusion process presents the promise of being able to form sophisticated ceramic parts much less expensively, just as metals and plastics are inexpensively and routinely extruded today.

Some of the most important work in advancing the technology of hot extrusion has been supported by NASA, particularly the early development of processes for hot extrusion and the identification of optimum parameters for hot extruding refractory oxides. These contributions follow.

o Development of a process for the hot extrusion of carbides (03-17-01).

The first process to successfully hot extrude high-temperature ceramic materials, such as carbides, was developed at NASA/LeRC in the early 1960's. For description of this process, see NASA contributions to advanced ceramic processing techniques, 03-01-04.

o Identification of optimum parameters for hot extruding ceramic oxides (03-17-02).

For a discussion of NASA-supported efforts to identify the optimum parameters for hot extrusion, and the effect of such a forming process on final materials properties, see NASA contributions to advanced ceramic process techniques, 03-01-05.

o Development of a process for the high-temperature hydrostatic extrusion of ceramics (03-17-03).

A process to hydrostatically extrude ceramics using soft solids as the hydrostatic medium was developed by Boeing and Nuclear Metals under a NASA contract. For a discussion of this process see NASA contributions to advanced ceramic processing techniques, 03-01-06.

#### Hot Forging (03-18)

The process of hot forging of ceramics, which is in the early stages of development, has been successfully applied to both densify and shape high-temperature refractory materials: uranium dioxide-molybdenum composites, friction materials in which ceramics are dispersed in metallic matrices, yttria, and alumina, are some examples of materials on which the process has been demonstrated. In recent efforts, hot forging has demonstrated the feasibility of producing preferred orientation in the microstructure for 99.9 percent pure alumina and magnesia. Forging conditions vary from high temperature (3,275 F.) and low pressure (6,000 psi.) to low temperature (2,200 F.) and high pressure (100,000 psi.).

As with many other advanced ceramic fabrication techniques, the potential inherent in forging is in obtaining properties that cannot normally be attained by conventional sintering techniques. Hot forging has been shown to improve the mechanical and physical properties of ceramic materials, including improved density, controlled levels of porosity, enhanced grain growth, improved grain orientation, higher strength and increased transmittance and transparency.

The full potential of hot forging has not yet been exploited, mainly due to its early stage of development and the favorable economics of other forming processes. However, it appears that many intractable ceramics might be formed by this process.

While several parties have supported research and development to bring the hot forging process to successful realization, including DOD efforts to fabricate ceramic armor, NASA contributions over the past few years have been particularly important in helping advance the technology to the point where it is close to application for refractory ceramic oxides such as alumina. Most important of these contributions is the NASA-supported work now underway at Avco.

o Development of forging processes for refractory ceramic oxides (03-18-01).

Forging processes for refractory metal oxides are now being developed at Avco under NASA sponsorship. For a description of this effort, see NASA contributions to advanced ceramic processing techniques, 03-01-07.

# Rare Earth Permanent Magnets (03-23)

Rare earth magnetic materials with extremely high maximum energy-products (BH) are now being made out of materials such as cerium-cobalt, samarium-cobalt and praesidium-cobalt. The cerium and samarium rare

earth magnets are now commercially available and have BH products ranging all the way up to values of 23 million as opposed to the 1 to 10 million found with standard magnetic materials such as ferrites and Alnico. The outstanding characteristic of the samarium-cobalt compositions is their exceedingly high intrinsic coercive force (resistance to demagnitization). They have a very high maximum energy product (23) and a high Curie (transition) temperature -- about 1375 F. While the cerium-cobalt compositions have a much lower maximum energy product, they are now being produced in isotropic form, that is, full use of magnetic capabilities regardless of direction of magnetization.

The rare earth high-energy magnets are made through standard ceramic powder processing techniques, as well as by melting and casting. Properties are processing sensitive but are also directly related to the composition.

There has long been an obvious need in this field for smaller, higher strength magnets. Work done on the crystallographic properties of materials led to the present developments in rare earth magnets. Preliminary work on the properties of powders was supported by the Air Force, and Raytheon did some of the basic work on sintering and grain size control of the materials.

The new rare earth magnets are so strong that they should permit substantial reductions in the size of permanent magnet motors and other products that normally use permanent magnets -- latching devices, electron beam focusing systems, loudspeakers, etc. Rare earth magnets also promise to open up a whole new range of applications where magnet size as well as strength is critical, such as medical applications. For example, Maimonedes Medical Center is testing the use of samarium-cobalt magnets as a way to overcome ptosis (drooping of the eyelid). This set of property changes will allow engineers to redesign such items as electric motors which are now designed around the problems of packing magnets into the motor. Greatly increased magnetic properties will allow significant changes in design as well as cost.

NASA contributions to advancing and developing the technology of rare earth permanent magnets are limited and fall mainly into properties determination at NASA/LeRC and the start of a basic research program.

## Transparent Ceramics (03-27)

Recent developments in high-density sintering and hot pressing are producing optically transparent ceramics that retain all the characteristic properties of ceramic materials. Transparent magnesium oxide (MgO) for example has been fabricated by hot pressing at 4,000 psi. and maximum temperature of 2,000 F. To enhance density, materials such as lithium fluoride are often added before processing. Such additives aid in eliminating porosity which is the major cause of loss of transparency in the final materials.

Particle size as well as purity in the starting powder plays a significant role in the properties of the final product. Particle size is important in both the compactability of the final density of the material as well as its strength. Strength is related to final grain size — the smaller the grain, the stronger the compact. Since some grain growth takes place during hot pressing it is advantageous to start with relatively fine powder and toadjust pressing conditions to keep grain growth to a minimum.

In addition to magnesium oxide, transparent ceramics have been made out of such materials as aluminum oxide, zinc sulfide, cadmium sulfide and yttrium oxide, among others.

To achieve optical transparency in ceramic materials, it was necessary to discover how to sinter powder compacts to full density. Research to develop a better understanding of sintering processes, as well as work on new forming processes such as hot pressing, led to the development of transparent ceramics. The first commercially available transparent ceramic was an alumina produced by General Electric and sold under the trade name of Lucalux.

The major properties which make transparent ceramics of interest are their high infrared transmittance over a broad spectral bandwidth. This broad optical bandwidth makes these materials applicable for missile domes, cryostat windows, spectrophotometer windows, radiometer optics, coder discs, and filter substrates. Many important commercial instruments now in the design stages such as infrared plant stream analyzers, clear air turbulence detection systems, and intrusion detectors, will need the broad bandwidth infrared transmittance property of transparent ceramic materials. Lenses are also a likely application because today's glass lenses cannot withstand high temperatures and available refractory materials don't have the necessary optical properties. In addition, another major advantage transparent ceramics have for lens applications is that they can be pressed directly into a lens shape with no waste, reducing manufacturing costs.

While NASA has made several peripheral contributions to advancing the technology of transparent ceramics, such as efforts in hot pressing, understanding grain boundaries and sintering, there does not appear to be any readily identifiable direct contributions.

# Transparent Ferroelectric Ceramics (03-28)

Transparent ferroelectric ceramics exhibit the unusual properties of electrically variable birefringence, the key to their potential as display and memory devices. When an electric field is applied, the ceramic extracts color from a beam of white light projected through it. Since the transmitted color varies with the applied voltage, an image can be created by establishing a "mosaic" of voltages across the ceramic.

Once the field is established, the voltage can be removed and the material will continue to produce the image until a counter voltage is applied. Thus the ceramic imaging system has a memory.

Formulations such as a lanthanum-doped mixture of lead zirconate and lead titanate (PLZT) are hot-pressed materials that have a variety of electro-optic properties. These materials have two refractive indices (bi-refringent materials) depending on the relative amounts of materials used in the final product and the hot pressing parameters used. In the processes used, microscopic porosity is decreased by hot pressing in an oxygen environment. Residual pores that form during the hot pressing process then contain only oxygen which easily diffuses through the PLZT lattice.

The general process for making materials is as follows: slurried PLZT is oven dried and granulated then dry pressed at about 5,000 psi.; the pressed slug is heated and pressed simultaneously in a flowing oxygen atmosphere for several hours; finally, a core is drilled from the slug and is sliced and polished.

Work on transparent ferroelectric ceramics is directly related to basic work done on transparent ceramics (see development 03-27, above) and basic knowledge of the sintering process.

In transparent ferroelectric materials, the electro-optical behavior is a primary characteristic and their application will be in electro-optic devices such as multi-colored computer displays, computer memories with increased storage densities, light modulators and shutters and spectral filters. In addition to have a "memory," ferroelectric ceramics can also be formulated for binary rather than analog behavior; that is, they can be made to change from transparent to opaque when a voltage is applied. These materials have great promise in imaging devices, data displays and computer memories.

As in the case of transparent ceramics, NASA does not appear to have made direct contributions to advancing the technology of transparent ferroelectric ceramic materials.

## Better Understanding of Failure Mechanisms (03-29)

A wide range of advances have been made in understanding how brittle materials such as ceramics fail, and how this knowledge can be incorporated into the design of new ceramic materials in various types of applications.

Fracture is a structure sensitive property. It depends on the particular specimens studied and their history. The first explanation of brittle material failure is due to Griffith, who pointed out that a

crack could act as a stress concentrator so that the stress at the tip of a crack can be very much larger than the nominal stress on the specimen. And secondly, that a crack will propagate if the elastic energy released by its passage is greater than the surface energy needed to extend it.

Cracks can be introduced very easily into ceramics by surface damage; cracks can also be generated internally during plastic deformation. Porosity can reduce the strength of ceramic pieces very markedly and experimental results can often be shown to demonstrate an exponential dependence on porosity. The strength of ceramics increases with decrease in grain size and porosity but the effects are difficult to study in detail because of the large number of other complicating factors.

As a consequence of the random nature of the size and position of cracks in any real ceramics specimen, it's strength is somewhat uncertain. A statistical distribution of strength of nominally identical samples is thus observed and practiced. At present, researchers are close to understanding what a micro-crack really is, through a better understanding of the role grain boundaries play in brittle failures. Recent work has related the relationship of total stored energy in brittle materials such as ceramics, to the initiation and propagation of cracks; and new testing methods have been developed for evaluating the strength of brittle materials.

Grain boundary, micro-crack and fracture mechanics work done over the past 30 years is now starting to find application. For years there has been extensive work in the field dating as far back as the theories of Griffith and Weibel. Most of the earlier work dealt with the concept of the "weakest link" which explained the initiation of cracks, but recent work has emphasized the propagation of cracks.

Understanding failure mechanisms in ceramics will aid designers in two ways: (1) many design problems will be alleviated in knowing how to design with brittle materials to avoid failure; and (2) new ceramic materials will be developed which will avoid the problems of failure through proper control of microstructure. NASA contributions to the advancement of this understanding include the following, among others.

o Increased understanding of fracture nucleation and propagation in magnesia (03-29-01).

NASA-supported efforts at Boeing to determine the effect of hot extrusion on materials properties dealt with the relationship of strength to texturing, grain elongation, recrystallization and grain growth. Analysis of the strength of extruded materials provided information on fracture nucleation and

propagation and revealed the preference of fracture for texture directions. This effort also indicated that crack nucleation by dislocation pile-up at grain boundaries occurs in extruded magnesia and calcia and is probably the controlling factor in the strength of all dense magnesia and calcia of adequate purity and flaw content. For a description of the hot extrusion process, see NASA contributions to advanced ceramic processing techniques, 03-01-05.

o Identification of protective residual stresses in hot-extruded magnesia (03-29-02).

In the course of determining optimum parameters for hot extrusion, Boeing, under a NASA contract, found an increasing incidence of surface fracture origins in extruded magnesia at both elevated temperatures and room temperature after annealing or chemical polishing. Further testing of the effect of annealing revealed that extruded bodies, especially those without cracks, had residual stresses and that chemical polishing or annealing reduced these stresses. This indicates that work hardening of surface magnesia grains occurs during extrusion and that this protects the surface from minor flaws. For a description of the hot extrusion process, see NASA contributions to advanced ceramic processing techniques, 03-01-05.

o Advanced understanding of the effect of grain boundaries on mechanical behavior in polycrystalline ceramics (03-29-03).

Understanding of the role of grain boundaries in determining the mechanical behavior and strength of polycrystalline ceramics was advanced by research at NASA/JPL. Research was conducted to (1) directly observe the atomic structure and microstructure of the grain boundary in a typical polycrystalline ceramic (magnesia); (2) determine the chemical nature of the grain boundary; (3) develop suitable techniques for production and characterization of specimens; and (4) develop suitable mechanical test data and facilities.

o Development of a new theory of brittle fracture (03-29-04).

A new theory of how brittle materials fail was developed at NASA/JPL. This theory contends that, while crack initiation can be explained in terms of the weakest link concept, crack propagation depends on total strain energy. In all cases, previous explanations are either inadequate or incorrect.

For example, traditional explanations of fracture assume that nominal stress stays the same during crack propagation. But this happens only if the drop in total strain energy caused by crack propagation is negligible compared to the total strain energy stored in a specimen and testing machine. This condition prevails in large parts and specimens where strength is governed by the weakest link theory and size effects follow the statistical distribution of flaws.

However, a different situation prevails if the part/specimen is sufficiently small so that the decrease in strain energy during crack propagation is a significant fraction of initial strain energy. Here the nominal stress will decrease during crack propagation and a consequent tendency toward crack propagation and arrest. Although crack initiation in such small specimens agrees with the weakest link theory and its associated statistical size effects, crack propagation and strength are dictated by total available energy. Thus, since there appears to be a sharp transition between the behavior of small and large parts, this NASA-developed theory proposes that "transition size" be used as a material parameter.

o Identified role of grain boundaries in the hightemperature plastic deformation of brittle materials (03-29-05).

In the course of determining the hightemperature mechanical properties of polycrystalline high-temperature carbides (hafnium carbide and hafnium carbide containing 13volume percent hafnium diboride as a second phase) NASA/LeRC identified the mode of hightemperature plastic deformation as grainboundary sliding rather than lattice slip. When grain-boundary sliding is operable in a brittle material such as hafnium carbide, strength increases with increasing temperature. This is believed to be due to the stress-relieving action brought about by grain-boundary sliding. By grain-boundary sliding, stress concentrations can be relieved, preventing premature failure and thus allowing higher applied stresses to be reached before fracture.

o Development of a technique for determining the mechanical properties of ceramic materials (03-29-06).

In order to develop better data, and a more advanced understanding of how ceramic materials fail, NASA has supported development of several new testing techniques and machines. Included, among others, are the following.

A technique was developed at the University of Kentucky, under a NASA grant for the controlled propagation of cracks in a ceramic specimen with simultaneous observation of the path of the crack and the force changes. Crack behavior may be observed using optical microscopy, scanning electron microscopy and transmission electron microscopy. This technique can be used as a research tool to develop a better understanding of the interaction of cracks with ceramic microstructure.

In another instance, a machine for the creep testing of polycrystalline ceramic materials was designed and built by Stanford University under a NASA grant. This machine can be used to test cylindrical samples in compression at constant stress in controlled atmospheres up to 3,100 F. This machine can be used to develop basic data on the high-temperature mechanical properties of ceramic materials.

#### HIGH-TEMPERATURE METALS (04)

For many years, alloys high in nickel, chromium, and cobalt were known and used in the chemical industry. During World War II, an increased demand for alloys to withstand higher temperatures for such applications as exhaust superchargers in aircraft engines, resulted in the development of what are today known as high-temperature metals. These are broadly classified as superalloys (iron, cobalt or nickelbase, depending upon the predominating alloying element) or refractory metal alloys (columbium, molybdenum, tantalium and tungsten). Some of these metals are used in the cast state, others are wrought, and still others are fabricated through powder techniques.

While the term high-temperature service implies a wide range of different thermal conditions, a minimum temperature of 1,200 F. has been chosen for the alloys discussed in this section. Above this point, conventional alloys are either not applicable or they have serious limitations in mechanical properties. Between 1,200 and 2,200 F. the superalloys find service, and above 2,200 F. the refractory metal alloys are used.

The superalloys are readily available in the commercial market-place in all mill forms and can be formed, riveted, brazed and welded using essentially standard techniques. These alloys have good oxidation and corrosion resistance, although protective coatings are often required. The refractory metal alloys retain their useful strength from 3,300 F. (columbium) to over 4,000 F. (tungsten). They are generally difficult to fabricate and all require surface protection due to catastrophic oxidation at high temperatures.

Much of the recent impetus for the advancement of high-temperature metal technology has come from the design demands of high-performance aircraft gas turbine engines, due to the use of increasingly higher turbine inlet gas temperatures. This has generated a need for improved high-temperature alloys for engine components such as stator vanes, turbine buckets and discs, transition ducts, combustion liners and compressor components. Depending upon the engine component, alloys must operate at temperatures ranging between 1,200 and 2,200 F. Superimposed upon the temperature and time requirements are other factors such as stress, strain, thermal and mechanical fatigue, and the erosive, corrosive effects of high-velocity combustion gases.

While aircraft turbines are presently the single biggest market for high-temperature alloys, modern industry has many structural requirements for materials at elevated temperatures. Examples of industrial equipment operating at high temperatures may be found in the chemical industry, in the petroleum industry, in the cement industry, and in the electric power industry, where generators are often driven by gas turbines operating at extremely high temperatures.

The market for high-temperature metals appears to be growing: useage of high-temperature alloys in aircraft gas turbine engines should increase as engines go to hotter operating temperatures; gas turbines for long-haul trucks and buses are already here and the development of a low-cost, high-temperature alloy could help bring about turbine powerplants for passenger cars; and the chemical process industries are using larger quantities of superalloys and refractory metal alloys to combat higher temperatures and pressures so that they can turn out products of high purity and hold down rising maintenance costs.

Advances in the technology of high-temperature metal alloys are generally directed at the well-known fact that materials are substantially weakened by elevated temperatures. The loss of strength varies with time, so that the useful strength of metals at elevated temperatures is less for long-time service than for short times. Because of this, significant developments in the technology have dealt with the combined effects of time and temperature. The approach has been three-fold: development of metals with improved high-temperature properties; development of new or improved processes for producing or fabricating the metals; and development of design techniques to make better use of existing materials properties. A list of significant technological advances in high-temperature metals, as identified by technological leaders in the field, is given in Table 4. These advances emphasize new or improved materials such as dispersion hardened metals, nickel and cobalt-base superalloys, and refractory metals; forming processes such as grain-oriented casting, powder metallurgy and superplasticity; and design approaches, such as the use of fracture mechanics concepts and computer-based alloy design.

While aerospace application requirements generated by NASA's space missions are directly associated with NASA's technological efforts in helping to advance the field of high-temperature metals, the major impetus appears to come from NASA's involvement in aircraft turbine engine development. NASA contributions to the advancement of the technology of this field dates back to early NACA days, with large-scale efforts starting in the immediate post-World War II period and early 1950's. NASA has been directly and indirectly involved in advancing the technology of almost all facets of high-temperature metals including research and development efforts within its own laboratories, support of research and development in industrial and academic institutions, and the creation and support of needed technological facilities and experience in industry.

Significant developments in the field of high-temperature metals, as identified in Table 4, which NASA has helped to advance through technological contributions, follow.

# TABLE 4 SIGNIFICANT DEVELOPMENTS IN THE FIELD OF HIGH-TEMPERATURE METALS\*

- \*\* 1. Alloy design computer program (04-01)
  - 2. Auger spectroscopy (04-02)
  - 3. Consumable electrode melting (04-03)
- \*\* 4. Dispersion hardened metals (04-04)
  - 5. Gatorizing (04-04)
- \*\* 6. Grain-oriented casting (04-06)
- \*\* 7. High-purity superalloy powders (04-07)
  - 8. Hot corrosion resistant alloys (04-08)
  - 9. Improved casting techniques (04-09)
- \*\*10. Improved nickel-base superalloys (04-10)
  - 11. Improved thermal working of superalloys (04-11)
  - 12. Investment casting of reactive metals (04-12)
  - 13. Large rolled tungstun sheets (04-13)
  - 14. Mechanical alloying nickel (04-14)
  - 15. Nickel carbide coated cutting tools (04-15)
- \*\*16. Powder metallurgy advances (04-16)
- \*\*17. Refractory metal advances (04-17)
  - 18. Rene 95 alloy (04-18)
  - 19. Silver-infiltrated tungstum (04-19)
- \*\*20. Superplasticity (04-20)
  - 21. Transmission electron microscopy (04-21)
- \*\*22. Understanding of fracture machanics (04-22)

<sup>\*</sup> As identified by technological leaders in the field of high temperature metals.

<sup>\*\*</sup> Developments selected for identification of NASA contributions.

#### Dispersion-Hardened Metals (04-04)

Dispersion hardening and strengthening entails the dispersion of an inert material, such as thorium dioxide, throughout the bulk of the alloy itself. Unlike precipitation-hardened superalloys, which are strengthened through an intermetallic gamma-prime phase, the inert dispersed material does not go into solution at high temperatures and therefore retains its high-temperature properties far beyond that of the normally available precipitation-hardened materials.

Despite the superior high-temperature strength of dispersion-hardened metals (above 1800 F.), the strength of dispersion-hardened nickel, the most commonly available dispersion hardened superalloy, is relatively poor compared to conventional superalloys (precipitation hardened) at moderate and low temperatures. In addition, the oxidation resistance of dispersion hardened nickel does not compare favorably with conventional superalloys at the higher temperatures. To alleviate these shortcomings, numerous alloy modifications have been introduced to boost low and intermediate-temperature strength and to improve oxidation resistance.

Most methods of dispersion hardening have used a chemical process to disperse particles of chemically inert high-temperature capability substances throughout the alloy matrix. Several new processes have used thermomechanical tehniques in which the materials are ground, blended, cold pressed, sintered, hot rolled, and then put through several cold-roll anneal cycles. A new process has been developed by International Nickel, combining dispersion strengthening with precipitation hardening so that the alloy will have the low and intermediate properties of the precipitation hardened metal.

Dispersion hardening dates back to the original strengthening of tungsten filaments for incandescent lamps. One of the original products was known as SAP (sintered aluminum product) which was an alumina-strengthened, dispersion-hardened aluminum alloy, developed about 20 years ago by R. Irmann, a Swiss Professor. TD nickel was introduced by DuPont in 1962, and since then, the product has gone through extensive alloy and fabrication development and is commercially available from Sherritt Gordon and from Fansteel (the latter of which acquired the DuPont technology).

Dispersion-hardened metals provide a margin of temperature capability that, up to now, has not been available or possible through normal strengthening mechanisms. Dispersion hardened alloys open a new family of alloy design and range of alloying beyond the gamma-prime hardened superalloys.

This prospect has led to a wide range of industrial research into exploiting the properties of dispersion-hardened materials. For example, Siemens A. G., a German firm, has developed dispersion-hardened sintered copper parts giving good electrical conductivity, plus high strength at elevated temperatures; Sylvania Electric Products has also achieved high conductivities at high temperatures using a thoria dispersed copper; and investigations are underway into the use of dispersoids such as molybdenum and tungsten for intermediate temperature strength and aluminum and titanium for better strength at high temperatures.

NASA has had major interest in the development and use of dispersion-hardened metals and has contributed to the advancement of the technology by developing or improving processes for producing and working the metals, techniques for fabricating and joining the metals, and coatings and claddings for protecting the metals. These contributions follow.

o Development of a mechanical process for producing dispersion-hardened metals (04-04-01).

Most successful high-temperature, dispersionhardened materials have been nickel or nickel-base materials (containing thoria dispersions) that have been made by chemical methods. The chemical approach, limits the range of inert dispersoids and metal matrices that can be combined. A mechanical approach, on the other hand, can be readily applied to dispersion strengthen a wide range of simple and complex alloys.

A mechanical comminution and blending approach to dispersion-strengthened material production was developed at NASA/LeRC. This process entails: (1) Preparing the metal matrix and dispersoid powders by grinding and blending in a special attrition-type ball mill. Oleic acid is used as a surfacetint to aid in the grinding and heptane as the grinding fluid. The powders are reduced to less than -325 mesh and the dispersoid is in a colloidal form. The attrition mill is a commercial machine which has been modified to get a finer powder more rapidly. Since contamination is a problem, compatible materials are used for the shaft and the grinding balls. The mill is blanketed in an argon atmosphere. (2) Densification is carried out through cold pressing and then sintering at 2,000 F. (3) Sintered slabs are then suitable for thermomechanical working.

Using this NASA-developed process, dispersionstrengthened, nickel-base materials have been prepared with properties equal to or better than comparable commercially produced (chemical approach) materials. A variation of this process is being used by International Nickel.

o Development of oxidation resistant slurry coatings for TD-nickel and TD-nichrome (04-04-02).

Until 1967, designers were planning to use TD-nickel and TD-nichrome without protective coatings. In-house data, developed by NASA/LeRC, revealed that both of these materials had far greater oxidation rates at high-temperatures in high-speed gas streams than had been originally thought. These high oxidation rates are due to the relatively simple structures found in dispersionhardened metals which allow a rapid diffusion of oxygen. Therefore, dispersion-hardened alloys need diffusion barriers in the form of coatings. To overcome this, Sylvania Electric, under a NASA contract, developed the first coating ever designed for TD-nichrome. The nickel-chromiumaluminum coating which was developed is applied as a slurry and improves the oxidation resistance of the metal by about three times.

o Development of oxidation resistant alloy cladding for dispersion-hardened metals (04-04-03).

Because of the need, identified through NASA research, for oxidation resistant coatings for TD-nickel, TD-nichrome and other dispersion-hardened metals (as described in NASA contribution 04-04-02, above) NASA/LeRC started an alloy cladding development program at IITRI. IITRI's work on roll bonding, combined with in-house efforts on isostatic high-pressure bonding, resulted in an iron-chromium-aluminum (Fe-Cr-Al) cladding for dispersion-hardened metals which improved oxidation resistance about three times. The cladding is applied in thin foil form and is hot gas pressure bonded to the surface of the base metal.

o Development of manufacturing processes for TD-nichrome sheet (04-04-04).

Space shuttle design calls for the use of 0.010-inch gage TD-nichrome sheet in a thermal

protection system. Prior to application in the space shuttle, TD-nichrome sheet had only found application in military engines in much larger sizes (0.050-0.060-inch gage). To fill this need, NASA contracted with Fansteel, Inc. to develop the necessary manufacturing technology to produce TD-nichrome sheet in the required sizes and thicknesses, and at the required quality levels. Also included were property improvement studies directed at elevated temperature ductibility and behavior on exposure to possible space shuttle environments. Changes and improvements in the manufacturing processes, should lead to better yields and better sheet quality.

o Development of a solid-state welding process for joining dispersion-hardened metals (04-04-05).

Dispersion-hardened metals present a difficult joining problem. Fusion welding and brazing methods result in weldments having only 50 percent of the parent metal strength, while previous attempts at solid state welding have proven to be weak at elevated temperatures (2,000 F.). Engineers planning to use dispersionhardened metals therefore have had to alter designs to get the desired joint strength (for example, by designing for a double load). To overcome this deficiency, NASA/LeRC developed a solid-state diffusion process which uses a special two-step welding process. The joint is first welded at 1,200 F. in a vacuum hot press welder at 30,000 psi. for one hour, then again at 2,175 F. and 2,000 psi. for two hours. During the second step, diffusion and recrystallization occurs. All welds are post-heated at 2,300 F. for one hour to relieve stresses and to insure complete recrystallization. This solid-state welding technique may be applied to any alloy that undergoes recrystallization upon heating. When recrystallization occurs, the original weld interface is removed by grain growth across it.

o Development of a process to reduce chromium oxide content in nickel matrix, dispersion-hardened metals (04-04-06).

The successful processing of dispersionstrengthened materials requires that contamination

be held to a minimum. Matrix oxides are serious contaminants which must be reduced or eliminated to produce thermally-stable microstructures. oxides of copper, cobalt and nickel are easy to reduce at low temperatures with hydrogen but chromium oxide is much more difficult. Reducing agents that have been tried for chromium oxide include hydrogen at high temperatures, carbon and hydrocarbon vapors. Each approach to reduction has potential drawbacks but the halides offer the potential for reducing chromium oxide at low temperatures. Based upon this, a process was developed at NASA/LeRC which uses hydrogen plus hydrogen chloride or hydrogen bromide to reduce the chromium oxide content. The process is effective in reducing "oxygen excess" to less than 100 ppm. at temperatures as low as 1.675 F. although greatest reduction occurs at 2,200 F. The presence of the hydrogen halides also reduces the carbon level of the metal, which might improve the stability of the material -particularly in terms of long-time, high-temperature exposure.

All nickel-base superalloys have the problem of chromium contaminations. Since chromium oxide is the main contaminant removed, this process should also be applicable to other chromium-containing superalloy powders.

o Development of thermomechanical processes to produce elongated grain structures in dispersion-hardened metals (04-04-07).

NASA-supported research at Battelle indicated that thermomechanical processing influences the microstructure, crystallographic texture and strength of dispersion-hardened metals. The major microstructural feature which affects high-temperature strength is the grain aspect ratio, i.e., grain length divided by width, and that yield strength and creep strength increased linearly with increasing grain aspect ratio. Close control of the thermomechanical processing step, which was worked out by Battelle, can be used to increase the aspect ratio in dispersion-hardened metals, resulting in significant increases in strength. (See NASA contribution 04-04-09 below for a discussion of thermomechanical working processes of mechanically prepared dispersion-hardened metals.)

o Development of a process for the controlled cleaning and stabilization of dispersion-strengthened materials (04-04-08).

One of the reasons that dispersion-hardened materials has not been made by mechanical instead of chemical methods is the problem of removing impurities. Difficulties in cleaning mechanically produced blends prevents retention of the fine blend during consolidation. Furthermore, small quantities of impurities cause detrimental reactions in subsequent processing and use. Most attempts at mechanical mixing approaches to the production of dispersion-hardened metals use reduction-type cleaning processes. These processes have a number of inherent difficulties which limit the efficiency and completeness of the methods and yield dispersion-strengthened materials with microstructural instabilities.

In efforts to develop a mechanical approach to produce stable dispersion-hardened metals at NASA/LeRC (see NASA contribution 04-04-01, above) attention was given to thorough cleaning of the alloy materials. This cleaning was accomplished by using a partially densified powder in a shape sufficiently thin to permit easy access of the cleaning gas to the interior of the product and escape of the reaction products. Slow heating permits elimination of the impurities prior to final and complete densification. The process yields a highly dense product which does not readily recontaminate on further handling or processing.

While this process was developed to remove impurities such as sulfur it may be applicable to the removal of other types of impurities in a nickel matrix. Materials such as carbon can also be removed, or any other impurity that can be made either volatile or gaseous as a reaction product. The method is also believed to be applicable to matrix materials other than nickel, which have a low stability oxide. For example, a chromium-bearing material containing large quantities of chromium oxide impurities (see NASA contribution 04-04-06, above).

o Development of thermomechanical processes for working dispersion-hardened metals prepared by powder metallurgy techniques (04-04-09).

One route to achieving higher-strength systems is to combine dispersion-hardening with thermomechanical working of the material. Thermomechanical working procedures, such as extrusion, swaging, rolling and annealing, had been successfully applied to achieve high-strength, in a chemically produced dispersion-hardened nickel material, but no such procedures existed for mechanically produced materials prepared by powder metallurgy techniques.

To complete development of a mechanical process for preparing dispersion-hardened materials (see NASA contribution 04-04-01, above) NASA/LeRC developed thermomechanical working procedures for the powder products which produced a stable microstructure and gave desired material strength in the 2,000 F. range. In general, the strength of the mechanically produced dispersionhardened products increase with the degree of thermomechanical working. The tensile and stressrupture properties of materials which are processed this way are equal to or better than comparable commercially available products produced by chemical processes. For a discussion of the role of grain structure in the thermomechanical working of dispersion-hardened metals see NASA contribution 04-04-07, above.

o Improved understanding of dispersion-strengthening mechanisms (04-04-10).

To improve the quality of dispersion-hardened metals, NASA has supported a wide variety of basic studies of dispersion-hardening strengthening mechanisms, which has led to an improved understanding. Included among others, are studies of: modes of deformation and fracture in oxide dispersed metal systems (M.I.T.); determination of the qualitative and quantitative nature of the substructure and elastic strains retained in cold worked and annealed dispersion-strengthened alloys (Ilikon Corp.); high-temperature creep behavior of recrystallized nickel-thoria alloys (Battelle); and elastic strains associated with the particle-matrix interface in dispersion-strengthened alloys (Ilikon Corp.).

o Identification of alloys suitable for development of new dispersion-hardened materials (04-04-11).

While nickel-matrix materials have gained the greatest amount of attention in the development of dispersion-hardened materials, a number of other alloys offer promise. To investigate this potential, NASA has contracted with a large number of industrial firms to systematically identify and explore the development of new dispersion-hardened base materials. Included, among others, are investigation of: dispersion-strengthened tantalium-base alloys for use in advanced space power systems (Westinghouse); cobalt-base dispersion-strengthened alloys (Sylvania); dispersion-strengthened chromium alloys (G.E.); and dispersion-strengthened nickel-molybdenum alloys (Teledyne).

o Identification, evaluation and development of new processes for producing dispersion-hardened alloys (04-04-12).

Almost all commercial dispersion-hardened materials are produced through a chemical process. The Dupont-developed approach which led to the production of commercially available TD-nickel uses chemical precipitation. NASA/LeRC efforts in the dispersion-strengthening field have been mainly concerned with mechanical communition and blending. In addition to this effort at Lewis, NASA has supported contract efforts in a number of industrial laboratories to identify, evaluate and develop new processes for producing dispersion-strengthened materials. Included, among others, are investigation of the suitability of the following types of processes: spray drying and selective reduction (Sylvania); splat cooling (M.I.T.); flash drying and selective reduction (Curtiss-Wright); vapor plating in a fluidized bed (Melpar); high-intensity arc processes (Vitro Labs); aqueous and fused salt electro-dispersion techniques (General Technologies); and ultransonic energy (West Virginia University).

# Grain-oriented casting (04-06)

Since alloys used at high temperatures tend to fail at grain boundaries, casting processes with controlled solidification patterns have been developed to improve high-temperature properties by aligning the grains along the major stress axis. Failure due to thermal fatigue occurs at the grain boundaries in the transverse direction. By aligning the grain boundaries along the major stress axis, the grain boundaries are eliminated in the transverse direction and the site or origin of thermal fatigue cracking is therefore eliminated.

The columnar grain structure is produced by pouring molten metal into a mold that is heated differentially so that metal solidification advances as a uniform front along the axis of the casting. In this way the grain boundaries line up along the major stress axis. By modification of this technique, monocrystalline castings can also be made with no grain boundaries at all. In this system, columnar grain boundaries are trapped as they encounter the mold walls in a circuitous gating system so that only one grain is allowed to grow into the body of the casting.

The original concept of this casting process was developed at General Electric. Pratt and Whitney subsequently developed the directional solidification process based upon the G.E. work, and gradual upgrading in casting technology. Improvements in ceramic molds and the general melt environment also led to the improvements which have allowed the development of this process. In addition, basic knowledge about grain boundaries, crystal growth and single crystals was important. Research into single crystal structures has come from a desire to determine the inherent properties of the material that were not affected by grain boundaries and polycrystalline interactions. Much of this emphasis came out of the semiconductor business. This single crystal work led to attempts to develop truly directionalized structures which have resulted in the processes cited above.

High-temperature metals for high-load uses generally have to be forged. Choice of metals is therefore limited to those that are forgeable at high temperatures, which is an obvious limitation upon materials selection. Directional solidification processes opens up a whole new range of alloy designs in that it allows use of alloys which have suitable high-temperature, load-bearing capabilities which cannot be formed through conventional forging processes, but which can be formed through casting processes. Columnar grain structures are particularly desirable in turbine buckets that are primarily under centrifugal stress. Because of this, controlled solidification processes leading to grain-oriented castings can be a powerful tool in extending the high-temperature capability of superalloys for turbine applications.

NASA's contributions to the advancement of this development have been aimed at developing a better understanding of the properties of directionally solidified alloys, and the processes by which they are made, and applying the process to new materials. Specific NASA contributions follow.

o Advanced understanding of directionally solidified alloys (04-06-01).

Because of the increased strength potential of grain-oriented alloys, NASA has sponsored a number of basic investigations into the structure and properties of directionally solidified materials. These efforts have increased basic understanding of the process and the materials and include efforts such as research on binary eutectic alloys produced by unidirectional solidification, including mechanical behavior at elevated temperatures, galvano-thermo-magnetic effects, and ambient temperature fatigue and fracture behavior (Lehigh University); and research on the control of properties of unidirectionally cast structures, including the role of atomoc ratios, the role of working and the role of microconstituents (Case Institute of Technology).

o Application of directional solidification techniques to produce superalloys with improved high-temperature properties (04-06-02).

Directional solidification techniques were applied to three NASA-developed nickel-base superalloys, resulting in improved high-temperature properties and new potential applications.

In the case of WAZ-20 (see NASA contributions to improved superalloys, 04-10-01, for a description of this material) the application of directional solidification techniques by NASA/LeRC increased intermediate temperature tensile strength and generally increased ductility and stress-rupture life. The improved strength of this alloy in the 2,000 to 2,200 F. range, made possible through directional solidification, raises its potential for application to stator vanes of advanced gas turbine engines.

By applying directional solidification techniques to a complex NASA-developed nickel-base superalloy, TAZ-8B (see NASA contributions to improved superalloys, 04-10-05) NASA/LeRC produced materials with a columnar grain orientation, eliminating grain boundaries transverse to the major stress axis. This processing resulted in substantial increases in ductility, ultimate tensile strength and stress-rupture life. The application of directional solidification to this alloy resulted in alloy properties which are of interest for potential advanced gas turbine applications where inlet gas temperatures are in excess of 2,000 F.

In the third instance, under NASA contract, Case Western University developed a cast directionally solidified cobalt-tungsten alloy with stress rupture characteristics that compared favorably with similar types of cast superalloys.

#### High-Purity Superalloy Powders (04-07)

Past powder metallurgy attempts with superalloys, using blended elemental powders, were failures due to the segregating and clustering of individual ingredients during cooling. Such segregation weakened the solidified metal. Several new techniques for making high-quality, high-purity (low-oxygen) pre-alloyed superalloy powders have been developed. Pre-alloyed metal powders consist of particles of essentially similar composition to that of the melt from which they are formed. Each particle is a microcosm of the original molten alloy and grains are uniformly small because they have no time to grow. Result of using pre-alloyed powders is a more homogenious product with higher strength and less brittleness than one made from blended powders. This development dates back to early 1960's, with work done by Federal-Mogul Corporation and Air Force-funded work at IITRI.

In a typical powder process, nozzles squirt jets of inert gas into a falling stream of molten alloy, atomizing it into tiny particles that fall into water and solidify. The alloy particles are then used in a powder metallurgy process. Work is now underway at developing atomization systems to produce alloy powders with less than 50 ppm. oxygen of metals for use in the 1,600-2,200 F. service range that can't be formed using traditional forming methods of casting and forging.

Availability of high-quality, low oxygen superalloy powders provides an entirely new forming process for superalloys, and brings new flexibility into the powder metallurgical process. It is now possible to form certain alloys through powder metallurgical techniques that could not be formed any other way. For example, certain alloys compositions are not forgeable or castable. It is now possible to process them through a powder metallurgical technique by producing a powder of the alloy. It also makes possible the use of alloys with higher amounts of such hardening agents as aluminum, titanium, columbium and tungsten.

Using powder metallurgy techniques, superalloy components can be made without the heavy capital equipment required to reduce an ingot to a semi-finished wrought product. The yield of finished materials from a given weight of molten metal is also considerably higher with the powder route than by conventional techniques, an important factor with high-priced superalloy materials.

Superalloys normally are sold by specialty steelmakers, but a few powder makers are now shipping not just the powders but massive, 1,000-lb. forging billets made from them. So far, these materials are being used only in experimental programs for such items as disks in jet engines. But by 1980, some industry sources estimate powder metallurgy will capture 40 to 50 percent of what is now a \$200-million market in the U. S.\* Currently, superalloys powders cost \$15 to \$20 a pound. However, new plants for producing powders being built by firms like Federal-Mogul could reduce the price to \$7 per pound.

NASA contributions to the advancement of this development have taken the form of developing and evaluating new processes for making pre-alloyed powders, and stimulating industrial suppliers of pre-alloyed powders to improve their products and processes so as to meet NASA needs. These contributions include the following, among others.

o Development of the splat cooling process for the large-scale production of alloy powders (04-07-01).

Splat cooling is a method for producing alloy powders by rapidly quenching liquid metal droplets. This process is being investigated at M.I.T. under a NASA grant, and includes the development of continuously operating atomization units for large-scale powder production. Rapid quenching rates have been achieved and promising alloy systems are now being examined. This process appears to offer some promise for the large-scale production of alloy powders due to its excellent potential for structure modification and property improvements.

o Adaptation of the high-intensity arc process for the production of nickel-base alloy powders (04-07-02).

Nickel-based, dispersion-hardened alloy powders were produced by Vitro Laboratories under a NASA contract. These powders consisted of a four-component nickel-base alloy (Ni-20Co-10Mo-10W) with three percent thoria as the dispersion-strengthening oxide. They were prepared by a high-intensity arc process using a homogeneous consumable electrode. The electrode formulation was developed to withstand high thermal shock, and vaporizes efficiently to yield a submicron oxide product of the desired composition. This oxide is then reduced in hydrogen to yield the desired alloy powder.

<sup>\*&</sup>quot;Powdered Steel: A Tough Customer," Business Week, January 2, 1971, p. 37.

o Application of pre-alloyed powders to achieve superplasticity in a high-strength superalloy (04-07-03).

The use of pre-alloyed powders offers a means of overcoming the problems inherent in conventional casting and hot working of superalloys. Exploiting this concept, a high-strength superplastic superalloy (NASA TAZ-8A) was produced at NASA/LeRC by extruding pre-alloyed powders.

The specific process involved atomization of the molten alloy with inert gas jets. Each metal droplet so formed solidifies rapidly and a highly homogeneous structure is obtained both in the powder particles and in the final compacted product. TAZ-8 powders were not available at Lewis at the initiation of this program. Federal Mogul (FM) and Cabot Industries modified the formulations for this alloy and produced the needed powders. While FM was already producing U-700 pre-alloy powders, the production of TAZ-8 powders represents one of the more highly alloyed superalloys to be made this way.

The powders were then extruded into bar steel which showed higher strength values than those obtained with any known cast or wrought nickel base alloys, and 1-1/2 to 2 times greater than for the same alloys when cast. Due to its extremely fine grain size, the material also exhibited superplastic behavior, which can be used to advantage in forming. The fact that superplasticity can be achieved with this alloy by means of pre-alloyed powder techniques, contributes to the attractiveness of the alloy for expanded commercial use. For a further description of this process, see NASA contributions to improved superalloys, 04-10-06, and to superplasticity, 04-20-01.

### Improved Nickel and Cobalt-Base Superalloys (04-10)

Superalloys offer all-around strength properties with reasonable stability in the 1,000 to 1,800 F. temperature range. As a group (cobalt or nickel-base, depending upon the predominating alloying agent) they offer excellent high-temperature properties.

General upgrading and development of superalloys has been directed at increasing the high-temperature properties of the materials through classical alloying procedures, such as advances in creep-rupture strength

through alloy additions of materials such as tungsten, and increased oxidation resistance through additions of materials such as rare earth metals. The approach to improvement has been through "enlightened empiricism," with small but steady incremental advances so that superalloy development has reached the point where significant advances have been achieved. Capabilities of superalloys have also been extended through departures from conventional alloying technology. For example, an approach used by NASA to develop an advanced, high-temperature, oxidation-resistant, nickel-based superalloy by reducing the number of alloying agents used — in some alloys as many as ten — to one principal alloying constituent, such as tungsten, with only limited amounts of aluminum, zirconium and carbon.

In general, improvements can be expected to continue in the mechanical physical properties of superalloys through a combination of:
(1) a better understanding of the basic, metallurgy of the materials;
(2) the application of various new processing techniques to improve microstructure; and (3) the development and use of new alloying concepts and agents.

Since the development of Nimonic 75 in the late 1930's, the high-temperature strength of superalloys has continued to improve. The most important of the nickel-base superalloys are precipitation hardened. That is, they are strengthened through precipitation of a gamma-prime intermetallic phase. Inconel X developed by the International Nickel Company, was the first commercial alloy using a gamma-prime intermetallic strengthening concept. All told, there are now some 50-odd nickel-base superalloys commercially available. Still among the most important are the Inconels, especially Inconel 718, along with Hastaloy X, Rene 41, Udimet 500 and 700, Waspaloy and In-100.

Cobalt-base superalloys received their initial development impetus from the materials needs generated in aircraft turbosuperchargers in the late 1930's. In today's aircraft gas turbine engines, cobalt-base superalloys are used mainly for first-stage turbine vanes, blades in the lower-temperature position of the turbine, and in sheet applications such as exhaust case assemblies and burner liners.

No intermetallic compound which possesses the same utility as does the gamma-prime precipitate in nickel-base superalloys has yet been found in cobalt-base systems. This lack of an effective strengthening mechanism for high-temperature strength has been a major limitation on widespread application of cobalt superalloys. Between 1961 and 1967 nine new cobalt-base superalloys were introduced in the U. S., while 23 new nickel-base superalloys appeared in the same period. The principal commercial cobalt-base superalloys are S-816, L-605, HS-21, HS-31, HS-151, and Wi-52, among others. Several new cobalt-base superalloys have been developed recently and, as in the case of nickel alloys, are in various stages of development.

Superalloys are the prime materials used for high-temperature applications in aircraft turbine engines, a market that is estimated to be greater than \$200 million per year. Advances in the high-temperature properties of superalloys have permitted jet engine operation at much higher temperatures, with attendant greater operating efficiency. In the next 20 years, engines will most likely be developed which are two or three times more powerful, with inlet temperatures of 2,500 to 3,000 F. Aircraft turbines are now 30 to 40 percent by weight superalloys. Because of this trend toward higher operating temperatures, the proportion of superalloys in jet engines can be expected to increase to 50 percent by weight by 1980. As a result, although some of the well-establishing superalloys will continue to see wide use, some will be replaced by newer, superior alloys now in various stages of development.

In addition to the use of superalloys in aircraft turbines, several new commercial markets are starting to emerge. Stationary gas turbines for power generation are already a growing outlet for superalloys as are shipboard turbines. Gas turbine engines for long-haul trucks, buses and possibly even passenger cars appear to be prime potential markets for superalloys.

NASA contributions to the advancement of the technology of superalloys include the development of new nickel and cobalt-base superalloys, the development of processing techniques for properties improvement, the development of forming techniques, and the development of protective coatings for oxidation resistance. Specific contributions, among others, follow.

o Development of a series of nickel-base superalloys using a limited number of alloying agents (WAZ-20) (04-10-01).

Available nickel-base alloys drop off sharply in strength above 1,900 to 2,000 F. This tendency toward a sharp decline in strength detracts from the desirability of using available nickel-base alloys as stator vane materials which are exposed to temperatures above 2,000 F. in advanced turbine engines. In order to develop a new series of nickelbase alloys to meet these needs, NASA/LeRC took a radical departure from current nickel-base alloy formulations. Nickel-base alloys in use today are metallurgically very complex and contain as many as 10 or more alloying agents. Substantial improvements have been made in these materials but development had reached a point where apparently little more could be achieved without increasing problems in casting and corrosion. NASA's approach was to drastically reduce the number of alloying

constituents and select one principal alloying constituent (tungsten) which would tend to raise rather than lower the alloy melting point. result of this effort is the start of a new allow series, WAZ-20, with an incipient melting point of 2,375 F., approximately 100 to 165 F. higher than conventional highly alloyed, nickel-base materials in use today. This alloy has high tensile strength over the entire temperature range and its strength of 20,000 psi. at 2,200 F. is approximately twice the strength of the strongest available nickel-base alloys. The alloy is amenable to controlled solidification techniques and, in the directionally solidified form, exhibits substantially improved tensile and stress-rupture properties over the random polycrystalline form. For a description of directional solidification, see NASA contributions to grain-oriented casting, 04-06-02.

o Development of a series of cobalt-base superalloys with extended high-temperature capabilities (04-10-02).

Cobalt-base alloys, due to the lack of a potent strengthening mechanism such as the gamma-prime phase, have lower strength than nickel-base alloys over most of their useful temperature range. However, above approximately 2,050 F., the maximum solutioning temperature of gamma prime, cobalt-base alloys tend to have higher strength than nickel-base alloys, when oxidation can be controlled.

NASA/LeRC has developed a series of cobalt-based superalloys using low-volatility alloying agents, principally tungsten (Co-25W), which give extended high-temperature capability. These alloys, due to their low chromium content, are significantly different in composition from currently used cobalt-base alloys. While they do not suffer from catastrophic oxidation, they do require adequate protective coatings.

Although originally designed for space power system applications, their excellent high-temperature strength should lend them well for use in advanced turbine engines. This material also has potential applications in aluminum die casting and is under consideration by the U. S. Army for use as gun barrel liners.

o Demonstrated improvement in intermediate strength and ductility of nickel-base superalloys by thermomechanical processing (04-10-03).

Many metallurgists believe that we are approaching a limit in properties improvement through alloy additions and that further advances will be brought about by processing to change microstructure, rather than by changes in alloy composition. Special processing techniques have already shown potential for improving the mechanical properties of nickel-base alloys, including thermomechanical processing (TMP). TMP consists of the application of combined mechanical working and heat treatment. The purpose of TMP is to provide extensive strain hardening of the alloy in conjunction with a precipitation hardening reaction.

The potential for improving the strength of nickel-base superalloys by TMP was demonstrated by NASA/LeRC, using Udimet 700, a commercially available material. This material is a high-strength, commercially available, nickel-base, precipitation hardening alloy with good high-temperature strength. At temperatures of 1,000 to 1,200 F., the yield strength of the alloy after TMP was as much as 100 ksi. greater than the yield strength after conventional heat treatment. also improved the 1,200 F. stress-rupture life. At a stress of 120 ksi., the rupture life of thermomechanically processed U-700 exceeded 1,500 hours. Conventionally heat-treated U-700 lasted only 400 hours in a comparable situation. The key to this improvement is the TMP schedule developed by Lewis and the cold working of the alloy.

o Development of a high-temperature cobalt-base superalloy with high-temperature magnetic properties (04-10-04).

In studies of high-strength alloys for use at high temperatures, NASA/LeRC developed a cobalt-tungsten alloy that, in addition to high strength, has high magnetic induction. This alloy retains its magnetism at least 10 times longer and to a temperature about 100 F. higher than standard commercial magnetic alloys at equivalent loads and at temperatures as high as 1,400 F. This alloy is the result of efforts to combine the ferromagnetic and high-temperature properties of cobalt. The addition of tungsten for high-temperature strength, unfortunately, detracts from the magnetic properties of the alloy. This alloy's tungsten content

is modified so as to obtain the best combination of temperature and magnetic properties. The alloy is finding application in alternators which are used in compact power generation equipment for spacecraft. However, its unique properties should find nonaerospace uses in applications requiring the development of electric power at high temperatures, such as in stationary turbine equipment and more efficient electric generators and motors.

o Developed a series of complex nickel-base superalloys with extended high-temperature oxidation resistance (TAZ-8 series) (04-10-05).

A series of complex nickel-base superalloys with high-strength and oxidation resistance was developed by NASA/LeRC. Designated the TAZ-8 series, these alloys combined: (1) high-temperature strength comparable to the strongest commercially available cast alloys; (2) demonstrated workability potential which allows the alloy to be rolled from cast slabs to sheet form; (3) high-impact and thermal-shock resistance; (4) excellent oxidation resistance; and (5) long time microstructural stability.

The original alloy in the series, TAZ-8, had suitable strength for elevated temperature service but the oxidation resistance of the alloy was somewhat less than that of a representative fully workable nickel-base superalloy. By modifying the chemistry of the TAZ-8 alloy and using vacuum melting techniques, TAZ-8A was developed, which provided improved oxidation resistance for use up to 2,200 F. The most recent alloy addition to the series, TAZ-8B, lends itself to directional solidification techniques, which provide significant improvements in intermediate and high-temperature ductility as well as stress-rupture life over a wide range of temperatures.

The very good high-temperature oxidation resistance of these alloys, good high-temperature strength, and workability potential, suggest applications in advanced gas turbine engine components, although these alloys need not necessarily be limited to gas turbine applications. Haynes Satellite, a division of Union Carbide, has an exclusive license for production of alloys in this series and some are now commercially available. There has been some interest shown by industrial firms in the use of TAZ-8 alloys in land-based nonaircraft turbines.

o Demonstrated improvement in intermediate-temperature strength of nickel-base superalloys by use of pre-alloyed powders (04-10-06).

To improve the intermediate-temperature (1,000) to 1,400 F.) strength of nickel-base superalloys, NASA/LeRC developed an approach using pre-alloyed powder techniques. Using NASA alloy TAZ-8 (see NASA contribution 04-10-05, above) and a commercial alloy 713C, pre-alloy powders were prepared by vacuum melting and then pouring the melt and atomizing the molten metal, using inert gas jets, into small droplets which solidified into powders. These powders were then compacted by hot pressing and extrusion. Finally, the compacted powders were subjected to heat treatment. The grain size of the final product is of the order of microns, much smaller than in typical cast materials, and the microconstituents are finer and more uniform than in cast materials. This gives a better, more homogeneous distribution of the strengthening phases throughout the powder compact.

At 1800 F. the TAZ-8A powder product exhibits superplastic behavior as did the alloy 713C powder product. The significance of this behavior is that it permits the pre-alloyed powder product to be readily formed into any required shape.

The advantages of the pre-alloyed powder approach developed by Lewis include: (1) substantial strength advantages over cast counterparts to intermediate temperatures of about 1,500 F.; (2) cast alloys, unworkable by conventional techniques, can be transformed into workable materials; (3) increased high-temperature strength can be expected due to finer distribution of strengthening phases; and (4) new alloys can be designed or tailored to contain higher alloy content than is possible using casting techniques. Also see NASA contributions to high-purity superalloy powders, 04-07-03, and to super-plasticity, 04-02-01.

o Demonstrated improvement in intermediate-temperature strength of cobalt-base superalloys by use of pre-alloyed powders (04-10-07).

Pre-alloyed powder techniques afford a means of overcoming the segregation and forming problems inherent in conventional casting and hot-working operations of superalloys. These techniques also can substantially improve superalloy strength and ductility. Earlier work at NASA/LeRC demonstrated that the tensile strengths of the as-extruded prealloyed powder products of two normally cast nickelbase alloys, Alloy 713C and TAZ-8A, are almost twice those of their cast counterparts up to 1,200 F. (See NASA contribution 04-10-06, above.)

Extending the original contribution, Lewis demonstrated that the technique of compacting and extruding pre-alloyed powders obtained by gas atomization was applicable to the commonly used cobalt-base alloy HS-31. Significant improvements in tensile strength over the as-cast condition are obtained with the as-extruded powder product up to 1,400 F. However, the pre-alloyed product has substantially lower elevated-temperature (1,500 to 1,800 F.) strength than its cast counterpart. By using heat treatments that exceed the solidus temperature and employ the application of pressure. substantial increases in the elevated temperature life of the pre-alloyed powder product can be achieved, as well as lives comparable to those obtainable with the cast product. The concept of heat treatment to restore the integrity of the material, thus closing voids formed as a result of minor-phase melting, appears to hold considerable promise for substantially increasing the hightemperature strength of compacted pre-alloyed powder products.

o Development of a series of complex nickel-base alloys with improved high-temperature strength properties (VI-A) (04-10-08).

Advanced aircraft turbine engine designs have been placing ever more requirements on materials to be used in blades, discs, compressor parts, and other "hot components." In particular, a need developed for turbine blade materials with a substantial improvement in stress-rupture life over commercially available alloys. In 1967, alloy VI-A was developed at TRW, Inc. under NASA sponsorship. This alloy represents one of the strongest high-temperature nickel-base alloys available today - for example, at 2,000 F. and at an applied stress of 15,000 psi., the average stress-rupture life is 63 hours.

The alloy uses substantial amounts of solid solution strengtheners, such as tantalium, tungsten and molybdenum in proper balance to achieve strength properties without decreasing oxidation resistance. The nickel, aluminum and titanium contents are also controlled. The addition of rhenium is used to improve strength and corrosive properties of the alloy.

This alloy is now undergoing tests for use in truck turbine engines.

o Development of an improved nickel-aluminide, oxidation-resistant coating (04-10-09).

Due to the high operating temperatures in aircraft turbine engines, the materials used in blades and vanes must be coated to resist oxidation and to extend their useful lifetime. Aluminide diffusion coatings are most commonly used to protect superalloys in current engines. By interdiffusion with aluminum, the superalloy surfaces are converted to complex nickel or cobalt aluminides. These coatings are presently used for thousands of hours at temperatures below 1,800 F., but must be improved for extensive use at higher temperatures.

Controlled composition modifications of aluminide coatings was an approach used at NASA/LeRC to increasing the oxidation resistance of the coatings. A two-step slurry plus pack aluminizing process was developed as a versatile way of depositing oxidation-resistant coatings on nickel and cobalt-base superalloys. The resultant coatings were 100 percent dense and were 0.05 to 0.10 mm. thick. The nickel-aluminide slurry coated nickel-base alloys demonstrated equal if not superior resistant than commercially aluminized materials.

o Evaluation of oxidation-resistant alloy claddings for nickel and cobalt-base superalloys (04-10-10).

To provide oxidation protection at temperatures significantly above the operating limits of simple aluminides (described above in contribution 04-10-09) coatings of more complex chemistry are required. For example, alloys such as FeCrAly and NiCrAl have been used as heating elements because of their excellent oxidation resistance at temperatures up to at least 2,200 F. Such alloys are much too weak for structural applications in aircraft engines at high temperatures but they are useful in the form of claddings on stronger but less oxidation-resistant superalloys.

The feasibility of protecting superalloys from cyclic furnace oxidation by cladding with such oxidation-resistant alloys was demonstrated by NASA/LeRC. Claddings of Ni-Cr-Si, Ni-Cr-Al-Si, and Fe-Cr-Al-Y were tested by pressure bonding foils to nickel (IN-100) and cobalt-base (WI-52) superalloy substrates. All three claddings provided excellent protection at 1,900 F. At 2,000 F. the Ni-Cr-Si and Fe-Cr-Al-Y claddings provided excellent protection for the cobalt-base substrates while the Ni-Cr-Al-Si cladding afforded good protection for the nickel-base substrates. The protection afforded by these claddings was many times better than the best commercial aluminide coatings.

o Evaluation of electron-beam vapor deposited coatings for superalloys (04-10-11).

The vapor deposition of oxidation-resistant coatings on superalloy substrates, using an electronbeam process, was scaled up from laboratory to fullsize by Pratt and Whitney (P & W) under a NASA contract. Traditional vapor-deposition techniques use a salt or compound of the coating, which is decomposed or reacted with a carrier gas and then passed over the substrate leading to a reaction on the surface. In the P & W approach, the coating material is prepared as an ingot, and then fed into a vacuum system. tip of the ingot is vaporized by an electron beam which then condenses on the substrate. Of the eleven compositions tested by P & W, four demonstrated oxidation resistance more than three times that of comparison aluminide coatings. Two of the coating compositions which were characterized by tensile and stress-to-rupture tests, demonstrated that the coatings produced no adverse effect on the mechanical properties of the base metal.

o Evaluation of commercial coatings for cobalt and nickel-base alloys (04-10-12).

As part of NASA's effort to advance high-temperature turbine engine development, it was necessary to characterize the useful upper temperature limits of commercial coatings. This effort, carried out by the Solar Division of International Harvester, under a NASA contract, entailed a systematic evaluation of the rate of failure of commercially available coatings on nickel and cobalt-base superalloy substrates, under turbine engine operating conditions. The coatings

evaluated were all basically nickel or cobalt aluminides formed by reacting the substrates with aluminum. Several were the duplex type in that elements such as chromium were applied either before or after the aluminum. Comprehensive analytical data on coated specimens were developed through extensive characterization of the coatings before and after test. This effort should provide turbine engine manufacturers who must use coatings on advanced engine design, with information necessary for evaluation of commercially available coatings.

o Development of a process to reduce chromium oxide content of superalloys (04-10-13).

Almost all superalloys contain chromium oxide as an alloying constituent, and as a result, also contain chromium oxide as a contaminant. For a complete description of the NASA/LeRC-developed process to reduce chromium oxide content of superalloys, see NASA contributions to dispersion-hardened metals, 04-04-06 and 04-04-08.

o Development of a tungsten fiber-reinforced nickel superalloy (04-10-14).

Fiber reinforcement of metals has been investigated intensely in recent years. One of the promising fiber composite types being studied is refractory metal fiber-reinforced superalloys. This type of material has the potential of combining the strength of refractory metals with the oxidation resistance of superalloys. The tungsten fiber-reinforced nickel-base superalloy developed by NASA/LeRC has excellent stress rupture strength at temperatures of 2,000 to 2,200 F. The matrix composition in weight percent is nickel 56, tungsten 25, chromium 15, titanium 2, and aluminum 2. The fiber composition is tungsten with 1 weight percent thorium oxide.

The 70 volume percent unidirectional fiber composite has a 26,000 psi. stress for rupture in 1,000 hours at 2,000 F. This is four times as strong as commercially available nickel superalloys.

The fiber composite may be substituted for superalloys where weight savings are desirable since it is twice as strong as competing superalloys on a stress-density basis.

o Evaluation and compilation of properties and design data on commercial superalloys (04-10-15).

To assist engineers in working with hightemperature materials, NASA critically and systematically
developed, evaluated and compiled properties and
design data on commercially available nickel and
cobalt superalloys. Included in this effort are
the following among others: a comprehensive
compilation of technical data on Inconel 718,
presented in a handbook form (Syracuse University
Research Institute); properties, data and metallurgical characteristics of Hastalloy X (Aerojet-General);
thermal fatigue data on 15 nickel and cobalt-base
superalloys (IITRI); and a literature survey on
the effects of hydrogen on nickel-base superalloys
(Atomics International).

o Advanced understanding of the metallurgy of superalloys (04-10-16).

Advances in nickel and cobalt-base superalloys will most likely be achieved by modifying current alloy systems. One of the major forces behind present and anticipated advancements is a better understanding of the basic metallurgy of superalloy materials. NASA has carried out and supported under contract and grant a wide range of studies and analyses designed to advance our understanding of these high-temperature materials. Included, among others are studies of: gamma-phase composition in nickel-base superalloys (NASA/LeRC); mechanical response of nickel eutectic composites (Lehigh University); local atomic arrangements and the effect of cold work on short-range order in a nickel-tungsten superalloy (University of Tennessee); solidification, structure and properties of eutectic alloys including consideration of properties control (Lehigh University); modified eutectic alloys for high-temperature service (Case Institute of Technology); and a study of internal oxidation of nickel-base superalloy systems (NASA/LeRC).

#### Powder Metallurgy Advances (04-16)

Powder metallurgy is commonly used to make precision components, and to fabricate exotic materials. Although sintered powder metal components are relatively brittle and can be used under low stresses, they have found wide applications in the automobile and light engineering industries which require large numbers of small, complex components made to accurate dimensional tolerances.

Many of the high-temperature metals are difficult to form through traditional methods. In many of these cases, powder metallurgy has been applied in fabricating such materials which are not amenable to conventional casting and forging techniques. In particular, refractory metal alloys such as tungsten and molybdenum are manufactured from powder compacts which are subsequently not worked to produce a fully dense product.

Over the past ten years, there has been a significant improvement in the technology of powder metallurgy, particularly as applied to the forming of high-temperature metals. Recent advances result from a combination of factors, including changes in processing as well as materials. The failure of many early attempts to form high-temperature metals, such as superalloys, by powder metallurgical techniques can be traced back to the lack of suitable powders, and the present upsurge of activity is closely related to new techniques for making high-quality pre-alloyed powder in bulk through gas-atomization techniques. Pre-alloyed metal powders consist of particles of essentially similar composition to that of the melt from which they are for need. They produce a more homogeneous product than the blended alloys, which are made up of a mixture of elemental powders.

Pre-alloyed powder compacts are formed by sealing the powder in a thin-walled can to protect it from oxidation during heating and then extruding or pressing the can until the powders are consolidated into a solid mass closely akin to the usual mill product.

Currently, excessive hardness makes many superalloys effectively unforgeable. But powder technology, using pre-alloyed powders, will make it possible to use alloys with higher amounts of such hardening agents as aluminum, titanium, columbium and tungsten. Powder techniques may become even more significant as more high-temperature alloys are developed with superplastic properties. With the use of superplasticity, usually intractable superalloys are easily deformed, and afterwards heat treated to produce high-strength levels by eliminating the ultrafine grain structure of the superplastic state.

Several other developments in powder metallurgy, including hot forging as well as processes using isostatic and hydrostatic pressure, are proving to be significant. Both of these techniques are based on the use of molds surrounded by pressurized gasses or liquids. The pressure acts equally on all parts of the mold allowing complex shapes to be produced with uniform density — a characteristic usually impossible to achieve on conventional presses. A variety of modifications on the isostatic principle have been developed including cold, warm and hot pressing, and wet-dry-bag tooling.

The advantages in powder metallurgy technology have been evolutionary and have resulted from advances in the production of powders, a growing understanding of basic phenomena, such as superplasticity, and the availability of new processing techniques.

The advances in powder metallurgy can have a profound effect on the technology of high-temperature metals by allowing production of metals that were not formable by conventional technology, by cutting production steps, and by reducing metal wastage. It has been projected that powder metallurgy technology might soon replace the usual ingot-pouring approach to making many important high-temperature metals. Estimates call for powder to capture 40-50 percent of what is now a \$200 million jet engine superalloy metals market.

NASA has been active in applying powder metallurgical technology to the field of high-temperature metals. In particular, NASA efforts have focused on producing pre-alloyed powders of many commercially available and newly developed high-temperature alloys and working to enhance the properties of compacts made from these powders through various processing techniques, such as extrusion and thermomechanical processing. Included among NASA contributions to the advancement of powder metallurgical techniques are the following.

o Development of a heat-treatment technique to enhance the properties of powder compacts (04-16-01).

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To develop good elevated temperature properties in hot-pressed or extruded compacts made from pre-alloyed powders, it is necessary to eliminate the fine grain structure associated with such compacts through controlled grain growth.

In the course of developing powder metallurgical techniques using extruded pre-alloyed powders of cobalt and nickel-base superalloys (see NASA contributions to improved superalloys, 04-10-06 and 04-10-07) NASA/LeRC developed a new heat-treating technique which enhances the high-temperature properties of powder compacts. The technique involves exceeding the solidus temperature and employs the application of high pressure. Result is a substantial increase in elevated-temperature life of the pre-alloyed powder product. This technique not only provides significant grain growth, but also a solidification structure at the grain boundaries, both of which may be necessary for high-temperature strength of pre-alloyed powder products.

The concept of heat treating above the solidus and applying high pressure to restore the integrity of the material, thus closing voids formed as a result of minor-phase melting, appears to hold considerable promise for substantially increasing the high-temperature strength of compacted pre-alloyed products.

o Demonstrated use of powder metallurgical techniques using pre-alloyed powders to improve the intermediate-temperature strength of nickel-base superalloys (04-16-02).

By producing pre-alloyed powders, extruding them and then subjecting the compact to thermomechanical processing, a substantial increase in the intermediate temperature strength of nickel-base alloys was achieved.

For a complete description of this process developed by NASA/LeRC, see NASA contribution 04-10-06 to improved superalloys.

o Demonstrated use of powder metallurgical techniques using pre-alloyed powders to improve the intermediate-temperature strength of cobalt-base superalloys (04-16-03).

Pre-alloyed powder of a commercially available cobalt alloy was produced by NASA/LeRC. By extruding the powder to form a compact and then processing the compact by thermomechanical working, the intermediate-temperature strength was increased substantially. For a complete description of this demonstration of the application of advanced powder metallurgical techniques to property improvement, see NASA contributions to improved superalloys 04-10-07.

o Development of a mechanical process for producing submicron powders for dispersion-hardened alloys (04-16-04).

A process for producing submicron size powders for dispersion-hardened metals was developed by NASA/LeRC. The powders are prepared by grinding and blending in a special attrition-type ball mill using heptane as the grinding fluid and oleic acid as a surfacetint. The powders are reduced to less than -325 mesh. For complete description of this process see NASA contributions to dispersion-hardened metals, 04-04-01.

o Development and evaluation of new processes for producing metal powders (04-16-05).

Due to a great interest in superalloy powders and dispersion-hardened metals, NASA has carried out and supported under contract a wide variety of research investigations into new and novel processes for producing metal powders. Included among these efforts are the following: production of chromium and nickel powders by reduction of their oxides with magnesium,

lithium or sodium vapors (NASA/LeRC); the development of continuously operating atomization units for large-scale powder production using the splat cooling process (M.I.T.); and the production of dispersion-strengthened nickel-base alloy powders using the high-intensity arc process (Vitro Laboratories).

o Development of a process for the production of ultrafine, ultrapure metal powders (04-16-06).

A process for making high-purity, finely divided, metal powders, using an electron beam, was developed at NASA/LeRC. The process involves relatively low temperatures and a simple system to produce powders with particle sizes ranging from 0.1 to 1.0 microns, that are almost completely free of oxygen and nitrogen. Powders of metals such as nickel, cadmium, thallium, tin, lead, cobalt, copper, zinc, iron, silver and antimony have already been produced, and numerous others are possible.

The process entails irradiating a dilute aqueous solution of a metal salt (generally the sulfate, acetate or chloride) containing a readily oxidizable organic compound. Radiation causes the metal to precipitate. The ultrapure powder product has a very low sintering temperature and due to its purity, no oxygen or nitrogen is contained in the grain boundaries.

The feasibility of using this process to make powders for dispersion-hardened metals, as well as for other high-temperature alloys, is now under investigation.

o Development of a process to reduce chromium oxide content of chromium containing powders (04-16-07).

Almost all superalloy powders contain chromium as an alloying constituent, and as a result, also contain chromium oxide as a contaminant. For a complete description of this NASA/LeRC-developed process to reduce the chromium oxide content of powders, see NASA contributions to dispersion-hardened metals, 04-04-06 and 04-04-08.

o Established industrial health guidelines to reduce health hazards associated with the operation and handling of ultrafine metal and metal oxide powders (04-16-08).

Facilities that handle ultrafine metal and metal oxide powders in powder metallurgical work have

difficult health problems since the fine dusts handled in such operations can be toxic. To establish guidelines for the operations and handling of these fine powder materials. NASA/LeRC contracted with Viles, Chamberlin and Boylin of Norwood Massachusetts to appraise the special health hazards associated with, and the control of ultrafine metal and metal oxide operations. Materials studied in this effort included nickel, tungsten, thorium oxide, aluminum and aluminum oxide, magnesium oxide, zirconium oxide, niobium, molybdenum, cobalt and chromium. Based upon this study, safe limits for occupational exposure to the ultrafine dusts are proposed, and control measures for all operations recommended. Suggested threshold limit values are from two to fifty times lower than current threshold limit values recommended by the American Conference of Governmental Industrial Hygienists. The proposed levels reflect the potential increase in toxicity of the materials due to their existence in the ultrafine powder state.

o Development of a method for controlling density and permeability of sintered, powdered, refractory metals (04-16-09).

An improved, relatively low-cost powder metallurgical method to produce porous refractory metals with predetermined pore size, pore spacing and density, was developed by Electro-Optical Systems under a NASA contract. The need for such materials exists for a number of applications such as contract ionizers, transpiration and ablative cooling, boundary control surfaces and fluid filters. With this method, the pore size, shape and spacing are predetermined by adding an inert volatile material of the desired pore size and geometry prior to pressing and sintering.

The objects are formed by pressing a mixture of fine refractory metal powder such as tungsten and fine inert metal powder such as copper or boron nitride. The resulting compacted body is then heated to a temperature above the sintering temperature of the pressed compact and below the melting point of the inert additive. The body is then heated above the melting temperature of the inert metal to evaporate the inert powder and sinter the refractory metal to the selected pore size.

### Refractory Metal Advances (04-17)

Refractory metals are arbitrarily defined by melting point, with a melting temperature of over 3,500 F. usually selected as the minimum for inclusion in this group. Of eleven metals with melting points above 3,500 F., four have been advanced significantly in recent years, as bases for structural alloys, namely columbium (Db), molybdenum (Mo), tantalium (Ta) and tungsten (W). The remaining seven have received lesser development: rhenium, hafnium, iridium, rhodium, ruthenium, technetium and osmium. Chromium, a metal melting just at 3,500 F., is sometimes classed with the refractory metals, but due to its brittle behavior and difficulty in fabrications it has not found widespread application.

Fifteen years ago, there was only one refractory alloy (Mo-0.5Ti) in commercial production. Currently, several dozen useful Cb, Mo, Ta and W alloys exist, with many obtainable in good quality as sheet, foil, tube, wire, bar and forgings.

Refractory metals are unique in terms of their properties at high temperatures but there is a continuing problem with the surface stability of such metals. It is now possible to obtain refractory metal alloys with generally good engineering properties, but the continuing problem of oxidation resistance still remains. The amount of work going on in developing coatings for high-temperature, oxidation-resistance of the refractory metals is therefore extensive. These developmental efforts on refractory metals and refractory metal coatings are being strongly pushed by the Navy, Air Force, AEC and NASA.

Work on coatings is directed not only towards the development of new coatings but also towards a better understanding of the interactions between the coating and the substrate. Many short-term applications have been developed which will work. Most of the successful coatings have been based upon silicides. However, the time limitations are still severe.

To improve reliability the standard method has been to take a coating system which has proved to be reliable for 100 hours and to use it for only 1 hour. Present work is being directed towards improving the oxidation resistance of the basic refractory metal system and to improving the life of the entire system by a combination of changes in the matrix as well as the coating.

Refractory metals offer the promise of sophisticated alloys with long-term thermal stability. These metals can find applications in power generation with particular long-range potential in nuclear and fusion power systems. In addition, usable refractory metal alloys can open up a whole new range of materials and alloys for use in blades and vanes for jet engines and other aircraft structural parts which work at high temperatures. The key to widespread commercial use lies in

extending service life. For example, NASA's requirements call for about a 100-hour life, and the Navy's requirements call for a 1,000-hour life, but commercial requirements call for anywhere from 10,000- to 30,000-hour life.

For economic reasons primarily, refractory metals seem destined for very specialized uses. However, they will probably play an important role in aerospace and nuclear power.

In the refractory metal area, NASA has concentrated its efforts on the development of high-temperature protective coatings and research into new alloy formulations. Contributions to the advancement of refractory metal technology include the following, among others.

o Development of high-strength tungsten alloys with improved ductility (04-17-01).

As the highest melting metallic element, tungsten is of interest for load-bearing components which must operate at extremely high temperatures. A disadvantage of tungsten is that it becomes brittle at temperatures below about 250 F. and alloying to increase the strength often increases this ductile-to-brittle transition temperature. A research effort at NASA/LeRC resulted in the development of an alloy which combines superior strength at elevated temperatures with improved ductility at lower temperatures. This is done by combining the strengthening effect of a hafnium carbide (HfC) precipitate with the ductilizing effect of 4 percent rhenium. The composition of the alloy in weight percent is W-4Re-0.35Hf-0.025C. At 3,500 F., the alloy has demonstrated a maximum tensile strength of 75,400 psi., an eightfold strength advantage over the 9,000 psi. strength of unalloyed tungsten. Sheet material of this allow can be bent at temperatures as low as 175 F... compared to 250 F. for unalloyed tungsten.

Although developed primarily for space applications, it is anticipated that this alloy may find use in some industrial applications where unalloyed tungsten is now employed, such as electrical components, die casting materials, and heating elements.

This alloy is the outgrowth of an extensive series of investigations at Lewis of the tungsten-hafnium-carbon system. An earlier NASA-developed alloy W-.20Hf-0.26C has tensile strengths of 88,200 psi. and 62,500 psi. in the worked condition at 3,000 and 3,500 F., respectively. The use of hafnium carbide

as a strengthening agent represents a substantial advance in the technology of tungsten. For a discussion of HfC-strengthened molybdenum alloys, see NASA contribution 04-17-02, below.

o Development of high-strength molybdenum-hafnium-carbon alloys (04-17-02).

A series of hafnium carbide (HfC), precipitate-strengthened, molybdenum alloys (Mo-Hf-C) were developed at NASA/LeRC. These alloys are significantly stronger than commercially available molybdenum alloys (Mo-TZC), contain 0.8 to 0.9 mole percent HfC in molybdenum, and are worked by thermomechanical processing (TMP) for high-temperature service. The TMP schedule calls for extruding at 4,000 F., the temperature of maximum carbon solubility, followed by warm swaging at 2,500 F. This produces a highly-worked structure stabilized by a fine HfC precipitate.

For a discussion of the use of HfC strengthening agents in tungsten alloys, see NASA contribution 04-17-01, above.

o Development of chromium alloys for high-temperature service (04-17-03).

As a high-temperature material, chromium has certain advantages over currently used nickel-base alloys, including higher melting point, higher elastic modulus, and lower density. Unfortunately, chromium suffers an almost complete lack of ductility below the ductile-brittle transition temperature, which for unalloyed recrystallized chromium of commercial purity is around 300 F. Further, chromium suffers from severe embrittlement resulting from nitrogen contamination during high-temperature air exposure.

Since 1965, NASA has been engaged in contractural efforts with Westinghouse and G.E. to develop chromium alloys suitable for high-temperature service. Early work dealt with solid strengthening agents such as tungsten, which proved to be deleterious to the metal's high-temperature properties. Efforts are now directed at developing precipitation-strengthening mechanisms.

If a useable chromium alloy can be developed, it would have a 300-400 F. service temperature advantage over other alloys with comparable strength/weight ratios, which would open up a wide variety of turbine engine applications.

o Better understanding of long-time creep behavior of columbium and tantalium alloys (04-17-04).

Columbium and tantalium will be used as contaminant materials in advanced space electric-power systems, particularly those of the nuclear turbogenerator type. In such applications, the creep strength of the containing material is an important design consideration. In order to generate the required design information, NASA/LeRC carried out an evaluation of the long-time, high-temperature (2,000 and 2,200 F.) creep behavior of several commercially available, fabricable columbium and tantalium-base alloys. The results of this effort should prove useful to designers planning to use these alloys in other long-term, high-temperature situations.

o Better understanding of the use of rhenium and other softening agents to improve the low-temperature ductility of chromium, molybdenum and tungsten (04-17-05).

Rhenium as well as several other metals are of interest as softening agents because of their potential to lower the ductile-brittle transition temperature of certain refractory metals, such as chromium, molybdenum, and tungsten. In effect, such a lowering would improve the low-temperature ductility of these metals and improve forming and handling properties.

Because of this potential, NASA/LeRC undertook a basic research effort to determine the effects of temperature and alloy composition on softening in Cr, Mo and W. Results showed that alloy softening was similar in all three systems and that the softening agent content required to produce a hardness minimum diminished rapidly in all three systems with increasing temperature. (For an application of the use of rhenium to improve ductility in a NASA-developed tungsten alloy, see contribution 04-17-01, above.)

o Development of equipment for the hot extrusion of refractory metals (04-17-06).

Development of refractory metal alloys having high strength at temperatures in excess of 3,000 F. requires methods of working at temperatures much higher than use temperatures. However, existing extrusion containers and other tooling melt in the temperature range 2,400 to 2,600 F. To overcome this, NASA/LeRC developed accessories for a 1,020-ton vertical extrusion press to permit extrusion at ultrahigh temperatures. These include a high-temperature controlled-atmosphere furnace, a rapid billet transfer device, an extrusion sharpener, and an eight-tube extrusion receiver. The furnace permits heating for extrusion in either inert gas or hydrogen atmospheres to 5,000 F. This equipment allows the use of some refractory metal alloys that could not be considered before because of forming and fabrication difficulties. This should be of interest to engineers concerned with nuclear plant design.

o Development of basic design data on commercial and experimental refractory metal alloys (04-17-07).

To aid engineers dealing with materials selection and application problems in high-temperature systems. NASA systematically developed, evaluated and compiled critical design data on commercially available as well as experimental refractory metal alloys. Typical data compilations include the following, among others: biaxial creep data on tantalium, molybdenum and alloys T-111, TZM and TZC (NASA/LeRC); long-time creep data on refractory alloys at elevated temperatures (TRW); thermophysical, mechanical and chemical properties of tantalium alloy T-11 (NASA/LeRC); long-time creep behavior of tantalium-10 tungsten in high vacuum (NASA/LeRC); stress-rupture and tensile properties of refractory metal wires at 2,000 and 2,200 F. (NASA/ LeRC); creep and tensile properties of molybdenumtitanium-carbon alloy (NASA/LeRC); biaxial creep strength of T-11 tantalium alloy (G.E.); and mechanical behavior of tantalium-base T-lll alloy at elevated temperatures (TRW).

o Development of a series of tantalium-base alloys with resistance to creep determination at elevated temperatures (04-17-08).

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Refractory metal alloys which were commercially available in the mid 1960's did not possess sufficient high strength and resistance to creep deformation needed for use in high-temperature advanced space power systems of the nuclear turbogenerator type. To meet these requirements, NASA/LeRC contracted with Westinghouse Electric Corporation to develop a series of dispersion-strengthened tantalium base alloys.

The resulting alloys are significantly superior to T-222, the highest strength tantalium-base alloy which was commercially available at that time. Possible applications for these alloys are structural materials for fluid flow components in chemical processing and nuclear reactor equipment.

o Development of a precipitation-strengthened tantalium alloy (ASTAR-811C) with superior creep resistance and good workability (04-17-09).

A carbide-strengthened tantalium alloy with better creep resistance than commercially available tantalium alloys and good fabricating and welding characteristics was developed by Westinghouse Electric Corporation under a contract to NASA/LeRC. This new alloy, called ASTAR-811C, (with a composition of Ta-8W-1Re-0.7Hf-0.025C) has ductility and fabricating characteristics similar to Ta-8W-2Hf, a commercially available alloy, yet the creep strength of the new alloy at 2,400 F. is approximately double that of the commercial alloy. ASTAR-811C has useful strength to approximately 3,000 F. and can be scaled up to larger ingots. For an earlier series of Westinghouse-developed, tantalium base alloys, see NASA contribution 04-17-08, above.

o Development, evaluation and compilation of design data on the properties of coating materials for refractory metal alloys (04-17-10).

To provide up-to-date compilations of design data on refractory metal coatings, NASA systematically developed, evaluated and compared data on the properties of commercially available and experimental systems. Included are the following, among others: literature review on coating materials (Battelle); protective coatings for refractory metals (IITRI); evaluation testing of protective coatings on refractory metals (TRW); evaluation of mechanical properties, oxidation resistance and structure of slurry silicide coated T-222 (Westinghouse); evaluation of coated molybdenum and columbium alloys in high-temperature supersonic airstreams (NASA/LeRC); and a refractory coatings bibliography (NASA/MSC).

o Development of a technique for applying silicide coatings to refractory metal alloys (04-17-11).

Silicide coatings have been used with some success to protect refractory metal alloys used at temperatures up to 3,500 F. Conventionally, silicide coatings have been produced by the chemical reaction of the refractory metal parts with a silicon halide vapor. A metal silicide coating is formed. However, such coatings provide limited protection under varying operating conditions because they are thin and variation of the chemical composition of the coating is not feasible. Under a contract with NASA/LeRC, the Solar Division of International Harvester developed a two-step technique for producing a silicide coating of predetermined depth and selected composition which provides improved protection to the original metal substrate over a wide range of operating temperatures.

This technique provides coatings that protect both tantalium and columbium alloys at temperatures to 2,400 F. for up to four times as long as conventional silicide coatings, including the critical intermediate temperature range between 1,450 and 1,800 F. where simple silicide coatings tend to fail catastrophically.

#### Superplastic Metals (04-20)

Superplasticity is the ability of an alloy at high temperature to stretch drastically under very small stress without nicking or cracking. Certain high-temperature alloys, made from powders, have exhibited superplastic behavior, that is, they have been made to deform under a small applied stress.

This behavior is associated with the fine grain size of the powder billet, which inhibits recrystallization during hot forming. An equiaxed grain structure is one of the requirements for superplastic forming. There is a strong possibility that this equiaxed structure can be maintained through the entire forming process. Properties would thus be isotropic and there would be no cross grain weaknesses.

The real promise of superplasticity is as a new forming process. If a usually intractable alloy can be easily deformed, it can be formed into the shape that is needed; afterwards, it can be heat treated to produce a large grain structure in place of the ultrafine grain structure of the superplastic state, bringing the alloy back to its high strength levels.

Some of the most intransigent nickel and cobalt-base alloys might prove amenable to hot pressing because of the superplastic nature of the powder metal billet, provided that their normal mechanical properties can be restored by economical heat treatment cycles. Superplastic characteristics have been found in only a few alloys to date and most of these are experimental materials, but research indicates that such behavior is general and eventually will be produced in many alloy systems.

This development is based almost entirely on the well-known theory of superplasticity. Superplasticity is developed by forming a microstructure at a specific strain rate and at an appropriate temperature. The phenomena of superplasticity has been known for quite some time and it was the observation that some materials exhibited high creep rate on tensile tests that lead to the development of this phenomenon into a potential metal forming process.

Processes that make use of superplastic phenomena allow the working of normally intractable materials that cannot be formed using conventional deformation technology. In addition, forming can be carried out at a much lower cost. The process has the potential for forming low-cost components exhibiting high levels of mechanical properties.

NASA's efforts to advance the technology of superplasticity have dealt mainly with the identification and development of superplasticity in several high-temperature alloy systems and the development of methods for forming these alloys using the superplastic behavior of the material. Included among NASA contributions are the following.

o Development of a method for forming superalloys by heating pre-alloyed powders to a superplastic state (04-20-01).

A method for forming high-strength superalloys using superplastic characteristics, was developed by NASA/LeRC. The process uses pre-alloyed powders of superalloys capable of developing superplastic characteristics. The powders are placed into a thin-walled, mild-steel can, and then heated and extruded into bar stock. Materials treated in this manner exhibits superplastic behavior at high temperatures. This superplastic property can be used to advantage in forming the alloy. By applying low strain rates and very low deforming loads while the material is held at high temperature, the alloy's superplastic behavior allows it to be readily shaped at very low processing cost. The superplastic properties are subsequently removed by heat treating.

This new processing technique is significant in that it not only provides a low-cost forming method but it also provides a readily useable technique for achieving substantially higher strength alloys. Since micro and macrosegregation are virtually eliminated in the final product, it is possible to formulate alloy compositions with higher quantities of strengthening alloying constituents than can be accommodated by conventional casting and wrought processing techniques. For a further description of this process see NASA contribution to high-purity superalloy powders 04-07-03, and to improved nickel and cobalt-base superalloys. 04-10-06 and 04-10-07.

o Identification and development of superplastic behavior in high-temperature metals (04-20-02).

The identification and development of superplastic behavior in a number of high-temperature alloy systems has been achieved by NASA/LeRC. Included among these systems is a NASA-developed superalloy TAZ-8A, a commercially available superalloy 713C, and a tungsten-rhenium alloy. Superplastic behavior was also identified in a Co-45W-0.05Zr alloy by researchers working under a NASA grant at Case Western Reserve University.

## Better Understanding of Failure Mechanisms (04-22)

The development of new high-temperature metals has exerted great pressure to develop a thorough understanding of the problems of structural failure caused by material fracture. Underpinning the development of fracture mechanics is the concept that when metals are fabricated into hardware or structures they will contain flaws, or will develop them within their service lifetime, and these flaws or cracks can drastically change metal behavior. Aircraft, ships, turbines and nuclear reactors are just a few examples of structures which rely on fracture mechanics in their design and long-term performance.

Most work in fracture mechanics has been directed towards the development of test methods for the qualitative evaluation of brittle crack propagation resistance, or fracture toughness, and towards the development of quantitative measures of fracture toughness which would be useful in design. Understanding has reached the point where concepts of fracture mechanics are being applied to actual problems and it appears to be having an effect upon design practices. The evolving technology is providing the tools to handle design problems related to the application of high-temperature metals.

While the field of fracture mechanics is quite old, present interest dates to problems with World War II liberty ships, and to the catastrophic failure of transmission pipelines and pressure vessels. Early work dealt with low-temperature problems, but present efforts have been extended to high-temperature metals to predict component life.

Understanding of how metals fail is already having an effect upon design practices and is being applied to existing problems. Design procedures have changed so as to improve the reliability of various materials and components. Knowledge of fracture mechanics is becoming useful in an engineering sense in that it is being translated into the types of tests and structural mechanics familiar to the metal producer and designer, and is having and should continue to have an increasingly larger impact upon component reliability.

Fracture mechanics has now matured into a key branch of stress analysis and structural design. Numerous structural design problems are being solved in industry using these concepts, in such areas as developing new alloys, designing thick-walled structures for nuclear reactors and pressure vessels, designing turbine generator rotors, analyzing structural failures, and generating a British Standard Test Method for Fracture Toughness.

NASA's contributions to advancing our understanding of how materials fail, date back to its pre-space-age predecessor, the National Advisory Council on Aeronautics (NACA). While these contributions relate to the entire problem area of structural failure, discussion in this section is limited to the field of high-temperature metals. In this field, NASA efforts have clearly contributed to a better understanding of the failure mechanisms of high-temperature metals and have had a major effect on the rate and direction of technological advancement, specifically, the development of fracture toughness test methods and their application to high-temperature design problems. As a result of these efforts, designers using high-temperature materials are now able to relate applied loads and flaw size to material properties, thereby accurately predicting the integrity of a structure for its intended high-temperature application. In addition to the development of fracture toughness test methods, NASA has played a major role in the application of the theoretical concepts of fracture mechanics to hightemperature structural design practice. Major NASA contributions to this development follow.

o Development of test methods for the quantitative measurement of fracture toughness (04-22-01).

Conventional design engineering practice has relied on several established criteria for assessing a material's ability to resist brittle fracture. Percent elongation and reduction in area—both measures of the amount of deformation in the

vicinity of a fracture—are widely accepted indices of ductility. Another criteria, based on the Charpy impact test, has found broad application in determining the temperature at which a material becomes brittle. Unfortunately, neither the percent elongation nor the Charpy impact strength can deal with sharply reduced crack strength of the material.

To overcome this, NASA has supported a major effort directed at two closely related objectives:
(1) the development of a quantitative measure of fracture toughness to assist in structural design and proof testing; and (2) the development of test methods for qualitative evaluation of crack propagation resistance that would be useful as a screening tool in alloy development. The result of extensive research to achieve the first objective was the plainstrain fracture toughness test, developed by NASA/LeRC.

This test provides a quantitative measure of a material's resistance to fracture, called the critical stress intensity factor ( $K_{\rm IC}$ ). Using this factor, a designer can directly relate flaw size to the load-carrying capability of a structural member, something that was impossible to do before. This NASA-developed technique has gained industry-wide acceptance as the prime method for assessing the fracture toughness of high-strength materials and has been incorporated as an ASTM standard (ASTM E 399-70T).

o Development of test methods for the qualitative evaluation of fracture toughness (04-22-02).

For many ductile materials it is impossible to determine a K<sub>TC</sub> value because of the prohibitive thickness required for test specimens. Furthermore, testing in accordance with ASTM method E-399-70T is expensive and requires relatively sophisticated laboratory practice. In many instances, there is a need to economically determine comparative measures of fracture toughness. A particularly difficult problem of this type was the screening of materials for use in solid propellant rocket motor cases. was overcome by development of the ASTM Test Method for Sharp-Notch Tension Testing of High-Strength Sheet Materials (E-338-68). Two types of screening specimens are suggested for use in this test. The first was developed by the Naval Research Laboratory; the second, developed by NASA/LeRC, is a sharp edge notched specimen, and is the more regularly used of the two.

This ASTM test, using the NASA-developed specimens, can be applied to the development of new high-temperature alloys, where a large number of samples are required to optimize heat treatment, alloy purity and temperature sensitivity. These kinds of screening tests do not replace the need for  $K_{\rm IC}$  measurements, but do provide a rapid and economical means for determining the relative toughness of metals.

o Development of industry standards for linear crack stress analysis (04-22-03).

The wide industry acceptance of both the quantitative and the qualitative measures of fracture toughness is heavily due to the activities of the ASTM E-24 Committee on Fracture Testing of Metals. NASA's contributions to the effort of this committee have been major, with NASA engineers providing the primary driving forces for both technical achievement and administration of the committee.

The ASTM standard (E-399-70T) for quantitative determination of fracture toughness was written by NASA engineers and has been accepted by the Society of Automotive Engineers, MIL Handbook-5, and the British Independent Steel Producers Association. The engineering community has generally adopted the NASA-developed K<sub>IC</sub> designation as the primary way of describing fracture toughness. NASA has sponsored an ASTM conference on this subject as well as distributed ASTM literature.

While most experience has been confined to relatively brittle materials, application for the use of plane-strain fracture toughness, K<sub>IC</sub>, have been growing rapidly, especially in the design of thick section structures. In the electric power industry, for example, many structures require the use of heavy sections made from low-to-intermediate strength steels. Pressure, vessels, turbine and generator rotors and nuclear reactor shells are examples of such structures. Westinghouse has conducted extensive research related to the application of linear elastic fracture mechanics technology to the design, inspection and maintenance of large turbine-generator rotors.

The rapid rate of acceptance is due not only to the ASTM activities of NASA engineers but also to activities directly related to aiding industry in accepting and using these methods. Linear crack stress analysis tests are now required by NASA on appropriate contracts and are written into RFP's. To help contractors meet these requirements, NASA/LeRC personnel provided technical assistance in the form of information as well as the use of their facilities, helping contractors to develop their own test facilities and to analyze and apply test results.

o Development of the concept of strain-controlled fatigue failure (04-22-04).

As the name implies, fatigue represents a tiring or weakening of a material with time. More specifically, fatigue deals with the influence of repeated cycling on crack growth. Since fracture mechanics considers the energy required for crack initiation and growth as well as the effects of geometric factors on crack propagation, fracture mechanics and fatigue are interdependent disciplines. Aircraft, rockets, turbines and reactors are just a few of the structures which are deeply concerned with fatigue failure.

Because of the expense and difficulty involved in long-term fatigue testing, a method for estimating the fatigue characteristics of a material from properties measured in simple static tensile tests was developed at NASA/LeRC in the early 1950's. Using this technique fatigue properties of metals can be estimated from data on tensile strength, ductility, fracture stress and elastic modules. This test is based upon several major NASA contributions leading to a more advanced understanding of fatigue failure in engineering materials, and leading to the concept of strain-controlled fatigue failure of materials, that is, that fatigue failure is related more fundamentally to strain than to stress. This is an almost universally accepted concept today.

o Development of techniques for estimating high-temperature, low-cycle fatigue (04-22-05).

Low-cycle, strain-controlled fatigue is recognized as an important failure mode in some components of gas turbines, nuclear reactors and other high-temperature systems. While reasonably valid procedures have been devised for obtaining estimates of fatigue behavior at low and intermediate temperatures, these procedures are

not directly applicable at high temperatures where creep processes begin to interact with fatigue. Research efforts at NASA/LeRC were instrumental in explaining the nature of this interaction and in developing a number of techniques for estimating low-cycle fatigue behavior in the creep range. One such procedure, called the "10 percent rule" is now used by designers as a rule-of-thumb. While this rule doesn't apply over the entire range of cyclic frequencies, it is still quite useful. Other simplified techniques have been developed at NASA/LeRC and are providing valuable guidelines for the estimation of the high-temperature fatigue behavior of existing as well as new materials.

o Development of techniques for prediction of the longtime static behavior of materials (04-22-06).

The need for correlating creep-ruptive data and for extrapolation of such data to long ruptive times has become increasingly important due to the many new applications that have developed in recent years involving extended service of materials, such as in nuclear power generation, the long-time properties of which are now known. Even if service time is moderate, it is important to reduce the test time in order to make possible the evaluation of numerous materials and to expedite the time required to make a decision regarding basic suitability.

Extensive efforts by NASA/LeRC, to devise estimating techniques using time-temperature parameters, has resulted in the development of several which enable prediction of long-time creep behavior of metals and which can be used to design structures for long life at elevated temperatures. These techniques are based upon a NASA/LeRC-developed generalized formulation using a station function approach. This technique is now under evaluation by the NATO AGARD committee for possible use as a way to develop materials standards.

### INTEGRATED CIRCUITS (05)

The computer industry, the military, aerospace, and the electronics industry as a whole, have place ever-increasing demands on the semiconductor manufacturers for smaller size, more economical, and more reliable solid state devices. This has forced the semiconductor device manufacturers to create a new technology which has been entitled "microelectronics" and from which a new group of sophisticated devices has emerged called "integrated circuits." Microelectronics has been classified into three separate areas based primarily on fabrication techniques: thick-film hybrid microelectronics, thin-film microelectronics and monolithic integrated circuits. The first group, thickfilm hybrid microelectronics, is based upon a thermomechanical approach to attaching individual active semiconductor devices and passive circuit elements, (e.g., capacitors) to a ceramic substrate preprinted with a conductor circuit pattern. Thick-film hybrid microelectronics have an added advantage over standard printed circuits in that most of the resistors can be printed, saving the cost of soldering, increasing reliability, and reducing space requirements. The second group, thinfilm microelectronics, uses methods based on vacuum deposition of the conductor, resistor and capcitor materials, onto the ceramic substrate. The third group, monolithic integrated circuits, is based upon fabrication techniques where the silicon semiconductor material itself is the substrate. The active and passive circuit elements are formed by either chemical or physical junction forming processes.

Each of the above three circuit-producing approaches have a number of practical advantages and disadvantages, related to cost, size, and end application. Monolithic integrated circuits, for example, have as their prime advantages low individual device cost, small size, performance and reliability. A list of significant developments, as identified by technological leaders in the field of integrated circuits, is given in Table 5. Many of these developments are related to improving the advantages mentioned above. For example, multilayer metallization was developed to allow vertical and overlapping interconnections between circuit elements; photomask technology advancements made it possible to achieve greater line and space resolution; and ion-implantation offers a method of accurately controlling the penetration and concentrations of the necessary impurity ions. All of these processes are making possible the large-scale integration of many hundreds of electronic circuits on a single tiny piece of silicon.

The military and aerospace applications of semiconductor devices in varied and extreme environments imposed reliability requirements on semiconductor components manufacturers which required extensive investigations into improving the device's stability. Instabilities were found to be related to both bulk imperfections and surface impurities. Stability has been improved by better manufacturing techniques, use of purer materials, more effective coating and passification procedures, and better control techniques to detect potentially unstable devices.

Growing circuit complexity, reduced size, and increasing costs have prompted circuit-design concepts which are a radical departure from classical design approaches. The advent of the computer as a design tool and its ability to create circuits from only input/output parameters has resulted in the production of custom circuits without the traditional dependence on space consuming resistors and capacitors.

The computer has also come to the aid of those faced with the task of isolating the newly generated complex multi-element devices which do not meet operational requirements.

In 1970 the monolithic integrated circuits portion of the semiconductor industry amounted to \$431.7 million, or 34 percent of the total semiconductor industry sales volume of \$1.266 billion. Market estimates prepared in early 1971 indicate that by 1974 integrated circuits sales will reach \$848 million and will exceed 50 percent of total semiconductor sales.\* These figures do not tell the whole story, however, in that the modern electronics industry is almost wholly dependent on the semiconductor industry in satisfying market needs. Total electronics industry sales (including military and aerospace, consumer, and industrial sectors) was \$24.6 billion in 1970; \$25.1 billion in 1971; and will reach an estimated \$32.2 billion in 1974. NASA electronics expenditures, although declining (\$1.5 billion in 1960, \$1.21 billion in 1970; and an estimated \$1.11 billion in 1974) represent sizeable expenditures, with a significant portion of the research funds devoted to advancing integrated circuits technology.

WASA's space activities would be severly curtailed without access to sophisticated electronics and, in turn, integrated circuits. Size and reliability, two of the three major advantages of the integrated circuits technology, are key prerequisites to the vehicular-borne communications, guidance, data collection, and biomedical surveillance systems which NASA requires for effective aerospace research and operations.

The high-volume uses of integrated circuit devices in the decade of the '70's will most likely be in: computer terminals, miniature calculators, mini-computers, and data transmission. The major challenges facing the industry today include the need to develop high-volume testing techniques for increasingly complex devices, and the need to develop automated circuit modification and fabrication techniques.

Selected significant developments in the field of integrated circuits (as listed in Table 5) and NASA contributions to the advancement of these developments, follow.

<sup>\*&</sup>quot;U. S. Markets 1971 Forecast," Electronics, January 4, 1971.

#### TABLE 5

# SIGNIFICANT DEVELOPMENTS IN THE FIELD OF INTEGRATED CIRCUITS\*

- 1. Amorphous semiconductors (05-01)
- 2. Beam lead technology (05-02)
- \*\*3. Circuit concept design (05-03)
  - 4. Complimentary metal oxide integrated circuits (05-04)
- \*\*5. Computer aided circuit design (05-05)
- \*\*6. Deposition of thin films of silicon on insulating substrates (05-06)
  - 7. High speed integrated circuits (05-07)
- \*\*8. Improved stability of metal oxide semiconductor transistors and integrated circuits (05-08)
  - 9. Increased integrated circuit yields (05-09)
- \*\*10. Ion Implantation (05-10)
  - 11. Large scale integrated circuit memories
     (05-11)
- \*\*12. Large scale integration (05-12)
  - 13. Linear integrated circuits (05-13)
- \*\*14. Multilayer metallization (05-14)
  - 15. Magnetic bubble technology (05-15)
  - 16. New device structures (05-16)
- \*\*17. Operational chip testing (05-17)
- \*\*18. Oxides and protective coatings (05-18)
- \*\*19. Photomask technology (05-19)
  - 20. Plastic packaging (05-20)
  - 21. Very thin epitaxial layers (05-21)

<sup>\*</sup>As identified by technological leaders in the field of integrated circuits.

<sup>\*\*</sup>Developments selected for identification of NASA contributions.

### Circuit-Concept Design (05-03)

Circuit-concept design is the technique of designing complex integrated circuits based on the properties of the semiconductor chip, and black-box input/output requirements. Circuits created by these techniques do not necessarily have a discrete component counterpart. The traditional approach to designing circuits to fulfill specific functions has been dependent upon the empirical modification of existing circuit designs, optimizing individual circuit elements such as resistors, capacitors and semiconductor components, until desired results are obtained. Circuit-concept design approaches allow the designer to consider the demands of the total function and adjust the overall parameters such as gain, switching speeds, impedance, capacitance, etc., to meet those demands.

Advanced circuit concept design techniques provide device manufacturers with greater design flexibility and are based on the knowledge that passive elements such as resistors and capacitors can be replaced by easily produced, easily altered, lower cost transistors. This is a major advance in circuit design philosophy.

Circuit-concept design has been actively used since 1965 by General Micro Electronics, General Electric, Fairchild, Motorola, RCA, and IBM. A number of currently available commercial devices have resulted from the use of this technique, for example, the Fairchild 709 operational amplifier, the read-only memory, and the MOS dynamic read-write memory. The application of circuit-concept design is becoming increasingly necessary in the electronics industry due to the increasing complexity of modern circuits and the attendant increases in design costs.

NASA's major contributions have been to refine and demonstrate the use of circuit-concept design techniques. At the Jet Propulsion Laboratory, the technique was used to create the epitaxial base resistor, making it possible to have a large resistance value in a small space. At NASA/GSFC, the "integrated inductor" was invented, using circuit-concept design techniques, demonstrating the power and usefulness of this approach. The inductor eliminated the need for conventional wire-wound inductors and transformers and allowed a substantial reduction in circuit size and at the same time improved performance. At NASA/ERC low-power multiplier arrays were designed and developed again using circuit-concept design techniques. This type of array has subsequently become a standard commercial product from RCA.

The aggregate result of the NASA contributions has been to advance the time scale for widespread acceptance and use of circuit-concept design; the impact to the development is estimated to be moderate. Hardware produced by these techniques should have a substantial impact. NASA contributions to the advancement of circuit-concept design include the following.

o Developed the mathematical models needed for advanced circuit-concept designs (05-03-01).

While involved in developing circuitry for the EXAM Multiprocessors which will be used for processing aerospace scientific data. NASA/ERC developed the most advanced technology in the large-scale integration (LSI) of both bipolar and complementary metal oxide semiconductor/field effect transistor (MOS/FET) circuits. To meet the requirements of high reliability, low power consumption, high processing capability, and fail-safe modes, circuit-concept designs far in advance of regularly available traditional approaches had to be developed. A group of multiplier arrays were designed using complementary MOS technology from models and mathematics developed by Solid State Scientific, Inc. under NASA contract. Although Fairchild Semiconductor Corporation had done similar work with high-powered devices, the development of low-powered devices would not have taken place without the NASA requirement and funding. Major potential commercial use of the arrays will be in portable calculators. These calculators are developing into one of the more significant industries of the 1970's.

o Demonstrated the application of mathematical modeling techniques to produce low-power multiplier arrays (05-03-02).

Using the circuit concepts, mathematics and models developed to design special circuits using MOS-technology (see contribution 05-03-01 above), a series of multiplier arrays were designed for use in the EXAM Multiprocessor. RCA is now commercially exploiting a line of products for computing applications which appear to be based upon the same mathematical models and circuit-concept designs as those developed by NASA/ERC and Solid State Scientific, Inc., and demonstrated in application in the multiplier arrays.

o Developed the concept of the epitaxial base resistor, or "pinched resistor," making it possible to have a large resistance value in a small space (05-03-03).

In the process of designing a metallized semiconductor large-scale array with 116 logic gates for the Grand Tour mission, a circuit concept was developed for generating high resistance values in much smaller space than possible using conventional integrated circuit fabrication techniques. These resistors. developed by Harris Semiconductor Corporation under contract with JPL, are now known in the semiconductor industry as epitaxial base resistors, or, more commonly, as "pinched resistors." Such devices should find extensive use in low-power applications such as minicomputers, calculators, mobile communications systems, etc. 1971 sales of these systems will approach \$500 million.

o Demonstrated the potential of circuit concept design by developing the "integrated inductor" (05-03-04).

The major obstacle to exploiting the advantages of microelectronics for reducing the size and power consumption of communications equipment has been the continuing use of wirewound inductors and transformers for tuning elements and rf coupling. "Inductorless technology," invented at NASA/GSFC, represents a major advance in communications technology in that it overcomes the need for such components, and offers the advantages of small size; low dc power consumption; lower cost through circuit simplicity and the use of common transistors; improved selectivity; and lower circuit noise. Goddard, working in conjunction with the Orlando Division of Martin Marietta, developed the equivalent circuit models and mathematics which made it possible to synthesize the integrated circuit receivers without wire-wound inductors. The design procedures were an outgrowth of NASA-sponsored work at the Gaertner Research Corporation which was also involved in the NASAP computer-aided circuit analysis program. (For a more complete description of NASAP, see computeraided circuit design, development 05-05.) The potential areas of use for integrated inductors

will be in consumer electronics such as AM and FM radio and television; police communications; commercial airlines; and military and aerospace. The total sales volume potential of these markets in 1974 is estimated to be between \$5 and \$6 billion.

## Computer-Aided Circuit Design (05-05)

Because of the growing variety and complexity of integrated circuits, computer-aided circuit design is playing an increasingly important role in the design and development of new and improved integrated circuits. Custom designed devices, which are becoming increasingly complex, are closely tied to the equipment manufacturer's program. Therefore, fast design completion time is essential.

In using computer-aided circuit design techniques, the design program is read into a computer. The designer then selects components or building blocks from a component library and specified their interconnections. Computer inputs are power supply values, temperatures, input and output loads, along with indicators for the specific performance curves the designer wishes to evaluate. The computer analyzes the circuit, and performance plots are displayed on a cathode ray tube. Modifications are made until the proper performance is obtained. Various programs are used to determine the physical interconnections and component layout. When the designer's needs are satisfied, the final design is recorded on magnetic tape.

The primary significance of computer-aided circuit design is fast circuit design completion time and more efficient use of engineering talent. The end results are more complex circuits, higher yields and reduced costs.

Computer-aided circuit design has only recently come into wide-spread use, but has already had a large impact on both integrated circuit and discrete component design. The increased availability of the computer, lower computational costs, a better understanding of how to use the computer for circuit design, and the need to design complex reliable circuits as quickly as possible, has prompted the development of computer-aided circuit design. All of the major integrated circuits manufacturers now use computer-aided design techniques for creating complex integrated circuits.

All of the NASA research centers with electronic circuits design groups, have sponsored programs to advance the use of the computer to aid in the design of complex circuits for electronic instrumentation. In addition to developing the models and necessary mathematics and techniques, NASA also prepared computer programs and made the programs available to all circuit designers. A number of new circuits were developed by NASA using computer-aided design techniques, some of which

have attained commercial acceptability. The significance of these contributions lie in advancing the time scale in which computer-aided circuit design has been developing. The estimated impact of these contributions to the advancement of computer-aided design is moderate. However, the products produced using these techniques, should have a substantial impact. Included among NASA contributions to computer-aided circuit design are the following.

o Demonstrated use of computers to synthesize circuits from input-output (black box) specifications (05-05-01).

NASA contributions to the advancement and development of new circuit-concept designs (see circuit concept design, development 05-03), specifically the development of models and mathematics to produce low power multiplier arrays at NASA/ERC (contribution 05-03-01), the development of the "pinched resistor" at JPL (contribution 05-03-03), and the development of the "integrated inductor" at NASA/GSFC (contribution 05-03-04) were made possible by using a computer to analyze, adjust and create the unique circuits and devices. These applications demonstrated the power and importance of computers as a design tool in synthesizing circuits from input-output specifications, and significantly advanced the acceptance of computer-aided design procedures.

o Developed and made readily available to design engineers a computer-aided circuit analysis program (05-05-02).

NASAP, an acronym for "network analysis for systems applications program," is a computer-aided circuit analysis program which can be used by electrical engineers without any knowledge of computer programming.

Many organizations and individuals participated in the development of the NASAP program, including the Automated Techniques Branch of NASA/ERC, Stevens Institute, General Precision Corporation, UCLA, University of Santa Clara, University of Missouri, and Gaertner Research, Inc.

NASAP is continuously being expanded at a number of research centers and it is expected that increasingly more powerful versions will be released to users from time to time.

### Improved Stability of MOS Transistors and Integrated Circuits (05-08)

Metal oxide semiconductor (MOS) devices, first introduced in the early 1960's, have the advantage over discrete conventional devices in requiring fewer processing steps, and in having smaller geometries, greater device densities and lower costs. MOS devices are a necessary prerequisite to the development of large scale integration of complex circuits. Until recently, such devices could not be used extensively, because they were electrically unstable. This instability was primarily due to contamination from sodium ions, which came in contact with the surface of the silicon dioxide layer during the manufacturing process. The sodium ions migrated along the silicon dioxide layer under prolonged bias, and caused the threshold voltage of the device to change.

The problem was eliminated by extensive research into developing a better understanding of semiconductor surface physics; the use of purer materials, cleaner processes and improvement in the chemical vapor deposition of dielectric materials; and ultimately the use of improved passivation techniques.

Most of the early work was done by RCA and produced limited results. In 1966, the Bell Telephone Laboratories introduced two techniques for passivating the active surface of the device. One technique was to coat the silicon dioxide layer with silicon nitride to prevent contamination. The second used polycrystalline silicon as a gate material in place of aluminum. The polycrystalline silicon prevents sodium ions from contaminating the silicon dioxide layer. The aluminum metallization is then placed on top of the polycrystalline silicon.

Practical devices using stabilization techniques such as those mentioned above, were not manufactured until 1968. Numerous organizations have contributed to the development of acceptable techniques and these are now extensively used as a routine part of manufacturing process. Included are firms such as Intel, Fairchild, RCA, IBM, Texas Instruments, Motorola, General Micro Electronics, and Hughes. MOS devices are now being extensively used in military, aerospace, and industrial applications, instead of conventional discrete devices, to increase reliability, reduce size, and reduce weight. Total sales of MOS devices were \$68 million in 1970 or 16 percent of the total monolithic integrated circuits market of \$432 million. MOS sales are estimated to increase by 65 percent in 1971 to \$102 million.

NASA has long been concerned with overcoming the stability problem of MOS devices to increase their long term reliability, so as to exploit their advantages for critical space applications. NASA efforts led to an increased understanding of the semiconductor material-oxide coating interface; aided in identifying the failure modes in MOS devices and ISI integrated circuits; brought about the development of techniques to deposit clean oxides; improved the reproduceability of the oxide coatings;

developed methods to restore devices made defective by extended exposure to radiation; and helped several businesses in improving their products by providing them with technical assistance to meet stringent NASA requirements.

These contributions helped increase the stability of MOS transistors and integrated circuits at a time earlier than it would have occurred otherwise, and the impact of the contributions is estimated to be moderate to high. Among NASA's contributions to advancing the development of more stable MOS transistors and integrated circuits are the following.

o Developed a technique to restore defective devices after breakdown in an extended radiation environment (05-08-01).

Oxides and structures of metal oxide semiconductor/field effect transistors (MOS/FET). Which are extremely sensitive to extended exposure to radiation, were modified by NASA/GSFC by implanting boron atoms at the interface between the oxide layer and the silicon substrate. Devices so treated are self-healing at room temperature, and further, allows ground-based engineers to restore defective devices in space vehicles. While non-aerospace applications are limited, the ability to remotely restore defective devices after breakdown in an extended radiation environment represents a significant technical advance for the semiconductor industry in their attempts to produce radiation-resistant devices.

o Advanced the understanding of semiconductor material/oxide coating interfaces (05-08-02).

One of the major areas affecting device stability is the interface between the oxide coatings and the semiconductor base material surface. Significant contributions to the overall understanding of the physical properties of the silicon-silicon dioxide interface were made by Philco-Ford under contract with NASA/ERC, particularly those factors that affect the fundamental electrical properties of the silicon-silicon dioxide interface. This understanding represents a major contribution to determining the failure modes of multilayer large-scale integrated circuits and served as the basis for developing techniques to improve the reliability and performance of LSI devices (see contribution 05-08-04).

o Assisted in development and early support of commercial market for specialized MOS integrated circuits (05-08-03).

Because of the many potential advantages of MOS technology, NASA/GSFC attempted to develop MOS devices in early 1964 but found they could not produce stable, highly reliable transistors. A contract was let to General Micro Electronics (GME) in late 1964 to produce MOS integrated circuit chips that would meet NASA specifications. GME was subsequently purchased by Philco-Ford and the program dropped. A contract was then placed with a newly formed company named American Micro-Systems, Inc. (AMI). The firm developed the technology required to produce stable MOS devices to meet NASA specifications, and became one of the early suppliers of specialized commercial MOS devices. AMI has subsequently gone on to become a major source of such devices throughout the country. For a description of the application of these devices see the field of telemetry, NASA contribution 12-07-02.

o Identified failure modes in MOS and LSI integrated circuits (05-08-04).

In NASA's efforts to develop a better understanding of semiconductor material/oxide coating interfaces (see contribution 05-08-02) they also determined failure modes in large-scale multilevel integrated circuits. Because of this better understanding of the fundamental electrical properties of the interface in multilayer microcircuit structures, developed by Philoo-Ford under contract with NASA/ERC, techniques have been developed for improving the reliability and performance of large-scale multilevel microcircuit arrays. These NASA contributions were prerequisites to further advances in large scale integration, and were instrumental in enhancing the advancement of the MOS technology.

o Developed techniques to locate the actual leak path in integrated circuit multi-lead devices (05-08-05).

One of the major instability mechanisms affecting the reliability of integrated circuits is moisture leaking into the fragile glass-to-metal seals used in the multi-leaded package containing

the semiconductor chip. A technique to absolutely locate the actual leak path in the package was developed by NASA/ERC. The package is back-filled with fluorocarbons which in turn can be detected as they escape with a halogen leak detector probe. Prior to this development it was impossible to detect all of the leak paths. This technique is now used by all of the major semiconductor manufacturing companies.

o Improved MOS and integrated circuit stability by developing techniques for depositing "clean oxide" coatings (05-08-06).

A method for preparing a stable, nonpolarizable oxide coating for use as passivation and dielectric for integrated circuits and MOS transistors was developed by NASA/ERC. The method consists of:
(1) forming the silicon dioxide layer on the silicon substrate; (2) depositing a phosphosilicate glass film on the silicon dioxide layer to serve as a "getter" for mobile ions; and (3) etching the layer of phosphosilicate glass to remove any mobile ions present in the glass or at the glass-silicon dioxide interface to prevent polarization effects.

o Improved MOS and integrated circuit stability by developing techniques for depositing reproduceable oxide coatings (05-08-07).

In the process of developing techniques for depositing clean oxides, measurement patterns and improved monitoring techniques were developed, making it possible to achieve greater reproduceability in the oxide coating thickness and greater consistency in coating thick layers across the whole critical area than was possible before. This work sponsored by NASA/ERC at Philco-Ford Corporation is the basis of testing procedures now used throughout the semiconductor industry.

o Improved MOS and integrated circuit processing capabilities by providing technical assistance to commercial manufacturers (05-08-08).

In order to develop more reliable sources of MOS and integrated circuit devices, NASA instituted a "NASA microelectronics reliability program." This program which set criteria for acceptance of semiconductor devices, required

NASA inspection of the production facilities of semiconductor suppliers. NASA provided assistance to those companies, not able to meet the acceptance criteria, in making the necessary changes to become acceptable. Semiconductor manufacturers, such as Motorola, Fairchild, Harris, and ITT, who desired to supply MOS devices to NASA programs had to comply with the NASA microelectronics reliability program specifications. Many of the major integrated circuits manufacturing companies participated in this program, working with NASA to improve their processing capabilities to meet NASA specifications, leading to subsequent improvements in their commercial capabilities.

o Established the criteria for acceptance of high-reliability microcircuits (05-08-09).

The basic standard for high-reliability microcircuits for non space applications, used by many commercial semiconductor manufacturers, is the "Line Certification Requirements for Microcircuits," MSFC Drawing No. 85 MO 3877 and the "Microcircuits, High Reliability Certified Lines List and Qualified Circuits List," MSFC Drawing No. 85 MO 2706. These requirements were developed as part of the NASA microelectronics reliability program and many of the major integrated circuits manufacturing companies having been qualified to this acceptance criteria, use these same standards for their commercial customers.

## Ion Implantation (05-10)

Ion implantation, first demonstrated in 1962 by T. Alvager and N. J. Hansen at the Argonne National Laboratory, provides the integrated circuits process engineer with a technique to precisely control the concentration, penetration, and location of the n- or p-type impurity ions which determine the electrical characteristics of semiconductor devices.

Diffusion is currently the most acceptable production method for doping (introducing impurity ions) semiconductor materials. In diffusion, the doping agents (dopants) are presented to the surface of the semiconductor, and under the proper temperature treatment, they diffuse into the bulk material. In the ion implantation process the dopants are ionized and then driven into the semiconductor material by

being accelerated to high velocities by a high-intensity electric field. This technique, however, did not become practical in the manufacture of semiconductor components until it was possible to produce very thin epitaxial layers.

In spite of the substantial capital equipment costs associated with ion implantation, the many potential advantages of the approach have prompted increasing efforts to better understand the process, to develop production-size fabrication technologies and, to determine its practical limitations. Among the advantages of ion implantation are the following: (1) the process offers a wider choice of dopant-substrate combinations; (2) the process does not depend on the solubility or diffusibility of the dopant; (3) the process allows very shallow and/or abrupt junctions; (4) the process allows submerged layers of opposite polarities to be made in one step; (5) the process allows direct electrical measurement of the doping dose; (6) the process allows changes and variations in the order of processing steps; (7) the process eliminates several production steps; (8) the process makes purer doping possible; (9) the process does not spread the dopants laterally; and (10) the process allows formation of junctions beneath existing passivated layers.

Probably the most significant advantage is the ability to focus the beam to a spot size on the order of 5 Angstroms in diameter. This makes it possible to create circuit patterns with component densities an order of magnitude greater than the light-dependent photolithographic techniques now used. Beyond this, it offers the ability to take full advantage of the computer as a design aid to create and vary circuits at will.

It appears that ion implantation has the potential of being one of the most significant processing developments in the semiconductor industry in the 1970's. Given the predictions of \$1.7 billion sales in this industry in 1974, the widespread acceptance of this development should have a significant dollar impact on the economy.

A number of companies are experimenting with ion implantation to produce devices. Hughes Aircraft Company has made MOS/FETS and IMPATT diodes, and has a continuing active program to make ion-implanted devices for digital applications. Ion Physics Corporations, the pioneer in ion implantation, has been selling phosphorous-implanted solar cells to the Air Force and WASA for a number of years. Isofilm International, a small company in Chatsworth, California, was the first to offer off-the-shelf commercial ion-implanted diodes and rectifiers. Fairchild Semiconductor has been producing their 2500 series core driver using ion implantation. KEV Electronics in Reading, Massachusetts, is producing voltage-variable capacitors and high frequency devices.

Since the process has not yet moved into large-scale commercial use, the total impact of ion implantation on the field of integrated

circuits has been minor. In the same manner, the impact of NASA contributions are still to be felt. The general significance of these contributions has been to advance the rate of development of the ion implantation process, and to reduce the time when the process will eventually find widespread application. Because of this, the potential impact of NASA's efforts are estimated to be moderately high to high. NASA contributions to the technology of ion implantation include the following.

o Advanced the understanding of ion-implantation process (05-10-01).

In the Venus Lander program the NASA/ERC found it necessary to develop devices which could operate at approximately 400 C., two times higher than the normal operating temperature of silicon semiconductor devices. The use of diffusion techniques to make junctions in high-temperature semiconductor materials such as gallium arsenide (GaAs) and silicon carbide (SiC) were out of the question because of the need for extremely high-processing temperatures, which are well beyond today's processing know-how. Ion implantation offered the only method for creating p/n junctions in such materials. Much of the early work on use of ion implantation for this purpose was done under NASA contract at Hughes Semiconductor Corporation.

High-temperature devices produced from GaAs and SiC using the ion implantation process could have a high economic impact in that they would eliminate the need for expensive air conditioning of electronic systems. A major prerequisite for such devices, however, will be the ability to grow single crystals of GaAs and SiC with consistent impurity distributions and structures.

o Developed and demonstrated use of ion-implantation process for microwave devices (05-10-02).

The use of the ion implantation process to improve the frequency response of GaAs microwave transistors, was developed and demonstrated by Hughes Semiconductor under a contract with NASA/LeRC. Base regions in the order of 1,000 Anstroms have been achieved and now transistors which will operate at 20 gigahertz (GH<sub>2</sub>) have been produced using the ion implantation approach of doping. These attempts to use ion implantation for specialized microwave devices advanced the time when such devices would

be commercially produced by the ion implantation method. It is believed that these devices will find widespread use in commercial microwave communications within the next year. (See microwave integrated circuits in microwave systems, field 09, NASA contribution 09-17-01.)

### Large Scale Integration (05-12)

Following the development of discrete semiconductor components, simple integrated circuits, and simple integrated logic functions, large scale integration (LSI) was the next logical step in the progression of semiconductor technology. LSI has become a practical and economical development because of the advances in circuit and system design, layout and mask generation, photolithography, multilayer metallization, packaging and testing. This fourth generation of semiconductor products, as its name implies, integrates an ever increasing number of complex functions on single miniature silicon chip; for example, monolithic chips with greater than one hundred gates are no longer uncommon.

LSI is presently in its infancy. Circuit complexity of bipolar and MOS devices will increase as manufacturers learn how to overcome the high costs associated with manufacturing small quantities of custom-designed, highly complex, integrated circuits. To do this a fast design turn-around time is essential, which means developing techniques to provide more efficient utilization of engineering and fabricating facilities. Computer-aided design methods have already had and will continue to have a substantial impact on circuit and system design, layout and mask generation, and device testing. Ion-implantation and electron beam lithography will make it possible to modify existing circuits and generate new circuits quickly, and at minimum cost.

The important advantages of LSI are reduced costs, improved reliability, reduced size and weight, and the possibility of computerized fabrication. LSI should have a large effect on consumer products, industrial products, and military systems. All of the presently active semiconductor manufacturing companies interested in integrated circuits including Motorola, Fairchild, Signetics, National Semiconductor, and Texas Instruments, have been actively engaged in advancing the technology of LSI. LSI devices are now beginning to be used extensively in miniature calculators, data terminals, computers, etc. and should become a major portion of the semiconductor market in the mid 1970's.

NASA has contributed to the advancement of LSI through many of its efforts carried out in computer-aided circuit design (development 05-05), photomask technology (development 05-19), operational chip testing (development 05-17), and ion-implantation (development 05-10), with the

net effect of helping move LSI from a laboratory concept into early aerospace and commercial use. Direct NASA contributions to LSI include (1) the development and improvement of LSI fabrication techniques, such as the simultaneous diffusion of doping agents; and (2) improvements in our understanding of LSI, particularly the physics of interfaces, resulting in devices with improved performance. NASA contributions to each of the developments cited above, as well as directly to LSI, will have a moderate impact upon the maturation of large scale integration. Included among these contributions are the following.

o Developed techniques for the simultaneous diffusion of two doping agents (05-12-01).

Techniques were developed by NASA/ERC to diffuse two dopants simultaneously from an oxide layer to form p and n channel MOS transistors for complimentary integrated circuit functions. The presently used process requires separate diffusion of impurity agents, generally requiring different temperatures and either separate furnace zones or separate furnaces.

Since this development reduces the number of process steps required in large scale integration, it is only a matter of time before the semiconductor industry will be diffusing both n and p dopants in a single step, and the NASA contributions will probably have advanced the time scale in which this will occur.

o Advanced development of LSI devices by determining the relationship between the metal and dielectric layers and its effects on electrical characteristics (05-12-02).

The effects of a second layer of metal and dielectric on the electrical parameters of MOS and bipolar integrated circuits were determined by Philco-Ford Corporation, under contract with NASA/ERC, resulting in a much more advanced understanding of the effects of variations in the metal layers and the dielectric layers (see improved stability of MOS transistors and integrated circuits, NASA contribution 05-08-02). This scientific understanding of the physics of interfaces between thin layers of metals and dielectrics led to LSI devices with improved performance, which enhanced the acceptance of LSI devices in many applications.

### Multilayer Metallization (05-14)

As circuit complexity increased, a need developed for a method of making the required connections between the increasing number of circuit elements on a single semiconductor die, without increasing the die size. Multilayer metallization was the technique developed by integrated circuit manufacturers to make the necessary interconnections without increasing the chip size.

The process entails first depositing and etching a first layer of metallization. A dielectric layer, such as borsilicate glass, is then deposited on the metallized conductor pattern. Contact windows are formed by a process of sputter etching. A second layer of metallization is deposited on the dielectric using a sputter deposition technique, with the interconnections being made through the previously etched windows.

Multilayer metallization did not become a practical process until manufacturers of integrated circuits were able to deposit imperfection-free insulating layers of dielectric materials.

Multilayer devices require more processing steps than devices produced by other means. However, multilayer devices use less silicon area and permit more circuits to be manufactured on a single wafer, thus increasing yield and reducing costs. All of the integrated circuit manufacturing companies (Fairchild, Texas Instrument, and Motorola, to name a few of the larger companies) are exploring and utilizing the advantages of the multilayer fabrication of complex large scale integrated circuits.

NASA contributions to the development of multilayer metallization processes, and the use of multilayer devices, have resulted in much wider and more extensive acceptance of the process and the product among manufacturers and consumers of integrated circuits. contributions include: (1) advances in the metallization process, such as the development of a high-temperature system that does not have the temperature and current density problems associated with systems presently in use; (2) improvements in multilayer products, such as a technique to make direct interconnections of analog and digital silicon chips into beam leaded matrices, thereby increasing the reliability of complex integrated circuit chips; (3) improvements in the commercial marketplace, such as the development of competitive suppliers and the improvement of commercial processing capabilities; and (4) advances in our knowledge of the process and the product, such as a basic understanding of the effects of a second layer of metal on the properties of MOS and bipolar integrated circuits.

The significance of these contributions has been to bring about the acceptance and use of multilayer devices earlier than it would normally

have occurred. The impact of these contributions is estimated to be moderate to high. Included among the NASA contributions to multilayer metallization are the following.

o Developed a method for directly interconnecting analog and digital silicon chips into beam leaded matrices to increase packaging density, reduce interconnections and increase reliability of complex chips (05-14-01).

A technique to make direct interconnections of uncased analog and digital silicon chips into beam leaded matrices was developed by ITT/Semiconductor under a contract with NASA/LeRC. making direct interconnection, component density within the chip was increased and the overall number of interconnections reduced, resulting in increased reliability of complex integrated circuit chips. The process technology consists of additive plating and subtractive etching, using fine detail photoresist techniques for control of the conductor pattern. Considerable quantities of interconnections can be fashioned using a single piece of a double-clad dielectric laminate. The only physical terminations needed are to the exit terminals from the package and the metallized electrodes on the integrated circuit chip.

It appears that from this work, Motorola was able to create a new ISI product line using what they call the "spider concept." When NASA/LeRC ended the program at ITT, the ITT engineer involved with the development moved to Motorola to help them establish an ISI product line. This Motorola ISI product line uses concepts similar to those developed at ITT under NASA sponsorship. Sales of these devices have reached the \$3 million level.

General Electric is now using a similar processing technique which they call their "H-film" process.

o Advanced the use of multilayer metallization by determining the effects of variation in metal and dielectric layers on the electrical characteristics of MOS, LSI, and integrated circuits (05-14-02).

The problems associated with metal and dielectric layer interfaces (described in improved MOS and integrated circuit stability, NASA contribution 05-08-04, and large scale integration, NASA contribution 05-12-02) are directly tied to the development of multilayer metallization. By increasing technological understanding of the interaction of metal and dielectric layers, the use of multilayer metallization as a fabrication process was advanced more rapidly.

o Developed a high-temperature metallization system for high-temperature devices (05-14-03).

Under a contract with NASA/ERC, Motorola developed a high-temperature metallization system to meet NASA's needs for devices which did not have the temperature and current density problems associated with the aluminum metallizing systems presently in use. High-temperature metallization systems are a prerequisite to effectively building beam-leaded device circuits. Both gold-molybdenum and titanium-platinum-antimony systems were investigated. At the same time as the NASA-Motorola project, Texas Instruments, a major competitor, initiated a parallel in-house effort to develop high-temperature metal systems.

In conjunction with the NASA efforts to use high-temperature metal systems and to further improve the reliability of aerospace electronic components, ERC was interested in developing new metallurgical techniques for the analysis of microelectronic device structures. From this work, a new method for measuring the thickness of metal films on silicon was developed, leading to improved control of the metallization process. This new method depends on x-ray spectrometric technology, and makes possible measurement accuracies of 6 Angstroms for very thin aluminum layers.

o Assisted in the development of a competitive commercial market in the multilayer metallization field (05-14-04).

The Jet Propulsion Laboratory sponsored the development of a metallized multigate array silicon chip with 116 interconnected logic gates, to form a universal chip which could perform any logic function. To optimize this chip and make it small and reliable, a two-layer metal system was used: the first layer isolated the components into individual gates, while the second layer determined the specific logic function. Although this type of work was already being done by other semiconductor companies, Harris Semiconductor was awarded the contract so that NASA would not become overly dependent upon a limited number of suppliers. The acquisition of multilayer metallization capabilities by Harris in order to meet the NASA contract requirements brought one more company capable of performing this type of fabrication into the marketplace, and assisted in the development of a competitive commercial market.

o Improved the processing capabilities of commercial firms multilayer metallization (05-14-05).

To determine undetected causes of failure and deficiency in multilayer semiconductor devices and to assist firms in improving their multilayer metallization processing capabilities, JPL provided individual commercial firms with access to costly laboratory instrumentation. In particular, JPL's scanning electron microscope was used by semiconductor device manufacturers to determine heretofore undetected causes of failure. This use of NASA's instrumentation capability to improve commercial multilayer metallization processing was well-publicized by the microscope manufacturer, Cambridge Instruments, Ltd., in an advertisement which appeared in several trade publications.

# Operational Chip Testing (05-17)

As the manufacturing volume of integrated circuits and discrete components increased, a need arose to reduce finished device testing costs. The development of computer-controlled probe testers has enabled semiconductor manufacturers to test chips while they are in

wafer form. A single, one-inch-diameter wafer, may contain as many as 400 integrated circuit chips or 12,000 discrete transistors.

In 1965, it was possible to test about 8,000 transistors per hour. Today manufacturers can test up to 15,000 transistors per hour. The tester marks defective chips with magnetic ink and defective units are removed using magnetic techniques.

This technological development has lead to substantial price reductions in integrated circuits as well as in discrete semiconductor components. Semiconductor component manufacturers built the first test sets for automatically testing chips while in wafer form. As the demand for computer-controlled test sets increased, existing and newly founded test-equipment manufacturing companies started to build them. All major semiconductor manufacturing companies now use some form of automatic chip testing to increase testing accuracy and speed, and to minimize costs resulting from packaging defective devices.

The test probes need great accuracy and their manufacture would have been impossible without the development of photolithographic technology. The seriousness of the testing problem has become particularly obvious with the advent of LSI. The development of these sophisticated components make computer-aided chip testing an absolute necessity.

Because of the almost astronomical task of manually testing multielement LSI chips, and the need for 100 percent inspection, NASA has advanced the development of procedures, equipment and systems using automatic techniques for operational chip testing, including procedures and hardware to electrically stimulate complex integrated circuit chips to make it possible to test individual circuit elements; and also a practical, high-speed optical technique to test such chips.

While operational chip testing has been a regular control procedure in microcircuit manufacture, the NASA contributions are advancing the state-of-technology much more rapidly than would have occurred otherwise, and are making advanced practical procedures and devices available in commercial operations at an earlier date. While the technological impact of these contributions is estimated to be small, the economic impact should be significant. Included among these contributions are the following.

o Developed the procedures and hardware to simulate complex integrated circuit chips to make it possible to test individual circuit elements (05-17-01).

With the opportunity to exploit the technological advantages of large scale integration, a major need developed to test such complex devices to assure

compliance with NASA requirements. NASA/ERC. working with RCA, Telpar Corporation, and Martin-Denver, developed the philosophies, techniques and high-speed word generators, needed to stimulate large scale integrated chips in such a way as to be able to test each of the individual functions. Testing equipment was built by Telpar, under NASA contracts, using procedures developed with NASA funds. This equipment will be used by the Texas Instrument Company to test large scale integrated circuit devices. To date, test rates of up to 5 megahertz for MOS devices have been attained, and it appears that test rates of up to 10 megahertz for MOS memories are feasible. While the technology used for this development was not new, the application of this technology for the automatic testing of complex integrated circuit devices is new.

Since the testing of these new and complex circuits is of considerable importance to a wide variety of potential users, NASA was active in the formation of a committee to coordinate the efforts of the many parties concerned with the problems of testing LSI devices, including people from IBM, Princeton University, University of Missouri, Stevens Institute of Technology, RTI, etc.

o Developed the procedures and hardware for automatic visual inspection system for microcircuits (05-17-02).

Because of the problems associated with visually testing large scale integrated circuit chips for potentially unreliable conditions, NASA/ERC contracted with Arthur D. Little (ADL) to develop procedures and hardware for an automatic visual inspection system. The ADL system proved to be ineffective but was a forerunner of work now underway at JPL.

The MSA efforts are significant in that it will be economically impractical for LSI manufacturing companies to produce reliable devices without automatic electrical and visual test systems. NASA efforts are directly related to the Air Force activities in this same area. Both NASA and Air Force funds are supporting the development efforts for the extremely costly equipment needed for automatic testing of complex integrated circuit chips.

o Developed a practical high-speed technique to optically test complex integrated circuit chips for defective gates (05-17-03).

Because of the problems of testing multigate multilayer integrated circuit chips for the Grand Tour Mission, JPL instituted a program with the American Optical Company to develop an optical method to automatically screen for mechanically defective elements within the chip. The complexity of the devices needed in the Grand Tour Mission allows, at best, a production yield of 5 percent. Therefore, a total of 200,000 devices will have to be tested to yield 10,000 useable devices (1,000 devices per system and 10 systems for a total of 10,000 devices). Under presently available techniques it takes approximately six hours to test each chip. For 200,000 devices, this would amount to 2.4 million hours of testing time. Since such extensive testing time is impossible. automatic testing will be an absolute necessity to be able to exploit the advantages of LSI. Since automatic testing will also be an absolute necessity in fully exploiting LSI in commercial applications, such as minicomputers, calculators, and portable instruments, the NASA efforts should find widespread application.

### Oxides and Protective Coatings (05-18)

The reliability, performance, and yields of integrated circuit devices can be substantially improved by passivating the active surface by oxidizing the surface and/or coating it with such materials as silicon nitride, polycrystalline silicon, or phosphorus glass. Passivating protects the active areas from the detrimental effects of contaminant ions such as sodium ions. Improvements in surface passivation come directly from a better understanding of surface physics, the effects of contaminant ions on device performance, the control of chemical purity of coating materials, and chemical vapor deposition techniques.

Integrated circuits and discrete semiconductor devices have attained greater reliability and increased performance through improvements in surface passivation. All integrated circuits manufacturing companies employ one  $\alpha$  the other of the passivation techniques discussed above to protect the active surfaces of their products.

Because of compelling interest in the long-term stability and reliability of electronic system, NASA's main contributions to improved oxides and protective coatings for integrated circuits has resulted in improved device reliability. Contributions include: increased know-ledge about semiconductor materials and oxide coatings, which in turn helped identify failure mechanisms related to surface contamination; techniques to deposit clean oxides; and techniques for measuring the thickness of oxide coatings.

The significance of the NASA contributions is that the time-scale for improvements in the state-of-technology in oxides and protective coatings for integrated circuits was advanced, and the improvements occurred at a date earlier than they would have normally. The impact of these contributions is estimated to be moderate to high. Included among the NASA contributions are the following.

o Developed techniques for monitoring and measuring oxides and protective coatings (05-18-01).

See NASA contributions to improved stability of MOS transistors and integrated circuits (contributions 05-08-02, 05-08-04, and 05-08-07); large scale integration (contribution 05-12-02) and multilayer metallization (contribution 05-14-02 and 05-14-03) for a complete description of NASA/ERC efforts to develop techniques for accurately monitoring and measuring oxides and protective coatings. The NASA contributions resulted in improved consistency in the protective coatings.

o Developed techniques for depositing clean oxides on MOS, ISI, and integrated circuits (05-18-02).

See NASA contributions to the improved stability of MOS transistors and integrated circuits (contribution 05-08-06) for a description of NASA-developed techniques for depositing clean oxides. These techniques are now accepted procedures for passivating MOS, ISI, and integrated circuit devices.

# Advancements in Photomask Technology (05-19)

Within the past five years, significant improvements have been made in photomask generation and handling. Photoengraving masks, developed from the circuit topology layout provided by the circuit design engineer, are needed to photographically reduce the size of the areas for diffusion, contacts, and/or interconnecting patterns before transferring them to semiconductor substrates.

An automatic drafting machine is used to generate the artwork used for the mask. This machine is computer controlled and uses light to paint the lines on a photosensitive film. It is faster and more accurate than manual methods. The size-reduction ratio for mask generation is 100:1, instead of 500:1, substantially reducing drafting time and eliminating the first camera step. The artwork size is reduced with a minimum amount of distortion. Prior to 1965, line resolution was approximately 0.001 inch. Today, line resolution of 0.0002 inch is possible. In the future, electron beam lithography will result in line resolution of 0.0001 inch or less.

Other advancements in photomask technology include materials changes, such as the use of chromium masks, which have better wear characteristics than gelatin masks. Lasers are also being used to precisely position the mask, and air bearings are being used to reduce wear.

Some of the companies associated with advancements in photomask technology include Bell Labs, Gerber Instrument Company, IBM, Texas Instruments and Motorola.

Advancements in photomask technology have been evolutionary and have resulted from advances in mask generating and handling equipment. All of the major semiconductor companies (Texas Instruments, Motorola, Fairchild, Signetics, and National Semiconductor, to name a few) which produce complex integrated circuits depend on computer-aided mask generation to achieve error-free designs, eliminate layout drudgery, increase design efficiency, obtain high production yields, and reduce costs.

NASA made early contributions to the improvement of mask-making capabilities in the electronics industry which helped to advance the state-of-technology. Specifically, NASA's contributions included improvement of techniques to produce the finer lines and spacing required for microwave transistors and complex integrated circuits. These improvements, which follow, while not extensive, occurred early in the history of these devices and their impact is estimated to be moderate.

o Developed procedures to improve line and spacing definition (05-19-01).

MASA/GSFC contracted with semiconductor manufacturers in two separate programs to improve line and space definition in the photomasking process. The first program was in relation to an attempt to develop microwave power transistors at RCA where it was necessary to increase the power density of the device (see high-frequency power transistors in the section on microwave

systems, NASA contribution 09-06-02). The second program, at Westinghouse, was connected with a program to produce improved complex integrated circuits. Both RCA and Westinghouse developed new procedures to improve their facilities and capabilities because existing facilities could not produce sufficiently fine lines and spacing definitions to meet NASA's requirements for the devices. Other semiconductor manufacturers subsequently adopted the procedures.

#### INTERNAL GAS DYNAMICS (06)

Internal gas dynamics is that phase of fluid mechanics which deals with the flow of gases within a confined space. For the purpose of this study the scope of the field has been reduced to developments related to gas turbine aircraft engines and further, the use of such engines in nonaircraft applications. Gas turbines use hot gases directly from the burning fuel to turn the turbine wheels; steam turbines differ in that the burning fuel is used to generate steam which, in turn, drives the turbine wheels.

Gas turbine engines have three main sections: (1) a compressor, (2) a combustion chamber, and (3) one or more turbine wheels. The compressor is made up of one or more sets (stages) of specialized fans which suck in air through the front of the engine (air inlet) and compress it. The compressed air mixes with the fuel and burns in the combustion chamber. The rapidly expanding burning gases rush through the turbine section spinning the turbine wheels.

Since an aircraft depends on the hot gases rushing out of the engine exhaust to give it forward thrust, the prime function of the aircraft turbine is to rotate the compressor. In other than aircraft applications, however, design efforts are directed towards exploiting the hot gases before they escape and to use the exhaust gases for a variety of purposes, including, for instance, the generation of steam for combined cycle gas-steam turbines, which are now being explored for electric power generation.

Today's commercial aircraft are powered by "turbofan" engines, which have large fans at the front end of the engine driven by a second turbine wheel. The turbofan functions like an ordinary propeller and moves large volumes of air through the engines. The incoming air is divided into two streams, one stream going through the compressor, combustion chamber, and turbine sections, the other flowing around through an outer section, "by-passing" the engine. Turbofan engines have two major advantages over the turbojets described above. The first is greater power at lower speeds which results from the great quantity of air being moved through the engine; the second is that these engines produce a significantly greater thrust but without a proportional increase in fuel consumption.

Since the technology of turbines has existed many centuries, recent developments are the evolution of the continuing efforts to increase the efficiencies of gas turbine engines, e.g., to reduce fuel consumption, to obtain greater power from smaller size, etc.

Recent significant developments in this field are related to increasing efficiency and thrust without significantly increasing weight. To increase the thrust-output per unit of weight, while

reducing fuel consumption, designers have continually attempted to increase the pressure ratio of the compressors, and to operate engines at higher temperatures. This has prompted extensive efforts into developing base materials and coatings that will not deteriorate at the high operating temperatures. It has also made it necessary to develop techniques for cooling the turbine blades, since they are directly exposed to the hot burning gases. Over the past decade, the availability of the computer as a design aid has increased the designer's capabilities in improving engine materials needed to develop engines which can surpass present-day industry economic and technical demands. Significant developments in the field, as identified by technological leaders in internal gas dynamics, are given in Table 6.

In addition to aircraft turbine use, nonaircraft applications of gas turbine engines for ground transportation and for rapidly increasing electric power demands are becoming more extensive. Many of the private turbine manufacturing firms have been in the forefront in adapting gas turbine engines to nonaircraft applications, specifically in the areas of surface transportation and electric power generation.

Because the use of gas turbine engines is so widespread, the noise generated by commercial airliners and ground-based turbines is considered a major source of noise pollution. It has become of sufficient concern to generate a major socially significant effort into reducing the noise levels related to turbines used in aircraft engines and in ground machines.

The gas turbine engine industry is a significant portion of the American business scene. The total value of shipments of aircraft engines and parts in 1970 was \$4.9 billion, with approximately two-thirds attributable to gas turbine engines. The United States military has been a significant customer with approximately 25 percent of the purchases. Exports of gas turbine engines and parts had a value in 1970 of greater than \$293 million.\*

The major contributors to the advancements in the gas turbine engine field have been the Air Force, NASA, and the larger engine manufacturers such as General Electric, Pratt & Whitney, the Allison Division of General Motors and the Lycoming Division of the Avco Corporation. During the period 1958 to 1965, when the newly formed NASA shifted emphasis from an aeronautical mission to a space mission, much of the advanced work which had originated at NACA, who were

<sup>\*&</sup>quot;Aerospace." <u>U.S. Industrial Outlook 1971</u>. Washington, D.C.: U.S. Department of Commerce, Bureau of Domestic Commerce, 1971, pp. 388-389.

#### TABLE 6

# SIGNIFICANT DEVELOPMENTS IN THE FIELD OF INTERNAL GAS DYNAMICS\*

- \*\*1. Increased pressure ratio (06-01)
- \*\*2. Turbine cooling (06-02)
  - 3. Regenerative gas turbine (06-03)
- \*\*4. Advanced materials (06-04)
- 5. Inlet design (06-05)
- \*\*6. Coating materials (06-06)
  - 7. Gas bearing design (06-07)
- \*\*8. Nonaircraft power generation (06-08)
- \*\*9. Computer analysis (06-09)
- 10. Turbo-machinery (06-10)
- 11. Fabrication techniques (06-11)
- 12. Accessory improvements (06-12)
- \*\*13. Noise abatement (06-13)
  - 14. Diagnostic testing (06-14)
- \*\*15. Surface transportation (06-15)
  - 16. Acoustical linings (06-16)
  - 17. Aerodynamics of duct flows for VTOL (06-17)
  - 18. High by-pass ratio engines (06-18)
  - 19. Ceramic cutting tools (06-19)
  - 20. Electrochemical forming (06-20)

<sup>\*</sup>As identified by technological leaders in the field of internal gas dynamics.

<sup>\*\*</sup>Developments cited for identification of NASA contributions.

traditionally the technical leaders in gas turbines, was exploited by private companies. Many of the gas turbine engine advances of the 1960's were an outgrowth of the NACA work done in the 1950's.

Significant developments as identified by technological leaders in the field of internal gas dynamics, are given in Table 6. These developments, for the most part, represent incremental advances in the field rather than specific significant developments; the field is so well established that improvements in the total engine are the result of many designers working on specific components, with improvements subsequently introduced incrementally into the total engine.

Selected significant developments in the field of internal gas dynamics, as listed in Table 6, and NASA contributions to the advancement of these developments, follow.

### Increased Pressure Ratios (06-01)

The developing interest in large commercial aircraft and the desire to make gas turbine engines practical for other than commercial aircraft applications by miniaturization, prompted research into methods for increasing the thrust output per pound of power plant weight. Because of the influence of pressure ratio on the overall performance of gas turbine power plants, increasing the ratio has been a major area of continuing investigation by the engine manufacturing companies.

The major factors affecting rotor pressure ratios are blade loading and rotor blade tip speed. Higher pressure ratios can be obtained by increasing blade loading and/or tip speed. However, increases in pressure ratio are complicated by the requirements of high efficiency and a broad range of operation. Increased blade loading increases the potential of blade-row stalls; as blade tip speeds increase and exceed the supersonic range, severe shock losses result, with a corresponding decrease in efficiency.

In the 1950's engines were generally designed to have many stages with pressure ratios per stage of approximately 1.4:1, and overall pressure ratios in the order of 8:1 or 10:1. The demands for increased thrust output per weight and low specific fuel consumption have forced design changes which allow a greater by-pass ratio and stage pressure ratio. Increasing the by-pass ratio and the stage pressure ratio makes it possible to reduce the number of stages necessary and still increase the overall pressure ratios. Overall pressure ratios of 24.5:1 and by-pass ratios of 5:1 are being achieved.

Small engines, 24 inches long and 12 inches in diameter weighing approximately 65 pounds with thrusts of 430 pounds, and thrust to weight ratios of 7:1, have achieved overall pressure ratios of 8:1 with a conceivable increase to 14:1 in the next few years.

Much of the fundamental technology to increase the pressure ratio was advanced by NACA programs during the 1950's through research contracts with the major engine manufacturers. Since reinvolvement in 1966, the goals of the NASA fan and compressor technology program at Lewis have been to obtain increased pressure ratios per stage, while maintaining acceptable efficiency and operating range, allowing a reduced number of stages for a given application and resulting in lighter and more compact fans and compressors. Since their reinvolvement in 1966, NASA contributions to increasing pressure ratios have not been extensive, but some have already resulted in major achievements, such as a simple method for improving stall conditions in rotor blades.

The total impact of NASA contributions since 1966 is estimated to be moderate but the potential impact of these contributions should be substantial. Included among NASA contributions to increasing the pressure ratio of gas turbines are the following.

o Provided the technology base for advances in increased pressure ratios achieved by manufacturing companies in the decade of the 1960's (06-01-01).

Prior to 1958 NACA was heavily involved in advancing the state-of-the-art in gas turbine engines. In general, NACA developments were approximately 10 years ahead of the work in private engine industry and, at that time, NACA research personnel were considered technical leaders in the field. Private industry was substantially dependent on NACA facilities and the efforts of the NACA workers to determine the direction of research in engine development. When NASA research priorities changed in 1958, placing greater emphasis on the space effort, engine manufacturing companies became more heavily involved in advancing engine design themselves, and exploited the technology base which had been developed at NACA. advancements in increasing the pressure ratio were heavily dependent on earlier basic research done at NACA facilities. Examples of such basic research include: investigating axial-flow compressor inlet stages operating at transonic mach numbers; developing diffusion factors for estimating losses and limiting loading in axial-flow compressor blade elements; determining the magnitude of shock losses in transonic compressors; and analyzing compressor rotor-blade wakes.

o Advanced industry understanding of the present state-oftechnology related to increasing pressure ratios (06-01-02).

When research priorities at NASA/LeRC changed from aircraft propulsion systems to space rocket propulsion in 1958, basic research and development in engine design

shifted to private industrial research facilities. With the reactivation of interest in aircraft propulsion in 1966, much of the knowledge and technology that had been developed during this eight-year period was proprietary, and not available for public knowledge. To "catch-up" to the state-of-technology in the field and to make the results available to all companies, NASA/LeRC undertook research efforts on centrifugal compressors and similar areas of engine development, despite the fact that parallel work was underway in private industry. Result of this effort is widespread knowledge about the present state of compressor and engine design among all engine manufacturers.

o Developed a method for minimizing the onset of stalls in rotor tip regions (06-01-03).

Two methods for obtaining greater ratios are (1) by increasing the compressor blade tip speed; and (2) by increasing the blade loading, which corresponds to increasing the lift of the blade. There are disadvantages to both of these methods: increasing the blade loading increases the blade stall potential; increasing the tip speed into the supersonic range can result in severe shock losses and a corresponding decrease in efficiency. In addition, tip speed should be maintained at as low a speed as possible to reduce engine noise. To deal with this problem, NASA/LeRC instituted a program designed to obtain a better physical understanding of the nature of shock losses, and to find methods to predict losses. Since tip speed may have to be restricted for other than aerodynamic factors. broad ranges of speed were investigated. During this research effort, it was discovered that having holes in the blade tip region minimized the onset of stalls of the rotor blade. The findings were significant enough to prompt an extensive NASA effort into optimizing the effect, and for two major engine manufacturers to modify new engines to accept this change, based upon final test results. Maintenance costs, resulting from the instabilities caused by blade stalls will be substantially lower with this adaptation.

o Increased understanding of airflow around blades (06-01-04).

NASA/LeRC has conducted extensive studies into the effects of variations of unsteady and unstable airflows on engine components of varied geometries. The results of these findings, which are too numerous to cite, have been widely published and have added significant new scientific information about the physical nature of airflows.

o Advanced the design of engine components in the supersonic range (06-01-05).

In an effort to increase the pressure ratio on engines for the Boeing SST, NASA/ARC developed and tested models of engine inlets with geometries designed to be effective at supersonic speeds. These designs were recommended to technical personnel at Boeing who, in turn, built full-scale inlets which were tested in the supersonic wind tunnels at ARC. Engine and airframe manufacturers did not have the supersonic test facilities to advance the designs of engine components to operate in the supersonic range. Without the use of NASA facilities, Boeing would have had to expand their own facilities.

## Turbine Cooling (06-02)

Since significant increases in the operating efficiencies of gas turbine power plants result from higher operating temperatures, techniques for cooling turbine blades and vanes to withstand the high inlet-temperatures are being continually investigated and improved.

There are four basic approaches to cooling turbine components:

- 1. Convective cooling, first introduced around 1960, allows operating temperatures up to 2200 F. This technique takes air from the compressor discharge region and allows it to pass radially through holes inside the blades, removing heat from the blade interior walls, and then discharging at the blade top or along the blade trailing edges.
- 2. Impingement cooling, originally used around 1965, depends on an accelerating low-pressure coolant airflow to impinge on the blade leading edge to provide an improved heat transfer coefficient in

that region. This method of cooling is practical to temperatures in excess of 2400 F.

- 3. "Film cooling," first effectively used in 1963, employs air flowing from small holes, located in areas of the blade surface where there is maximum heat load, to provide an insulating layer between the blade and the hot gas. Turbine components cooled this way can tolerate temperatures greater than 2600 F.
- 4. Transpiration cooling, the most efficient method of cooling, depends on air passing through a porous wall to establish a complete and continuous blanket of cool air on the outer surface of the blade while picking up the heat from the wall by convection.

The higher operating efficiencies which are possible with higher inlet temperatures, are dependent upon being able to effectively cool the turbine blades. Every degree of temperature gained provides an extra 100 pounds of engine thrust, with minimum increase in fuel consumption. Furthermore, the use of cooling permits increased allowable stress levels resulting in increased turbine reliability. Another significant attribute of effective cooling is an increase in time between replacement of turbine components. It appears that future turbine may operate at temperatures of 4000 F, which increases the importance of cooling research. Until the airbus (Boeing 747, DC-10, etc.) became a factor in commercial air transportation, engines which operated with turbine inlet temperatures in the 1600-2000 F range effectively fulfilled most of the commercial demands. Now, to meet the power needs of the airbus, engines typically operate in the 1900-2400 F range.

The gains which can be attained by operating at higher temperatures and the necessity to cool the turbine components brought about the development of a whole section at NASA/LeRC devoted to this area. This group discontinued its efforts in 1959 and its work was summarized in an ASME report (Journal of Heat Transfer 8, 1959, pp. 79-85). The NASA contributions to turbine cooling provided the basis for many of the advances achieved by engine manufacturing companies in the 1960's. In 1966, the turbine cooling section at LeRC was reactivated, with its prime objective to once again become the leader in the field of turbine cooling. Present efforts are directed at developing turbine cooling techniques for application to aircraft gas turbine engines at gas temperatures up to 4000 F and gas pressures up to 600 psia. Included among NASA contributions to advancing the development of turbine cooling techniques are the following, among others:

o Reduced redundant research in turbine cooling techniques by systematically compiling and evaluating material and turbine designs (06-02-01).

Since 1967, the propulsion turbine section at NASA/LeRC has been compiling and evaluating information on materials of construction and turbine designs already actively in use, and is preparing a handbook of design data for turbine design engineers, which will be published shortly. This handbook will cover areas such as heat transfer mechanisms, materials and structures and the aerodynamics of cooling, and should minimize redundancy of design efforts.

o Provided the technology base for turbine cooling advances during the decade of the 1960's (06-02-02).

The search for more efficient and higher powered aircraft engines prompted extensive research into the turbine cooling field in the 1950's at Lewis. NACA was a major contributor to this field of knowledge during that decade. In addition to general cooling studies, efforts included studies of air and liquid cooled heat transfer mechanisms, fluid flow and heat exchangers. Cooling studies were directed at determining the effectiveness of various methods for cooling solid turbine blades; as well as the study, design and testing of turbines cooled with air. Broad knowledge was developed in gas-to-blade and blade-to-coolant heat transfer, fluid flow, and heat exchangers. This information, developed by NASA/LeRC prior to their heavy involvement in the space effort, was the basis for much of the engine cooling technology advancements generated by private engine research facilities during the 1960's. The full development and application of convection cooling techniques, for example, which are still the most prevalent method of turbine blade cooling, is based upon early NACA work.

o Aided in the developments of widespread turbine cooling research facilities and capabilities (06-02-03).

Prior to 1959, most of the fundamental research on turbine cooling was done by NACA/LeRC. Engine companies had neither the facilities nor the trained research personnel necessary for

advanced work. Between 1959 and 1965, private industry surpassed Lewis's research capabilities in turbine cooling. Since 1966, Lewis has been attempting to catch up and regain their leadership. As part of this effort, Lewis has sponsored the development of numerous new research facilities programs in turbine cooling at major technical institutions and industrial research centers, such as the University of Minnesota, Stanford University, General Motors Corporation, University of Cincinnati. Arizona State University, General Electric Company, AiResearch Manufacturing Company, Aerojet Liquid Rocket Company, Newark College of Engineering, Shell Development Company, Battelle Memorial Institute, and the Bendix Corporation. The result of this development of widespread facilities and capabilities is increased interaction between the researchers in the turbine cooling field and an acceleration of advancements related to this field.

## Advanced Materials (06-04)

High temperatures, extreme variations in corrosive environments, high mechanical strength, lightweight, and cost are major considerations of the gas turbine designer. These requirements have created an increasing demand for new materials. Alloys with increased high-temperature strength have been developed, along with coating materials to protect these alloys.

Efforts to increase the thrust/weight ratio of the very large engines has prompted extensive developmental work in composite materials to be used both for structural and moving components. To date, experimental fan blades made from boron fiber are being tested; and boron-epoxy composites are being considered to reinforce titanium compressor disks. Composite materials using boron or carbon fibers in a titanium matrix can also be used in many compressor stages. The prime advantage of carbon fibers is that they can be aligned in the direction of maximum stress. Eventually the turbine may be made using an unidirectional matrix, permitting the development of greatly improved high-temperature properties.

Composite materials represent a major advance in structural materials technology in that they offer the potential of a significant decrease in material weight while attaining strengths equivalent to or better than that of currently available structural materials. Extensive tests are now underway to improve durability and improve the cost effectiveness of these materials so that they can be incorporated into commercial engines. In addition to economic and technological advantages, there are important social gains, specifically, a reduction

in the demands of dwindling natural resources, and the potential transfer of these new materials concepts to other structural applications. The investigations into composite materials has had a significant impact on our understanding of the properties of metals and have created a new fabrication technology.

While the Air Force has played the major role in composite material development, early NASA work demonstrated the feasibility of using fiber-filled material to attain increased structural strength. Since 1965, extensive NASA efforts have been directed at developing a better understanding of mechanisms for improving the structural properties of metals and composites for high-temperature applications. (For a fuller description of NASA's contribution to the use of advanced materials in gas turbine engines see the fields of high-temperature ceramics, and high-temperature metals.)

Among the NASA contributions to the design and development of advanced materials, are the following.

o Demonstrated the use of fiber-filled materials for aircraft engine structural components (06-04-01).

In the 1950's, NACA efforts at Lewis demonstrated the basic concepts of composite materials and the feasibility of using such fiber materials to increase the structural strength of aircraft engine components. Since 1965, numerous contributions to improve the structural properties of metals for high-temperature applications have been made by NASA/LeRC personnel. Over this period, efforts have been towards the development of new or improved fibers, because of the lack of fibers which could be used at high temperatures. This resulted in the development and improvement of: refractory metal fibers; macroscopic oxide fibers, e.g., alumina; and carbon fibers. purpose of these efforts was to develop hightemperature metal matrices which have better impact strength, better nondirectional strength, and greater ductility. The major problem was to find their fiber materials which would not react at the high temperatures. Research at NASA/LeRC made possible the production of materials with six times the strength to weight properties of the base metals at 2000 F.

The NASA efforts have significantly increased the understanding of the properties of metals and should have an impact on economies of air travel by aiding in increasing the operating efficiencies of gas turbine engines.

o Advanced basic knowledge about high-temperature materials (06-04-02).

NASA efforts to advance the use of fiber-filled materials and metal alloys in high-temperature applications, have significantly increased our knowledge of the properties of materials. Because of this increased knowledge it has been possible to produce metals and composites with a substantial increase in their strength to weight capabilities. (For more detailed information see contribution 06-04-01 above, and NASA contributions to high-temperature metals, field 04.)

o Developed new and improved refractory metal fibers (06-04-03).

A number of improved refractory metal fibers were developed by NASA/LeRC, making it possible to produce materials with high-temperature strength properties six times higher than the base materials. Composite materials made from these fibers are being considered for aircraft engines. (For a fuller description see NASA contribution 06-04-01, above.)

o Developed macroscopic oxide fibers (06-04-04).

See NASA contribution 06-04-01, above.

#### Coating Materials (06-06)

Hot corrosion, oxidation, sulfidation, and attack by lead and vanadium are major problems facing turbine engine designers. Of these, sulfidation presents the biggest problem. While sulfidation does not interfere with turbine performance, except in the most advanced stages, it does limit the operating temperature-time relationship of turbine blade materials. Earlier gas turbines, which had lower operating temperatures, used nickel alloys with sufficient chromium content to impart good corrosion resistance. With the demands for greater efficiencies and higher operating temperatures, new superalloys with better high-temperature strength were created by reducing the chromium content and adding alloying agents such as tantalum, tungsten, and molybdenum. This reduction in chromium content resulted in reduced sulfidation resistance. However, the higher aluminum content in the new alloys provided an increased oxidation resistance.

Modern day, high-temperature, gas turbine engines would not have been feasible without the development of effective coating materials.

The coating materials have made it possible to reduce the effects of sulfidation, thereby increasing the temperature-time relationship of the turbine blade materials, and in turn reducing maintenance costs. All of the turbine engines produced within the past few years use coating materials in critical areas to increase the temperature-time capabilities of the base materials and thereby reduce costly down time. However, at the current stage of development, there are definite limitations on the use of coatings because of the loss of coating integrity through erosion, diffusion, mechanical, and thermomechanical damage. Coating materials which are finding turbine applications are generally aluminum and chromium-plus-aluminum type materials.

The long-term solution to hot corrosion problems appear to be the proper combination of improved coating materials and techniques for applying the coating to more corrosion-resistant base alloys.

NASA contributions to the advancement of coating technology for gas turbines have been directed at improving both materials and application methods to protect superalloys and dispersion strengthened materials from the effects of oxidation, corrosion, and erosion. As operating environments become more severe, future engines will benefit from, if not depend on, surface protection systems for their successful long-time operation. NASA has been taking three major approaches to improve protection systems: (1) characterize the protection ability of currently available coatings; (2) develop improved coatings for current alloys; and (3) develop new protection systems for advanced alloys.

These contributions include characterizing existing currently available coating materials; and the development of several promising new systems, including vapor-deposited cobalt-chromium-aluminum-yttrium coatings, alumina enriched aluminide coatings and metallic claddings. (See NASA contributions to high-temperature metals, field 04, for a fuller description.)

The significance of these NASA contributions has been to advance the time when new, more effective coating materials can be effectively used on advanced base materials. The actual and potential impact of these contributions is estimated to be moderate. Included among NASA contributions are the following.

o Characterized and evaluated the protection ability of currently available coatings (06-06-01).

Since 1965, NASA/LeRC has been involved in research into advanced coating systems for gas turbine engines to protect superalloys, dispersion hardened metals, refractory metals, and chromium alloys, and has sponsored research at major engine companies such as Solar, Pratt & Whitney, and General Electric. The protective systems being

developed are now in the stage of advanced development and have not yet found widespread application. In conjunction with these efforts, Lewis has characterized the protective abilities of currently available coatings and has made this information available to design engineers throughout the industry. This effort has minimized the need for extensive redundant research and development of protective systems. (For more detailed information see NASA contributions to high-temperature metals; dispersion hardened metals, development 04-04; improved nickel-base superalloys, development 04-10; and refractory metal advances, development 04-17.)

o Developed advanced coating systems (06-06-02).

A number of new coating systems such as vapor-deposited cobalt-chromium-aluminum-yttrium, alumina-enriched aluminide coatings, and metallic claddings, have been developed by NASA/LeRC through contracts with such companies as General Electric and the Solar Division of International Harvester. These systems for protecting superalloys and dispersion strengthened materials will probably not find use in currently available turbine engines but will be extensively applied to meet the higher operating temperatures that will be found in advanced engine designs.

## Computer-Aided Design Modifications (06-09)

Prior to the availability of computers, design modifications of engines came about through extensive empirical "cut-and-try" methods which were costly in both money and time due to a substantial amount of redundant effort. It is now possible to simulate varying operating conditions using mathematical models. These simulations and analytical techniques, in turn, help pinpoint problem areas in inlet design, compressor design, or turbine design where the design engineer can best spend his time. The use of the computer in design has substantially reduced design costs by minimizing unnecessary experimentation. The key to effective results by using a computer, however, has been the preparation of computer programs by qualified design engineers.

The availability of computers, and the growing number of people experienced in turbine design who are able to relate design requirements to the computer, have made it possible for the gas turbine design engineer to increase his analytical capabilities. It has also given him greater insight into effects of varying compressor and turbine geometries on air flow. Computers have made it possible to gain a greater understanding of the characteristics of unstable and unsteady

flow, and how these distortions affect engine performance. NASA and most of the major engine manufacturing companies have used computers to pinpoint areas where design dollars can be most effectively spent. Consulting facilities are now available to provide expertise in computer-aided design.

Since 1965, NASA/LeRC has been actively engaged in developing computer techniques for analyzing air flow through a gas turbine engine. Using mathematical models and computers in their analysis they have been able to relate the air flow characteristics to variations in the parameters of the engine components. A large number of computer programs have been developed at Lewis and have been made available through NASA publishing channels to aid turbine engineers in their design efforts. These contributions have served to advance the use of computers and mathematical models for research and design of engines, and have reduced the time when these advances would have otherwise occurred. The impact of NASA contributions to the development is estimated to be moderate. Included among them are the following.

o Developed analytical techniques using mathematical models and computers to aid compressor, turbine, and noise suppressor design engineers (06-09-01).

NASA/LeRC developed mathematical models and analytical techniques for use in compressor, turbine, and noise suppressor design. These models were developed both in-house and through contractors such as General Motors Corporation, Allison Division, Northeast Research Corporation, Iowa State University and MIT. Extensive investigations into the effects of air flow, on variations in geometries of the internal engine components made it possible to develop the techniques needed to effectively use the computer as a design aid. These NASA efforts have significantly increased the acceptability of computer-aided design in this area.

o Developed computer-aided design programs (06-09-02).

Computer programs used by engine manufacturing companies actively engaged in computer-aided design efforts were considered proprietary and were not generally available. NASA/LeRC, aware of the advantages of computer-aided design and the growing availability of computers, developed a substantial number of programs which were made available to all interested parties. As a result of the NASA/LeRC efforts, a large number of these programs are now being used throughout the industry.

o Widespread availability and use of computer-aided design programs (06-09-03).

The large number of computer-aided programs developed by NASA were put into the Cosmic System to increase their availability to design engineers. Based upon these efforts, these programs are now available and being used by industry. (See contribution 06-09-02 above.)

o Demonstrated that new knowledge could be added to the existing technology base through the use of computers (06-09-04).

The effectiveness of the computer as a tool for adding significant new information to the existing base of knowledge was demonstrated by NASA/LeRC in a program dealing with the characteristics of unsteady and unstable air flows resulting in improved compressor and turbine designs. The computer was used in the same way to increase the understanding of noise propagation and develop a theory which predicts an optimum wall impedence for which a maximum sound-power attenuation is obtained.

## Noise Abatement (06-13)

Increased use of jet transports along with a growing concern for the quality of environment has resulted in considerable emphasis on noise reduction efforts in the past five years. Since noise is primarily related to human reaction, two major areas of investigation are being studied. The first area under consideration is investigation of the effects of varying the flight profile and flying procedures during landings and take-offs (e.g., reduced power and increased glideslope angles). The second area is the modification of the gas turbine engines themselves to reduce noise.

Cas turbine engine noise can be divided into two general categories: internally generated noise, usually associated with the rotating machinery, and externally generated noise, or jet noise. The primary sources of internally generated noise are the fan, the compressor, and the turbine. In most high by-pass-ratio engines, the dominant source of the internally generated noise in the fan, since the noise propagates out of both the engine inlet and the fan discharge duct. The compressor noise and the turbine noise propagate out of the engine inlet and the jet exhaust nozzle respectively.

The primary approaches for reducing internally generated noise have been: increase the by-pass-ratio to remove energy from the core jet; reduce the speed of the fan; proper design of the fan and the relationship between the blades and vanes; and the use of sound absorption materials in critical areas.

There are two sources of externally generated noise in a turbine engine. One is located downstream of the fan exhaust duct and the other at the nozzle exhaust. The most effective method for controlling the externally generated noise is to keep the jet velocities as low as possible.

The increasing use of gas turbine power plants for aircraft and nonaircraft transportation and for stationary applications, such as electric power generation, coupled with the growing reluctance of the populace to accept the rising noise pollution related to this increasing use has prompted extensive research into methods of noise abatement. Eliminating the sources of noise is an important prerequisite to increasing the markets for gas turbine engines.

Since a better understanding of the mechanisms of noise generation, propagation and attenuation provides the scientific and technological tools needed for substantial advancements in noise abatement, several NASA centers -- Ames, Langley, Lewis and Marshall - have been involved for a number of years in such research. NASA's contributions have dealt with (1) national leadership in directing research dealing with this important social problem, such as the coordination of various fragmented research efforts, determination of future directions for basic noise research, and identification of areas of research that appear to be most pressing; (2) changes in the design of engines such as increased by-pass-ratios; and (3) suppression of noise by sound absorption materials.

Much of the early work done at LeRC in the 1940's became the basis for noise reduction research done by the engine manufacturing companies during the late 1950's and early 1960's. Noise reduction research was reactivated at LeRC in 1966 and the impact of these new efforts should be high. Included among the NASA contributions are the following.

o Development of techniques for using wind tunnels in noise research (06-13-01).

NASA demonstrated that wind tunnels could be used for noise research, thereby eliminating the need for the traditional low-yielding practice of conducting noise experiments in isolated outdoor facilities or in anechoic chambers. Both of the traditional methods have disadvantages, specifically in lack of control of environment and experiments. The outdoor method suffers from the

difficulty in obtaining accurate measurements, while the chamber method does not give an accurate representation of flyover noise because the effects of forward speed (such as compressibility, asymetric loading, inflow turbulence, and Doppler shift) are not represented.

In conjunction with work on vertical take-off and landing aircraft, techniques were developed at NASA/LeRC to allow use of wind tunnels in noise research. These efforts included techniques for cancelling out the ambient background noise generated by the wind tunnel air flow and drive system, and correcting for sound reflections and reverberations from the wind tunnel walls. Use of a wind tunnel as a noise-measuring range permits the investigation of the effects of flight parameters and aircraft configurations independent of the aircraft's handling qualities. Independent investigation of variables, such as advance ratio and mach number, are easily investigated in a wind tunnel, but would be costly and extremely difficult to control in a flyover situation.

As a result of the studies conducted at Ames, the Bell Helicopter Company is now conducting noise studies in the Ling Vaught wind tunnel in Texas, and other firms are expected to follow suit.

o Developed a quiet engine capable of meeting FAA Noise Standards (06-13-02).

Many years of NACA and industry experiences in jet-exhaust noise suppression work have indicated that little reduction in jet noise can be expected through the application of mechanical devices. In late 1966, a quiet engine program was initiated at NASA/LeRC "directed toward selection of thermodynamic cycle characteristics and mechanical design features which will produce a truly quiet airplane power plant."

NASA/LeRC initiated a number of contracts with the major engine manufacturers to study the effects of varying the engine configurations on noise reduction. Major emphasis of this effort was in the following areas: the effects of increasing the bypass-ratio; the relationship between the blades and vanes; the proper design and speed of the fan; and, the suppression of sound with sound absorption materials. The results of this work indicate that engines can be designed with noise levels 20 decibles lower than equivalent operating engines.

The intent of the effort is to develop a practical replacement for engines currently being used on subsonic aircraft such as the Boeing 707 and the Douglas DC-8, to allow them to meet the regulations of the FAA as authorized in Public Law 90-4. To date, it appears that retrofit costs will be in the order of \$1 million per aircraft and a 6700 pound weight penalty could be anticipated. Benefits to be anticipated, in addition to noise reduction include shorter takeoffs, lower specific fuel consumption, increased range, and increased altitude.

Despite the high retrofit cost, the results are sufficiently encouraging to justify continuation of the program.

o Aided engine manufacturing companies in evaluating modifications of engine components to reduce noise (06-13-03).

Both NASA/ARC and NASA/LeRC have wind-tunnel facilities to test the noise characteristics of engines. (See contribution 06-13-01 for a fuller description of capabilities and activites at Ames.) The efforts at Ames are primarily directed at vertical take-off and land aircraft (VTOL) while Lewis is set up to test conventional gas turbine engines in which the component geometries are varied with the expressed purpose of reducing noise. Lewis is working directly with General Electric, aiding them in testing fans, turbines, noise reduction devices, and acoustical linings for the cold and hot ducts. The resulting modified components will be incorporated in an engine to be fabricated at General Electric and sent to Lewis in late 1972 for testing. The Ames and Lewis facilities have aided engine manufacturing companies by giving them the opportunity to test engines under close to actual use conditions, and to control and vary those conditions at will. These facilities are regularly used by industry to evaluate modifications of engine components to reduce noise.

o Established the design constraints to meet FAA standards for safety, load factor, and noise (06-13-04).

The growing concern of the public over the noise related to the increasing use of jet transports has

prompted the FAA to set noise standards and to consider imposing noise certification requirements for the airlines industry. The commercial carriers and engine manufacturers, knowing that such certification was inevitable and that the costs of retrofit would be staggering, have been looking to NASA to determine the design constraints to safely attain the basic noise attenuation goal. In 1968. NASA/LeRC supplied a list of quiet engine design constraints to two of the major engine manufacturers, Allison and Pratt & Whitney, and asked them to design quiet engines within these limits. The set of constraints included: by-pass-ratio -- 5-6; take-off thrust -- 22,000 lbs.; cruise thrust -- 4900 lbs.; number of fan stages -- one; fan blade tip speed --1000 ft./sec. at take-off and 1,100 ft./sec. at cruise; fan cruise pressure ratio -- 1.5:1 to 1.6:1; number of compressor rotors -- one or two; maximum pressure ratio/compressor rotor -- 12.5:1; turbine inlet temperature -- 2000 F at take-off and 1775 F at cruise.

o Advanced the understanding of noise phenomena (06-13-05).

In order to advance the understanding of noise phenomena, NASA funded a number of universities and industrial research facilities to investigate basic unresolved questions concerning the interpretation of jet noise. Such investigations were prerequisites to the development of an effective noise abatement technology. Examples of such research include: studies of the relation of the convective decay of turbulent eddies to their far-field sound pattern, by Stanford Research Institute; supersonic jet noise theory and experiments by GE and the University of Massachusetts; noise reduction from interacting coaxial supersonic jet flows by Syracuse University; and the effect of duct heating on jet and fan noise by Lockheed Georgia Company.

o Demonstrated the use of analytical models and computers in developing and evaluating sound suppressors (06-13-06).

The problems associated with developing and analyzing a theory regarding the propagation of waves in an acoustically lined duct were overcome at NASA/LeRC by constructing a computer-based analytical model. The model eliminated all of the effects of air flowing around compressor and turbine blades and planes, the

varying diameters, and the varying wall impedances, making it possible to develop the mathematics related only to variations in the acoustical liner itself. Using the model and the associated mathematics, it is now possible to optimize acoustical liners for broad-band noise from mach zero to infinity.

o Provided leadership and coordination in noise reduction research (06-13-07).

Several NASA centers (Ames, Langley, Lewis, and Marshall), NASA headquarters and various contractors have been involved in programs to obtain a better understanding of the mechanisms of noise generation, propagation, and attenuation. These will ultimately provide the scientific and technological tools needed for substantial advancements in aerodynamic noise abatement. To coordinate the efforts of the large number of interested parties, to review the status of the university and industry basic noise research programs, and to survey the programs at the NASA centers, NASA held a meeting in July 1969, called the Basic Aerodynamics Noise Research Conference. The purpose of the conference was to determine future directions for basic noise research related to aircraft propulsion systems, to determine those areas of research that appear to be most pressing, and to discuss various avenues of research that appear to be most promising with regard to the understanding of noise generation and propagation. National priorities for basic noise research were established as a result of this conference.

The field of materials machining and forming entails the technology of removing material from a workpiece to shape, form or finish it as required. This field encompasses a broad complex of methods, processes and equipment, including materials cutting and forming machinery, cutting tools, coolants and lubricants.

The metalworking machinery industry represents a significant proportion of American industry, with U. S. shipments in 1971 amounting to \$4.2 billion. By 1980, this value is expected to increase to \$7.2 billion. The three major sectors of the industry -- metal cutting and metal forming machinery, tool and die products and metal cutting tools -- are all expected to show sizeable growth over the next decade. In 1971, the value of metal cutting and metal forming machinery amounted to \$1.5 billion. Shipments of these products are expected to reach a value of \$2.1 billion by 1975 and \$2.75 billion by 1980. Shipments of tool and die products (dies, jigs, fixtures and molds) are estimated at \$2.0 billion in 1971 and are expected to grow to \$2.7 billion by 1980. Metal cutting tool shipments in 1971 had an estimated value of \$0.7 billion. This sector of the industry is expected to reach \$1.7 billion by 1980.\*

The most common types of cutting and forming machines include: various types of drilling machines (drill press, jig bore, radial drill); boring machines (horizontal, vertical); lathes (engine, bench, screw machine, turret, vertical); milling machines (bench, automatic, vertical, universal); planers; shapers; grinders (surface, centerless, cylindrical); broaching machines; power saws; nibbling machines; electric-discharge machines (EDM); electrochemical machining devices (ECM); and laser machines. The operations performed on these machines involve turning, grooving, undercutting, radiusing, boring, facing, shaping, polishing, honing, planing, milling flat and formed surfaces, drilling, reaming, counterboring and countersinking, broaching, threading, sawing, grinding, controlled erosion, melting or dissolution.

Cutting tools used in these machines may be single-point, as in lathe bits, fly-cutters, drills, reamers and countersinks; may have single or multiple abrading surfaces, as in saw blades, broaching tools, grinding wheels, and various honing stones and abrasive belts; or, may make use of various advanced shaping procedures, as in electrochemical, electric-discharge, ultrasonic, and laser cutting or erosion devices.

The workpiece may be a section cut from raw stock (bar, sheet, slab, tube, extruded); a casting, forging, extrusion, or composite; or

<sup>\*&</sup>quot;Metalworking Machinery." <u>U.S. Industrial Outlook 1971.</u>
Washington, D. C.: U.S. Department of Commerce, Bureau of Domestic Commerce, 1971, pp. 239-246.

already partly machined in prior operations. The cutting tools, speeds, feeds, coolants, lubricants, and machine tools must be matched to the material to be machined. The machine surfaces (internally/externally cylindrical, flat, contoured, grooved, curved, shouldered, beveled, etc.) are generated by some combination of holding stationary, traversing, rotating, indexing or adjusting the workpiece relative to corresponding cutting tool movements.

The machinability of various materials is a function of their ductility, hardness, and tensile strength. Cutting tools, which of course have to be harder and tougher than the material to be machined (or, as in EDM, ECM, ultrasonic and laser machining, accomplish the same relationship) may be made of low-cost carbon steel for low-temperature machining of soft metals; more expensive high-speed tool steel for fairly high-temperature, rapid cutting of harder and tougher materials; various types of expensive carbide or coated carbide for high-temperature, high-speed cutting of quite hard materials; diamond, cermet, or cemented oxide for very hard materials; and, finally, ECM, EDM, ultrasonic or laser cutting for the most difficult to machine materials such as superalloys, tungsten, tantalum, and beryllium.

For most cutting tools, a coolant or lubricant is required to control the considerable heat generated at the cutting edge. These include air, freon-mixtures, methonol-mixtures, straight or mixed mineral and fatty oils, water soluble oils, water, and other coolant, lubricant, and anti-oxidant additives which increase cutting speeds, reduce tool wear, and improve surface finish of the workpiece.

Numerically controlled (N/C) machining is a relatively recent development in response to the increased material, parts and system performance requirements of the aerospace and advanced commercial sectors. The development of numerically controlled machining was brought on by machining problems due to use of new materials such as superalloys, beryllium, and various composites; stringent tolerances; exceptionally difficult shapes; and high material, parts and labor costs. In conventional machining constant surveillance, adjustment and correction by the machinist is required, leading to high unit-costs, human error, and distinct feasibility limits to the workpiece complexity and materials allowable. N/C machines are controlled, instead, by a punched tape. Appropriate feeds, speeds, and tool paths required to produce the machining operation are coded onto this tape. Tape-controlled machining is especially useful for short run production and prototype work because setup time is drastically reduced. N/C parts can be made faster, using more difficult materials, to closer tolerances and of configurations laboriously if at all possible by hand methods. Even the most elaborate shapes can be reliably reproduced.

Direct numerical control (DNC) is an evolutionary N/C advance which enables the computer to bypass the tape and instruct the machine directly. DNC advantages include quicker tool changeover, greatly

increased point-to-point cutter feeds, part-family programming, and direct engineering intervention to modify a process or part. Other computer-dependent or related activities associated with N/C-DNC include computer aided design (CAD), where engineering information is displayed graphically and computer-manipulated by means of a hand-held light pen by the engineer sitting at a display console; and computer-aided manufacturing (CAM), or the automation of essentially all factory operations.

One of the most significant developments in recent years has been the discovery and explication of the importance of surface integrity — the effect of processing and fabrication history on a materials microstructure. Machining and processing operations produce profound surface layer and subsurface effects, such as plastic deformation, high-temperature gradients, rehardening, overtempering, and residual stress. Data are being developed to measure and analyze the effects of machining and processing operations, which result in stress corrosion, distortion, and fatigue failure.

Significant developments in the field of machining and forming, as identified by technological leaders in the field, are given in Table 7. Principal support for the advancement of many of these developments has come from the Air Force. Since the massive research and development effort of the Air Force cuts across the entire field of machining and forming, it has rarely been necessary for other agencies to undertake or support significant R & D programs in this field. As a consequence, NASA's principal contributions to the advancement of the technology of the field fall into two broad categories: (1) developing materials analyses and theory, as for example, stress corrosion causes and effects; and (2) developing the appropriate tools and techniques when existing technology has not been sufficient to meet space program needs, as for example, in advancing the technology of electric-discharge machining.

One major complicating factor, in addition to the dominant role of the Air Force, is the fact that virtually all NASA production machining, and most development machining, is performed at contractor facilities. Since the contractor facilities used to service NASA's machining and forming needs are often the same facilities used by the contractor to service other customer's needs, it is difficult to determine the source of funding for many of the technological advances achieved by the various contractors. Because of this, most advances in machining and forming technology are considered company-proprietary information, and are rarely documented as well as advances in other fields of technology. Included among the significant advancements in the field, to which NASA contributions have been identified, are the following.

#### TABLE 7

# SIGNIFICANT DEVELOPMENTS IN THE FIELD OF MATERIALS MACHINING AND FORMING\*

- 1. Cast alloy advances (07-01)
- 2. Cold forming steel (07-02)
- \*\* 3. Computer aided manufacturing (07-03)
- \*\* 4. Cutting tool improvements (07-04)
- \*\* 5. Electrochemical machining (07-05)
  - 6. Engineering metrology advances (07-06)
  - 7. Gatorizing and powder metallurgy (07-07)
- \*\* 8. High speed grinding (07-08)
  - 9. Hot forming ceramics (07-09)
- \*\*10. Hydrostatic extrusion (07-10)
  - 11. Isothermal forging (07-11)
- \*\*12. Numerical control direct numerical control (07-12)
  - 13. Product liability (07-13)
- \*\*14. Surface integrity (07-14)
  - 15. Tungsten alloy advances (07-15)

<sup>\*</sup>As identified by technological leaders in the field of materials machining and forming.

<sup>\*\*</sup>Developments selected for identification of NASA contributions.

## Electric-Discharge Machining (07-05)

Both electric-discharge-machining (EDM) and electrochemical machining (ECM) are nontraditional, nonmechanical approaches to the machining of materials. The EDM process can be described as the shaping of parts by the controlled erosion or removal of metal by rapidly recurring spark discharges striking the workpiece surface. Metal removal in ECM, on the other hand, is by electrochemical reaction or dissolution, brought about by the passage of an electrical current through a suitable electrolyte between the workpiece (anode) and a shaped tool (cathode).

Practical development of the electric-discharge-machining (EDM) process by Soviet engineers goes back about 30 years. However, much of the more productive research and development work has been carried out during the past 10 years in the United States and abroad. During this period, great advances have been made in machine design, power supplies, electrode-tool materials, etc., all of which have contributed to the establishment of EDM in industry as one of the most widely used of the nonmechanical machining processes.

There is, as yet, no universally accepted theory for the exact mechanism of metal removal by EDM. It is rather generally accepted that erosion or metal removal is brought about by melting and possibly by some vaporization of the metal. A spark discharge will occur when the voltage difference across the gap between the tool electrode and the workpiece electrode becomes large enough to break down or ionize the dielectric fluid and make it act as an electrically conductive channel.

For example, when the voltage between two electrodes separated by a gap of about 0.001 inch containing a dielectric fluid, e.g., hydrocarbon oil, reaches about 70 volts, the dielectric becomes ionized and a discharge occurs. At this time a flood of electrons, which constitutes the current pulse or discharge, flows through the narrow ionized channel formed in the dielectric. The initial breakdown is assisted by microscopic foreign particles in the dielectric fluid. The electrons striking the workpiece change their kinetic energy into thermal energy. The heat produced causes surface temperatures to rise above the melting point, resulting in the formation of a liquid phase and. possibly, some vapor phase and ions. The melted or vaporized metal particles are violently blasted away or ejected by the impact of the discharge. As the great flow of electrons occurs, the voltage between the tool and workpiece drops to about 20 volts, which is low enough to stop electron flow. The ionized channel collapses, the surrounding dielectric fluid takes its place, and the cycle is completed. The time required to accomplish ionization is about 1 microsecond.

Some unique features or advantages of the EDM process are (1) ability to generate complex configurations with single-axis tool travel; (2) ability to provide burr-free machining; (3) ability to cut high-strength

materials; (4) no tool-to-workpiece contact, which permits machining of fragile parts or structures; and (5) good machining-gap control, which contributes to accuracy and maintenance of close-tolerance ranges on parts.

These desirable features of EDM are used for fabricating parts that are difficult to make by conventional machining methods because of their shape or because of the hardness or toughness of the metal or alloys. The greatest single application of EDM is the production and finish machining of dies. Other important uses of EDM are for: drilling small-diameter holes; drilling multiple-holes; producing irregular-shaped holes or slots; and blanking parts from sheets. EDM's ability to machine dies or parts in their fully hardened condition is an important feature for the above applications.

Unlike EDM, which uses an electric discharge, ECM can be likened to an electroplating process operating in reverse. At the start of the operation, the tool is brought to the desired gap distance (e.g., 0.002 to 0.015 inch) from the workpiece surface. Then, the voltage is applied causing current to flow. As the operation proceeds, the workpiece dissolves and the tool is steadily advanced to maintain a constant machining gap. Electrolyte is pumped down through the tool and leaves through the space between the tool and the workpiece wall. Insulation on the outside of the tool minimizes side cutting and helps produce shape with the desired dimensions.

The general procedures described above can be used for drilling, die-cavity sinking, trepanning, broaching, and other shaping or contouring operations. For contouring and die-cavity work, the flow-past type (i.e., flow roughly parallel to the electrode surfaces) of electrolyte flow, is often used rather than the flow-through type. Three-dimensional cavities can be produced by ECM using a single-axis movement of the tool electrode, which closely resembles the reverse image of the desired cavity shape. ECM is especially suited for drilling of multiple holes or irregular-shaped holes.

Some unique characteristics or advantages of ECM for machining or shaping are: (1) burr-free machining; (2) stress-free machining; (3) no thermal damage to workpieces; (4) no tool wear; and (5) no intrinsic effect of material hardness on ECM rates and performance. Electrochemical machining in its various forms is well-suited for machining the hard and tough stainless and high alloy steels. This is particularly so for operations such as: production of complex shapes or cavities, such as turbine blades, broaching, drilling, or trepanning of round or irregular-shaped holes; deburring, etc. In general, good surface finishes are obtained by ECM on stainless and alloy steels.

It is expected that electrochemical machining, which is already being widely used in industry, will be even more extensively used for machining steel and other tough alloy parts in the years to come. This is because ECM processes are readily adaptable for production work and automation, and do not require highly skilled personnel for routine production operations.

NASA's contributions to EDM, although in some respects quite significant, are difficult to document because so much of the technology is disseminated by personal communication rather than by documentation, conferences or other such formal channels. NASA contributions to the advancement of EDM, therefore, have often taken the form of support and assistance for non-NASA facilities, rather than the actual development of technology. Because of this, the economic and technological impact of the NASA contributions must be considered substantial. Among the NASA contributions are the following.

o Development, support, and assistance to EDM job shops (07-05-01).

Since the early years of this nation's space program, NASA/LeRC has developed and tested new flight and space vehicle configurations. Every such vehicle begins life as a model attached to various force-measuring strings and strain-gage balances in its wind tunnel tests. These devices are exceptionally precise and have complex internal geometries requiring extensive machining.

Faced with more fabrication demands than in-house EDM facilities could accommodate. Langley instituted a comprehensive country-side survey to uncover potential outside contractors with either adequate EDM facilities or skills. Since EDM had been first introduced into this country in 1956, the search proved negative. Although various jobshops had expressed interest in attempting to fabricate the intricate force-measuring devices, none were found to have suitable capabilities. To develop these needed capabilities, open-ended, risk-free development contracts were awarded to the most promising of such contractors, and NASA EDM experts were sent to the shops to help them acquire, set up and operate EDM equipment. These NASA experts, then remained in the shops for extended periods as on-line consultants, or on-call as required.

The contractors involved in this first large (i.e., \$100,000 each for three years) EDM contract award were Allied Pacific Manufacturing Company, Parent Tool and Die Company, Microcraft Incorporated, Astrospace Corporation, Speed Ring Corporation, and Modern Machine and Tool Company. Some of these six pioneer EDM contractors, and other subsequent contractors, have developed not only advanced EDM skills and equipment, but also added considerable

staff and now routinely perform EDM work for other One shop now employs a section of customers. approximately 60 persons which was developed in response to NASA/LaRC demands for advanced EDM fabrication techniques and quality control. these job-shops now represent some of the most advanced EDM fabricators in the nation, the advanced level of EDM technology demanded by LaRC is available to commercial industry. The practice of providing facilities, information and NASA experts to contractors does not appear to be restricted to EDM but is apparently a normal day-to-day practice in many other areas of technology at LaRC as well as other NASA centers. For similar type of assistance, see NASA contributions to numerical control. contribution 07-12-02.

o Development of an on-site EDM electrode resurfacer (07-05-02).

Electric discharge machining operations severely erode the shaped electrodes employed, requiring frequent resurfacing. To perform the necessary regrinding, the electrodes must be removed from the machine, causing costly downtime and repeating tool setup procedures. North American Rockwell, under a NASA contract, developed a portable, removable resurfacing device which can be temporarily mounted to the T-slots on the transverse EDM carriage. This device performs necessary resurfacing without tearing down a setup and is immediately useful for any shop employing EDM machine tools.

o Commercial applications of NASA-developed EDM technology (07-05-03).

Electric Discharge Machining (EDM) was apparently introduced into the United States in about 1956 by the ELOX Corporation. One of the first machines was purchased by the NASA/LaRC, which subsequently has acquired many additional EDM machines. Because of the advanced instrumentation requirements of NASA/LaRC's 50 major wind tunnels, the Center has become one of principal developers of EDM technology in this country. ELOX and other EDM manufacturers meet frequently with Langley personnel to exchange information and when in need of technical assistance. Other industry representatives and consultants also routinely visit the Center and exchange information by telephone on a daily basis.

Based upon the advanced EDM techniques in operation at Langley, one ELOX representative began manufacturing EDM machines in Switzerland which could perform the innovative operations he had observed and learned about in pilot-type setups. In another instance in 1960-62, the Cincinnati Milling Machine Company (now Cincinnati Milcron) developed an EDM machine with a hydraulically operated quill specifically for NASA/LaRC. The company is now marketing such an EDM machine commercially.

As possibly the most advanced EDM technologists in the country, Langley personnel are an important source to industry of raw and evaluated EDM data and information.

o Preparation of reports on the technology of EDM machining (07-05-04).

The Battelle Memorial Institute prepared a seven-volume series of reports for NASA describing the technological practices and problems in removing metal from ultra-high-strength steels, stainless steel alloys, beryllium, titanium, titanium alloys, nickel-base alloys, and cobalt-base alloys. EDM, as well as other common machining methods are discussed at length in these reports, including the tools and auxiliary equipment recommended for specific operation; the causes of fabrication problems and precautions for avoiding them; optimum machine settings for various metal removal rates; and metallographic and microphotographic techniques to be used for comparison and failure analysis.

These comprehensive reports are of great value to designers, tool, production, and quality control engineers, and to the man in the shop, who is either using or contemplating using EDM as a forming and machining process.

## Numerical Control/Direct Numerical Control Machining (07-12)

Demonstration of the first numerically controlled (N/C) machine was in 1952, involving a Cincinnati Milicron, Hydro-Tel Vertical milling machine. This three-axis hydraulically controlled mill was adapted to electronic supervision/direction by having drawing and machining data (dimensions, tolerances, feed, speed, positioning) of parts programmed on a punched tape, which in turn was digitally analyzed

by the electronic control system for translation into machine (hydraulic) commands. Tape (numerical) controlled machining is especially useful for short-run production and sophisticated prototype work because setup time is drastically reduced and part modifications can be easily performed simply by punching a new tape. Direct numerical control (DNC) is a more recent computer technique to control a number of machine tools, by-passing the tape when required for purposes of sequence rearrangement, tool changing, prototype development and other atypical requirements.

Important dates in the development of numerical control include:

- 1949 Air Force Materials Laboratory (AFML)
  Wright-Patterson Air Force Base, Ohio,
  awards N/C contract to John T. Parsons,
  Traverse City, Michigan.
- 1951 AFML contracts Massachusetts Institute of Technology to further N/C development.
- 1952 Cincinnati Milicron demonstrates its numerically controlled Hydro-Tel milling machine.
- 1959 AFML contracts with MIT to develop computer-aided design (CAD).
- 1960 AFML and the Aerospace Industries
  Association reduce their automatically
  programmed tools (APT) language to
  practice.
- 1962 AFML contracts with Bendix Corporation to develop adaptive (feedback) control.
- 1963 AFML sponsors IBM to develop Air Forcedeveloped automatically programmed tool (ADAPT) computer language compatible with APT.

N/C parts can be made faster, of more difficult materials, to closer tolerances, and of configurations only laboriously, if at all, possible with conventional hand-controlled machining techniques. Developments subsequent to N/C, such as adaptive control (sensor feedback on position, spindle deflection, tool chatter), computer-aided manufacturing/computer-aided design (CAM-CAD), and many other sophisticated computer techniques, are revolutionizing the manufacturing environment by lowering costs, and improving quality and efficiency. N/C-DNC is generally considered to be a revolutionary advance in fabrication technology.

Among the NASA contributions to advancing the technology of N/C-DNC are the following.

o Support and direction for APT long-range planning user group (07-12-01).

Developed originally at MIT under Air Force sponsorship and with the cooperation of the Aerospace Industries Association, the three-dimensional programmed tooling machine language system called automatically programmed tooling (APT) is now the most widely used such system of computer/machine compatible languages. The APT family of programming systems are run on a variety of computer sizes and types. APT is a technique for automatically programming part-geometry descriptions, tool-movement instructions, and other data by means of a computer which then processes the information and produces a punched tape to be fed into the tape reader of a numerically-controlled machine tool; which in turn executes the appropriate feeds, speeds, cutting actions, and other processes to produce the part that is programmed on the digitally coded tape. The APT language is exceptionally flexible in mancomputer communication, and is in constant evolution. APT is now available for national and international commercial use and is under the administration of the Illinois Institute of Technology Research Institute (IITRI) in joint sponsorship with industry to expand and improve its capabilities, and disseminate the results to the various APT membership groups, who pay a yearly fee for APT programs, assistance and information. Virtually every major commercial and aerospace manufacturer is a member of this association. Typical members include Ford, Chrysler, Westinghouse, TRW, GM, IBM, Pratt & Whitney, Bendix, G.E., all machine tool manufacturers, and numerous European, Canadian, Japanese and other foreign firms.

NASA personnel have helped support research at IITRI since 1962, have participated in major membership groups, identifying problems and needed research. Each major computer has its own version of the APT language, and its array of users, who meet at long-range planning symposia to discuss common problems and potential, suggest research for IITRI, and assist in utilization of APT. For users of Computer Development Corporation (CDC) machines, NASA personnel have served as chairman of the CDC computer-users sub-group of the APT long-range planning symposia (ALRP).

o Assistance to N/C Contractors (07-12-02).

Since successful low bidders on a NASA request for proposals are not always those having the most advanced technological capabilities in all areas of the efforts, NASA personnel often provide these contractors with technical assistance to aid them in bringing facilities and personnel up to desired levels. This assistance is often provided through informal telephone consultation. In those cases where adequate industrial skills are either lacking or inconveniently remote from the contract-awarding NASA facility, NASA technicians and engineers are made available to the contractor at their own facilities to help them work out the contract program and to aid them in developing the basic skills to successfully perform the work.

One example is a continuing requirement at NASA/LaRC to fabricate numerous complex wind-tunnel models, best machined on N/C equipment. Since local job shops were inadequately staffed and furnished, NASA systematically helped them to acquire the necessary tools and skills to perform these advanced fabrication functions. Where there were originally only five firms in the local area responding to these NASA requests, there are now twenty-two business firms. This exchange is typical of NASA-contractor relations in virtually every field. For similar assistance, see NASA contributions to electric discharge machining, contribution 07-05-01.

o Development of a combined measurement N/C data recording system for free-formed surfaces (07-12-03).

Because NASA researchers are continuously developing advanced wind-tunnel models with very complex free-formed, sculptured surfaces, their mathematical measurement and explication in three-dimensional machine language is both difficult and time-consuming using conventional methods. No system existed which could provide a point-by-point measurement of such free forms while simultaneously recording the critical points on computer-compatible punched cards. NASA therefore contracted with the Bendix Industrial Corporation in 1970 to develop such a system. The resulting system, which is now being marketed commercially by Bendix, consists of the adaptation and marriage of a Bendix Sheffield Cordax 3000 Series Measuring Machine and an IBM 523

Data Card Puncher. In practice, the measuring machine lightly senses a point on the model surface, and records the position of this point on a data card. The card is then fed into a computer for fairing or smoothing between the points, and developing a punched tape to be used for larger or full-scale prototype fabrication on N/C machines. This system is an important incremental advance in N/C state of the art, and should find use in aircraft, ship, boat, and auto manufacturing firms. Lockheed Aircraft is already using the system.

o Development of a method for thread cutting with a three-axis N/C milling machine (07-12-04).

Some thread-cutting requirements while necessary are often excessively expensive because they exceed the capability of ordinary shop machines; require elaborate tooling, jigs and setup time; or must be made with difficult materials. Since innovative devices with special threads are in routine demand at NASA facilities, it was necessary to develop some convenient alternative to conventional thread-cutting methods. NASA personnel therefore developed TAPDIE, a generalized thread-cutting program written in FORTRAN for the APT numerical control system using a 6000 series CDC computer (see NASA contribution 07-12-01, above) which computes the foot path and N/C tape necessary to cut almost any desired thread. Advantages of this technique include a significant reduction in time, and increased flexibility and convenience. The thread diameter is limited only by the size of the cutting tool. It is equally easy to cut inside or outside threads, left or right-handed threads, and straight or tapered threads with any pitch or number of threads per inch. This important addition to the array of special N/C techniques would be of significant value to any shop employing N/C tools, but particularly for experimental or prototype facilities.

o Development of N/C capabilities in industry (07-12-05).

It is a routine practice to provide contractors with technical assistance by sending NASA personnel to contractor facilities to assist them in developing quality control standards, to inform them of recent technological advances, and to assist them in working out the fabrication, processing and inspection procedures (see NASA contribution 07-12-02, above).

In order to meet NASA standards, many small firms have had to develop inspection facilities and personnel far more sophisticated than they had in the past. To meet these needs, many firms need experienced personnel on their staff. In one center, an assistant section head, a foreman, and eight firstclass experimental machinists moved from NASA to contractor employment or set up their own "spin-off" shops. All of these shops now have larger, more diversified, more sophisticated plants as a consequence of hiring former NASA employees and having to meet demanding space program standards. As an example, there are at any given time between 100 and 150 jobs under contract between NASA/LaRC and various job shops, and a constant dialogue is underway between present and past NASA personnel concerning job optimization.

o Preparation of an N/C machine data manual for programmers (07-12-06).

When numerically controlled machine tools were first introduced in the late 1950's and early 1960's, there were few publicly available documents for programmers. Under contract to NASA in 1962, North American Rockwell developed an early N/C machine data manual for programmers which contained specific information on the then-existing types and sizes of N/C machine tools and auxiliary equipment. The manual includes photographs, isometric and orthographic drawings, machine dimensions, coordinate systems, indexing pallets, specifications, standard tooling, a programmer's check list, and machine-language instructions, among other items. This early document was a significant contribution to advancing the proper use of N/C systems.

o Development of a digital simulation model of N/C production systems (07-12-07).

The University of California at Los Angeles (UCLA), under NASA contract, investigated the nature of numerically controlled production systems to determine the basic principles of their operation. Using these findings, UCLA researchers developed a method of digitally simulating the generalized functions and their interrelationships were formally expressed in an N/C control production systems simulator. The UCLA effort resulted in a computer-based generalized model which illustrates

the methodological rationale with which one can conduct experimental investigations in system behavior with varying system typologies or under different operational disciplines. This basic research is particularly useful to N/C-DNC designers attempting to automate industrial operations.

o Development of a method for contouring with a numerically controlled punch press (07-12-08).

Because production schedules for the Saturn I and IB boosters were being hurt by the unavailability of required specially shaped brackets, Chrysler Corporation personnel developed a technique to produce the brackets in a N/C turret punch press. While developed for specific purposes, the technique is readily adaptable for experimental shop use. It involves programming contours on an automatic press, with accuracy to .003 in., which does not overload the machine, and which significantly reduces programming time. The N/C programmer defines the part geometry, outlines the cutting sequence, and selects the appropriate punches that will produce the desired periphery in a minimum number of steps. Part costs are reduced, and the need for tooling and sheet-metal layout eliminated. This information is particularly valuable to the small, owner-operated job-shops which routinely perform complex and highly varied operations, but which cannot afford special tooling for each new innovative program.

## Surface Integrity (07-14)

It has long been known that mechanical properties and service performance of materials are a function of surface as well as bulk properties. However, it was not until the aerospace-generated need developed for highly reliable components to operate at high stress in severe environments, that much research was done on the history, properties and performance of the surfaces of machined materials. Shot peening, tumbling, and stress-relief heat treating have been traditional responses to improve residual surface stress problems due to grinding burn and cracks, creation of untempered martensite in drill holes, stress corrosion, cracking, tears, laps, plastic deformation, recrystallization and other post-fabrication surface phenomena. Some in-service effects of machining and process history include part distortion, cracking, short life, microstructure alterations, and chemical alteration, thus subverting design requirements and introducing an unpredictable liability factor.

Knowledge of in-service effects resulting from the fabrication history of a part will generate far more sophisticated quality control processing specifications during the manufacturing regimen. Inexplicable in-service failures and shortened service life should be brought under prediction and control, through a better understanding of surface properties, resulting in increased performance and reliability, particularly in critical environments.

A major consequence of the surface characteristics resulting from many machining and processing operations is the phenomenon known as stress corrosion. Stress corrosion failure is due to residual surface tensile stresses, resulting from machining, which can cause cracking and therefore lead to failure in a corrosive environment. Two historical examples are the seasonal cracking of brass cartridge cases, and embrittlement failures of locomotive boilers during wet weather. For more sophisticated applications such as aircraft and space vehicles, the stresses may be both residual and environmental, and the corrosive elements might be fuels, gases or air.

Considerable research is underway to determine the causes and consequences of stress corrosion in various structural materials employed in high reliability components exposed to severe stresses and environmental conditions. NASA has conducted many studies into the basic nature of this phenomenon, as well as its effect on such materials as titanium, various aluminum alloys, and nickel alloys. Since the nature of the NASA contributions are incremental, no single contribution can be identified as having a major impact. However, the collective impact of these and other basic contributions on advancing the state-of-technology in surface integrity must be rated high. Included among the NASA contributions are the following.

o Development of standardized test procedures for stress corrosion cracking (07-14-01).

Specific methods or special test specimens did not exist for evaluating the stress corrosion cracking characteristics of common structural materials, such as exist for determining mechanical properties. Since neither the type of specimens nor the corrosive environments for conducting stress corrosion tests were standardized, conflicting results were obtained on the phenomenon, leading to designer confusion in appraising the susceptibility of various materials to stress corrosion. To overcome this handicap, NASA developed standardized test procedures, including specimen preparation and design. Other uses for this standardized procedure include the development of new alloys, the maintenance of uniformity of products, and the determination of new applications for specific materials.

o Better understanding of stress corrosion crack initiation and growth in aluminum alloys (07-14-02).

Because both the short and long-term structural integrity of materials for man-rated space systems must be stringently analyzed and controlled, NASA and NASA-contractors have performed considerable research to develop a better understanding of stress corrosion phenomena. One such study by the Tyco Laboratories investigated the mechanism of stress corrosion in high-strength aluminum alloys, such as 7075-76 and 2219-7851, using electrochemical, mechanical, electron-microscopic, and nondestructive testing techniques. The Aluminum Company of America (ALCOA) investigated the initiation and development of stress-corrosion of cracking in heavy plates of the same two alloys. The ALCOA study identified the effects of such cracking on metallurgical structure. its propogation path characteristics with respect to the orientation of applied stress and other contributing factors. North American Rockwell (NAR) Corporation conducted an extensive theoretical and experimental investigation which resulted in a proposed new model or mechanism which describes stress corrosion cracking in 7075 aluminum alloy. In another NAR study, the effects of crystal lattice point defects on the susceptibility of 7075 aluminum to stress-corrosion cracking were determined. Defects were induced into specimens by neutron radiation and the growth of stress-corrosion cracks observed and analyzed.

The collective significance of such fundamental research has been to advance the understanding of stress corrosion phenomena explicitly in the materials studied and implicitly in related structural materials. The effects of machining can then be related to the in-service effects of stress-corrosion site-nuclei generation and growth. This information is useful to materials researchers, metals manufacturers and fabricators, design and maintenance engineers.

o Protecting aluminum alloys from stress corrosion cracking (07-14-03).

Under contract to NASA, researchers at ALCOA's Research Laboratories developed a technique to minimize stress-corrosion cracking in aluminum alloys subjected to sustained surface stresses and corrosive environments by applying appropriate surface treatments

and protective coatings. The most effective protection determined in this effort was the application of a coating of epoxy-polyamide paint to either a shot peened or 5 to 7-mil-thick metallized surface of the aluminum alloy. Although satisfactory temporary protection is possible with a 3 to 4-mil-thick electroplated galvanic coating or a coating of epoxy-polyamide or polymethane resins, the former treatment is clearly superior. Anodic films and zinc-rich paints are less effective. This stress-corrosion control information should be useful to primary aircraft manufacturers, airline maintenance engineers and materials researchers, among others.

o Degreasing of titanium to minimize stress corrosion (07-14-04).

Cleaning agents such as trichloroethylene, methanol, methyl ethyl ketone and acetone, have been commonly used as degreasing agents in processing titanium. However, personnel at General Dynamics-Convair determined that trichloroethylene and highpurity methanol contribute heavily to titanium stress corrosion at elevated temperatures. Even residual perspiration stains from fingerprints are implicated. Solution to this problem was the development of a technique for handling titanium, including: diluting the methonal with 2 percent distilled water, using cotton gloves, and keeping the titanium from being exposed to even the fumes of trichloroethylene degreaser. This approach should be useful to both producers and users of titanium, such as aircraft manufacturers, and to those concerned with investigating the mechanism of stress corrosion.

o Better understanding of welding effects on aluminum alloys (07-14-05).

Cracking was observed in the heat-affected zone of 6061 aluminum alloy welded by the tungsten inert gas (TIG) ac weld technique, despite the best known TIG welding procedures and controls. To overcome this problem, North American Rockwell Corporation, under a NASA contract, determined that the basic cause was due to uncontrolled high-frequency current alone. The problem could be overcome by removing erratic and superimposed ac line loads. The analytical and experimental methods and techniques, as well as the conclusions, should be of value to those concerned with aluminum welding, including materials researchers, metals manufacturers and fabricators, design and maintenance engineers.

o Understanding of electrical-discharge machining surface effects (07-14-06).

Because nickel-base alloys are unusually difficult to machine with conventional methods, electric-discharge machinings (EDM) has found increasing use (see NASA contributions electricaldischarge machining). However, the EDM process produces a hard, brittle layer of melted and redeposited material on the machined surface which was presumed to importantly affect the metallurgical properties and thus the service performance of the machined part. North American Rockwell (NAR), under a NASA contract, determined that the EDM layer resulted in ambient-temperature property losses as great as 81 percent in ductility, 15 percent in yield strengths, and 27 percent in ultimate strengths. This surface-damaged layer would be especially detrimental to components subjected to cyclic loading by providing initiation sites for fatigue failure. NAR experiments showed the EDM-layer could be removed in such superalloys by abrasive-grit blasting or electro-polishing at room temperature, and inspection to insure complete removal by etching. This information should help to advance the rate of application of EDM processes in industry.

o Better understanding of stress-corrosion mechanisms and detection (07-14-07).

Although the development of high-strength aluminum alloys have allowed use of materials with significantly improved strength-to-weight ratios over those that were available before, these alloys are highly susceptible to stress corrosion cracking. Because the mechanism is complex and only poorly understood on a microscopic basis, and no method was available for determining stress corrosion "damage" prior to actual failure, NASA undertook to: (1) develop a better understanding of the mechanisms involved; (2) develop a practical means of evaluating stresscorrosion susceptibility; and (3) nondestructively evaluate stress-corrosion related material property changes. Measurements of electrical conductivity, ultrasonic surface wave attenuation, and internal friction loss were made on such common aluminum alloys as 7079-T6, 2219-T3 and 2219-T81. This effort demonstrated the viability of these NDT techniques to detect and compare relative stress corrosion in

these materials. The techniques and results of this study constitute an incremental addition to materials technology and thus to further understanding of surface integrity effects.

o Evaluation of stress-corrosion cracking of precipitation-hardened stainless steel (07-14-08).

Processing and heat treating operations significantly affect the stress corrosion resistance of precipitation hardened (PH) stainless steels. Because available data were limited, NASA personnel performed a stress-corrosion resistance evaluation program on ten commonly used PH stainless steels subjected to tensile stress in a corrosive environment. Three types of specimens were fabricated to test the materials in at least two directions of grain orientation: flat tensile specimens stressed in direct tension; and c-rings loaded by constant deflection for testing transverse direction of bar stock. The methods, results, and analysis constitute another incremental addition to the state-of-materials technology and therefore also of surface integrity. This evaluation should be of value to designers using precipitation hardened stainless steel, to quality control engineers, and primary metals manufacturers.

o Development of a method to increase aluminum alloy resistance to stress corrosion (07-14-09).

Residual tensile stresses remaining in the surface of high-strength aluminum alloys from processing or machining operations are often reduced by shot-peening or careful machining. Both of these techniques distort the surface grainboundary structure. Because stress corrosion occurs along grain boundaries, it cannot begin before this distorted layer is penetrated by pure corrosion. Since both pure and stress corrosion start at particularly favorable sites, if the initiation of both types of corrosion at those sites was interrupted in a controlled manner, before re-exposing the alloy once again to a corrosive environment, these sites became far less favorable for renewed stress corrosion activity and the net rate to failure therefore appreciably reduced. Techniques were developed by Tyco Laboratories, under a NASA contract, to retard the rate of stress corrosion based upon this approach.

These techniques should prove valuable to aircraft, chemical and boat building manufacturers, metallurgists, oceanographers and designers of high-strength aluminum structures.

o Development of a glass-bead shot peening system to induce residual compressive surface stress (07-14-10).

Because titanium tanks required for space missions were developing stress corrosion cracks, NASA personnel conducted an analysis to determine causes, consequences and cures. It was determined that the cause of premature failure was simultaneous corrosive action and mechanical stress. Result was the development of a rigidly controlled shot peening treatment of the titanium surface to induce residual compressive surface stress in the material.

#### MATERIALS JOINING (08)

Advances in the technology of joining materials have accelerated so rapidly that the field is difficult to describe adequately. Most of the 37 major welding processes classified by the American Welding Society have been advanced in part or in whole over the past decade and many new joining methods added in response to developments that have occurred in materials technology. A list of significant developments in materials joining, as identified by technological leaders in the field, is given in Table 8. Of immediate interest are major processes such as gas, arc, resistance and electron beam welding; brazing and soldering; and diffusion and adhesive bonding.

Gas welding is an inclusive nomenclature for all joining processes employing a gas flame as the welding heat source, with or without the application of pressure or filler metal to the parts to be welded. The regulated and mixed gases are burned at the nozzle or tip of the welding torch, producing an intense flame. Gases employed may be oxyacetylene, oxyhydrogen or other mixtures. While gas welding is simple, flexible, and economical, it is highly dependent upon operator skill.

Shielded-metal-arc welding (MIG) involves generating an electric arc between a covered (coated) metal electrode and the workpiece, with the electrode being melted and deposited in the weld. The consumable electrode wire is coated with minerals and chemicals that protect the weld puddle from oxidation and contaminants. MIG welding is used for a wide variety of metal thicknesses and alloys, is very fast, produces relatively low workpiece distortion and excellent quality welds.

Inert-gas-metal-arc welding (TIG) also employs an arc, but uses a nonconsumable tungsten electrode operating in an inert gas atmosphere, usually argon or helium. The gas provides a path for the arc, and protects the electrode and the molten puddle from atmospheric contamination and attack. Pressure and filler metals may or may not be used. Since the inert gas precludes the necessity for using a flux, and is a cleaning agent, virtually no oxide is produced in such difficult-to-join metals as aluminum, magnesium, stainless steel and beryllium copper. TIG can be used as a hand or automated process. Production costs are low, weld quality and rate are high.

Resistance welding is one of the most commonly employed metals-joining techniques. In this process, the fusing temperature is generated at the weld joint by the electrical resistance of the material when either alternating or direct current is passed through it. The parts to be joined are clamped between electrode-dies and forced together while heated to the plastic condition. The best welds are made in high-resistance metals. It is a high-speed production process, requires no filler or flux, produces low distortion and warpage, requires only semiskilled operators, and produces a smooth joint, even on

#### TABLE 8

#### SIGNIFICANT DEVELOPMENTS IN MATERIALS JOINING (08)\*

- \*\*1. Aluminum welding techniques (08-01)
  - 2. Automated welder (08-02)
  - 3. Brazing General Electric superalloys (08-03)
- \*\*4. Brazing techniques (08-04)
- \*\*5. Composite bonding techniques (08-05)
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<sup>\*</sup>As identified by technological leaders in the field of materials joining.

<sup>\*\*</sup>Developments selected for identification of NASA contributions.

dissimilar metals. The commonly used types of resistance welding which have found widespread application in auto, aircraft and appliance manufacturing, are spot, seam and projection welding.

Electron-beam welding (EB) differs from most other welding processes in that its heat source is generated by electron-beam bombardment of the workpiece rather than by an arc or flame. The beam can be focused to a very small area with the heating effects correspondingly minimized. Until recently, EB welding was restricted to in-vacuum environments; now, however, out-of-vacuum welding is in the early development stages, which would thus free the process from vacuum chamber dimensions. EB welds are quite narrow, deep, have very low distortion and stress, and are of exceptional quality.

Brazing is defined by the American Welding Society as a process "wherein coalescence is produced by heating to suitable temperatures above 800 F. and by using a nonferrous filler metal having a melting point below that of the base metals. The filler metal is distributed between the closely fitted surfaces of the joint by capillary attraction." Brazing is an oxide-free process of bonding of similar or non-similar metals which produces a stress free bond, can be used with base workpieces of different thicknesses, and results in a finished fine-line joint. Brazing may be accomplished with a torch for non-oxidizing materials, or those easily protected by flux; in a controlled atmosphere furnace (usually hydrogen or vacuum); in a bath of molten flux (usually for aluminum assemblies); or by induction brazing.

Diffusion bonding requires that the part surfaces are atomically clean, i.e., consisting of parent metal only, and subjected to sufficient pressure and heat to cause interatomic migration. Heat levels used are one-half to two-thirds the base metal melting point. In diffusion bonding it is necessary to control not only heat and pressure, but also time, atmosphere, joint surface finish, and cleanliness. Principal techniques to accomplish this type of joining are hot-press (heated platen) bonding; isostatic (high-pressure gas or fluid) bonding; friction (one part held stationary, the other spun and rammed into the fixed part) or inertia welding; extrusion bonding; explosive bonding; roll bonding (employs conventional rolling mills). Common to all these methods are the principal parameters described above. Diffusion bonding is dissimilar metals, such as aluminum to ordinarily employed to join stainless steel, which would be impossible by conventional welding or brazing techniques. The joint consists of parent metal(s) only.

In response to aerospace requirements for sophisticated, lightweight, and strong composite structures, adhesive bonding has evolved into a complex and rapidly growing technology. Adhesive bonded joints may be classified as continuous surface (both adherend faying surfaces relatively large and of the same size and shape), and core to core (lightweight sandwich structures using honeycomb core bonded to thin face sheets). Since there is no univeral adhesive applicable to any

material or environment, more than 2500 commercial adhesives have been developed. Adhesive bonds are used for mechanical fastening; sealing and insulation; fatigue, corrosion and vibration resistance, and contour smoothing. Such bonds permit great flexibility in designing.

Complete descriptions of selected developments in the field of materials joining, as identified in Table 8, together with analyses of their significance and NASA contributions to the advancement of these developments, follow.

#### Aluminum Welding Techniques (08-01)

Aluminum alloys have found extensive use in aerospace shipbuilding and various structural applications because of excellent strength-to-weight ratios. The use of these alloys has been accelerated by advances in joining technology which allows these alloys to be routinely joined by welding, brazing, soldering, and solid state bonding methods for even the most critical uses.

Prior to the space program, the technology of aluminum alloy welding was relatively primitive and unreliable. As a consequence fasteners and rivets were the primary joining processes. It was unclear how to predictably produce a weld with known characteristics; inspection techniques could not adequately evaluate a completed weld; process controls were rudimentary; aging effects and stress-corrosion cracking were poorly understood; the relationship between time, temperature, and weld quality was largely a matter of intuition rather than scientific-control; the types and effects of filler materials were not adequately defined; and a whole constellation of welding effects, such as porosity, cracking, incomplete fusion, and lack of penetration, were largely undetectable without destroying the weld in the process.

Considerable progress in aluminum alloy welding technology is directly attributable to the numerous research and development programs of aerospace organizations, research institutes, primary metals and welding components manufacturers. However, the single most significant contribution to the advancement of aluminum welding technology appears to be the massive, NASA-funded "Aluminum Welding Development Complex" program. This effort systematically investigated and advanced the entire spectrum of aluminum alloy welding technology. These and other such extensive NASA contributions, listed below, cumulatively represent a major contribution to the field.

o Aluminum alloy welding development program (08-01-01).

Space vehicles require the high-integrity welded joints with predictable behavior under extreme temperature, chemical, and mechanical conditions. Because existing aluminum welding technology could

not meet these standards, NASA undertook a nation-wide survey to identify the welding problems, practices, and on-going research, and to develop a systematic assessment of the state of welding technology. The aim of this survey was to establish a coordinated effort to solve common welding problems, avoid duplication of effort, and define those areas requiring further research. All NASA facilities were investigated, as were those of North American Aviation, Douglas, General Dynamics, Martin-Marietta, Boeing, Republic Aviation, Grumman Aircraft, Lockheed, and other such organizations.

The survey revealed a wide diversity and duplication of effort in programs, and little knowledge of the scientific bases underlying welding technology. This appeared to be due to the fact that there had never been a comprehensive aluminum welding research and information dissemination program and aluminum alloy welding was still an art rather than a science. Problems identified in the survey were analyzed, summarized, collated, and grouped, such as: welding parameters and techniques; welding equipment and instrumentation; inspection and defects; materials and material preparation, and others.

In 1964 NASA held a symposium, attended by representatives of 75 major business firms, research institutes, and other government agencies, to discuss these problems and to agree on approaches to solutions. A general outline for a comprehensive aluminum alloy welding development program was generated. Based upon this program, NASA awarded 15 major R & D contracts to transform the art into a science. research efforts included: weld-base metal investigation (Battelle Memorial Institute); mechanisms of porosity formation (Douglas Aircraft); gas analysis (Boeing); weld defect vs. joint performance (Martin-Marietta); transferability of setup parameters (Lockheed); Hydrogen Gettering (IITRI); base-metal relative to commercial plate (Battelle); time-temperature effects (MSFC); welding study on 2219 al. alloy (MSFC); material preparation (for welding) and instrumentation (IITRI); time-temperature control (Harvey Aluminum); arc shaper (AIRCO); power output wave shape (AIRCO); and non-vacuum electron beam welder (Westinghouse -see NASA contributions to EB welding, contribution 08-07-04 above); and residual-stress measurement

(Benson Associates). As a consequence of these and other studies, aluminum alloy welding is today a scientifically predictable practice, which is increasingly applied in industry to designs and structures that would otherwise be impractical. The data, devices, and theoretical understanding developed in this effort form a base used in developing aluminum alloy welding from an empirical art to a science.

o Analysis of thermal stress and movement during aluminum alloy welding (08-01-02).

A major problem in the fabrication of space vehicle structures is distortion caused by welding. These structures include numerous components which are fabricated to close dimensional tolerances. Resulting welding, distortion was often beyond correctional methods, caused delaterious joint tension and reduced joint strengths. A comprehensive literature survey revealed no systematic research had been conducted on distortion control in such situations. To deal with this problem, NASA initiated a weld distortion research program at Battelle Memorial Institute which led to the development of techniques for predicting and controlling distortion. A three-dimensional computer analysis, aimed at predicting total metal movement, was carried out. This analysis describes the relationship between width of the heat zone and rigidity of the material, and can be used to design better jigs and fixtures. This is the only analysis that has been made of the dynamics of welding distortion while the process is underway.

o Development of a comprehensive aluminum alloy welding textbook (08-01-03).

A comprehensive aluminum alloy welding textbook, titled "Integration of NASA-Sponsored Studies on Aluminum Welds," is being prepared at M.I.T., in conjunction with APT Associates, under a NASA contract. This will be the first, and most definitive, text on the subject and is therefore of interest in engineering and physics curricula to basic researchers, and to applied welding engineers. Much of material used in this text is based upon NASA aluminum welding development efforts described in contribution 08-01-01, above. For example, some materials to be included in the text which are based on NASA aluminum welding efforts include:

effects of shielding gas contamination on porosity; effects of base and filler metal contamination on porosity; mechanisms of porosity formation; use of scavenger elements to reduce porosity; effects of porosity on weld-joint performance; time-temperature effects and control; and transferability of welding parameters in the GTA process.

o Development of a sensitive hydrogen detector (08-01-04).

Hydrocarbons are one of the principal sources of aluminum weld contamination. These come from unfiltered factory air, fingerprints, and contiguous erosion courses such as vacuum roughing pumps, and elsewhere. Since hydrogen gas from these absorbed and surface contaminants is evolved during the weld process, a suitably sensitive detector could be used to indicate the probable character of the finished weld during the welding process. Under a NASA-supported program, Boeing developed a supersensitive hydrogen detector which indicates hydrogen to one part in 25 billion. Boeing is patenting the device and marketing it to those welding facilities requiring superior quality weld control. This spectrometer analyzer may also be applicable to air pollution monitoring.

o Compilation of critical data on aluminum alloy joining (08-01-05).

Although the number and variety of NASA in-house and contractor performed research programs in many technological areas is large, it often happens that information of particular interest to anyone scientific or technological discipline forms only part of any given study report. Retrieving such data from numerous sources is often beyond the resources of the interested party. To deal with this problem, NASA compiled basic and advanced data on joining aluminum and other light metals and published the compilation in SP-5064, "NASA Contributions to Metals Joining." Some of the data in the light metals section includes: mechanical property tension test results from vertical position MIG and TIG welds in 20-14-T6 aluminum alloy; required arc current as influenced by pressure and wire feed speed; macro and micro section photographs of desirable welding arcs and welds; notch sensitivity and cryogenic properties of Al-Cu alloys; and mechanical properties of 6000 and 5000 series aluminum alloys.

o Development of a weld intelligence system and controls (08-01-06).

Unlike many other manufacturing technologies, welding has defied true adaptive automation. Even with advanced control devices for those few weld variables amenable to automatic control (e.g., arc voltage, current, travel speed and wire feedrate). the human operator still provides the only effective feedback loop between direct weld characteristics and controllable variables. Since advanced automation is desirable both for economy and reliability, NASA contracted Merrick Engineering to investigate the feasibility of developing a transducer (sensor) which would directly or indirectly monitor one or more of the direct weld characteristics. and to develop a prototype automatic feedback control system that would use the transducer signal to make the necessary modifications in the welding process. Merrick developed hybrid thermocouple probes and an adaptive controller and successfully verified the system performance. This system represents an important incremental advance in welding technology. The system is insensitive to radiated arc interference and variations in puddle emissivity, provides an essentially instantaneous response, and is free from interference due to surface oxides.

o Advanced understanding of aluminum welding technology (08-01-07).

To advance the state of aluminum welding technology, NASA carried out and sponsored several studies of welding designed to develop a better understanding of the process. Included were studies of time temperature effects on aluminum welding; variables affecting the occurrence of weld defects; a theoretical model based on nucleation and growth theory for the porosity formation in aluminum welds; transferrence of inert-gas welding settings and data spun machine to machine and facility to facility; and the correlation between porosity and other defects and the mechanical behavior of a weld joint. These and the numerous other similar NASA studies and compilations contributed segmentally, to developing the scientific base for aluminum alloy welding science. For a description of a major planned program designed to advance aluminum welding technology, see NASA contribution 08-01-01, above.

o Development of welding techniques and filler metals for high-strength aluminum alloys (08-01-08).

High strength aluminum alloys are considerably more difficult to weld than conventional alloys. yet because of their good strength-to-weight ratio are desirable for critical applications. NASA therefore contracted with the Southwest Research Institute (SWRI) to conduct an extensive investigation and development program to develop practical joining techniques. From early-1963 to mid-1966, SWRI investigated such parameters as: the phases formed in weld joints of 2219-T87 A6 with 2319 filler; variations in mechanical properties across welded joints; failure mechanisms in both uniaxial and biaxial testing; alternative ways to improve the strength of welded aluminum alloys; new welding techniques and filler metal alloys; and the properties of MIG and TIG weldments on 2014-T6 and 2219-T87 Al alloys under biaxial and uniaxial loading. Some of the important findings of these studies include the fact that TIG weldments as a group exhibit higher uniaxial tensile properties than those of MIG weldments (other studies have also amply verified this finding), and that residual stress is distributed with 57 percent in the weld, 8 percent in the heat affected (i.e., crack-prone) zone, and 13 percent in the parent metal.

o Development of methods for controlling aluminum alloy welding distortion (08-01-09).

Although space vehicles consist of lightweight structures, total structural weights are quite large and the geometric integrity of these structures must be held to close tolerances. Since most of the structures are welded, and welding characteristically induces heat-generated distortion, it was necessary for NASA to find corrective measures. Under a NASA contract, North American Aviation (NAA) investigated the feasibility of controlling aluminum alloy welding distortion by the application of mechanically or thermally induced preloads. Results indicated that mechanically applied preloads resulted in approximately 30 percent less residual stress and substantially less distortion; with related results for thermally applied constraints. A model was developed for predicting preload magnitudes and patterns related to residual stresses.

o Development of weave bead techniques for MIG welding of aluminum alloys (08-01-10).

The orientation of parts to be welded (e.g., flat plates lying horizontally flat, vertically upright, or horizontally upright) requires a variety of positions for the welding head, each presenting unique problems of gas entrapment, weld bead shape. and contamination. Vertical welding, as is required for many space vehicle assemblies, is particularly difficult for a manual operator due to the continuous pull of gravity. NASA, therefore, contracted with IITRI to define the parameters of a machine-controlled wide-weave technique for performing vertical-up welds in thick aluminum plates, and to develop an automatic machine to perform such welds. To be assured of a technique of sufficient latitude to reproduce high quality welds, IITRI researchers studied the movements of skilled human welders, which defined the necessary oscillation parameters, and constructed from this data an automatic machine which successfully simulates the correct movements to produce reliable welds.

o Improved quality of commercial aluminum alloy weld wire (08-01-11).

Because inexplicably defects were developing in 2219 aluminum alloy welds, Lockheed Missiles and Space Company under contract to NASA investigated the problem and determined the cause was due to the wire used in the welding process. The weld wire imperfections consisted of surface pits and foreign particulate matter adhering to the wire, which Lockheed determined to be the cause of the weld contamination. The weld wire producer was notified and he improved the wire and modified the wire reel, which was source of the foreign matter. This relatively indirect, but expensive and important investigation would probably not have been undertaken were it not for space program quality standards. Defective welds in 2219 aluminum produced by inadequate wire might have remained problematical for commercial users of the 2319 aluminum alloy welding wire from that firm.

o Development of new weldable aluminum alloys (08-01-12).

The Aluminum Company of America (ALCOA), under contract to NASA, developed two new series of weldable aluminum alloys with mechanical properties superior to

those commercially available. The first series based on 2219-T87 has alloys with ultimate tensile strength, yield strength, percent elongation, and notch-tensile ratios from 8 to 14 percent higher than commercial counterparts. These alloys have good resistance to stress corrosion cracking, unchanged strength and formability after room temperature storage, can be pre-aged, stretched, and aged, are well suited to cryogenic temperatures, and have better mechanical properties at -423 F. than at room temperature.

The second series of alloys were developed specifically for cryogenic temperature applications. This new 7000 series of wrought aluminum alloys has mechanical properties superior to standard 7000 series alloys, which are relatively low in notch toughness and weldability.

These improved alloys are useful in such high stress environments as air frames, submersibles, structures, high-speed surface craft, and cryogenics.

o Preparation of materials data handbooks for aluminum alloys (08-01-13).

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Researchers at the Syracuse University Research Institute, under NASA contract, compiled a series of materials properties handbooks. Two of these handbooks cover aluminum alloys 6061 and 7075 and include data on the properties of the alloys at cryogenic, ambient, and elevated temperatures; procurement information; metallurgical characteristics; production and manufacturing practices; static mechanical dynamic, time-dependent, and physical properties; corrosion resistance and protection; surface treatment; and joining techniques. These compilations provide necessary data for the successful welding of these aluminum alloys.

o Determined causes of cracking in ac-TIG welding of aluminum alloys (08-01-14).

Cracking was a frequent occurrence in the heataffected zone of aluminum alloy 6061 welded with
ac-TIG equipment. Although these cracks are only
0.015-inches deep, they lead to serious deleterious
effects when subjected to environmental stress such
as static tension, fatigue, and salt spray corrosion.
Under a NASA contract, North American Rockwell
determined that the cracks developed when the super-

imposed high-frequency current was improperly adjusted, leading to adaption of the semimolten metal in the heat-affected zone. Lack or depletion of boundary constituents combined with rapid freezing and normal shrinkage stresses resulted in cracking. Control of the high-frequency current is therefore of paramount importance for high quality ac-TIG welding.

o Determination of weld joint strength and mechanical properties of aluminum alloys (08-01-15).

The weld joint strength and mechanical properties of a variety of aluminum alloys were determined by NASA. Among these alloys analyzed was 2219-T81, by General Dynamics, which resulted in a better understanding of the welding properties of this material including the following: welding significantly reduces the yield strength of 2219-T81, but only modestly reduces ultimate strength; base metal sheet and plate toughness, plane stress and plane strain fracture toughness of the heat-affected zone all increase with decreasing temperature; toughness behavior becomes unpredictable after three "repair" welds; cyclic flow growth rates are generally higher on the weld metal than elsewhere; and other such data of utility to materials researchers and welding engineers.

o Study demonstrates weld improvement by bead removal (08-01-16).

To assure full weld penetration, aluminum alloy plate is welded from both sides leaving a high-tension area caused by the resulting weld beads. The effect of removing one or both beads on the mechanical properties of the plate was not known. Under a NASA contract, Boeing determined that removal of either bead increased ultimate strength by 5 percent and removal of both beads increased ultimate strength by 10 percent. Ductility increased by 14 percent when the "top" bead was removed, by 29 percent when the "bottom" bead was removed, and by 50 percent when both beads were removed. Yield strength, on the other hand, decreased 3 percent with removal of the top bead, 8 percent with removal of the bottom bead and 11 percent with both removed. In designing such a system for a particular environment the trade-offs becomes one of balancing improvements in strength, ductility, fatigue properties, and burst pressure against the slight drop in yield strength. Information of this nature is invaluable to the design and structural engineer.

### Brazing Techniques (08-04)

Unlike welding, where process temperatures are high enough to melt the parts to be joined, brazing requires melting of a filler material below the melting temperature of the parts to be joined. This is a distinct advantage in joining dissimilar metals or metals which crack easily upon melting and resolidification. Although all brazing processes — dip, induction, resistance, torch and furnace brazing — are employed extensively in aerospace and advanced industrial applications, vacuum furnace and induction brazing are probably the most widely used in these facilities. If the filler alloy is to be melted in air, a flux is necessary to remove surface oxides in order to present a clean base metal to the molten filler metal and to prevent oxidation during heating.

Furnace brazing is done in a reducing (hydrogen), inert (nitrogen, argon), or vacuum atmosphere. Furnace brazing is suited to high-production runs, joining of large and small parts, and parts with multiple or hidden joints. Induction brazing involves surrounding the potential braze joint by an induction coil field which heats the desired area by means of its resistance to the induced current. The filler metal is ordinarily flux-free and the joint may be made in air, inert gas, or vacuum. Induction brazing is suitable for in-place brazing of joints (a very important space-program application) and other applications requiring close control and localized heating.

o Development of a welding, brazing and soldering handbook (08-04-01).

In applied technological fields, such as brazing and soldering, there are usually few handbooks or texts that combine pragmatic information of immediate utility with professional acceptability. To overcome this problem, NASA contracted with Boeing to prepare a comprehensive survey of techniques for the selection, evaluation and application of welding, brazing and The Handbook contains a summary description soldering. of the joining processes; criteria for selection of the various joining processes for specific alloys, types of joints, structural configurations, and material thicknesses; and details the advantages and disadvantages of the different joining methods for various structural designs and applications. Some of the joining methods discussed include: fusion welding (arc, electron beam, electro slag, laser) resistance welding, solid state welding, brazing, and soldering.

o Preparation of a comprehensive survey of ceramic to metal joining technology (08-04-02).

Battelle Memorial Institute, under a NASA contract, prepared a comprehensive survey on ceramic to metal joining technology for electronics application. Included is a review of ceramics, metals, and brazing filler metals; joint configurations and surface preparation; theory of ceramic-to-metal joining, fusion, solid phase, gas-pressure, and solid-liquid phase joining; and fusion and nonfusion joining of graphite.

o Development of a brazing and brazing alloy bibliography (08-04-03).

Critical evaluations and compilations of ordinarily diffuse reference material are important aids to scientists and engineers working in a field of technology. Among the many such compilations prepared by or for NASA is a selection of materials from a retrospective literature search of NASA's Scientific and Technical Information Division's data bank on brazing and brazing alloys which are thought to be particularly useful to industry. The bibliography includes alloys, basic and technical papers, fabrication and development methods, and testing and evaluation.

o Evaluation and compilation of reference materials on joining of nickel and nickel-base alloys (08-04-04).

An extended and critical evaluation and compilation on the state of current technology of joining nickel and nickel-base alloys was prepared by Battelle Memorial Institute, under a NASA contract. The Battelle effort covers the techniques of joining by welding, brazing, and soldering; and discusses preparation methods, specific joining process, joining dissimilar metals and joint quality. These alloys are used extensively in high temperature and corrosive environments and are particularly difficult to fabricate reliably. This compilation is therefore of interest to engineers concerned with the design and use of these metals. For a more complete description of advances in the use of nickel-base alloys, see NASA contributions to improved superalloys in the field of high-temperature metals.

o Development and evaluation of braze alloys for vacuum furnace brazing (08-04-05).

Aerojet-General conducted a program to develop an alternative braze alloy to the very expensive commercial copper-gold alloy widely used in space vehicle applications. Two of the new alloys developed on this program are equal in all respects to the expensive commercial variety. Use of these brazing alloys in preference to the off-the-shelf variety reduced the gold content by 57 percent and braze material cost by 50 percent. In the enormous quantities these alloys are used on large Saturn thrust chambers, for example, this represents significant savings. These new alloys should bring equal economic benefits to present commercial consumers of copper-gold alloys.

o Identification of a low-cost substitute for expensive high-temperature brazing materials (08-04-06).

An inexpensive, commercially available material was identified for use in critical high-temperature furnace brazing by NASA. To replace expensive gold-palladium alloys, NASA found that commercially available nickel-copper wire containing 6 percent and 12 percent could be successfully used to braze nickelbase alloys and stainless steels at 2070 F. and 2610 F., and also to replace gold-copper alloys for lower temperature (1400 F.) brazing applications. The brazed joints using this material have properties comparable to or better than those brazed with the expensive alloys, and cost savings have been substantial. This brazing alloy should find application with industrial users of high-temperature costly braze alloys. The inexpensive nickel-copper wire is ordinarily sold for common electrical uses and probably never have been considered for sophisticated high-temperature applications on critical joints.

o Improvements in joint properties (08-04-07).

NASA has supported a wide variety of efforts designed to improve joining materials and methods. These efforts, which have generally been directed at improving properties between specific material combinations, include the following, among others:

(1) Development of a technique to promote flow of filler material or brazing alloys, by pretreating the parent material with a thin coating of pure titanium

in a vacuum deposition system. Parts so treated can be safely stored for considerable periods and still produce a superior weld with excellent weldability (M.I.T.). (2) Development of a brazing process that produces a ductile, high strength, corrosion-resistant bond between stainless steel and aluminum for service temperatures ranging from -320 F. to 1,000 F. Formation of the usual brittle intermetallic compounds in the joint is prevented by vapor depositions titanium in conjunction with an aluminum-zirconium-silicon alloy. This process allows a designer to take advantage of the high strength to weight ratio of aluminum and the strength and corrosion resistance of the stainless steel (North American Aviation). (3) Development of a method for producing high-strength braze joints between copper and steel by first plating the surface of the copper with a very thin (.0004 inch) gold layer. The gold plating reduces porosity in the joint considerably and the joint strength is greater than the parent copper itself (North American Aviation). Numerous contractor research and development efforts for NASA contains such incremental advances in joining and brazing technology, but because the purpose of the total effort is often directed at some specific space program application, these important contributions often go unnoticed by other workers in the field.

o Development of an ultrasonic scanning system for in-place inspection of brazed tube joints (08-04-08).

A miniaturized ultrasonic scanning system was developed under contract by Spaco, Inc. for the in-place evaluation of brazed tube assemblies on the Apollo telescope mount thermal-conditioning system. This scanning device is capable of detecting defects as small as 0.008 by 0.010 inch, which exceeds the usual 0.015 inch diameter defect resolution called for in specification documents. It is a fast, reliable, accurate, and diversified brazed tube inspection system capable of scanning 1/4 to 5/8 inch diameter joints (in the prototype size) with limited side, bottom, and access clearances to unions, tees, elbows, and cross configurations. This distinctly innovative NDT device should be of considerable interest to personnel at refrigeration, chemical, petroleum and other such facilities, as well as the aerospace industry.

#### Composite Bonding Techniques (08-05)

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A 1959 article by Claude Talley in the <u>Journal of Applied Physics</u> describing the development of exceedingly strong boron rods, may be considered one of the prime precursers to today's research and development efforts in composite technology. By 1965, the U. S. Air Force was supporting massive programs to "get composites into the air." Composite materials may be made of ceramic or graphite fiber matrices, metal wire matrices, plastic matrices, or combinations of these and many other materials. Composites are particularly interesting to spacecraft and aircraft designers because they offer such unique advantages over conventionally homogeneous materials as low density (i.e., lightweight), extremely high strength, high stiffness, low thermal stress, and exceptional formability. By tailoring a material to the environmental forces it will encounter, composites provide elaborate design options, allowing an engineer to create structures that would be otherwise impossible.

Composites research already accounts for well over \$100 million of government and industry support and shows every indication of becoming a major new technology. As with any promising new discipline there are of course problems in the midst of demonstrable advantages. Composites are difficult to inspect with conventional NDT methods. Materials properties of individual components are by themselves difficult to assess. When bonded to other dissimilar materials to make a heterogeneous aggregated composite, the cumulative properties are exceptionally resistant to conventional methods of analyses. Bond strength between laminates, for example, are difficult enough to determine in the laboratory, let alone in a routine production inspection.

Composites are already being employed in aircraft, ship and building construction for gears and other components, in appliances, in sporting goods and elsewhere. They hold the promise for widespread acceptance and use throughout industry in the near future. More rapid and widespread acceptance is held back, primarily, by the present high cost of advanced fibers, by difficulties in analysis and inspection and by a still-incomplete technological data base.

NASA's role in advancing the technology of bonding composite materials has been in supporting research on adhesive bonding technology materials, diffusion bonding (see NASA contributions to diffusion bonding below) and in developing inspection and analysis techniques and equipment (see NASA contributions to the field of nondestructive testing).

Specific NASA contributions to composite bonding technology include the following, among others.

o Advanced understanding of time-dependent strength of adhesive joints (08-05-01).

Time-dependent failures of elastic and viscoelastic solids joined by polymeric bonding such as in composite materials adhesive joints, was analyzed by the California Institute of Technology. The problem was analyzed in relation to microscopic processes and macroscopic stress fields acting on the joining system and covered: stress analysis of imperfect bonds; equations governing time-dependent joint fracture; descriptions of time-dependent effort and transition from adhesive to cohesive fracture; and influence of fracture path on joint strength.

o Evaluated and compiled current information on the deformation characteristics of adhesive-bonded joints and metal-adhesive interfaces (08-05-02).

Battelle Memorial Institute systematically evaluated and compiled current information on the deformation characteristics of adhesive bonded joints and metal adhesive interfaces. Theoretical and experimental analyses were detailed for elastic-stress distributions in adhesive-bonded joints, with consideration given to the rheology and fracture mechanisms and the effects of loading and environmental conditions on deformation and joint fracture.

o Developed improved adhesive-bonded structural joints (08-05-03).

The Whittaker Corporation, under a NASA contract, investigated methods for obtaining uniform shear stress distribution in adhesives used in double-lap bonded joints. These joints are usually designed from assumptions about rivet and weld-joint technology. As a result, only 20 percent of the available adhesive strength potential is used. The object of the Whittaker effort was to develop methods to increase the use of such strength potential. Whittaker studied boron-epoxy composites, aluminum, titanium and steel in their tests of alternative lay-up joint designs, and materials properties. Result of the effort is more effective adhesive-bonded joint designs and a procedure for characterizing adhesives based on analytical results.

o Development of an improved primer for bonding polyurethanes to metals (08-05-04).

North American Rockwell (NAR) developed for NASA a primer to ensure effective bonding integrity of polyurethane adhesives to metal surfaces at temperatures ranging from -423 F. to 120 F. The primer-adhesive system provides greater metal surface protection and bond strengths over this interval than other such systems; it also reduces gas permeability and provides a film of lower surface tension, thus facilitating adhesive application; and it can be applied directly by spraying or brushing.

o Explosive bonding of metal matrix composites (08-05-05).

NASA has supported considerable research into explosive forming and bonding. One effort, directed at making metallic composite sheets, resulted in the development of a process for producing sheet composites of aluminum alloy reinforced with interstitial stainless steel wire —a system with tensile strength of 67,000 pounds per square inch. Another explosive—bonded aluminum composite reinforced with a modular—filament sheet of stainless steel has a tensile strength of 94,000 pounds per square inch. The bonds formed by this process are metallurgically excellent and no external heat is required. In addition, dissimilar metals can be joined simply and inexpensively.

o Development of a flowmeter to determine the mix ratio for viscous adhesives (08-05-06).

One of the persistant problems in adhesive bonding technology is identifying and maintaining the precise ratio and mix rate of adhesive resin and hardener in the initial synthesis of the compounds. Even slight variations can seriously affect the resultant bond characteristics. Under a NASA contract the Douglas Aircraft Company developed a continuous flow mixing machine which produces a high viscosity adhesive. This adhesive is formed from high viscosity resin and aliphatic amine hardener pumped through separate lines to a rotary blender. Malfunction or flow irregularity are detected instantaneously by differential strain gages placed in the gummy materials which relay a

signal to a microammeter and control circuitry. Since adhesive bonding is rapidly being adopted in industries such as appliance, auto, truck, boat, aircraft and homebuilding, this device or some evolutionary successor will probably figure importantly in future applications.

o Developments of a bonding method for large structures (08-05-07).

It is frequently necessary to attach doublers, brackets and other fittings to the surface of large structures. In those cases where conventional fasteners cannot be used, bonding becomes a problem because the size of the structure precludes the use of normal bonding techniques. North American Aviation developed a novel technique employing a radiant heat source in conjunction with a heat-resistant transportable vacuum bag and a black heat absorbing cloth. This system forms a flexible, portable bonding oven. The item to be bonded to the structure is temporarily held to the large structure surface by any convenient means. It is covered by the black cloth, which itself is covered by the vacuum-pumped bag, the edges of which are sealed with suitable vacuum sealant to the large surface. The vacuum bag provides an even pressure to all assembly elements, and the radiant heat source is provided a short, dense path for conduction from the black cloth to the bond interface. This technique should find application in composite structures or adhesive bonding situations as described above, such as in shipbuilding, automotive, truck, boat, and appliance manufacturing.

## Advances in Diffusion Bonding (08-06)

Diffusion bonding is a technique for joining metals through the controlled application of heat and pressure, which results in the interdiffusion of atoms across mating surfaces in either the solid or liquid state. Joint surfaces must be atomically clean and brought within atomic distances by applied pressure if solid state diffusion is desired, or by thoroughly wetting both surfaces with a continuous film if a liquid phase is called for. Heat is applied to increase the rate of the atomic migration, with temperature, usually 1/2 to 2/3 the melting temperature of the base metal. Intermediate filler materials are occasionally used to facilitate the diffusion process. The joint resulting from this technique is usually as strong as the parent material. Other major advantages of the process are: the production of very close tolerance joints; the ability to join dissimilar metals;

the production of virtually flawless stress-free joints; the ability to produce laminated structures and complex shapes otherwise impossible with conventional methods; considerable savings in machining costs and material wastage; smaller radii at joints; and a reduction in lead time through the use of sheet and plate stock rather than forgings.

Some principal developers of diffusion bonding technology have been the Battelle Memorial Institute with AEC, Air Force, and NASA support; and North American Aviation, Boeing, McDonnell Douglas and Lockheed with in-house, DOD and NASA support. The process is only at the beginning of its potential and is currently employed in fabricating parts for nuclear reactors, turbine blades, composite structures, joining or cladding dissimilar metals, bonding bellows and turbine impellers, and other similar applications.

NASA's contributions have been primarily to advance both theoretical understanding of the process and to generate state of the art improvements in diffusion bonding itself. Among these contributions are the following.

o Development of a low-temperature technique to diffusion bond 347 stainless steel tubing connectors (08-06-01).

As part of an overall NASA effort to develop improved connectors for tubing, a research program was undertaken at the Battelle Memorial Institute to develop diffusion bonding techniques to produce leaktight connector joints. Each space vehicle employs many thousands of such joints, the reliability of which must be assured. Previous Battelle studies had indicated such joints could be produced at bonding temperatures below 1000 F. The technique developed by Battelle employed first machining an oxide free surface, which was then electroplated, which itself was then diffusion bonded to its correlative electroplated film on the mating surface. The intermediate metal system, which was demonstrated far superior to other tested, consisted of a gold-copper-gold composition. Strong leaktight joints were produced at 700 and 500 F. with tensile strengths greater than 20,000 psi. in about 15 minutes. The significance of this successful program is that superior joints can now be made in the field, in the open air, and at moderate to relatively low temperatures. This important innovation in diffusion-bonding state of the art is not only theoretically significant but can also be empirically useful to maintenance and research personnel working in aircraft, ship, reactor, and numerous laboratory situations.

o Discovery of unanticipated bond strength problems in TD-nickel (08-06-02).

In evaluating materials for potential space shuttle at NASA/LeRC, it was learned that the diffusion bond of TD nickel was weaker at elevated (1600-2,000 F.) temperatures than predicted by theory. Further investigation of the problem revealed that the dispersed thoria in TD-nickel was inert in the diffusion process, and that the bond was due primarily to nickel diffusion. The thoria only provided mechanical atomic interlocking. and did not migrate across the joint. In-service use would have permitted the joint to creep, and thus with repeated cycling, to ultimately fail below its predicted strength. Because of this discovery the primary metals manufacturer launched a development program to correct the unsuspected anomoly. For a discussion of the development of an effective process for diffusion-bonding TD-nickel. see NASA contribution 08-06-07 below.

o Development of roll diffusion-bonded titanium panels (08-06-03).

In 1965 NASA awarded a contract to North American Aviation (NAA) to advance the technology of roll diffusion bonding. Lightweight titanium structures in the form of T-stiffened panels were designed, and fabrication techniques for titanium alloy roll diffusion bonding developed. Panels manufactured under this program were tested and revealed that roll-diffusion bonded titanium structures possess excellent strength characteristics under tensile and compressive loads. This effort represents a significant contribution to both the theory and practice of diffusion bonding technology and therefore should be of interest to materials researchers and designers of aircraft. submersibles, and aerospace vehicles, among others. The process has been purchased by U. S. Steel.

o Development of techniques for diffusion bonding beryllium to beryllium (08-06-04).

Harvey Engineering Laboratories, under a NASA contract, developed process techniques for roll bonding (i.e., a variety of diffusion bonding) beryllium to beryllium, and beryllium to titanium for use as rib-stiffened panels for space vehicle

applications. Beryllium was bonded to beryllium at 1500 F. and 40-60 percent reduction, with best results obtained when the covering steel jacket was degassed, gettered and evacuated prior to sealing off just before rolling. Beryllium to titanium bonding proved unfeasible. Although the process needs further work, the program successfully demonstrated the feasibility of roll bonding beryllium to beryllium. Since beryllium is a health hazard and also exceptionally difficult to machine, the process offers designers and production engineers new, safer, and more flexible design options.

o Development of a practical method of weld diffusion bonding steel plate in air (08-06-05).

NASA personnel have developed a relatively simple method of diffusion welding AISI 1020 steel plate in a standard air atmosphere furnace using only a 5 psi. deadweight loading. Surfaces are ground flat, degreased, and aligned in ambient conditions, then seal-welded around the periphery. The sealed unit is then furnace heated at 2200 F. at 5 psi. loading for two hours. This step involves a combined autogeneous vacuum cleaning of occluded surface oxides and absorbed gases from the surfaces to the interior, and the diffusion migration across the interface.

Welds produced by this method are nearly undetectable metallographically and tensile specimens break in the parent materials instead of at the joint. This simple technique eliminates many expensive procedures and devices ordinarily employed in such bonding and is economically superior to the standard submerged arc and electroslag processes. It is applicable to carbon steels, alloy steels, stainless steels, reactive and refractory materials, ceramics, glass-to-metal joints and many variations of these materials. Since no vacuum furnace, hot press or welding machines (other than for the seal-bead) are required, the process should be of interest to any industry employing weld processes, such as auto, appliance, structural steel, and ship manufacturers.

o Advanced understanding of the joining of dissimilar metals by diffusion bonding (08-06-06).

One of the principal advantages of diffusion bonding techniques is to join dissimilar materials.

Since the chemical, magnetic and physical properties of two dissimilar materials mitigate against a permanently stable interatomic or intermolecular bond, careful matching of the thermophysical properties is required. In the early 1960's, NASA contracted with the Boeing Company to conduct a development program in the area of joining dissimilar metals. The Boeing efforts included compilation of bibliographies and reviews of various solid state welding techniques, such as press bonding, roll-bonding, gas pressure bonding, explosive bonding, and resistance diffusion welding; assessment of effects of critical parameters such as time, temperature, surface preparation pressure, intermediate materials (diffusion aids) and atmosphere; development of a simple bonding method using differential thermal explosion tooling; and demonstration of the feasibility of diffusion bonding stainless steel to aluminum alloys.

These early research efforts represented an important incremental advance in the technology by adding significantly to the data base of diffusion bonding and by developing techniques applicable to actual use in industry.

o Development of a diffusion-bonding process for joining dispersion-hardened metals (08-06-07).

Dispersion-hardened metals present a difficult joining problem with standard welding and brazing methods resulting in joints with only 50 percent of parent metal (see NASA contribution 08-06-02, above). A solid-state, welding process was developed at NASA/LeRC which gives good welds through diffusion and recrystallization. This technique is applicable to welding any alloy which undergoes recrystallization upon heating. For a complete description of this process, see NASA contributions to dispersion-hardened metals in the field of high-temperature metals, contribution 04-04-05.

## Electron Beam Welding (08-07)

Electron beam (EB) welding is unlike conventional weld methods in that it employs not an arc or flame but a highly collimated beam of electrons to effect the joining process. Since the electron beam is collimated and focused on the workpiece, it has to be protected from collision with ambient gas molecules to prevent destructive dispersion of the electron energy density, and from contaminating the weld.

Because of this, EB welding has had to be carried out in a high-vacuum environment, which therefore limited the size of the welded object to vacuum chamber dimensions. Recently, however, welds comparable in quality to those made by inert gas-shielded arc welds have been generated in only a rough-pump vacuum (75-100 microns). Today, manufacturers are offering nonvacuum EB weld machines. Although these welds are not comparable in depth or quality to EB vacuum welds, they are still superior to conventional welding methods in production welding speeds and ratios, and in having a minimum heat-affected area.

EB welding is widely used in the aerospace industry, where it has developed from laboratory to practical application, in the automobile industry, and increasingly in routine industrial welding.

In the automotive industry EB welds are made on ball joints, steering column tubes, distributor covers, frame members, gears, fly wheels, and elsewhere. Very thick sections can be welded with ease and precision; filler metals are usually not required; welds can be made through a solid part to a joint beneath; ordinarily only one rapid pass is required; the weld strength often exceeds that of the parent material; virtually any metal can be welded except gaseous metals such as cast iron and high carbon steels; and various nonmetallics such as alumina, beryllia, magnesia, Pyrex, Vycor and various ceramic-metal combinations, can also be welded. EB welders cost from \$35,000 (unusually small) to \$80,000 (normal), to well over \$1 million.

As a pioneering consumer and developer of EB state of the art, NASA's contributions range from basic materials properties studies to the development of EB component devices and an important new type of EB weld system. Specific NASA contributions to EB welding, among others, follow.

o Provided technical assistance to contractors (08-07-01).

NASA technical personnel familiar with electron beam welding routinely respond to inquiries from vendors and contractors for technical assistance. In one case, in point, a major southern California aerospace contractor was experiening considerable difficulty in building a hypersonic research engine, which required joining of parts made of many different materials by electron beam and conventional welding and brazing. To aid the firm, NASA personnel were sent to the contractor's facility to provide technical assistance. The NASA team learned that outdated welding specifications were being used. Also that much of the NASA work had been subcontracted, making it difficult to evaluate when returned for quality control inspection by the prime contractor, and that the EB weld operators were

unfamiliar with joining magnetic to non-magnetic materials. NASA personnel remained at the contractor and subcontractor facilities for over five months providing technical instructions on the application of EB welding techniques and on the development of welding specifications. Personnel from this same NASA field center also host a 42-member monthly meeting of the local American Welding Society during which considerable additional information is exchanged. This type of assistance to commercial firms aided them in acquiring and successfully applying electron beam welding technology long before they might have been able to otherwise.

o Development of suppressor plates to eliminate EB arcing (08-07-02).

EB welding of aluminum, copper, titanium alloys and other strongly vaporizing materials have always presented a serious problem. When the high-velocity electron beam strikes these materials, secondary emission of ions and electrons generate a plasma in the weld region which causes arcing between the gun and the plasma, thus greatly reducing the beam energy of the weld site and introducing difficult weld quality control problems. To overcome this, NASA developed suppressor plate designs which either collect the plasma or redirect the secondary emission back to the workpiece. This system increases beam efficiency, eliminates the necessity for grounding screens over viewpoints (thus enhancing visibility) and significantly increases filament life, thereby reducing maintenance.

o Determined capabilities of equipment for high-voltage EB welding of aluminum alloys (08-07-03).

As noted above (contribution 08-07-02), aluminum vaporizes easily in even low-energy electron beam densities. In high-voltage EB applications, aluminum vapors will readily contaminate the filament, lens and other components, cause arcing, and for some aluminum alloys, draw exceptionally high currents and generate high heat. Shortly after the inception of practical EB systems in the early 1960's, NASA contracted with North American Aviation (NAA) to determine the capabilities and limitations of high-voltage EB weld equipment for joining high-strength aluminum alloys. NASA determined the reciprocal

effects of the alloys on the EB system and vice versa; thickness limitations; the effects of varying lens current; travel speeds; and cleaning procedure effects on weld depth to width ratios.

o Determined relationships between weld quality and nonvacuum EB weld procedures (08-07-04).

The need to use a vacuum chamber in the EB welding process limits the size of the workpiece, and also imposes time-consuming and inconvenient pump-down and remote-control manipulation processes. Because of this, manufacturers have been attempting from the beginning to free the workpiece from the vacuum constraint. A NASA-supported EB program conducted by the Westinghouse Electric Corporation involved identifying and analyzing the critical procedure details for nonvacuum EB welding and relating these to undercut, bead width, contour, and porosity. Westinghouse developed critical machine setting data, quantified the welding parameters produced radiographically determined sound welds comparable to or better than TIG welds in 1/4-inch aluminum, investigated the relationships of porosity to speed and energy density, and developed other basic data. This early (1965) program was a pioneering effort to advance EB welding technology to out-of-vacuum welding.

o Development of the first practical out-of-vacuum portable EB welder (08-07-05).

Because the y-support rings for the Saturn vehicle are 33 feet in diameter, they would not fit any existing EB vacuum chambers, yet NASA required the integrity of an EB weld for this application. It was therefore necessary for NASA and contractor personnel to develop an entirely new type of practical, portable EB welding gun and associated subsystems which could operate out-of-vacuum. Although the Air Force had attempted to develop such a gun, its performance was inadequate for any practical purposes. Several commercial manufacturers of EB welding machines were also unsuccessful in attempting to develop such a gun.

To meet their needs, NASA contracted with Westinghouse in the early 1960's to develop just such a portable machine. Result was a lightweight (61 lb. as against 15,000 lb. for a conventional EB welder),

small (21 x 12 inches, as against 200 cubic feet for a conventional system), battery and power-operated, gun having self-contained electronics and evacuation features, and operating for as long as 5 minutes continuously without battery drain.\*

The technological achievement embodied in this EB system development program is extremely high. In reducing the volume of practical EB systems many times, it required a very difficult miniaturization of the power supply; development of a new, rugged, long life electron emitter; very sophisticated optical advances and orificing; and the development of a low-molecular weight shield gas system. The welder operates at 140 inches/minute and requires only one pass, rather than repeated "cosmetic" passes; offers much greater control ease; and reduces heat input more than 60 percent over that of the TIG process, thereby practically eliminating shrinkage.

This step-wise addition to EB welding technology promises to have a major impact on the welding industry as the system and its advantages become known and available. Any medium to large welding shop, using either EB or conventional welding, will ultimately use or contract-out for the use of such portable EB welders.

<sup>\*</sup>It should be noted that Westinghouse was already conducting inhouse research and development programs on various aspects of such a system, and some of this work was used in the EB system delivered to NASA.

#### MICROWAVE SYSTEMS (09)

Microwaves, which are very short radio waves between 0.03937-inch and 12.00000-inch long, first came into widespread public notice through World War II application in radar. The frequency range of these wavelengths falls between the UHF (ultrahigh frequency) used for television, and the IF (infrared) light bands. Microwaves have characteristics similar to light in that they travel in straight lines, can be reflected, and can be focussed and directed in a beam. They have advantages over and above those of light in that they pass easily through rain, smoke, fog, and materials such as plastics and wood. These characteristics make microwaves attractive for applications such as long distance, line-of-sight communications and control of navigation.

Until quite recently, the most active users of microwave systems have been the military, aerospace, the telegraph and telephone industry. Increasing availability of microwave semiconductor devices, improved fabrication techniques, and most important, declining military markets, have prompted a revolution in engineering and marketing attitudes and a marked increase in efforts to apply microwave technology to commercial and consumer needs. The major new areas now being considered and explored are:

- Industrial heating -- the directive properties of microwaves can be used for selective heating such as glueing wood products. Industrial heating sales are expected to exceed \$100 million by 1975.\*
- 2. Civil aviation -- microwave systems are being used to overcome the air traffic control crisis, for instrument landing systems, for enroute identification, surveillance, and automated processing systems, and for visible displays of runways in zero visibility landings. The Department of Transportation alone plans to spend between \$600 and \$800 million annually over the next ten years to develop the systems needed, and anticipates a total of \$2 billion in sales in new electronic equipment in this industry.\*\*
- 3. High volume data, video, and voice communications -- these applications will depend on millimeter-wave equipment to overcome the

<sup>\*&</sup>quot;Putting Power Where It Counts Economically," Microwaves, September 1970, p.44.

<sup>\*\*&</sup>quot;Air Traffic Demanding More Accurate Control," Microwaves, September 1970, p. 48.

communications log jam that is developing. The data communications market alone, exclusive of terminals, is estimated to exceed \$2 billion by 1975, with microwave relays playing the most significant part in the transmission of computer traffic.\*

- 4. CATV -- community antenna television represents an industry with unusually large potential which is expected to open up new markets for short-haul microwave equipment, the size of which is as yet unpredictable.\*\*
- 5. Laser systems -- these systems can be used for welding of miniature parts, cutting and aligning. For example, laser systems can be used for cutting or chilling hard materials such as diamonds. Again, the market potential is unpredictable but it appears unusually large.\*\*\*
- 6. Low-cost radar -- radar systems costing in the order of \$800 per unit are expected to be produced. The uses of such low-cost items are also unpredictable, but appear significant. For example, such devices could be used to apply car brakes automatically, or in police traffic control work.\*\*\*\*

The growing microwave systems field appears to have been most significantly affected by advancements in the microwave semiconductor industry. The most significant developments in the microwave systems field and those having the greatest impact include, among others: the development of high-power, high-frequency transistors; the development of small-signal transistors; the use of hybrid and monolithic processes to produce low-cost microelectronic devices for microwave systems; the development of a variety of high-power microwave diodes; and the discovery of semiconductor laser diodes. In addition to general advancements in the semiconductor field, two other technological developments have had an impact on this relatively new field. The first of these

<sup>\*&</sup>quot;Relief from Telephone and Spectrum Jams," Microwaves, September 1970, p. 56.

<sup>\*\*</sup>Ibid.

<sup>\*\*\*&</sup>quot;Light to Drill, Cut, and Communicate," Microwaves, September 1970. p. 52.

<sup>\*\*\*\*&</sup>quot;Doppler to Stop a Car and Catch a Thief," Microwaves, September 1970, p. 60.

was the development of carbon dioxide lasers to provide high-power, choerent, light sources. This development overcomes the inability of semiconductor coherent light sources to fully exploit the advantages of lasers for medical research, industrial processing, communications and optical data processing. The second development is an outgrowth of the need for low-noise receiving systems in interplanetary tracking and astronomical radio research, which led to ultimate improvements in maser communications systems and calibrations. A list of significant developments, as identified by technological leaders in the field of microwave systems, is given in Table 9. Selected significant developments, and NASA contributions to the advancement of these developments, follow.

### Carbon Dioxide Laser (09-02)

Laser devices use the principle of amplification of electromagnetic waves by stimulated emission of radiation in the infrared and optical regions. "Laser" is an acronym for "Light Amplification by Stimulated Emission of Radiation." It is not practical to construct amplifiers or oscillators by scaling down conventional electronic devices, for these short wavelengths (from 1 x  $10^{-1}$  to 4.0 x  $10^{-5}$  cm or less). However, atoms, ions, molecules, and crystals have resonances throughout this region. These molecular resonances can be used as generators of coherent radiation in the infrared, visible, and ultraviolet regions.

All conventional light sources are basically hot bodies. Once excited, they give up this excitation energy by the spontaneous radiation of light. This spontaneous emission from each excited atom or electron takes place independent of the emission from the others. Thus, the overall phase of the emitted light fluctuates randomly from moment to moment and from point to point on the wave front. That is, the light from conventional sources is incoherent.

Coherent light arises when the atoms are stimulated to emit radiation which is in-phase with the wave, rather than independently. The output power is substantially increased because the atoms can be stimulated to emit much faster than they would spontaneously. Coherent light is very nearly monochromatic, because stimulated emission is a resonance process, which occurs most rapidly at the center of the range of wavelengths that would be emitted spontaneously. Since the atoms are stimulated to emit in phase with the existing wave, the phase is preserved over many cycles. Thus, the laser radiation has a high degree of time coherence.

Gas lasers make use of nonequilibrium processes in a gas discharge, and laser action can occur at many wavelengths in any of a large number of gases under suitable discharge conditions. Gas lasers have been developed which are almost totally monochromatic, have wavelength stability, and narrowness of beam. Carbon dioxide is one of the gases which has been an effective source of coherent infrared light.

#### TABLE 9

# SIGNIFICANT DEVELOPMENTS IN THE FIELD OF MICROWAVE SYSTEMS (09)\*

- 1. Better understanding of ultrasonic waves on semiconductors (09-01)
- \*\*2. Carbon dioxide laser (09-02)
  - 3. Computer-mided design (09-03)
  - 4. Broad-band sweep generators (09-04)
- \*\*5. Gunn effect devices (09-05)
- \*\*6. High-frequency power transistors (09-06)
  - 7. Yttrium-iron-garnate filters (09-07)
  - 8. Electron beam bombarded semiconductors (09-08)
  - 9. Far infrared lasers (09-09)
- 10. Hybrid junction devices (09-10)
- \*\*11. IMPATT diode (09-11)
- \*\*12. Improvements in maser systems (09-12)
  - 13. Improved traveling wave tubes (09-13)
  - 14. Increased accuracy in microwave measurements (09-14)
  - 15. Increased reliability of maser systems (09-15)
  - 16. Laser mode locking (09-16)
- \*\*17. Microwave integrated circuits (09-17)
- \*\*18. Optical data processing (09-18)
- \*\*19. Semiconductor lasers (09-19)
- \*\*20. Small signal microwave transistors (09-20)
- \*\*21. TRAPATT diode (09-21)
  - 22. Tunable lasers (09-22)

<sup>\*</sup>As identified by technological leaders in the field of microwave systems.

<sup>\*\*</sup>Developments selected for identification of NASA contributions.

The carbon dioxide laser was proposed by Charles Townes, the inventor of the laser, and first demonstrated at Bell Labs in 1961. Within the past five years, high-power carbon dioxide lasers have been developed by increasing the flow of the gas and replacing electrical excitation with chemical excitation.

Bell Labs, Hughes Aircraft, Raytheon, United Aircraft, Sylvania, DOD, and NASA have been primarily responsible for developments and advancements in carbon dioxide laser technology.

The carbon dioxide laser is expected to find future applications in medical research, industrial processing, communications, optical data processing and display systems. Carbon dioxide lasers are already being used in medical research; for cutting of nonmetals and plastics; and in the microelectronics industry for the high-speed trimming of thick-film resistors. Major factors which have created the laser technology explosion and its growth in commercial applications have been the growing availability of more powerful, rugged, and reliable lasers, coupled with a better understanding and control of laser energy.

The concept of laser communications has interested telephone companies and others in the communications industry, and research is underway to develop laser communications networks. Since at least 2.5 million voice channels could be carried on a single link, a laser telephone link would offer 100 times the capacity of any present method of transmission.

Carbon dioxide laser communication systems are an attractive alternative to microwave and millimeter wave systems for a number of space applications, specifically for synchronous satellite-to ground communications, low-altitude satellite-to-synchronous satellite communications, synchronous satellite-to-synchronous satellite communications, and Mars probe communication to either a ground station or a synchronous satellite. This potential has prompted a significant effort by NASA/GSFC, with NASA funding from 1964 to date between \$10-20 million. All of the major companies involved in carbon dioxide laser development and improvement have been conducting research with NASA funding including, Sylvania, Airborne Instruments Lab, Aerojet-General Corporation, Hughes Aircraft, Honeywell, etc.

NASA contributions to the advancement of the technology of carbon dioxide lasers include the following, among others.

o Developed the first space-qualified lasers (09-02-01).

NASA had two primary concerns with regard to carbon dioxide lasers for space applications: sealed-off lifetime and amplitude and frequency stability of the output beam. Design operating lifetime goals of 10,000 hours and a shelf life of three years have been achieved. Carbon dioxide lasers

with output power in excess of 1 watt and frequency stability of one part in 10<sup>10</sup> have been produced.

o Advanced basic understanding of carbon dioxide lasers and developed the technological base for carbon dioxide laser advances (09-02-02).

Since the discovery of carbon dioxide lasers in 1964, NASA has provided more than \$10 million of research funds in an effort to exploit carbon dioxide lasers for deep-space communications systems. In 1965, an in-house program to demonstrate the feasibility of such systems was initiated at NASA/GSFC followed late that same year by a contract with American Instrumentation Laboratories, Inc. (AIL) to develop a gigahertz bandwidth infrared receiver. Early in 1966, NASA funded Sylvania to develop a stabilized carbon dioxide laser and also to develop a ground-based communications system. These efforts continued into 1967. In 1968, NASA/ERC issued contracts to Sylvania for a space-qualified carbon dioxide laser and the first synchronous-to-synchronous satellite laser communications system. NASA/MSFC issued contracts to Honeywell for a carbon dioxide laser communications system. In 1969, Aerojet-General Corporation, received a contract for a laser communications system for the ATS-F satellite. Hughes Aircraft received the contract for the ATS-G laser experiment in June of 1971. The major immediate interests in the NASA effort will be for communications between synchronous satellite-to-synchronous satellite, low-altitude satellite-to-synchronous satellite, lunar-orbit-to-ground, and lunar-colonyto-ground. Other activities will be applying the NASA work to specific programs such as for the data relay satellite, the earth resources technology satellite, DOD, and COMSAT. Although the major effort has been space-related, NASA efforts have significantly enhanced the advancements in carbon dioxide laser technology.

o Defined the emission frequencies of carbon dioxide lasers (09-02-03).

Research efforts by NASA to develop information about the basic characteristics of carbon dioxide lasers (see 09-02-02 above) led to a definition of emission frequencies and demonstrated that individual lasers had characteristic emission patterns or "signatures."

o Aided in making research results and literature on carbon dioxide lasers readily available (09-02-04).

In order to "reduce considerably the time required to search the literature on carbon dioxide lasers or to find the value of a particular parameter," NASA/GSFC performed a literature search on carbon dioxide lasers and made this search available through twenty-three scientific and technical journals and the NASA Scientific and Technical Aerospace Reports (STAR) in two volumes. The first, "A Guide to the Literature on Carbon Dioxide Lasers (1 January 1964 - 30 June 1968)" was published in November 1968; the second covering a period between 1 July 1968 - 30 June 1969 was published in February 1970. To date NASA has received over one thousand requests for copies of these documents.

#### Development of Gunn Effect Devices (09-05)

In 1963 it was discovered that a small cube of N-type gallium-arsenide could generate microwave energy when a low dc voltage is applied across it. This discovery was made by Dr. J.B. Gunn of IBM and devices exhibiting this effect are called "Gunn effect devices." Such devices created considerable interest at the time because it then seemed that conventional transistors could probably not deliver useful power levels in the microwave region, above 1 gigahertz. Since then, however, transistors have been developed which will operate at frequencies an order of magnitude higher.

The bulk negative resistivity exhibited by gallium arsenide, which is responsible for the effect that Gunn observed, is obtained by transferring electrons heated by high electric fields from high-mobility to low-mobility conduction subbands. For N-type gallium-arsenide, the differential drift velocity of conduction electrons is positive for electric fields less than about 3 kilovolts/cm. and is negative for electric fields above that level. When the proper conditions are satisfied, traveling spacecharge dipole layers form in the device, and its steady state is oscillatory. The fundamental frequency of operation is approximately equal to the transit-time frequency. The above mode of operation has prompted the generic name of such devices "transferred electron oscillator," or TEO. Such devices have also been used in an amplification mode and when used as such are called "transferred electron amplifiers," or TEAS.

The development of bulk effect devices has resulted in reliable, inexpensive sources of microwave power, and are actively being used in aircraft altimeters, mobile-vehicle and man-pack radars and transponders.

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Low-power doppler radar systems offer unique marketing opportunities with relatively little capital investment. The key to developing these radars for commercial use is the solid state source. Gunn effect devices are hot contenders for this activity.

In 1968, NASA/LaRC funded a program at RCA to explore the potential of Gunn effect devices for transponders to be used in the L-band region. This contract effort, initiated RCA's efforts in this new technology. From this effort much new knowledge was obtained about III/V compounds, resulting in several RCA patents and new products for commercial exploitation. Specifically, NASA contributions to the advancement of the technology of Gunn effect devices include the following among others.

o Advanced the understanding of Gunn effect devices (09-05-01).

NASA/LaRC initiated a program at RCA in 1967 to exploit the properties of Gunn effect devices for L-band transponder applications. In conjunction with this effort, basic research was conducted on growth systems for growing III/V compound crystals and epitaxial layers. New knowledge about III/V compounds was added to the existing bank of technology, most important of which was the effects of varying phosphorous concentrations on the threshold of Gunn effect devices. In June 1971 these investigations were extended into the s-band region to acquire additional information on gallium-arsenide and phosphorous epilayers. In the event Gunn effect devices find widespread application, the NASA effort will probably have a moderate effect in accelerating the rate at which the devices became useful.

o Aided in the development of a competitive marketplace for Gunn effect devices (09-05-02).

Prior to the NASA-supported effort on Gunn effect devices (see NASA contribution 09-05-01 above) RCA was not actively involved in producing Gunn effect devices. The NASA requirement stimulated RCA to enter the market and become a supplier of such devices sooner than they would have on their own, with the net result increasing competition in the field by increasing the number of suppliers.

### Development of the High-Frequency Power Transistor (09-06)

Advances in the thin-film and monolithic integrated circuit technologies over the past ten years have lead to the development of transistors with output powers in the 20 to 40-watt range which can operate in the high-frequency (HF) and ultra-high-frequency (UHF) ranges. Factors that have contributed to the advancement of this development include, among others: Improvements in mask generation; improvements in the photolithography process which have resulted in smaller geometries; the use of tungstun in place of aluminum as the metallization material to minimize metal ion propagation; improved techniques for removing the heat from the device; and new packaging concepts. Ion-implantation techniques (see NASA contribution 09-17-01) and electron beam lithography offer the potential of further improvements in the performance of such devices in the future.

High-power microwave transistors generally consist of many medium-power transistors in parallel with positive temperature-coefficient nichrome film resistors placed on the surface in series with each of the emitters of the individual medium-power transistors. The resistors regulate the current in the medium-power transistors and prevent secondary breakdown in these devices.

To date it has been possible to achieve operating frequencies up to 100 megahertz in 20 to 40-watt devices and up to 3 gigahertz in 5-watt devices.

The use of transistors in microwave communications had been limited because, until recently, transistors were unable to operate effectively at microwave frequencies. Now high-performance transistors are used in microwave communications systems for all applications where amplification is required below about 1 gigahertz. The ability to provide substantial gain with relatively low-power consumption makes the microwave power transistor the most desirable amplification device for microwave repeaters and for FM terminals.

Availability of such solid state devices has resulted in the introduction of a new line of microwave equipment, superseding older vacuumtube versions, and providing improved performance, increased reliability, smaller size, lower costs and reduced power consumption. The single remaining tube, a traveling-wave tube, is also being threatened by solid state replacements. Once this has been achieved microwave communications systems used for long-distance telephone communications, and data transmission will be entirely solid state.

Operating revenues from telephone and telegraph, radio and television broadcasting, and other communications services are expected to exceed \$28.7 billion in 1971. Revenues from international telephone and telegraph services, the fastest growing segment of the communications industry, are expected to total \$664 million in 1971.\* The new segment of the communications industry, data communications, in which microwave relays will play the major transmission role, is predicted to exceed \$1 billion by 1971.\*\*

NASA has played a major role in the development of high-frequency power transistors. A NASA requirement for a transmitter with 20 watts output to operate in the 2 gigahertz frequency range for satellite communications applications led to a NASA-funded program at RCA in 1963 to develop a high-performance, high-frequency spectrum. From this effort RCA was able to develop improved processing techniques and to generate new information about solid state materials.

O Developed the most advanced high-frequency power transistor (09-06-01).

In order to develop a satellite communications transmitter, NASA/GSFC required a transistor with an operating frequency capability of 2 gigahertz and an output power of 20 watts. Until this requirement, it was possible to fabricate devices which would operate up to 400 GHz. NASA contracted with RCA to fill this need, resulting in the development of an RCA line of commercial devices for microwave applications. The NASA contribution advanced the time when such devices would have been commercially available through the normal evolutionary cycle and the impact of the NASA contribution is estimated to be high.

• Advanced the manufacturing technology for high-frequency power transistors (09-06-02).

In a program to develop a high-frequency power transisistor for NASA/GSFC (see NASA contribution 09-06-01 above) RCA had to make significant improvements in their processing techniques. Obtaining finer lines and better control of the spacing between the lines in their monolithic chips was a prerequisite to increased power density. To do this, the problem of electrical breakdown between circuit elements, resulting in decreasing the device geometry to increase power-density and frequency-response, had to be corrected by improving oxide coatings and coating procedures.

<sup>\*&</sup>quot;Electronic Equipment and Components." <u>U.S. Industrial Outlook</u> <u>1971</u>. Washington, D.C.: U.S. Department of Commerce, Bureau of Domestic Commerce, 1971, p. 298.

<sup>\*\*&</sup>quot;U.S. Markets 1971 Forecast," <u>Electronics</u>, January 4, 1971, p. 44.

Since these improvements in oxide coatings, coating procedures, and other manufacturing techniques, were necessary in order to produce the high-frequency power transistors described above, the impact of this NASA contribution is estimated to be quite high.

## Improvements in Maser Systems (09-12)

The "maser," an acronym for "microwave amplification by stimulated emission of radiation," is a device which can amplify or generate electromagnetic waves by use of the excitation energy in resonant atomic or molecular systems. Eelctromagnetic waves can stimulate an unstable ensemble of atomic or molecular particles to radiate excess energy at the same frequency and phase as the stimulating wave, thus providing coherent amplification. This type of amplification is not limited to the microwave region: amplifiers and oscillators using maser mechanisms have also been produced to operate in both the audio and optical range of the frequency spectrum.

Maser amplifiers and oscillators are inherently low-noise devices which make them particularly useful for the detection and reception of very weak signals typically found in radio astromony, microwave radio-metry, long-distance radar, and long-distance microwave communications. Such low-noise receiving systems have been essential in the interplane-tary spacecraft tracking and radio research activities of NASA.

Continuous efforts to improve the component parts of maser systems and calibration techniques have increased the applications of this communications technology.

To date, the major use for maser technology has been for space applications and the primary impact of this technology has been in such areas as radio astronomy specifically: absolute flux density calibration of radio stars; measurements of atmospheric attenuation; and the absolute of cosmic background noise. The commercial impact of maser systems, to date, have been minimal.

The NASA need for a deep-space tracking and communications network prompted the expenditure of substantial amounts of funds to aid in the development of maser technology. Techniques developed at JPL, for example, make it possible to achieve microwave insertion loss calibration accuracies of 0.25 percent. This type of contribution, of precisely calibrated microwave thermal noise standards, has contributed greatly to the improvement in system calibration accuracies. Included among NASA efforts to improve maser systems are the following contributions, among others.

o Developed a technique for determining the input noise temperature of low-noise reflection-type amplifiers (09-12-01).

Demands of the Venus probe program for improved communications systems prompted NASA/LaRC in 1961 to initiate in-house as well as contract development programs with the Micromega Corporation. Objective of these programs was to improve the output power at low-noise levels of reflection-type amplifiers such as masers, parametric amplifiers, and tunnel device amplifiers. Reflection-type amplifiers, however. have reached a state of such low excess noise levels that input noise temperature determination is extremely inaccurate if the influencing effects of the network elements before and after the amplifier under test are not taken into account. NASA/LaRC developed a method for determining all effects which significantly influence measurement of the output-noisepower ratio. In addition, this method can be used to estimate the maximum error propagation which occurs in the derivation of the input noise temperature. This NASA effort was significant in advancing the development of maser systems. Since maser systems are not yet a significant factor in the private sector of the economy, the overall impact of this effort is small.

o Advanced the development of a display system to analyze negative resistance amplifiers (09-12-02).

Adjusting masers, and other reflection-type amplifiers (negative resistance amplifiers) for optimum operation is normally very tedious and time consuming. In 1967, NASA/LaRC developed a method of extending the conventional Smith chart into the negative resistance and admittance region by using the vector concept of the transmission and reflection coefficient. This effort has made it possible to now display the negative impedances or admittances on an oscilliscope for a direct measurement. A NASA award was made for this effort and a NASA patent is pending. Based upon this contribution, Hewlett-Packard has developed a commercial display system for analysing negative resistance amplifiers.

#### Microwave Integrated Circuits (09-17)

The need for increased reliability, versatility, small size, and low overall system costs, have prompted efforts to adapt thick-film

and monolithic microelectronic technologies to meet the needs of solid state microwave technology. Within the past five years a limited number of microwave circuits have been developed. The majority of microwave integrated circuits are fabricated by using thick-film and thin-film hybrid techniques. Conduction paths, mounting pads, and resistors are deposited on a ceramic substrate; active semiconductor chips and passive components are then added by thermomechanical bonding techniques.

There are basically three different monolithic approaches for fabricating microwave integrated circuits: silicon dielectric, which use the silicon substrate as the microstrip dielectric and the material for the active devices; dielectric on silicon, which uses the deposited material as the microstrip dielectric and a conventional silicon wafer for the active devices; and silicon on sapphire, which uses an epitaxially grown layer of silicon for the active devices and a sapphire substrate for the microstrip dielectric.

Microwave integrated circuits have been developed which can generate up to 10 watts in the 1 to 10 gigahertz range of the frequency spectrum. The major developers are Texas Instruments, Bell Labs, Hewlett-Packard, RCA, Microwave Associates, Watkins-Johnson, and Motorola.

The development of microwave integrated circuits technology brings to the microwave field the well-known advantages of existing low-frequency integrated circuitry such as lower cost, reduced size and weight, increased ruggedness and reliability, greatly simplified and reduced geometries and precise tolerance control. These factors are most dramatic because microwave hardware in the past has been characterized by massive costly assemblies of precision-machined, brass-block components, connected together with either wave guides or coaxial cables, and complex electric field problems which dictated empirical rather than theoretical design approaches with resulting long development times. Microwave integrated circuits lend themselves readily to theoretical designs which eliminate the need for expensive cut-and-try design procedures.

Microwave systems have traditionally been very expensive items. Microwave integrated circuits will enable these systems to be produced at reduced prices and increase their usage for existing applications. In addition, new applications will be considered which were previously considered impractical because of cost. The major impact of microwave integrated circuits will be in applications in inexpensive local radar systems, microwave long-distance telephone repeaters, and in rapidly expanding computer data-link systems.

NASA requirements for space communications initiated several programs to develop solid state microwave components. In efforts to develop microwave frequency transistors, improvements were made in photomask and coating technology, and programs are presently underway to develop microwave integrated circuits for S-band communications systems. The use of ion-implantation for reducing the base region in microwave transistors has demonstrated that this technique can be effectively applied to

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the production of microwave integrated circuits. In addition to programs directly related to microwave integrated circuits, NASA also has applied integrated circuits concepts to overcome microwave problems. For example, a technique was developed by NASA to electronically focus microwave antennae using standard integrated circuits. Among the specific NASA contributions to the use and advancement of microwave integrated circuits are the following.

o Demonstrated the use of ion-implantation techniques to produce microwave integrated circuits (09-17-01).

The use of ion-implantation techniques by NASA to improve the control of base thickness, thereby increasing the frequency response of n-p-n transistors, demonstrated that these techniques are applicable to microwave integrated circuits. These efforts at NASA/LaRC and under contract at Hughes Aircraft, the leader in the use of ion-implantation methods for doping semiconductor devices, advanced the time when such techniques would find widespread use and therefore has had a moderate impact on the advancement of the entire development of microwave integrated circuits. (See the development of ion-implantation in integrated circuits, field 05, NASA contribution 05-10-02.)

o Developed a high-gain, 1.5-watt power amplifier for S-band transmitters (09-17-02).

NASA/GSFC funded programs at RCA and Spectrum Microwave, Inc. to improve processing techniques and apply microwave integrated circuits technology to S-band systems. Result was the development of a high-gain, 1.5-watt power amplifier. These efforts improved RCA's processing techniques and helped to add a new product to Spectrum's line of products. Present users of this amplifier are the Air Force and the Navy, and it appears to be of interest to COMSAT.

o Developed an integrated circuit phase-shifter to electronically focus microwave antennae (09-17-03).

Prior to a development program at NASA/GSFC, which demonstrated the use of integrated circuits to improve the design of microwave antennae, microwave antennae had to be focussed mechanically. In space applications, narrow-band microwave antennae are difficult to focus because of the mechanical overshoot and lag time of the servo-motors. This was corrected by the development of an integrated circuit phase-shifter to electronically focus the microwave antenna. The system has found use in

satellite-to-earth communications, and has also been used for the radar mapping of Venus. This contribution has the potential for replacing mechanically driven radar antennae, leading to potentially high economic significance.

#### Semiconductor Lasers (09-19)

Semiconductor lasers, developed in 1962, are devices which operate by using stimulated emissions to produce very intense monochromatic coherent light beams. When minority carriers recombine with majority carriers in a semiconductor, energy is released in the form of light, heat, or kinetic energy to other carriers. Although minority carriers can be introduced in several ways, the most effective way is carrier injection in a pn junction, the basis for semiconductor light sources.

Any pn junction, under forward bias, can emit some light, but before reasonable conversion efficiencies can be obtained, a great many factors must be optimized. For instance, light output can be drastically reduced by competing nonradiative recombination processes, as well as by internal absorption and reflection losses. These losses can be minimized by choosing the right material with a high degree of purity and crystal perfection, which has been properly doped, and by using geometrical structures and other methods to reduce absorption and reflection losses.

Single crystals of gallium arsenide and mixed compounds such as Ga(AsP) and GaAlAs, are some of the materials which have been selected as a promising material because of their high probability of light emission and ability to be tailored to a desired wavelength by varying composition. A variety of other III/V and II/VI compounds are being continually investigated to increase emission efficiencies in different color bands.

Because light generation takes place almost entirely in the p-region and is of a slightly longer wavelength than the absorption edge of n-type material, thin, flat sections of GaAs will emit light through the n-region without excessive absorption. Hemispherical domes, placed over the junction, can eliminate the internal reflection, and thereby increase the light output by a factor of 25.

A very significant market is developing for light-emitting semiconductor devices in the area of visual displays from simple numerical readouts, as in desk-top calculators, to highly-sophisticated, multi-color displays, with thousands of light-emitting diodes of different colors, placed side by side in arrays and excited by many sets of lines driven by integrated circuits. This approach could lead to the development of the much talked about picture-frame TV receiver.

NASA/LaRC has been managing programs, which were begun at NASA/ERC, to investigate the possibilities of developing green light (5150 Angstroms)

emitting injection semiconductor laser devices by attempting to increase the mobility by p-type carrier injection. They have been investigating both III/V compounds, such as gallium-arsenide, and II/VI compounds, such as cadmium-sulfide and zinc-selinides. One of the major difficulties has been to accurately locate the p-type carriers. It appears that ion-implantation offers the best approach to overcome this problem. These NASA contributions are described as follows.

o Advanced the basic understanding of III/V and II/VI compounds for use in semiconductor lasers (09-19-01).

In an effort to develop effective green light (5150 Angstroms) emitting injection semiconductor laser devices NASA/LaRC has funded research to find ways to increase the mobility by p-type carrier injection. In these programs they have investigated the properties of both III/V compounds such as gallium-arsenide and II/VI compounds such as cadmium sulphide and zinc selinide. Numerous other organizations have been conducting parallel work in similar areas.

o Demonstrated use of ion-implantation methods to increase control of p-type carrier locations (09-19-02).

Ion-implantation approaches to more accurately control the location of p-type carriers were investigated in conjunction with NASA efforts to develop semiconductor laser devices (see NASA contribution 09-19-01 above), and were found to be extremely effective. While ion-implantation techniques were already in use for other types of devices, the NASA effort demonstrated the power and effectiveness of the technique in another area of concern.

#### Small Signal Microwave Transistors (09-20)

Small-signal microwave transistors, which have been developed over the past five years, are capable of operating at frequencies up to 12 gigahertz (GHz). The development of these transistors is an outgrowth of advancements in a number of already existing technological developments, including: photomask making, photolithography, epitaxial growth processes, diffusion processes, thermal dissipation, and packaging. All of the business firms involved in developing solid state microwave devices have been involved in developing small signal microwave transistors.

Microwave semiconductor components, such as microwave transistors, make possible microwave systems with increased reliability, decreased size and lower costs. Prior to the development of these devices, microwave equipment depended on complicated, expensive-to-manufacture, and not too reliable vacuum-tube components which consumed substantial

amounts of power. The advantages of reliability, size and cost of microwave transistors make microwave systems attractive in many new applications.

While NASA has not contributed significantly to the actual development of microwave transistors, the reliability of such devices has been advanced by NASA-funded programs, as has manufacturing techniques through a demonstration of the use of the ion-implantation techniques.

o Demonstrated the use of ion-implantation techniques to produce small-signal microwave transistors (09-20-01).

In 1970, LaRC initiated a program at Hughes Aircraft, the leaders in the use of ion-implantation techniques, to investigate the use of the ion-implantation method to improve the frequency response of gallium-arsenide microwave transistors (see NASA contributions to microwave integrated circuits, development 09-17). Through this program, NASA demonstrated the feasibility of reducing the base region to 1,000 angstroms to produce npn Ga/As transistors which will operate in the 20 GHz region of the frequency spectrum. Although these devices have not yet found commercial application, it appears that they should find widespread use in the microwave communications industry in the near future and the impact of the NASA contribution should therefore be moderate.

#### TRAPATT Diode (09-21)

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When an IMPATT diode (a semiconductor pn junction device, in a microwave cavity and driven into avalanche to generate microwave power) is installed in a resonant cavity whose resonant frequency is approximately one-half, or less, the frequency at which the device would normally operate, very high-peak powers and efficiencies can be obtained. Such devices when operated as indicated are called TRAPATT diodes (an acronym for "trapped plasma avalanche triggered transit"). These devices are also called "anomalous mode," "avalanche diodes," or "high efficiency mode" diodes. Experimental devices have been operated at ultra-high frequencies (UHF) with peak powers of 350 watts.

A number of companies are now offering off-the-shelf delivery of IMPATT devices which can be used to operate in the anomalous (TRAPATT) mode, including RCA, Monsanto, Sperry Microwave, RAytheon, and Hughes Aircraft, both in the evolutionary cycle to determine the impact of such devices. It is anticipated, however, that such devices will find application in transponders and low-power local radar.

Since such devices are quite new, NASA has been interested in staying abreast of the state of technology of the development, and has advanced understanding of TRAPATT diode characteristics.

o Advanced understanding of TRAPATT diode characteristics through development of computer models of avalanche oscillation modes (09-21-01).

A series of computer models of the various modes of oscillation of avalanche diodes which has added to the understanding of the characteristics of TRAPATT diodes have been developed at MIT under NASA/LaRC sponsorship. This increased understanding should enhance the practical application of such devices.

#### NONDESTRUCTIVE TESTING (10)

Although nondestructive testing (NDT) began in the United States fifty years ago with the installation of the first radiographic laboratory at the Watertown Arsenal, it has remained solely a quality control technique until recently. Within the past decade, however, rapid advances in materials technology and increasingly stringent aerospace and commercial specifications, have moved NDT out of its singular role of quality control, into a new and still unexplored role as a diagnostic and predictive tool to be used in all phases of design, development, test and field evaluation. The potential inherent in NDT techniques has stimulated a high level recommendation by the National Materials Advisory Board (NMAB) to encourage the transformation of qualitative NDT technology to the quantitative science of nondestructive evaluation (NDE). The objective of this recommendation and subsequent research is to:

"...initiate a long-range interdisciplinary research program to insure that all possible energy-material interaction phenomena are recognized for their potential to nondestructive evaluation, e.g., to screen and qualify candidate materials to meet stringent design criteria and to monitor production processes and service performance."\*

To test and evaluate materials, engineers have historically relied upon (1) simple qualitative nondestructive testing; (2) destructive testing to analyze the physical properties of a representative test sample, destroying the part in the process, or (3) forensic analysis to predict performance. For many pre-space-age applications, these standard approaches were satisfactory. But the greatly increased performance requirements of today's aerospace, commercial, and research systems, particularly those which are man-rated, demand corresponding advances in analytical techniques to assess current and projected performance characteristics without destroying elaborately expensive hardware.

The application of physical properties for the analysis of material properties without impairing the properties is termed nondestructive testing (NDT). The virtually concomittant step of evaluating the material thus analyzed is termed nondestructive evaluation (NDE).

<sup>\*</sup>National Research Council, National Academy of Sciences - National Academy of Engineering, Nondestructive Evaluation, A Report of the National Materials Advisory Board, Washington: June, 1969 (NMAB-252).

Engineering materials start with imperfections, and in their subsequent manufacturing or process development into useable components and systems, they accumulate cracks, flaws, inclusions, surface and body strains, impurities and other deficiencies. The testing problem, therefore, is one of detecting, analyzing and then evaluating these imperfections with respect to the function, i.e., how much imperfection is acceptable?

Many of the sensitive techniques and tools employed in NDT can detect imperfections far beyond the ranges of concern, so the principal difficulty is to scientifically evaluate the significance of the data. Considerable materials research is now in progress, therefore, to compile this basic data from which specifications and tolerances can be developed.

NDT/NDE tools and techniques are employed in flaw and crack detection; process monitoring and improvement; measurement of physical, mechanical and metallurgical properties; and in various control systems. The purpose of NDT/NDE is to identify and assess the present condition of a material and to evaluate its life expectancy or potential performance, and from these operations accept, reject or rework the material or object.

Virtually every commonly accessible form of energy is employed in the main NDT methods, which include, among others: sonic and ultrasonic acoustics; holography; neutron, x- and gamma ray penetrating radiography; eddy currents; visual fluid penetrants; magnetic leakage fields; pressure and leak detection; and thermal testing. Recent advances in the discipline include acoustic emission, computer enhancement of radiographs; acoustic holography; excelectron emission; and real-time x-ray and neutron imaging. Ultrasonic and radiographic methods are used for in-depth analysis; while holography, eddy current, magnetic particle and thermal methods are used for the detection and identification of near surface anomolies. A complete listing of significant developments in NDT, as identified by technological leaders in the field, is given in Table 10.

The rapidly advancing technological base of the field, coupled with the increasing testing and evaluation needs of government and industry, has resulted in a rapidly growing market for NDT equipment and supplies. Sales volume in 1969 is estimated to have been \$85 million, and the average annual growth rate is expected to exceed 10 percent per year. By 1980, the market for NDT supplies and equipment is expected to reach a level of \$250 million.\*

<sup>\*</sup>Zubrick, John R., "Nondestructive Testing," McGraw-Hill Yearbook of Science and Technology, New York: McGraw-Hill, 1971, pp. 34-35.

NASA in-house and contractor facilities are, of course, major consumers of NDT/NDE state-of-the-art tools and techniques. Where an NDT need existed for which existing services or knowledge was inadequate, NASA and space program contractors have advanced the state of technology in the field by stimulating NDT equipment manufacturers to meet these needs, or by developing and producing the required articles or data themselves. Some of these efforts include delta scanning, axial transverse laminography, computer enhancement of radiographs, and the solid state radiographic image amplifier, among many other systems. The space program has also served as a principal developer and compiler of NDT data and instruction materials.

Selected significant developments in the field of nondestructive testing (as listed in Table 10) and NASA contributions to the advancement of these developments, follow.

### Acoustic Emission (10-01)

When materials are deformed, they emit high-frequency acoustical noise. When properly evaluated, these noises can detect flaws in materials, such as porosity, inclusions, incomplete weld fusion, and stress corrosion cracking. Efforts are presently underway to develop techniques using acoustic emission to predict or detect the onset of failure, as for example, monitoring the structural members of in-flight aircraft.

H. L. Dunegan is credited with developing the initial laboratory instrumentation (1964) to identify signals of interest out of undifferentiated background noise, making acoustic emission detection a practical reality. Since the stress waves given off by deformed materials can provide information on impending failure and fracture mechanics behavior, acoustic emission has considerable potential for the monitoring of crack initiation and growth in in-flight aircraft, nuclear reactors, and pressure vessels, as well as for the monitoring of welds during cool-down.

NASA's efforts to develop fundamental state-of-the-art data and devices in acoustic emission, starting in 1964, contributed importantly to subsequent developments in the field. The actual and potential technological impact of these contributions is estimated to be moderate. These contributions include the following among others.

o Development of an ultrasonic (acoustic) emission detector for evaluation of the strength of bonded materials (10-01-01).

While new applications of composite structures were rapidly accelerating within NASA, NDT equipment which could adequately

#### TABLE 10

#### SIGNIFICANT DEVELOPMENTS IN NONDESTRUCTIVE TESTING (10)\*

- \*\*1. Acoustic emission (10-01)
  - 2. Color radiography (10-02)
- \*\*3. Computer enhanced radiography (10-03)
- \*\*4. Delta scanning (10-04)
- \*\*5. Real-time x-ray imaging (10-05)
  - 6. Eddy current techniques (10-06)
  - 7. Exoelectron emission (10-07)
- \*\*8. Holography/interferometry (10-08)
  - 9. Liquid crystals (10-09)
- \*\*10. Neutron radiography (10-10)
- \*\*11. Nondestructive testing/nondestructive evaluation data improvement (10-11)
  - 12. Piezoelectric generation of x-rays (10-12)
- 13. Proton radiography (10-13)
- \*\*14. Solid-state radiographic image amplifier (10-14)
- 15. Stray magnetic flux perturbation (10-15)
- \*\*16. Thermal nondestructive testing techniques (10-16)
  - 17. Ultrasonic imaging (10-17)
  - 18. Ultrasonic techniques (10-18)
  - 19. Water wash fluorescent penetrant (10-19)

<sup>\*</sup>As identified by technological leaders in the field of non-destructive testing.

<sup>\*\*</sup>Developments selected for identification of NASA contributions.

evaluate bond strength did not exist. To fill this need, a contract was awarded to the General American Transportation Corporation (June 1964) to develop NDT devices and techniques that could be used to evaluate adhesion bond strength in such composite structures. The ultrasonic emission detector developed under this program is applicable to metal-to-metal bonds, and metal-to-nonmetal bonds, such as in laminate and sandwich core structures; is portable; requires no couplant; evaluates large areas at the same time; requires only nominal skill; is adaptable for automatic test evaluation; and is sensitive enough to detect low bond-strength. This equipment deals directly with increasing the reliability of bonded joints -- one of the most difficult technological problems in the use of composite materials -- and should, therefore, find applications in organizations that are starting to use composite materials, such as airframe, boat and appliance manufacturers.

### Computer Enhancement of Radiographs (10-03)

For a number of years, digital computers have been used at the Jet Propulsion Laboratory (JPL) to improve resolution and contrast on filmed images received from the Ranger, Mariner and Surveyor space craft, by correcting various photometric, geometric, and frequency-response distortions in the pictures. These methods are now being used to enhance medical x-ray, biological and infrared photographs, photomicroph scintillation and scanner displays. The procedure involves first converting a picture into a form suitable for computer input. This is accomplished by scanning the film with a cathode ray tube device with a light beam on a line-for-line basis. Each line is a series of points, with typically 500,000 such sample observations for a l-inch square transparency. This information is placed on magnetic tape and then subjected to computer absorption and modification by either filtering to restore fine detail, or by subtracting to show changes over elapsed time between two snapshots of the same object.

Many industrial and medical radiographs when developed are too poor for reliable interpretation, which can be due to imperfections in equipment, technique, processing, or the original film. Rather than summarily scrap these films they are often interpreted on a best-effort basis and filed away. Computer enhancement may be able to extract the large amount of now unavailable information they contain, and make comparisons of changes over time or against new films, thus possibly relieving the ordinary industrial, medical or dental interpreter from seeking experts on poorly made film.

Computer enchancement of radiographs is a technique first developed at JPL. While the present impact of this development is still slight, the potential technological, scientific and economic impact is much higher, increasing over time.

o Computer enhancement of radiographs (10-03-01).

NASA has a very real need to maximize the amount and reliability of information contained in the large production of T.V. and other images telemetered back from space and lunar spacecraft. To meet these needs, JPL developed the original techniques used to enhance radiographic images by means of computer assistance.

### Delta Scanning (10-04)

The Delta scanning technique is an ultrasonic inspection method which uses redirected energy for flaw detection. It is a reliable, accurate and rapid method for weld evaluation (NDE), and is virtually insensitive to detector-to-flaw orientation.

Conventional ultrasonic weld inspection requires the beam to encounter a void, crack, fracture or inclusion in a nominally normal orientation. If, in scanning, the ultrasonic beam does not traverse the anomoly or flaw in a near-normal orientation, the probability is high that it will go undetected. With Delta scanning, two transducers are used, arrayed so that the workpiece and transducers form a geometrical configuration approximating a triangle, or the Greek letter delta. object of this configuration is to make the sending-receiving units insensitive to the orientation of the anomoly or flaw, thereby increasing the number and quality of detections. Sound energy is transmitted into the material at an angle that produces shear wave energy in the material, and propagates until it strikes an interface, i.e., an inhomogenity such as a crack, lack of weld penetration, lack of fusion, inclusion or porosity. Three distinct ultrasonic waves can then occur in consequence of this transmitted shear wave: reflection, mode converted longitudinal and re-radiated waves. The receiver search unit transducer, which is oriented normal to the surface, detects and trasmits this redirected energy for display and analysis.

Delta scanning is a rapid and accurate weld inspection technique having the important characteristic of being relatively insensitive to detector-to-flaw orientation, which has until now been a major quality control problem in all metal fabrication facilities, such as in automotive, aircraft and ship manufacturing. Because of its increased detection capability the Delta technique will lower inspection costs and improve quality assurance.

NASA was one of a number of early supporters of this development at Automation Industries in Boulder, Colorado.\* Because of space program needs for high reliability welds, NASA's efforts were directed at moving the device and technique from laboratory to production application. The potential technological and economic impact is estimated to be moderate to moderately high.

o Development of Delta scanning technique (10-04-01).

A major problem in weld inspection using standard ultrasonic techniques is the possibility of failing to detect a flaw, inclusion, crack or other anomaly because of unfavorable positioning of the ultrasonic transducers. The Delta technique is a unique, multi-crystal inspection method that is relatively insensitive to defect orientation. Internal weld defects such as lack of penetration and lack of fusion are readily detected with this technique. In addition, this technique is capable of rapid scanning rates (50 ft./hr.) while providing a simultaneous and permanent record of the test results. The Delta technique will detect up to 80 percent of penetration defects, while radiography detects only 36 percent.

Although a number of organizations supported laboratory development of Delta scanning, this technique was transformed from laboratory to practical application due to NASA's requirements for a reliable production weld inspection method. This system is valuable for nondestructive evaluation of production welds and should therefore find application in metal fabricating firms such as automobile, aircraft, ship and machinery manufacturers.

## Real-Time X-Ray Imaging (10-05)

Because of the expense and delay involved in generating and developing radiographic (x-, gamma and neutron ray) film in medical and industrial use, considerable effort has been expended in recent years to develop and improve real-time imaging techniques which obviate the

<sup>\*</sup>Other supporters include the Martin Company, Air Force, Navy, G.E., General Dynamics, and in-house funds of the developer, Automation Industries.

use of such film. Conventional fluoroscopic screens have been improved, and various image intensifier tubes and scanning T.V. systems developed. One commercially available unit uses a television-type camera tube with a pick-up surface directly sensitive to x-rays, rather than light, thus bypassing the fluoroscope.

Real-time viewing of remote or obscured processes greatly increases the volume and sophistication of data and therefore has improved analysis as its most immediate consequence. Another advantage is the selective elimination of the expense and delay of film making and developing; selective because the option still exists for taking a conventional snapshop or Polaroid photograph when a permanent record is called for. While real-time imaging offers many advantages over conventional methods, the image quality is still generally inferior to filmed radiographic images, except on very thick specimens.

There are many industrial processes which require direct, real-time viewing for their interpretation, particularly moving systems, such as hot steel deformation during rolling, casting metals, fluid flow and control, heat pipe studies and various process controls. Radiographic movies of blood flow, respiration, muscle movements and other biological phenomena are another class of important applications of real-time imaging.

NASA has supported development of a solid state image amplifier (see NDT development 10-14) as well as a real-time system using closed circuit television. The solid state image amplifier is now being marketed commercially by Westinghouse and is under evaluation by medical and veterinarian organizations. The potential technological and economic impacts of these contributions are estimated to be moderate.

o Development of a continuous scanning x-ray laminograph for multilayer printed circuit boards (10-05-01).

One of the basic needs in electronic assembly was for the development of techniques for making compact, reliable, high-density, circuit interconnections with a minimum of human error. Although the multilayer printed circuit board answered these needs it introduced a difficult inspection problem: how could the interior volume of a stacked array be interrogated in detail? Conventional x-ray images would be masked by layers above the layer being examined; high-current stress testing often creates marginal joints; and simple continuity testing gives no information about the quality of the joint.

NASA therefore contracted the Illinois
Institute of Technology Research Institute (IITRI)

to investigate candidate methods for nondestructively testing such laminated circuit
board arrays. IITRI developed a process for
this testing using axial transverse laminography. This technique involves photographically
smearing unwanted images while the image of
interest remains sharp throughout the exposure,
by synchronously rotating the film (or prism,
of the real time system) and sample. The
technique is a real-time, continuous, automatic
scanning, closed-circuit T.V. system. The
system provides magnifications of 0.2, 1.0
and 4.0 times with resolution to about 0.001inch, and is capable of 100 percent inspection
in any volume of the sample.

This system could be of value to electronic manufacturers requiring mass inspection of multi-layer circuit boards. Permanent records are available by photographing the image of interest. This NDT inspection tool may also be used in the examination of solid homogeneous or nonhomogeneous bodies.

o Development of a solid-state radiographic image amplifier (10-05-02).

A solid-state radiographic imaging system for use in real-time radiographic systems was developed by Westinghouse under a NASA contract. For a complete description of this contribution see NDT development 10-14, solid-state radiographic image amplifier.

### Holography/Interferometry (HNDT) (10-08)

Often described as three-dimensional lensless photography, holography is the technique of imprinting coherent light reflected from an object onto a photographic plate by means of another coherent reference beam. The resulting photograph, when developed and illuminated by laser light, revives the image coded on the plate and is seen by an observer as appearing in the space formerly occupied by the real object as a three-dimensional reconstruction. The photograph is now called a hologram.

By combining holography with optical interferometry, the powerful NDT tool of holographic interferometry is created. Interferometry is a technique of superpositioning two beams of monochromatic light at a given point. When the two beams are of equal amplitude, they will

reinforce each other if in phase, or cancel each other out if not. Splitting the coherent light from a laser, and causing the two halves of the split beam to interfere at the hologram surface, produces the coded image, i.e., the hologram, discussed above. When illuminated from behind by another laser beam, the hologram reconstructs for the viewer the original scene. Because only two wave sets of coherent light per image are imprinted on the plate, it is possible to store hundreds of pictures on a photographic plate ordinarily capable of storing only one.

Three nondestructive test techniques based upon this development are in common use: (1) Time-lapse methods which involve superimposition of a hologram taken before an event with another taken after. The difference between the two is indicated by interference fringes caused by physical disturbance due to the event. For NDT purposes this usually means deformation effects due to the applied stress of heat, shock, vibration and pressure. (2) Real-time methods, where coherent light is bounced off the object and directed onto the original hologram. This allows the object reaction to be observed as the test occurs. (3) Time-average methods, which involve long exposures to record an object's motion. These are usually employed in vibration studies.

Holographic interferometry is a major advance in NDT and can be used to assess deformation due to the applied stresses of heat, pressure or vibration; locating and identifying flaws, voids, inclusions, disbonds, or fatigue cracks. It is currently in use for testing automobile tires at Uniroyal, composite structures at Boeing, strain-gaging at Bell Telephone Laboratories, vibration studies at the University of Michigan, nuclear engine component testing at Aerojet General, and in numerous other applications.

In holographic interferometry, NASA's contributions primarily involve developing precise methods of measuring very small displacements and improving the detail in holograms. The consequence of this work has been to advance the development more rapidly than it might otherwise have occurred, and the collective technological and scientific impact of the NASA contributions upon the development is estimated to be moderate. The NASA contributions include the following, among others.

o Developed techniques for measuring small linear and angular displacements with holographic interferometry (10-08-01).

Under contract with NASA, Sperry Rand developed techniques for accurately measuring linear and angular displacements using realtime and double-exposure holographic interferometry. This effort resulted in the construction of a general theory accounting for both angular and linear motion of the

test object, and an error model for the two largest sources of such error: (1) measurement of interference fringe spacing and (2) measurement of experiment geometry. The objective was to determine the accuracy of measuring the motion of laboratory standard angle and displacement generators using holographic interferometry. In addition to verifying the viability of this technique for such measurements, Sperry Rand determined that calibration of laboratory standards was possible by refining fringe-space measuring techniques with a microdensitometer; and that by extrapolating the theory to three dimensions, complex staticdisplacements could be measured with greater ease and simplicity, and with an accuracy comparable to present optical tooling methods.

o dethod of improving hologram brightness by fringe stabilization (10-08-02).

Even slight vibrations received by the photographic plate during exposure of a hologram will disturb the standing interference fringe pattern and thus degrade the brightness of the holographic reconstruction. This occurs because darkened fringes are created by destructive addition of interfering waves. Because of this, holographic test apparatus is ordinarily placed on a thick granite slab with pneumatic supports to dampen out any immediate or background vibrations. This means that continuous-wave holographic interferometry is dependent upon laboratory-type optical supports, which seriously constrains the range of possible NDT applications. Even small changes in the air density caused by temperature or pressure variations will affect the location of the fringes if the optical paths are of the different lengths. To reduce these fringe motion effects, NASA developed a fringe stabilization system for use with low-power, continuous-wave lasers. The system employs a fringe monitoring photodetector and fringestabilizing piezoelectric crystal servoelectronics feedback control. This method of fringe stabilization should find application in gas lasers where superior fringe stabilization is required.

o Technique for holographic stress analysis of solder joints and prediction of circuit board lifetime (10-08-03).

Because printed circuit board failures cost the government large sums each year, and reliability requirements for man-rated space systems are so stringent, NASA developed a laser system for nondestructive testing of solder joints, and for correlating stress with load, to predict printed circuit board lifetime. The system employs a spatial filter to increase transmitted beam coherence by screening out unwanted modes and by passing the reference beam through a beam steerer into a metallograph having magnification of 1 to 2,000 times. In the double-exposure technique, a hologram is taken with the circuit board unstressed and again when thermally, mechanically or electrically stressed. The fringe spacing displacement indicates the relative motion of each component. which is then correlated with the amount of induced stress, and thus projections of lifeexpectancy can be made. The second, real-time technique involves illuminating the test sample with a no-load hologram and observing fringe growth under stress. The object in these methods is to assess solder joint reliability and predict joint lifetime from a simple NDT measurement. Magnification permits detailed assessment of any suspicious area.

o Development of an instrument for measuring very small displacements by combining a holographic interferometer and digital counting system (10-08-04).

Attitude and flight control systems for space vehicles operate to very close tolerances and therefore must be reliably tested. Under contract with NASA, M.I.T. developed an instrument to measure small linear displacements with extreme accuracy and sensitivity, with digital indications of magnitude and direction of displacement. This instrument combines a digital electro-optical fringe counting system with a Twyman-Green amplitude splitter interferometer employing a fixed wavelength laser. Output signals are amplified and fed into digital logic circuits, providing a digital read-out of the magnitude and direction of each displacement.

The instrument will measure a linear displacement over a two-inch range with an accuracy of ± 4.25 x 10<sup>-9</sup> inches, making it quite useful to automatic (numerically controlled) machine tool manufacturers and users, as a seismograph standard, vibration monitor, film thickness gage, and gyrotesting service. (See also NASA contribution to holographic interferometry 10-08-01: development of techniques for measuring small linear and angular displacements with holographic interferometry.)

### Neutron Radiography (10-10)

It was obvious at the time neutrons were discovered 30 years ago that they could be used to see through objects much as do x-rays. However, until recently, practical applications were virtually impossible because low-priced neutron sources were unavailable. In a number of ways, neutrons and x-rays compliment one another. X-rays are significantly attenuated by dense materials, where neutrons are virtually unaffected, passing through thick sections of many materials with relative ease. Alternatively, x-rays pass through hydrogenous materials with considerable facility, where neutrons are almost completely absorbed. In practice, this means that for many applications neutrons "see" what x-rays cannot, and vice versa.

Thus, neutron radiography could make visible what is lost in an x-ray radiograph, e.g., such hydrogenous materials as explosives, lubricants, tissue, elastomers and drugs. Moreover, neutron radiographs display a range of grays and blacks, unlike the far less informative stark black and white contrasts of an x-ray radiograph, thus providing indications of density, volume and position.

The crucial constraint on widespread efforts to apply neutron radiography has been the unavailability of an inexpensive source of slow neutrons. Californium-252, a synthetic element with a half-life of 2.65 years, has been the most commonly used source. However, at a cost of \$100 for a 0.1 microgram sample, the level of nonspecialist interest has been limited. To overcome this cost problem, the Atomic Energy Commission is now making the element available in bulk form for \$10 per microgram, thus opening the door for widespread commercial applications of neutron radiography.

Because neutrons are so readily attenuated by hydrogenous substances, and so little effected by the mass of many other materials, the technique is being used in such diverse applications as detecting drugs, firearms and explosives within suitcases, packages and vehicles; checking castings for hydrogen embrittlement; investigating fluid flow

in control systems (turbulence and cavitation analysis); detecting voids, cracks and density variations in composite materials; and determining the diffusion characteristics of solids and gases.

Neutron radiography was used as an important NDT tool in the Apollo missions, to accurately determine the reliability of explosive bolts used for staging separations. By categorically specifying neutron radiographic inspection of all pyrotechnic devices, such as the explosive bolts cited above, as well as in numerous other types of inspections, before neutron radiography was available as part of normal industrial NDT capabilities, NASA helped stimulate the widespread use of the technique. The potential technological and economic impact of this reliability requirement is estimated to be high.

o Early support of the market for neutron radiographic nondestructive testing equipment (10-10-01).

The space program uses numerous pyrotechnic devices, such as explosively actuated bolts. The reliability specifications for these critical devices are so high that NASA required neutron radiography as the inspection technique to be sure they contained the proper amount and distribution of explosive. The reason for choice of this NDT technique is that neutrons are strongly attenuated by hydrogen-bearing materials such as explosives, gaskets, seals, and fluids, yet pass through most other materials freely, while x-rays are not attenuated by such hydrogen-bearing materials and therefore do not "see" them. requirement for the relatively new NDT method of neutron radiography, over standard methods such as x-ray analysis, was instituted before the technique was commonly available. Since NASA contractors were required to use neutron radiography, the net effect was to stimulate the growth and development of neutron radiographic equipment manufacturers and trained personnel to meet the growing market needs. This early market stimulus and support underlies, in part, the increasingly widespread availability of neutron radiography for NDT purposes today.

# Nondestructive Testing (NDT)/Nondestructive Evaluation (NDE) Data and Personnel Improvements (10-11)

The past two decades have produced many new materials, combinations of materials, and improved versions of pre-existing materials. Concurrently, greatly increased demands upon materials by the space program,

the defense effort, and private industry have created elaborately sophisticated design requirements to increase efficiency, reduce wear, maximize strength and minimize weight. The increased performance, reliability and safety requirements have outstripped conventional quality assurance methods, and have created an urgent demand for more effective and comprehensive approaches to nondestructive testing and evaluation.

In its 1969 report (see description of the field of nondestructive testing), the National Materials Advisory Board (NMAB) of the National Academy of Sciences recommended that the term nondestructive testing (NDT) no longer appropriately described either the empirical practices or the increased scope of the discipline, and suggested instead that NDT be subsumed under the more comprehensive and accurate description of nondestructive evaluation (NDE). This shift in terminology is more than just semantic, reflecting as it does an evolution from mere detection and measurement, to materials evaluation and prediction. In practice, this meant that NDE should be deliberately incorporated into every phase of the research-design-production-service cycle as a screening, qualifying and predicting methodology. To do this, a very significant increase in the information data base was required on: materials properties; fracture mechanics; surface integrity; and residual stress analysis of complex bodies such as composites, cermets and alloys. In addition, corresponding advances were needed in NDT detection and measurement hardware, software, processes and techniques.

Development and improvement of NDE data, theory and processes, and NDT techniques and hardware, affects virtually every significant manufacturing sector: automobiles, appliances, aircraft, machinery, communication, etc. Advanced designs and increased performance, safety and reliability requirements are predicted upon commensurate advances in NDE/NDT technology.

Space program specifications for man-rated flight and space systems have required considerable advances in the NDE/NDT data base, test techniques, hardware, software, and personnel skills. These advances have been developed by NASA or by NASA contractors, or where the technology was already developed elsewhere, the advances were incorporated into contract specifications for quality control. The general character of NASA's contributions has been to develop and disseminate fundamental NDE/NDT data, standards and training manuals, as well as initially create or augment existing NDT devices, procedures and techniques. In many cases, NASA's requirements have shortened the time scale for these advances to be developed, demonstrated and disseminated; in others, the space program's needs created advances which still have only potential nonaerospace applications. Each contribution to the advancement of this development represents a small incremental step. The actual and potential technological and economic impact of individual NASA contributions, therefore, is estimated to be only moderate.

However, the overall impact of NASA contributions on the improvement of NDT/NDE data and personnel, is estimated to be high. Included among NASA contributions are the following.

o Improved commercial NDF capabilities through direct technical assistance (10-11-01).

Because NASA personnel are simultaneously involved in advancing the state of NDT technology, and assuring proper application of NDT techniques on NASA contracts, they are in daily professional contact with corresponding workers in other government offices, aerospace companies and in commerce and industry. With each awarded contract, NASA personnel review and assess the NDT capabilities of the contractor, recommending changes necessary to conform to contract requirements. It is not unusual for a contractor to develop quality control problems for which NASA personnel often have solutions. For example, Westinghouse experienced anomalous spotting (contamination) of image amplifier panels they were making under contract. NASA personnel traced the cause to the unfiltered plant air supply; filtering the production environment air removed the contaminants. In another case, the manufacturer of a high-pressure oxygen bottle for Skylab was experiencing leakage through welds, yet his dye penetrant inspection failed to indicate cracks. NASA personnel determined that the contractor had failed to first acid-etch the weld areas to remove smeared metal laminates which were obscurring the flawed areas beneath. Etching solved the mystery.

Acting as middlemen, NASA personnel routinely bring together people with problems and others with potential solutions. Inquiries for such assistance occur daily. This continuous consultative role, over and beyond developing and funding advances in NDT technology, has served to advance NDT capabilities in business, industry and other government organizations.

o Development of a radiographic interpretation guide for aluminum alloy welds (10-11-02).

Interpreting radiographs from NDT x-rays has been a difficult problem since the beginning

of industrial radiography. To improve the accuracy of radiographic interpretation of aluminum alloy welds, NASA conducted an inhouse program to correlate specific images with corresponding discontinuities, and to standardize the terminology used. Radiographs were made of welds containing various frequently encountered discontinuities. Subsequent metallographic studies were made to confirm the image types and shapes as depicted in the radiographs. Very precise welding techniques were required to produce these "discontinuities" and exacting controls required to generate truly representative radiographs and their metallographic verification. To eliminate differing uses of terminology as a source of interpretation errors. NASA also produced a standardized glossary of commonly used radiographic terms. This guide supplements, rather than replaces actual experience, and is an attempt to place radiographic interpretation on a scientific basis.

o Development of NDT test standards for detecting defects in advanced filamentary composites (10-11-03).

The appearance of high-strength, highmodulus, low-density filaments in the late 1950's ushered in the development of advanced composite materials. These materials have found wide use in aerospace structures, and as a class of materials, have the potential for extensive use in automobiles, architectural structures, appliances, aircraft, ships, and other vehicles. Two principal problems associated with the further development and application of composite materials are the high cost of fibers and the inability to adequately test composites for correspondence to design specifications and quality control requirements. Until engineering reliability can be assessed during fabrication and while in service, these promising materials are unlikely to receive widespread application. Without widespread application there will probably not be any substantial reductions in fiber cost.

Advanced composite materials develop many variations not found in conventional homogeneous materials, such as the number, orientation and

character of plies, accidental inclusion of foreign matter during lay-up, and strength and cohesion of bonds. All of these variations can affect the integrity of the structure. To overcome these problems, NASA supported a program at the Lockheed-Georgia Company which resulted in the development of designs for composite materials test standards, and a systematic comparison and evaluation of established NDT methods to detect the anomalous conditions.

o Demonstrated techniques for radiographic threshold detection of subsurface and surface cracks in aluminum welds (10-11-04).

The increasing demands by government and industry for high-strength hardware of known reliability has accelerated interest in flaw detection. Although the types of flaws most likely to be generated in welds prior to service are well known, the detection of very small flaws, such as cracks, is extremely difficult. To overcome this problem and to provide reliable quantitative data useful in fracture mechanics and therefore in evaluating and estimating the service life of critical components, NASA contracted with General Dynamics to determine the effectiveness of x-radiography to detect and measure crack and hole sizes in aluminum welds.

The primary objective of the test and study program was to design and fabricate special graduated aluminum penetrameters\* to evaluate the threshold detection capabilities of a fixed radiographic technique to detect surface and subsurface cracks and holes in aluminum welds. Although tests for thicker aluminum welds had been developed, there were no known techniques for weld thicknesses of 1/4-inch or less, and none with a precisely graduated series of penetrameters. The threshold detection capabilities

<sup>\*</sup>A penetrameter is a thin plate of the same material under investigation, and is used to determine radiographic resolutions. The thickness of the plate is in proportion to the test object, usually 1 or 2 percent, and ordinarily contains precisely formed holes one to five times the thickness of the penetrameter sheet.

of fixed radiographic techniques were defined and compared in terms of minimum detectable width, depth, and length of cracks, and maximum radiographic sensitivity.

This effort demonstrated the feasibility of using thin, precise penetrameters as test standards capable of quantitatively defining minimum detectable crack dimensions in thin aluminum welds. These techniques should find application in organizations performing critical aluminum welds on thin cross sections, such as manufacturers of aircraft, boats, and road vehicles, and cryogenic tank fabricators. (For similar NASA contributions to the development of a scientific data base for NDT through systematic evaluation and comparison of techniques, see NDT contributions 10-11-06, 10-11-12, 10-11-14 and 10-11-16.)

o Preparation of NDT training manuals for inspectors and technicians (10-11-05).

Adequate NDT texts did not exist for classroom and home instruction of quality control inspectors and technicians. To overcome this deficiency and fill this need, NASA contracted with the Convair Division of General Dynamics to prepare a fourteen volume, three-thousand page, textbook series. This series contains information on: NDT techniques (1 volume); NDT radiography theory, safety, equipment and applications (6 volumes); liquid penetrant testing (2 volumes): magnetic particle testing (2 volumes); and eddy current principles, equipment and applications (3 volumes). This unique compilation is of fundamental use to all organizations using NDT methods and equipment, such as aircraft, ship, automotive, machinery, electronic, appliance and precision equipment manufacturers. It should also find application in vocational and technical education courses and programs.

o Evaluation of nondestructive techniques for low-density foam composites (10-11-06).

Standard ultrasonic techniques, such as pulse echo testing, cannot be used for evaluating low-density foam composite materials. This is because foamed materials, such as polyurethane,

have a low acoustic impedence, making it difficult to distinguish the foam from air. Since low-density foam composites are used for insulating the cryogenic tanks of the Saturn vehicle, NASA undertook to evaluate the applicability of other nondestructive techniques for testing these materials. A combination of radiographic and acoustic methods, using a NASA-developed "resonant foam coupler," and low audio frequency eddy current techniques, were found to be applicable. This evaluation of NDT techniques for low-density foam materials should be of value to industrial firms using this class of materials, such as aircraft, boat and appliance manufacturers.

o Developed NDT methods for determining residual stress and fatigue damage in metals (10-11-07).

Premature failure of a structure is often caused by a lack of knowledge of residual stresses, since the design can only account for the superimposed loads due to external forces. NASA contracted with R. W. Benson and Associates to develop NDT methods of stress analysis suitable for field application on actual stresses and ultrasonic wave propagation in bulk and near surfaces using rising shear and surface waves. The Benson effort resulted in the development of transducers and other equipment for determining relative ultrasonic velocities along principal stress axes, and determined that an increase in surface resistance preceded any visual evidence of fatigue damage and impending fatigue.

These contributions represent an advance in fundamental NDT theory and application techniques and should be useful to NDT researchers, and manufacturers of such critical items as airframes, automobiles, ships, machinery, and industrial structures.

o Development of NDT weld inspection system (10-11-08).

A triple-assessment NDT weld inspection system was developed by the W. V. Sterling Company under a NASA contract. This system uses: (1) weld pulse monitoring to determine weld joint resistance variation while the weld is in progress; (2) indirect

measurement of thermal radiation during welding; and (3) correlation of dynamic setdown rate and weld quality. The instrumentation to perform these functions was designed and developed as part of this effort, as were definitions of weld acceptance criteria.

o Compilation of NDT bibliographies on the evaluation of the strength of bonded metallic materials (10-11-09).

Since advances in the state of technology are aided by knowledge of work already accomplished, Lockheed-Georgia Company, under contract with NASA, developed bibliographic materials covering the state-of-the-art of nondestructive evaluation of the strength of bonded metallic materials. This bibliography is of value to fabricators of composite materials, such as airframe, tanktruck, tankcar, boat, and appliance manufacturers.

o Application of NDT measurement techniques to the analysis of residual stresses in metals and metal structures (10-11-10).

Distortions caused by welding are a major problem in fabricated structures such as space vehicles. Although empirical approaches to correcting distortion have been developed, these approaches do not prevent the distortions, nor do they give an understanding of the thermal and mechanical factors that caused them. To develop a better analytical understanding of the problem, NASA contracted with the Battelle Memorial Institute to: (1) provide fundamental information on residual stresses necessary to understand measurement techniques; (2) review methods of measuring residual stresses, including stressrelaxation, x-ray diffraction and ultrasonic techniques; (3) provide methods of measuring residual stresses during fabrication of metal structures, and (4) select and describe appropriate measurement techniques.

This basic approach to the measurement of residual stresses represents an important advance in developing a scientific base for the advancement of NDT.

o Development of spot test and galvanic reaction test techniques to rapidly identify metals (10-11-11).

The problem of materials identification is a common one in industry. For example, identifying symbols on raw metal stock often become illegible or are accidentally cut off, or machined parts that look alike may have different compositions due to aging in a storeroom or mixed batching. This identification problem often results in either expensive scrap or expensive reruns.

Two simple methods -- the spot test and the galvanic reaction test -- have been developed by NASA and contractor personnel to alleviate this problem, and thus bypass the tedious and expensive identification method of emission spectroscopy. The spot test is done on the metal surface, in spotplate depressions, or on filter paper, using standard chemical reagents. Qualitative identification is made by color comparison of reaction products using a specially prepared chart. test can be made semiquantitative by comparison with standard specimens. All common metallurgical elements, many alloys, high-temperature stainless steel, and high- and low-carbon steels are covered by the report on this work. Detailed instructions and description of expected experimental results have been developed and are provided by NASA.

The galvanic reaction test measures the potential difference produced by a galvanic reaction between a reference electrode and the test metal, using a water drop as an electrolyte. This test can be carried out on mounted components of an assembly as a go/no-go determination.

These techniques and the NASA data, tables and instruction should find use in metal fabricating ships.

o Evaluation and development of NDT techniques for analysis of honeycomb structure bond strength (10-11-12).

Due to the problems associated with evaluating composite bond strength, NASA contracted with North American Aviation to advance the state-of-technology in this area, including: a comprehensive survey of

the literature and the industry, with an index and annotated bibliography; the identification of candidate test methods; and a systematic NDT test and development program. Five promising ultrasonic methods were identified and corresponding composite specimens and breadboard test systems constructed and evaluated: echo interference, impedance, decrement, spectrum analysis, and intermodulation.

The impedance system successfully detected disbonds and was selected for further development. In addition, a number of candidate semiautomatic scanning and recording systems were developed to supplement the ultrasonic system. Since this system allows remote testing of large surfaces with minimum surface disturbance, it should find application in the nondestructive testing of composites materials in airframes, boats, and appliances.

o Preparation of a bibliography of the effects of noise on man and materials (10-11-13).

The effect of noise on man and materials is of considerable interest at Kennedy Space Center since personnel and equipment are periodically subjected to high noise levels. Since such information had not been adequately compiled, NASA compiled and prepared an annotated bibliography of the effects of noise upon men and materials. Although concerned primarily with audio acoustic noise, some materials cover electrical noise as well. This information should prove useful for NDT/NDE test purposes, for safety engineers, and medical use.

o Evaluation of NDT techniques for detection of weld flaws (10-11-14).

Since each Saturn V space vehicle requires over 5,000 feet of aluminum welds, all of which are critical, NASA required reliable techniques for the detection of weld flaws. To meet this requirement the capabilities of ultrasonic and radiographic NDT techniques were systematically evaluated and compared. Emphasis was on the detection of lack of penetration; lack of fusion, porosity, slag inclusions and weld bead interference. Except for porosity, the superiority

of ultrasonic methods over radiographic for weld flaw detection was demonstrated. This information should be useful in all aluminum welding facilities and should lead to improvements in the quality of weld inspection in areas such as aluminum airframe, ship, vehicle and tank and vessel manufacturers, and aluminum structural fabricators.

o Preparation of textbooks and classroom training procedures on ultrasonic NDT techniques (10-11-15).

Adequate training manuals and procedures dealing with ultrasonic NDT techniques did not exist. To overcome this, NASA contracted with the Convair Division of General Dynamics to prepare a series of textbooks and classroom training procedures dealing with the whole field of ultrasonic NDT. Comprising over 1,000 pages, the ultrasonic series of programmed instruction covers basic principles and theory; test equipment and accessories; procedures, methods, techniques, and application examples; and a post-instruction classroom training manual. (See NASA contribution 10-11-05, preparation of NDT training manuals for inspectors and technicians, for a related effort.)

o Evaluation and comparison of NDT techniques to inspect brazed joints (10-11-16).

The five F-1 booster engines clustered about the base of the Saturn V vehicle contain many brazed components and assemblies. Because the integrity of many of these brazed joints are critical, reliable inspection techniques were required. North American Aviation, under a NASA contract, systematically evaluated and compared the application of standard NDT methods, such as radiography, ultrasonics, thermography, and leak testing, to the inspection of brazed joints. The effort resulted in the development of unique equipment and test methods, and recommendations concerning the advantages and limitations of the various NDT methods for specific geometries, material compositions, and joint accessibility. (For similar NASA efforts developing a scientific data base for NDT through systematic evaluation and comparison of techniques, see NDT contributions 10-11-04, 10-11-06, 10-11-12 and 10-11-14.)

o Comprehensive survey and analysis of the state of NDT theory, techniques and equipment (10-11-17).

North American Aviation, supported jointly by the Air Force and NASA, compiled a comprehensive literature survey on NDT theory, techniques and equipment, with primary emphasis on ultrasonics. Also included were reviews on the current state-of-technology of honeycomb and sandwich construction, composites and bonding. This survey was an early attempt (1965) to integrate NDT and composites information, so as to provide a firmer base for the development, improvement and application of NDT techniques for this new class of materials.

## Solid State Radiographic Image Amplifier (10-14)

Space stations will require a variety of NDT systems, among which are radiographic devices. Because storing, developing and evaluating radiographic film in space presents difficult shielding problems, considerable effort has been invested in developing a solid state imaging system equivalent to a fluoroscopic screen or x-ray film. The system, which was developed by Westinghouse under a NASA contract, has image retention capabilities, is portable, has the detail and thickness resolution of ordinary film, and can be interpreted immediately following exposure. The radiographic amplifier panels were constructed on glass substrates and on plastic, and have a resolution of about 300-500 lines/inch, a storage (i.e., image retention) time between 5 minutes to one hour, and sensitivity similar to high-resolution radiographic film.

This recently (1969) developed system has many obvious advantages in industrial NDT, as well as medical and dental work: (1) Because an image with excellent resolution can be retained for up to one hour, it is not necessary to make time-consuming and expensive radiographic film records, although a film can still be made if a permanent record is desired. (2) Because the system is portable it can be used in the field. (3) Because of the system's superior sensitivity, interpretation is more precise and efficient. (4) Because it is almost a real-time process, it offers significant time and cost savings over radiographic film processes. (5) Because the process employs far less radiation, it is less hazardous than conventional systems. (6) Because the system is capable of thousands of rapid erasures, it is economical. The U.S. Public Health Service has already expressed considerable interest, and industrial and medical/dental application appear imminent. Westinghouse has already marketed 20 laboratory-built versions and is expected to announce a product line of such panels in the near future.

This particular development is an entirely NASA-supported effort by the Westinghouse Corporation. The idea was initiated by NASA

personnel, who also made substantive contributions to the designs, particularly in recommending incorporation of the British Thorn image retention panel. Although the present economic and technological impact of this NASA development is still low, the estimated technological, economic and scientific impact should significantly increase with time.

o Development of a solid state radiographic image amplifier (10-14-01).

This equipment was developed by Westinghouse, under NASA contract, as described above.

## Thermal NDT (10-16)

Since every material object above absolute zero emits thermal radiation, its thermal "signature" (spectrum and intensity) can be detected and analyzed, as, for example, the increased temperature of a tumor. Alternately, an object may be artificially heated and its differential absorption may be analyzed for some internal feature of interest, such as a disbond in a composite structure. Applied heat will flow uniformly through an object unless some discontinuity or inhomogeneity impedes its flow, in which case a "hot spot" will occur at the surface. The distribution and intensity of total emissivity and/or absorption can be detected, measured, and analyzed to give information about the state or response of the body to its environment.

Detection can be direct, by contact, or indirect by remote sensing. Direct detection of surface temperature can be accomplished: by using radiometers, thermocouples, or resistance thermometers, which are inefficient for large surfaces; by applying temperature-sensitive paints and phosphor coatings, which do not offer a permanent record and are useful for only near-surface defects; and by using liquid cholesteric crystals, which pass through a spectrum of colors with fine sensitivity to temperature changes. Indirect detection can be accomplished by using infrared remote sensing of surface temperatures. For most NDT applications remote sensing is preferred because the sample is not disturbed, there is little atmospheric attenuation of the infrared radiation, and rapid scanning of large areas or fast moving samples is accomplished with little difficulty.

The two principal techniques employed today are liquid crystals and infrared scanning. Principal developer/manufacturers are AGA Aktie (Swedish), Automation Industries and the Barnes Engineering Company.

Thermal NDT methods offer a fast, inexpensive, reproduceable, sensitive and relatively easy way to determine large voids, delaminations and other subsurface defects or discontinuities. The advantages of this technique have led to the development and commercialization of sophisticated infrared radiometers, microscopes and cameras.

can be measured and profiled for signatures of normal, bad and incipiently-failed circuits and components. However, measuring the heat dissipated in microelectronic devices is virtually impossible by contact devices because the interconnections are only a few millionths of an inch in size. Testing was therefore restricted to measurement of input and output, but not of components themselves. In such a testing approach, the performance of a marginal part may be obscured by a strong part absorbing the additional load.

NASA reliability requirements required proven assessment of all microelectronics parts, but no device existed to provide such reliability. To overcome this, NASA contracted with the Raytheon Company in 1964 to develop a fast-scan infrared microscope for measuring and plotting the infrared profile of semiconductor chips, transistors, and integrated circuits. The thermal maps that are developed are compared with the corresponding profiles of test standards, yielding a measurement of integrity with design values, and thus indicating what corrective steps must be taken in process and production control.

This system should find commercial application with semiconductor manufacturing firms who are concerned with the nondestructive testing of microelectronic circuits.

o Development of an automatic in-process NDT microweld monitor (10-16-03).

Since it is more effective to make in-process corrections than to inspect, reject and correct finished products, Martin Marietta Corporation, under a NASA contract, developed a system to monitor in-process microresistance welding of electronic assemblies on a 100 percent nondestructive inspection basis. The system automatically measures the infrared energy generated in the weld as it is made. It then compares the thermal signature against maximum/minimum allowable limits of infrared energy values, which are correlated with acceptable weld-strength tolerances. When a weld is out of tolerance, a signal warns the operator to take corrective action. It is possible to link this system with a computer to provide a closed-loop self-correcting system. This system should find application with electronic assembly manufactures.

Thermal NDT is widely used in aerospace, automotive, boat and appliance manufacturing.

NASA contributions to advancing the technology of thermal NDT techniques are estimated to have a moderate potential impact, and include the following.

o Development of equipment for thermographic testing of bonded structures with liquid crystals (10-16-01).

Thermal nondestructive testing with cholesteric liquid crystals is an economical, fast and simple real-time technique used to detect disbonds, voids. cracks, or inclusions in a structure. These organic, cholesterol-containing compounds behave mechanically like liquids but exhibit the optical properties of crystals. In practice, the surface to be tested is coated or otherwise covered by the cholesteric fluids. Heating or cooling the surface alters the colors of the liquid according to the differential thermal absorption gradients of the underlying structure, thus indicating subsurface anomalies. These crystals are sensitive to temperature differences as small as 0.1°C., and can be formulated into compounds sensitive through the visible spectrum with a temperature range as narro as 1 C. and as broad as 50 C.

To provide data for the evaluation and use of liquid crystals for nondestructive testing of bonded composite and laminate structures, NASA contracted with the Lockheed-Georgia Company to develop such data and the necessary testing techniques and equipment. The self-contained portable analysis system which was developed, contained photographic equipment for permanent recording of results, and special instrumentation for calibration and quality checking of the compounds themselves. This equipment provides a simple, fast and portable method for performing the difficult job of inspecting composite or laminated materials. It should also find further application in the inspection of heat exchangers, joints, and in making surface temperature measurements.

o Development of a fast-scan infrared microscope for testing microelectronic devices (10-16-02).

Within electronic components, wiring and connectors, some fraction of the electric current flow is dissipated in heat due to resistance. This heat

### SIMULATION (11)

The field of simulation is both broad and complex, and it involves developing and experimenting with models of reality, either physical objects or real but abstract systems. Simulation may have any of several purposes: to predict the performance of a system when subjected to various environmental conditions or stimuli; to analyze the function of a system when various changes are made in its components, as an aid to understanding; to gain knowledge for decision-making and problemsolving; to optimize the design of a system by experimenting with various changes in its parameters; or to train persons (e.g., managers, astronauts) by simulating the actual experiences they will face when operating in a real environment. Simulation is applied to the interactions of a variety of systems: physical, managerial, political and social.

Simulations generally involvesimplifications of reality which nevertheless adequately portray one or more important features of the real object or system. Simulation is particularly useful for the solution of problems of systems design and systems analysis, when the systems under consideration cannot be analyzed using direct, formal analytical methods. Frequently, system simulations take the form of mathematical representations of the interactions of the subsystems and components. Such mathematical models lend themselves well to computer manipulation, which provides the means for rapid and thorough experimentation and search for optimum solutions. Not only digital computers but also analog and hybrid computers are used for system simulation.

The value and power of simulation lie in its ability to impart the insight necessary for people to make more intelligent decisions than would otherwise be possible. To give answers directly, a simulation model must be validated, which is often difficult and sometimes impossible to do. Furthermore, it must be used under precisely the conditions for which it was validated, and this sometimes precludes its use for the intended purpose. However, valuable insight to aid decision-making can be gained even without rigorous validation.

Simulation techniques are extensively used in research and development, in management, and in education and training. Although simulation is more common in the technologically advanced sectors of the economy (e.g., the universities, the aerospace industry) its use is growing and spreading in more conventional industries. Within government, simulation has transferred from the military, which has long made use of it for weapons research and policy guidance, and the space program, which uses simulation in mission analysis, space vehicle design, technical training, and management. Now, more and more use is made of simulation by the Federal departments and agencies charged with domestic missions of social welfare and economic progress.

The economic significance of the field of simulation seems impossible to estimate. As a technique rather than a physical product, it does not appear directly in the national income accounts of gross national product. Yet it contributes to the effectiveness of nearly every sector of the economy. Simulation has a great impact on research and development and on industrial operations, promoting better design and management throughout the U. S. economy. Even qualitatively, the impact of simulation cannot fully be measured in economic terms. For example, what economic measure can be placed on a national policy decision affecting U.S. relationships with foreign governments, when the decision relies on knowledge gained through military and political policy simulation? What is the economic significance of the successful completion of the Apollo 13 mission, involving the lives of three astronauts, scientific findings, and the national prestige of the U. S.? The procedure for successfully coping with the Apollo 13 emergency was worked out on the NASA simulators during the crisis.\*

The space program has, in fact, stimulated many advances in the field of simulation. NASA's simulation interests span policy and mission analysis, technological research and development, design analysis, technical training, and program management.

The aerospace simulation advances have accompanied numerous other significant developments in the field of simulation in the last few years. A selected list of significant simulation developments, as identified by leaders in the field, appears in Table 11.

The field of simulation is growing at an exponential rate and will continue to do so into the foreseeable future, due almost exclusively to transfer of simulation technology to new areas of application. The aerospace sector, which has been the major area of simulation application, is considered by experts to be comparatively saturated with such applications. In view of current production and development cutbacks, little or no increase in the use of simulation can be expected in aerospace. However, the same cannot be said of the transfer of simulation technology to other sectors. The Federal Government, which has been slow to recognize the value of simulation, now is changing and contributing to the expansion of the field. The Department of Transportation, Environmental Protection Agency, and others are making greater and greater use of simulation. In addition, both public and private organizations are making increased use of simulation to study economic, political, social/urban, ecological, and other problems too complex for the unaided intellect to fully comprehend. These factors account for the exponential growth.

<sup>\*</sup>We are indebted to John McLeod, Executive Director of Simulation Councils, Inc. and Editor of <u>Simulation</u>, for these examples and for numerous valuable insights provided to us during the conduct of our research.

#### TABLE 11

### SIGNIFICANT DEVELOPMENTS IN THE FIELD OF SIMULATION (11)\*

- 1. Time sharing of computers, using remote terminals (11-01)
- \*\*2. Man-machine interactive systems (11-02)
- \*\*3. Hybrid computers for simulation of complex systems (11-03)
- \*\*4. Continuous systems simulation languages (11-04)
  - 5. Discrete systems simulation languages (11-05)
  - 6. Simulation of international affairs (11-06)
- \*\*7. Simulation of social processes (11-07)
- \*\*8. Simulation of human bodily functions (11-08)
- \*\*9. Optimizing models (11-09)
- \*\*10. Simulation of flight operations (11-10)
  - 11. Space vehicle logistics simulation
     (11-11)
- - 13. Modeling aeroelastic structures (11-13)
  - 14. Cost estimating and trade-off models (11-14)

<sup>\*</sup>As identified by technological leaders in the field of simulation.

<sup>\*\*</sup>Developments selected for identification of NASA contributions.

The major problems in the field of simulation are inadequate communication and education. As a result of these shortcomings:

- a. Many who could use simulation to advantage are not aware of its usefulness.
- b. Some who recognize the potential of simulation do not know how to construct and use a model.
- c. The purpose for which simulation should be used is often misunderstood. That is, too many people think that the purpose of simulation is to provide answers. It can, but only under very special circumstances.

Selected significant developments in the field of simulation as listed in Table 11, and NASA contributions in the advancements of these developments, follow.

### Man-Machine Interactive Systems (11-02)

Devices that permit interactions of men and machines (generally computers) extend the capabilities of persons to conduct complex tasks. Such interactive systems provide men immediate and direct access to machine outputs and permit them to immediately alter machine inputs. There are several types of interactive systems: (1) computer graphics. which aid planners and engineering designers by giving them the ability to "converse" directly with a computer during the development of engineering systems which are displayed visually on a cathode-ray screen; (2) mathematical systems which allow a scientist or engineer to solve mathematical problems on a computer without need for computer programming knowledge; (3) file manipulation interactive display systems, which permit instant access to detailed data from massive storage files, by project engineers or managers; and (4) educational interactive display systems, used as training devices. In all cases, the computer simulates the actions of a system or object, so the man can interact in a conversational, real-time mode.

Man-machine interactive systems are, almost by definition, necessary for the design and planning of high-performance aircraft and spacecraft as well as the training of pilots and astronauts. The more sophisticated the interface, the greater the realism and the better the design, flight planning and training.

Some early work in interactive computer graphics was done around 1963 at MIT, under the Air Force-sponsored Project MAC. The BIOMOD work, done at Rand under Air Force and later NIH sponsorship, adapted interactive techniques to biological modeling for biomedical research.

NASA has made major contributions in integrated display systems, simulation-aided design, computer mapping, and in development of a mathematical problem-solving system. These contributions include the following.

o Development of integrated, time-shared instrumentation display systems (11-02-01).

NASA's Langley Research Center, as well as numerous NASA contractors, have made substantial efforts to develop simulation instruments that realistically model the experiences of spacecraft and that display accurately the visual images of a dynamic space vehicle. An integrated, time-shared instrumentation display system patented at NASA/LaRC mixes static and dynamic elements from photographic transparencies. Other significant work has been done under NASA contract at McDonnell-Douglas, University of Southern California, the Franklin Institute, University of Michigan, University of California, Berkeley, Martin-Marietta, Hughes, and at NASA/LeRC and NASA/ERC.

o Computer-aided circuit design (11-02-02).

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A man-machine interactive display system developed at NASA/LaRC, known as LICCA (for Langley Interactive Computerized Circuit Analysis), enables a designer to draw electronic circuit diagrams on a cathode ray tube screen, using a light pen. The system analyzes the circuit during design, detects errors, and prints guidance messages for the designer. NASA also has sponsored research in simulation-aided engineering design at the University of California, Berkeley, which emphasizes the behavioral variables of designers using computer-aided techniques.

o Mathematical problem-solving technique (11-02-03).

The AMTRAN system, developed at NASA/MSFC, is an electronic simulation interface which simplifies and speeds up communication between a man and a computer which solves mathematical problems. AMTRAN permits a scientist or engineer with no background in computer techniques to nevertheless readily communicate with the computer by means of a keyboard. The computer immediately displays the mathematical equations (in alphanumeric or coordinate plot form) on a cathode ray oscilloscope, solves the equations, and prints results with a typewriter.

### Hybrid Computers for Simulation of Complex Systems (11-03)

Hybrid computers are combinations of digital and analog computers. If properly combined, hybrids can take advantage of the desirable characteristics of each while avoiding most of the undesirable characteristics. The availability of hybrid computers encouraged engineers and scientists to undertake more difficult simulations and to model much larger systems than could then have been accomplished with either digital or analog computers alone.

The earliest hybrid computers were installed by Air Force contractors (Convair and TRW) about 1956-57 for use in ICBM program simulations. There was considerable activity and experimentation with hybrids at that time, although the earliest hybrids used vacuum tubes and were somewhat limited in their reliability. In the early 1960's, when transistors came in, hybrid computers became much more reliable. The period 1963-67 was the peak of hybrid use and importance, when hybrids were used for very large systems.

Significant work in the development of hybrid computers for simulation use was done at the Universities of Arizona, UCLA, Southern California, and Minnesota, and at Lockheed and Honeywell. Much of the work has been done under Air Force and NASA sponsorship.

Since 1967, the use of hybrid computers has declined. Not a single large hybrid computer center has been installed in the last two years, although some small hybrids have been. The reason for the decline of hybrids has been the dramatic developments in digital simulation. Recently, the cost per computation of digital computers has been less than for hybrids, and the trend toward use of all-digital simulation is increasing.

NASA contributions to the development of hybrid computers have been significant, and include the following, among others.

o Sponsorship of early hybrid computer development efforts (11-03-01).

NASA has sponsored much of the significant work in hybrid development by Professor Granino Korn of the University of Arizona. Professor Korn was a pioneer in analog simulation, and the term "hybrid" first appeared in the title of his classic work, Electronic Analog and Hybrid Computers (1964). NASA sponsorship of Professor Korn's work since 1965 has significantly advanced the time of development of faster, more reliable, and lower-cost hybrid computer systems.

o Demonstrated the advanced simulation of control systems (11-03-02).

Auburn University researchers, under NASA sponsorship, in 1968 accurately simulated the attitude control system of the Saturn V booster stage, using hybrid simulation techniques. This contributed to the successful design of the Saturn control system for the Apollo Program which was one of the most advanced demonstrations of hybrid computer simulation applications.

o Development of methods for optimizing dynamic system parameters (11-03-03).

In 1963, NASA helped sponsor some early work at UCLA, which involved development of a computational strategy for the optimization of parameters in a nonlinear dynamic system. A hybrid computational strategy was formulated, including the best features of analog and digital computer solutions. This work led to an expansion of the role of hybrid computers in the simulation of complex systems.

o Development of automatic patchboards (11-03-04).

Automatic patchboards are recently developed devices that fit on the analog portion of a hybrid computer and allow components to be connected automatically under digital control. NASA/MSFC sponsored the development of these patchboards by Electronic Associates, which has made them available commercially. The use of patchboards greatly simplifies the amount of programming required to conduct hybrid computer simulations, thus reducing cost and adding flexibility.

# Continuous Systems Simulation Languages (11-04)

Simulation languages made it possible for the system scientist to directly perform the simulation of continuous, dynamic systems without having to work through a computer programmer who usually could not be as concerned or as well informed about the problem. The direct involvement allowed the responsible scientist to gain the insight which is so important in simulation-based decision-making.

Although all digital languages are discrete (i.e., only one computation is made at a time), continuous systems can be, and typically are, described by differential equations. These are transformed into

difference equations for digital solution, and the computer steps through them at a given rate with respect to time. Differential functions formerly were handled by analog and hybrid computers because it was very difficult to program them on digital computers. The development of these languages was the major advance that allowed continuous system functions to be simulated easily on the digital computer.

Among the continuous systems simulation languages are MIMIC, CSMP, and CSSL. MIMIC was developed at Wright-Patterson Air Development Center. CSMP was developed by IBM, with considerable cooperation and assistance from NASA/ARC. CSSL was a language originally specified by Simulation Councils, Inc. and further developed by Lockheed Sunnyvale and implemented by Spectrodata.

NASA's contributions to language development are somehwat fragmented, consisting of some assistance in development, in augmentation, and in dissemination of results.

o Assistance in language development (11-04-01).

During the development of CSMP, one of the three most used languages for continuous systems simulation, NASA/ARC provided cooperation and assistance to IBM, the prime developer. In another area, NASA also has sponsored development of Extendable Computer System Simulator (ECSS) at RAND. ECSS is a programming language for simulating computer systems, as an aid to the design and evaluation of such systems. This has been a notable contribution to the advancement of computer system design.

# Simulation of Social Processes (11-07)

Some of the potentially most significant applications of simulation techniques have been made in the area of social systems and processes. This work offers great hope in understanding the dynamics of social processes as an aid to improving them.

Notable pioneering work was done by Jay Forrester, first in Industrial Dynamics (1961) which simulated the production and marketing behavior of industrial enterprise, and next in <u>Urban Dynamics</u> (1969) which applied dynamic simulation techniques to such urban problems as unemployment and housing. Dr. Kenneth E. F. Watt and the staff of the Environmental Systems Group, University of California, Davis, have been engaged since 1968 in developing "A Model of Society." This is an ambitious, complex simulation of the human ecosystem in California, involving demography, agriculture, health, education, climate, and other subsystems.

### Simulation of Human Bodily Functions (11-08)

Biomedical research has been significantly advanced by the development of simulations of various normal and abnormal bodily processes. Digital and analog simulations have been developed of many humanphysiological systems, which add to medical understanding of the systems' operation and allow experimental research which could not be done on living patients. This research appears likely to lead to reliable predictive models that can simulate the potential effect of a medication on a specific human patient.

In 1963, John McLeod of Simulation Councils, Inc. prepared a paper on the computer simulation of the hydrodynamics of the cardiovascular system. In 1966, McLeod had developed PHYSBE, a physiological simulation for the study of the hydrodynamic transport of various substances (oxygen, CO<sub>2</sub>, nutrients, wastes, medications, and heat) by the circulatory system. Subsequently, many researchers have added to the wide variety of physiological processes that have been simulated.

Much of the simulation research on the human body has been conducted or sponsored by NASA as part of its studies of astronaut capabilities under the rigors of space flight conditions. NASA/ARC has developed a variety of physiological simulations. Among the NASA contributions are the following.

o Development of a phonocardiogram simulator (11-08-01).

In 1970, a phonocardiogram simulator was patented by NASA/KSC. This device produces electrical voltage waves that simulate the sounds of the human heart. The device is used to calibrate phonocardiograms and appears to have potential value for training physicians.

o Circulatory system simulation (11-08-02).

NASA has sponsored research at the University of Wisconsin to simulate venous blood pressure. Using an analog computer model of the cardiovascular system, the researchers studied mechanisms of venous blood flow under various gravity conditions. Other NASA-sponsored work at General Electric, Philadelphia, studied pulmonary blood flow by use of an analog computer simulation. Both of these simulations contributed to the knowledge of how to simulate complex bodily processes, using analog computer techniques.

In <u>Mathematical Models of Public Systems</u> (1971), Simulation Councils, Inc. described efforts to simulate ecological systems (air pollution, land use), transportation systems (freeway traffic, air traffic control) and socioeconomic systems (housing, communications).

NASA has sponsored research which has made a substantial contribution to the simulation of social processes; two such contributions are described below. Another potential contribution of great significance is the Earth Resources Technology Satellite (ERTS) program. ERTS will gather data on hydrologic, oceanographic, and biological activity which would greatly increase the effectiveness of environmental/ecological simulations.

o Simulation of urban affairs (11-07-01).

NASA has sponsored research at Drexel University in the simulation of urban systems. In Drexel's Urban Simulation Gaming Laboratory, a simulation game, BUILD, has been used to train residents and officials of a deteriorated community in the process of decision-making leading to community redevelopment. A second simulation, MISPA (Management Information Systems for Public Administration), has applied game theory to the management of city government programs. MISPA incorporates such elements as budget analysis, personnel management, project renewals, and priority evaluation, and constitutes a model for training city administrators. Both BUILD and MISPA have been pioneering efforts in the transfer of simulation/gaming from the scientific area to the urban scene.

o Police problem simulation (11-07-02).

Wayne State University, under NASA sponsorship, has developed a model of a metropolitan police department. The model was used to evaluate the operational effectiveness of the department in meeting its objectives, and to analyze such specific problem areas as communications, information transfer, car locations, and jurisdictional problems. The police model, one of the first applications to city government, demonstrated the applicability of the simulation technique to the pursuit of social objectives in public administration.

o Human performance capabilities in space (11-08-03).

In work directly applicable to astronauts, NASA has developed devices which simulate lunar conditions and has studied human self-locomotion (walking, running, jumping) to determine performance parameters under such conditions. This research has been done both at NASA/LaRC and under contract by Grumman. This work showed how physiological simulation could be used to test human reactions to extra-terrestrial conditions.

o Development of renal and nervous system simulations (11-08-04).

NASA/ARC has used computer simulations, validated by laboratory animals, to simulate the human renal system, the nervous system, and the inner ear. Ames has also built on earlier simulations of the cardio-vascular system. Such simulations have extended the technique of physiological simulation from the simpler to the more complex bodily processes.

## Optimizing Models (11-09)

Although scientists and managers have long been attempting to optimize solutions to problems, there have been some problems so complex that optimum solutions have been considered impossible. Several technological developments have changed this. One is the evolution of the digital computer which vastly speeds computational time. Another was the development of dynamic programming by Richard Bellman in 1957. Dynamic programming is a technique for analysis of complex problems by separating the total problem into sequential, interdependent stages. A third development was the 1963 publication of the Pontryagin maximum principle. These developments have led to solving the optimum control problem, and have permitted simulations which can give optimum solutions for the control of physical processes.

The need for extremely accurate guidance of missiles and spacecraft to their targets led to numerous advances in optimization routines in the aerospace sector. Within the last 10 years, many persons in aerospace have developed algorithms which differ in programming but result in more speed and a more accurate approximation of the optimization routine. These software applications, which improve the simulation of optimization and indicate how to change the parameters of a system, are now being transferred for application outside the space program.

A different type of computer simulation, not directly related in form to the system optimizing model but allied to it in purpose, is the simulation model used for optimizing operation of a complex management system. This systems model simulates the interactions of a system and is used both for analyzing how to optimize the system's operations and for training managers to optimize performance.

Examples of NASA contributions to the development of various types of optimizing models follow.

o Development of production operations simulation model (11-09-01).

The General Electric Company, under contract to NASA, published in 1971 a major study of aerospace manufacturing concepts, based on the Saturn/Apollo manufacturing experience. This contributed to the development of optimizing models by demonstrating application of simulation techniques to cost optimization of major industrial operations. A computerized simulation model was used to accumulate costs to show the impact of variations and interactions of factors on manufacturing cost. The conclusions of the study point to ways to reduce unit production costs and optimize production efficiency of conventional aerospace structure manufacturing.

o Optimization model for component redundancy (11-09-02).

The General Electric Company in 1967 developed an optimizing simulation for the Jet Propulsion Laboratory, as part of the design planning for the Voyager program. The model determined the optimum degree of redundancy in design components when balancing the increased mission reliability with the offsetting increases in weight and cost. The model used dynamic programming techniques to obtain an approximate optimum solution, among 10<sup>21</sup> possible solutions. This contributed to the field of simulation by illustrating the use of optimizing techniques in solving the most complex of design trade-off problems.

o Research Engineering Management Simulation (11-09-03).

NASA's Goddard Space Flight Center has developed a widely applicable computer assisted management simulation exercise. GREMEX (for Goddard Research and Engineering Management Exercise) simulates the functioning of an R&D project environment, including human and chance factors. It is used to train project managers to manipulate the various factors of time, cost, and technical performance to optimize completion of project objectives. GREMEX filled an important need in providing the necessary experience required for R&D decision-making. Although management gaming simulations existed, GREMEX extended the use of gaming to the accomplishment of scientific and technological programs having pre-established optimum goals.

o Generalized material handling simulation system (11-09-04).

A Pittsburgh University researcher, under NASA sponsorship, developed a generalized material handling simulation system (MHSS) applicable to the majority of industrial material handling systems. The computer program of the model simulates the interactions of the handling equipment with the requirement for material to be moved, thus permitting analysis leading to optimization of the system. The MHSS simulation contributed to the development of general-purpose simulation systems have relatively wide applicability to technological systems.

o Computer program control procedures (11-09-05).

Bellcomm, Incorporated, a NASA contract research center has developed formal, optimized techniques for management control to insure the production and delivery of working, usable computer programs. The techniques cover control of programs developed by outside contractors. The Bellcomm effort illustrated the use of simulation in developing improved management control techniques.

o Management forecasting and control system (11-09-06).

In 1965, NASA Headquarters published a description of its FAME (Forecasts and Appraisals for Management Evaluation) system. FAME, developed for the Apollo Program, uses four mathematical models as predictive tools to forecast trends in the weight of a spacecraft under development.

The entire forecasting, evaluating, and reporting system is potentially quite valuable to commercial industries for use in cost control, sales forecasting, financial reporting and prediction of variance from optimum program goals. FAME represents an ingenious application of statistical techniques for optimization and prediction to the needs of management information and control. As a demonstration of the wide applicability of computer simulation to management problems, FAME has contributed to the advancement of simulation techniques.

o Design R&D policy (11-09-07).

A laboratory simulation of research and development policy using the methodology of industrial dynamics was developed at MIT under NASA sponsorship. The computer simulation studies of the R&D model led to conclusions concerning the relative importance and desirability of management and government policies, and suggested ways to optimize such R&D policies. The MIT work contributed to the advancement of simulation techniques by demonstrating their applicability to complex technological policy optimization.

o Development of an optimized personnel utilization model (11-09-08).

Jet Propulsion Laboratory officials, faced with the dual management problem of manpower ceilings and employee turnover, found themselves unable to maintain a full effective complement of personnel. JPL's Advanced Development Studies group designed a computer simulation which found an optimum solution, allowing the personnel office to overhire yet stay below the hiring ceiling with 95 percent probability. This technique now has been applied by the airlines to permit systematic overbooking of passenger space yet leave a very small probability of an actual seat shortage.

### Simulation of Physical Environments (11-12)

For several years, firms have simulated certain aspects of the physical environment during product testing. Certain environmental conditions (heat, humidity, impact, noise, and vibration) are relatively easy to reproduce or to simulate. The space program, however, brought unusual challenges. It was necessary to test spacecraft and train astronauts in conditions simulating the hostility of outer space, in environments never before experienced, much less simulated. Among the numerous NASA contributions to the field of environmental simulation are the following.

### o Zero gravity simulation (11-12-01).

A scientist at NASA/MSFC invented an apparatus for simulating zero gravity conditions to permit determination of the effects of acoustic radiation pressure on liquids in a gravity less environment. Marshall also conducted a study in 1969 on the use of fighter aircraft, flying a descending parabola, to produce a zero g environment for space manufacturing experiments. A water immersion facility, also at MSFC, balances gravitational forces with buoyant forces to produce a zero g environment in which astronauts can simulate space travel. These contributions illustrated the ability to simulate environmental conditions which cannot exist on the earth.

### o Centrifuge mounted motion simulator (11-12-02).

NASA/ARC personnel invented a centrifugemounted simulator capable of simulating inflight
motions of spacecraft. A manned cockpit on the
simulator is able to perform reversible circular
horizontal movement, linear vertical motion and
angular translation around axes of roll, pitch,
and yaw. This simulation system was a significant
advance in the technique of environmental simulation
because of the extensive variety of dynamic motions
which could be simulated simultaneously.

#### o Spacecraft environment simulator (11-12-03).

The Jet Propulsion Laboratory developed a 10-foot and later a 25-foot space simulator, designed for environmental testing of unmanned spacecraft under simulated interplanetary conditions of extreme cold, high vacuum, and intense solar radiation. These simulators demonstrated that full-scale spacecraft could undergo final testing under simulated environmental conditions of outer space.

## Simulation of Flight Operations (11-10)

Simulation of flight operations had its rather primitive beginnings with the World War II-era pilot training devices which simulated airplane control responses. Flight simulators have been used since then for airline pilot training and have become exceedingly realistic.

Yet it was the space program, particularly NASA's Apollo program, which made the greatest use of simulation. John McLeod stated in Simulation (July, 1970, p. 5):

All who have any understanding of just how great an accomplishment [the moon landing] was agree that it could not have been done had it not been for simulation, the development and use of computer models of real-universe systems.

Simulation permeated and guided the program from concept through realization. It was used to make feasibility studies of competing overall concepts, to design systems and evaluate components, to train astronauts and ground support teams, and to evaluate both system and personnel performance . . .

Among the many contributions by NASA to flight operations simulation, the following are representative.

o Development of lunar landing simulations (11-10-01).

NASA/LaRC designed numerous lunar landing simulators to help develop astronaut flight procedures. These included approach and descent guidance procedures and lunar escape systems. Bell Aerosystems built for NASA a lunar landing training vehicle for astronaut training which simulated, in earth flight, the handling characteristics of the lunar module, in an environment with 1/6 the gravitational pull and no atmosphere. These simulations contributed to a substantial advancement in the development of simulators for man-machine interaction.

o Interplanetary guidance simulator (11-10-02).

General Motors developed for NASA a digital computer program that simulates the flight of a lifting-type vehicle during atmospheric entry.

The simulation formed the basis for a performance assessment program for aided-inertial entry guidance systems. The GE work contributed to the advancement of all-digital computer simulation systems.

o Space station mission simulation model (11-10-03).

Simulation specialists at NASA/LaRC developed in 1967 a set of digital programs for simulation of manned space station missions. The programs were used as analytical tools for design analysis and for evaluation of such factors as subsystem capability, crew size and skill mix, logistics system capabilities, and mission planning requirements. The space station simulation model demonstrated some considerations which have proven to be of importance in the construction of complex simulation models.

#### TELEMETRY SYSTEMS (12)

Telemetry is the technical discipline which deals with the presentation of measured data at a location remote from the source of the data and involves the following functions: (1) generation of a signal which measures the pertinent physical variables and is in a suitable form for transmission; (2) transmission of the information to the remote location; and (3) conversion of the data into a form appropriate for display, recording, or application to further data-processing equipment. In addition, the field of telemetry is becoming increasingly concerned with exploiting and analyzing the acquired data with a computer to make necessary adjustments in systems which are remote from central control points. Significant developments in the field, as identified by technological leaders in telemetry, are shown in Table 12.

Aerospace research would have been severely limited without the advancements in telemetry technology which were developed over the past ten years. Satellite experiments are activated and data collected through telemetry systems. Biomedical information is acquired in a similar manner. Space vehicle course corrections, orientation, deployment of antennae, and other apendages are all controlled from the ground with the telemetry equipment.

The major limiting factors in aerospace telemetry systems have been, and still are, limitations on available power to operate the systems and available space. As a result, most of the technical effort dealing with telemetry systems for space applications has been directed at increasing the efficiency of the systems. Information handling capacity, for example, has been increased by: compressing the data prior to transmission, increasing the transmission speed, transmitting a number of signals over the same channel, and using coding systems to minimize transmission of incorrect and superfluous data.

Many of the advances in aerospace telemetry systems would not have been feasible without recent advances in the technology of microelectronics. By using integrated circuits it was possible to create small, sophisticated systems which operate on very little power, and provide the additional benefits of reduced cost and increased reliability.

With increasing emphasis on automation and sophisticated process control in industry, there has been a resultant increasing dependence on telemetry systems and techniques for collecting and transmitting useable data, such as temperature, pressure, flow rates and volume, to remote process control centers. With such information, for example, a process control device, a railroad dispatcher, or a traffic controller, (either human or electronic) now can regulate complex processes or systems.

Transmission of information in most industrial activities is over telephone lines or over lines established specifically for transmitting such data. Since most nonaerospace applications do not have the same limitations of available space, much of the technology developed through aerospace telemetry research will not find immediate use. Without the power restrictions of space, more practical methods are available to increase the information handling capacity of a telemetry system. However, the commercial potential of space communications is developing so rapidly that aerospace developed telemetry technology should be finding large-scale use.

The virtually untapped industrial telemetry markets should experience significant growth over the next 5 to 10 years. Some of the areas being considered are: monitoring industry housekeeping functions such as air conditioning or plant environment; telemedicine for monitoring patients miles from a medical center for monitoring intensive-care patients in hospitals; pollution control and surveillance; or monitoring hydroelectric power.

Communications satellites will play an increasing role in sharing the 100 to 200 times increased communications traffic burden anticipated by the year 2000. Much of this increase will be in the form of data transmitted from computer to computer, or video signals for entertainment, teleconferencing, or instruction. NASA has had a significant cooperative involvement with all of the agencies, such as the COMSTAT Corporation, the International Telecommunications Union, the United Nations Direct Broadcast Working Group, and the Office of Telecommunications Policy, who are concerned with the problems of communications satellites.

# Computers for Satellite Control (12-06)

Computers have played a significant role in satellite control in all of the aerospace missions and have been necessary to control orientation, power systems, failsafe systems, course-correction maneuvers, data formatting, status reporting, analyzing scheduling messages for conflict, switch setting, and link verification.

A typical sequence of events is exemplified by the orientation problem. Optical and/or thermal sensors will sense that the vehicle is not properly orientated for optimum conditions. The analog signal from the sensor which could be a variation of current, voltage, or frequency, is "conditioned and normalized" or converted into a dc voltage ranging from 0 to 5 volts. Before transmitting the information over the RF link, it is encoded in some manner to minimize errors that might be introduced by transmission. Since a large number of parameters are usually transmitted over the same channel multiplexing techniques are used. At the receiving end the signal is demultiplexed to separate it from the other signals, the raw data is then decoded, and is then

ready for processing for computation. Extent of orientation is then calculated and steps to correct the condition are determined. The command information is then encoded and multiplexed for transmission to the vehicle which in turn must demultiplex and decode to effectively activate the orientation rockets. With the use of computers this whole sequence takes place in a matter of minutes and would be an improbable manual task. With the advent of sophisticated integrated circuits it is now possible to produce small, highly reliable computers which perform the above sequence on-board the vehicle, with the ground systems used only for periodic verification and back-up.

The technique described above is already used, and will probably find increasing use, in a variety of polyvariable interrelated processes where control must be maintained at a point remote from the individual processes. In the industrial case, the data link can be "hardwire" (telephone lines, etc.) microwave or other RF, or computer tape which is physically transported to the control station. Honeywell has introduced automation systems which can control up to 3,900 remote points, monitoring such process variables as temperature, pressure, flow, and humidity, as well as a large number of condition variables such as on-off, slow-fast, day-night, and manual-automatic. These systems are being used in modern buildings for automated building services to control air conditioning units, furnaces, elevators, etc.

NASA had a significant interest in computers for satellite control in that manual analysis and calculations of data for space vehicle control could have severely limited the space program. It appears that NASA efforts to advance this field, helped make scientific computers available long before they would have been through the normal evolutionary cycle. In addition, NASA's efforts to develop a scientific satellite network, including techniques for formatting, scheduling, status reporting, and link verification created a requirement for equipment to transmit taped data via telephone, which should find increasing use in commercial data-transmission activities.

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o Developed a scientific satellite network called STADAC (station data acquisition and control) which included techniques for formatting, scheduling, status reporting, link verification (12-06-01).

In 1965 NASA/GSFC began a program to develop the system and hardware to perform the following functions: data formatting; scheduling including receiving messages, messaging for conflicts, alarming operators; equipment status reporting; and link verification. This system will be operational in the beginning of 1973 and will be improved in that it will be able to accept telephone data. The new system will substantially reduce the costs and time of having to physically ship taped data. The primary user of the development will be the aerospace community. It appears to have some potential for use by airlines for flight scheduling.

#### TABLE 12

#### SIGNIFICANT DEVELOPMENTS IN TELEMETRY (12)\*

- 1. Application of space communication technology to commercial sectors (12-01)
- 2. Automatic equalization of digital pulses (12-02)
- 3. Coding advances (12-03)
- 4. Communication satellite system (12-04)
- \*\*5. Commutators (12-05)
- \*\*6. Computers for satellite control (12-06)
- \*\*7. Data compression (12-07)
  - 8. Direct digital data transmission in pulse code modulation systems (12-08)
  - 9. Earth resources satellite (12-09)
- 10. Electromagnetic conductivity measurements of earth resources (12-10)
- \*\*11. Error correction codes (12-11)
- \*\*12. Frequency sharing (12-12)
  - 13. Growth of pulse code modulation systems (12-13)
  - 14. Hardened communication systems (12-14)
- \*\*15. High speed data transmission via satellite (12-15)
  - 16. High speed printer for mobile communication (12-16)
- \*\*17. Increased information capacity in telecommunications technology (12-17)
  - 18. Inter Monitory Pulses satellites (12-18)
  - 19. Optical data processing (12-19)
  - 20. Partial response signalling (12-20)
- \*\*21. Phase locked loop systems (12-21)
  - 22. Remote visual display terminals (12-22)
  - 23. Statistical multiplexing in telecommunications (12-23)
  - 24. Study of man-made and atmospheric noise at assigned satellite frequencies (12-24)

- 25. Transfer of semiconductor technology to the consumer market (12-25)
- 26. Use of memories in pulse code modulation systems (12-26)
- \*\*27. Use of solid state devices in telemetry systems (12-27)

<sup>\*</sup>As identified by technological leaders in the field of microwave systems.

<sup>\*\*</sup>Developments selected for identification of NASA contributions.

o Developed equipment to transmit and receive taped data by telephone (12-06-02).

In order to develop a scientific satellite network (see NASA contribution 12-06-01, above) equipment was developed to transmit and receive large quantities of scientific data via telephone. Prior to this effort it was necessary to physically transport data by air, by car, by messenger to NASA/GSFC for processing. The new method of transporting the data to Goddard by telephone not only will reduce the transport time but will save in the order of \$1 million per year.

o Assisted in the development and early support of the scientific computer industry (12-06-03).

As part of the effort to develop guidance and control systems for deep-space vehicles, such as in the Mariner missions, JPL initiated development of computers capable of computing the proper adjustments necessary to keep a satellite on course from the many variables affecting its course. It appears that this effort was instrumental in bringing about and advancing the state of the scientific computer industry at a more rapid rate than it would have been developed in the normal evolutionary cycle. The scientific computer industry is very rapidly assuming a significant proportion of the electronic computing equipment industry with estimated sales of \$4 billion per year. The NASA contribution appears to have had a very high impact on the rapid development of this industry and should have a very high overall impact.

## Data Compression (12-07)

Data compression is one of the many techniques which has been explored and now are used to increase the efficiency of the transmission of the massive and growing quantities of scientific data to central computers for immediate processing or storage banks for later use. The primary function of data compression systems is to minimize the transmission of redundant data. In the more complex method this is done by transforming recorded data into another form prior to transmission, which is then decoded at the processing center for computation. In the simplest type of data compression system, only data that is different from previously transmitted data is transmitted.

Advancements in data compression technology were heavily dependent on advancements in integrated circuit technology, which were necessary for creating the sophisticated systems needed for data compression. Rapid advances in many regions of data transmission are necessary to accommodate an accelerating number of applications. Data compression technology which offers a method for reducing data by eliminating unnecessary data prior to transmission can decrease the burden on already overloaded microwave and hardwire data transmission links.

NASA's interest in data compression technology has been prompted by the need to transmit great quantities of data over a limited number of satellite-to-earth channels and with a minimum amount of power. All methods which increased transmission efficiencies had to be exploited. Many of the advancements in data compression developed for space telemetry systems are now being employed for commercial long distance data transmission systems.

o Improved data relaying efficiency by developing techniques for on-line pre-processing of data from planetary radar systems (12-07-01).

As part of its effort to increase the efficiency of transmission links in the Mariner satellite programs, JPL developed a technique for preprocessing data prior to transmission. The spacecraft system encodes raw binary data into a comma-free biorthogonal code that antipodally modulates a square-wave subcarrier. In turn, this subcarrier phase-modulates the downlink carrier. Since separate signals are not required for subcarrier, work, or symbol synchronization, all of the transmitted sideband power is available for data transmission. Major users of this NASA-developed technique will be scientists who have a need to determine signal significance in noisy data. While this contribution does not have major economic consequences, it will have a moderate to high scientific impact since it allows the acquisition of new scientific information.

o Developed highly reliable transistors for data compression systems (12-07-02).

In 1964, NASA found they were unable to effectively exploit the potential of metal-oxide semiconductors (MOS) devices for data compression functions on small scientific satellites because of their lack of reliability. NASA/GSFC first attempted to produce such devices in their own laboratories, and then contracted with American Micro-Systems, Inc. to produce them. From this effort, the technology was developed which could produce reliable MOS integrated circuit devices for data compression. This represents

a significant advance in data compression technology in that it helped established a source for highly reliable devices which in turn helped establish a more competitive market by adding a new major integrated circuits company. For a fuller description, see NASA contribution 05-08-03 in the field of integrated circuits.

## Communication Codes (12-11)

As discussed in the section on data compression (12-07), extensive efforts have been employed to increase the efficiency of the increasingly over-burdened data transmission channels. Early in the 1960's block coding techniques were used to improve the communication of analog data. In 1962 convolutional codes were created by Shannon at MIT; however, the advantages of convolutional codes over block codes were not fully realized until after 1965. Such codes, including sequential, maximum likelihood, and feedback, offer techniques for encoding data to minimize errors that might be introduced by the transmission. These error detection and correcting techniques are being used in satellite and terrestrial communication channels to increase the efficiency of available channels. To date, efficiency improvements of at least one-half an order of magnitude over what was possible early in the 1960's have been achieved.

Convolutional communication coding techniques should find increased use as demands on existing data transmission channels increase. Industrial and commercial applications of telemetry and data transmission for process control, and computer analysis are just now being appreciated, and should become significant activities in the latter half of this decade.

The availability of integrated circuits in the mid 1960's was a prerequisite for the practical application of convolutional codes for satellites. The first satellites to use such codes were the Lincoln Labs experimental satellite and the Pioneer 9. JPL was responsible for developing the encoders, software, and hardware for the first operational coding systems for space telemetry.

o Developed sensitive subcarrier modulation and synchronization equipment necessary for coded communications (12-11-01).

Since commercially available subcarrier modulation and synchronization equipment was not sensitive enough to meet the signal-to-noise requirements of the Mariner 69 program for coded communications, JPL extensively investigated long-constraint and short-constraint convolutional codes

and data formats and developed the subcarrier modulation and synchronization hardware needed for the Mariner mission. The equipment was built by Motorola under NASA contract. There is obvious COMSAT interest in this effort.

o Introduced the use of coding systems in space telemetry (12-11-02).

Encoders, software, and hardware for the Pioneer IX program were developed by NASA/ARC. providing the first operational use of coding systems in space telemetry. Coding was introduced into the Pioneer space communications telemetry link to permit more information to be transmitted at a given power within a maximum allowable error rate. If a fixed amount of power is to transmit a given information rate in an otherwise fixed communications system, the introduction of additional data reduces the available power per transmitted data bit. For this effort, the developer, Dale Lumb of Ames, received the 1970 National Telemetry Conference "Man of the Year" Award. To date the system has found application in the Pioneer/Jupiter program, the Helioz (German) program; the NASA/GSFC IMP Series, and COMSAT.

### Frequency Sharing (12-12)

The massive amount of information sent over transmission links requires that each link, whether a solid telephone wire or a microwave link, carry many messages simultaneously. The process of two or more communication channels sharing a common propagation path is called multiplexing and can be accomplished by: (1) frequency division, where the signals are arranged along a frequency scale; (2) time division, where the signals are arranged in time sequence; or (3) a combination of the two. In the approach using frequency division, each channel is assigned to a specific frequency band with the frequency bank corresponding to a sideband of a sinusoidal carrier frequency. At the receiving end, the channels are separated by filtering and then demodulated to recover the original information. Using the time-division approach, a number of messages are sent over the common transmission link by allocating different time intervals in sequence for the transmission of each message and by rapidly switching from one to the The receiver also has a switching device which must be synchronized with the transmitter to separate the messages properly.

Frequency-sharing techniques have made it possible to radically increase the efficiency of transmission media by allowing the transmission of hundreds of messages over individual links simultaneously. This is becoming increasingly important because of the communications explosion which will result from the need to transmit large amounts of data to and from computers in the coming decade.

NASA has had a very high interest in frequency and time-sharing transmission techniques, since it allows a number of activities to use a single transmission link to convey substantial quantities of information, thereby reducing the need for additional power sources and transmitters in a vehicle which has space and volume limitations.

o Eliminated the need for a number of transmitters to transmit a data link with multiple channels by using frequency-sharing and time-sharing techniques (12-12-01).

To overcome the problem of needing individual transmitters to transmit a data link with multiple channels to each of two satellites in close proximation to one another, a system was developed at JPL which employs frequency and time-sharing techniques. With this approach it is now possible to transmit many channels with a single transmitter. The system will be used in the next Mars flights. The inventor has applied for a patent for this development, and is avoiding publication until clearance is received. This system will have a significant impact in future space missions and will reduce the number of costly and power-consuming transmitters for multi-channel data links.

# High-Speed Data Transmission via Satellite (12-15)

Higher speed ground-to-satellite-to-ground information handling capabilities will be required to handle the increasing abundance of data that will have to be transmitted in the coming years. Transmission rates approaching 50 megahertz are being considered. Time sharing and special networks for "storage and forward" are being used to increase channel capacity. Advances in integrated circuits technology and microwave solid state devices are making it possible to develop the sophisticated circuits needed to increase the information handling rates of satellite data transmission links.

High speed data transmission channels via satellite and the resultant ability to handle more information in a given time period should result in reduced long distance telephone communications costs; an effective global communications system; and an improved navigational satellite system.

Both NASA/GSFC and the JPL have conducted research into using and improving data transmission links via satellite for handling data from aerospace experiments, for military communications, and to increase the data handling capabilities for commercial activities. Their efforts have made available both procedures and hardware for increasing the information handling capacities of satellite data links.

o Developed a practical, portable automatic, satelliteto-ground, picture transmission system (12-15-01).

NASA developed a weather satellite which continually circles the earth taking photographs, and then recording and transmitting these photographs to earth. To make the information readily available, a simple, low-cost "weather satellite picture receiving station" was developed at NASA/ GSFC using standard off-the-shelf components which can be built for \$500 and purchased for \$5000. Many of these units have been built as high school science projects, and many are in use by a number of industries and agencies throughout the world. To date more than 10,000 copies of the NASA publication SP-5080 have been sent to interested persons all over the world. The importance of this contribution is in the fact that it enables a large number of people in non-space related activities to exploit the capabilities of satellites.

o Developed a system to collect data from a remote moving transmitter via satellite (12-15-02).

NASA/GSFC developed the procedures and hardware to interrogate, record, and locate scientific information from a randomly moving instrumentated platform anywhere on the surface of the earth or above the surface. Such satellites make it possible to collect weather information from remote areas; track animals, birds, and fish; or locate ships, aircraft, ground vehicles, or persons, anywhere in the world. The ultimate impact of this contribution will be felt across many human activities.

# Phase-Locked-Loop-Systems (12-21)

A phase-lock-loop is an electronic servo-mechanism that operates as a coherent detector by continuously correcting the frequency of its local oscillator according to a measurement of the error between the phase of the incoming signal and that of its local oscillator. Although

automatic phase control systems were first discussed in the literature in the late 1920's and early 1930's the first serious application of the concept began as a horizontal-line synchronizing device for television in the late 1940's. Shortly thereafter, the use of a phase-locked-loop system as a tracking filter for a missile beacon was demonstrated, and the loop parameters for such a use was specified. The first analysis, including the effects of noise, appeared in 1955.

Phase-locked-loop concepts have been widely used for a variety of applications; however, due to the cost and complexity of such concepts in discrete systems design, the use of phase-locked-loop systems has been limited to precision applications requiring a high degree of noise immunity and very narrow band widths. With the advent of integrated circuits technology, it became possible to create phase-locked-loop circuits at a cost that makes them attractive for a variety of applications such as demodulators for commercial FM receivers; commercial TV sound IF and demodulators; tuned AM detectors; FM multiplex telemetry receivers; and frequency-shift-key telegraph receivers.

Phase-locked-loop systems have received much attention because of the many jobs they can do well, and should have a substantial impact on the consumer, industrial, and military electronics markets in the 1970's; a market which is anticipated to total \$32.2 billion by 1974.\*

Phase-locked-loop systems have been used by NASA for a variety of telemetry and aerospace applications where noise has been a factor and where it has been desirable to use very narrow bandwidths. Many of the individuals who have played a key role in the advancement of phase-locked-loop technology are now, or have been, associated with NASA.

o Developed a diversity-locked-demodulator which permitted receiving signals of opposite polarization from a singular satellite simultaneously (12-21-01).

Researchers at NASA/GSFC and through a contract with the Electronics Division of General Dynamics, developed a system called a diversity-locked-loop-demodulator, which allows the simultaneous reception of two signals with opposite polarities from a

<sup>\*</sup>Electronics. McGraw-Hill "U. S. Markets 1971 Forecast." January 4, 1971.

satellite. This system is now used in basic data receivers for aerospace work. Without the NASA requirement this demodulator would probably not have been developed. While present applications are limited to military and aerospace programs, it appears that applications in the commercial aviation industry will develop. A NASA patent has been granted to cover this device and several awards have been made. A new private business firm, called Electrac Corporation, was founded to exploit this technology and produce phase-locked-loop systems based on the NASA-developed concepts.

o Applied phase-locked-loop circuits in narrow bank communication systems to minimize noise to affect coherent communications, which was a prerequisite to space telemetry systems (12-21-02).

Phase-locked-loop circuits were first used in the Jupiter missile program as a tracking filter for the missile beacon. The radio inertial guidance systems required minimum noise in narrow band channels to affect coherent communications. modulation provided the most efficient method for telemetering coherent communications. Multiplexing has also been improved through the use of phaselocked-loop circuits because it allows a substantial increase in channel separation and a noise reduction of at least 3 decibels. Although this contribution has not as yet had a significant nonaerospace impact, the advent of integrated circuit phase-locked-loop components make this NASA-developed approach attractive for a growing number of commercial applications and an overall moderate nonspace impact is estimated.

## Use of Solid State Devices in Telemetry Systems (12-27)

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As discussed earlier, aerospace research would have been severely limited without the technological advances that have occured in the field of telemetry. Further, these aerospace telemetry systems would not have been feasible without parallel advancements that occured in semiconductor technology. Within the past five years, advances in integrated circuits technology have resulted in reliable miniature circuits for use in space and terrestrial telemetry systems. Small size, low-cost, low power requirements and increased reliability, make integrated circuit components ideal for remote airborne, airdelivered, or man-carried telemetry monitoring systems.

The availability of integrated circuits has enabled manufacturers of space and terrestrial telemetry systems to construct smaller, better quality, higher efficiency systems at substantially reduced costs. These systems are just now finding an increasing number of applications in the rapidly expanding industrial telemetry fields. For example, General Electric Company's aircraft equipment division has introduced miniature telemetry components including 3 watt S-band transmitters, 3 watt L-bank transmitters, and all solid state regulated dc-dc converters. These components are being aimed at a market whose estimated size is \$50 million a year.

The necessity of telemetry systems for aerospace experiments and missions required a considerable effort on the part of NASA to develop reliable solid state components for such systems. A number of the components developed for NASA missions are directly applicable for nonaerospace systems, for example, the integrated circuit developed at NASA/GSFC for electronically focussing microwave antennae; the telemedicine components developed at NASA/ARC; and multiplexers developed for the Apollo programs.

o Demonstrated use of solid state devices in space telemetry systems (12-27-01).

The use of solid state devices in space telemetry was demonstrated in a transmitter built under contract for NASA/GSFC for the NRL/Vanguard project. Complex discrete systems were continually replaced in a variety of programs by more sophisticated integrated circuit components which resulted in lighter weight, smaller volume systems. The NASA programs produced devices such as the following, among others: phase shifters to electronically focus microwave antennae; highly reliable MOS transistors for data compression systems for small scientific satellites; transformer and inductorless communications receivers; and metallized large-scale arrays of logic gates. In the use of solid state devices in telemetry systems, both DOD and NASA have been the major initiators, and therefore have had a significant impact on the advancement of the technology and the circuits which have been produced will find commercial applications.

o Developed and built a solid state transmitter for space telemetry systems (12-27-02).

Solid state transmitters, built in 1965 under a NASA/GSFC contract with American Electronics, Inc. were used for VHF band space telemetry systems.

(See NASA contribution 12-27-01, above). These transmitters are now only used for VHF, having been replaced for most applications by S-band transmitters. However, when introduced they represented a new approach using solid state components to UHF-band telemetry.

APPENDIX A
BIBLIOGRAPHY

#### CRYOGENICS

- Aerojet-General Corporation. Cryogenic Tensile Properties of Selected

  Materials. Washington, D.C.: U. S. Government Printing Office, 1964.
- Atomics International. <u>Investigation of Supercurrent Instabilities in Type II Superconductors Final Report</u>. Washington, D.C.: U. S. Government Printing Office, 1967.
- Bardeen, J., L. N. Copper, and J. R. Schrieffer. "Microscopic Theory of Superconductivity," Physics Review, 106, (April 1, 1957), pp. 162-164.
- . "Theory of Superconductivity," Physics Review, 108, (December 1, 1957), pp. 1175-1204.
- Birmingham, B. W. Superconductivity Program of the Atomic Energy Program. Washington, D.C.: Atomic Energy Commission, June 1967.
- Black, I. A. Basic Investigation of Multi-layer Insulation Systems.
  Washington, D.C.: National Aeronautics and Space Administration,
  Technology Utilization Division, October 30, 1964.
- Buchhold, T. A. "Applications of Superconductivity," <u>Scientific</u>
  <u>American</u>, March 1960, pp. 74-82.
- Bureau of the Census. Current Industrial Reports. Series M 28H (60-70)-13, Supplement 2. Washington, D.C.: U.S. Department of Commerce, 1970.
- Burkhalter, J. E., and V. L. Glasgow. Fluid Behavior Patterns Found in Subscale Geysering Study. Huntsville, Alabama: Marshall Space Flight Center, 1967.
- Clark, A. F. Technique for Predicting the Thermal Expansion Coefficients of Cryogenic Metallic Alloys. Cleveland, Ohio: Space Nuclear Pro-Pulsion Office, 1969.
- Cohen, M. H., ed. <u>Superconductivity in Science and Technology</u>. Chicago: University of Chicago Press, 1968.
- Coles, W. D., J. C. Laurence, and G. V. Brown. <u>Cryogenic and Superconducting Magnet Research at the NASA Lewis Research Center</u>. Washington, D.C.: U. S. Government Printing Office, 1969.
- "Cooling Down Electricity Cost," <u>Industrial Research</u> April 1969, pp. V6-V8.

- "Cryogenics: Key to Efficient Linear Accelerator," <u>Industrial Research</u>, January 1969, pp. V1 - V2.
- Essman, U. and H. Trauble. "The Magnetic Structure of Superconductors," Scientific American, April 1965, pp. 75-84.
- Flynn, T. M. and B. W. Birmingham. "Cryogenics and National Goals,"

  Advances in Cryogenic Engineering, 14 (1968).
- the United States. Boulder, Colorado: Institute for Basic Standards, National Bureau of Standards.
- Glaser, P. E. et al. Thermal Insulation Systems: A Survey. Washington, D.C.: National Aeronautics and Space Administration, 1967.
- . Thermal Protection Systems for Liquid Hydrogen Tanks. Washington, D.C.: National Aeronautics and Space Administration, 1962.
- Green, E. F. Fatigue Properties of Sheet, Bar, and Cast Metallic Materials for Cryogenic Applications. Huntsville, Alabama: Marshall Space Flight Center, 1970.
- Hoag, Ethan. Experimental Investigation of Advanced Superconducting Magnets. Superconducting Strip and Its Use in Magnets. Washington, D.C.: U.S. Government Printing Office, 1964.
- Hudson, W. R. The Mixed State of Superconductors. Washington, D.C.:
  National Aeronautics and Space Administration, 1970.
- Hust, J. G. et al. <u>High Precision Cryogenic Conductivity Standards</u>. Cleveland, Ohio: Space Nuclear Propulsion Office, 1970.
- Institute of Gas Technology. Proceedings of the First International Conference on LNG Liquified Natural Gas. Chicago: Illinois Institute of Technology, April 1968.
- Kahn, Aman. "LNG-2 Report: Growing World Trade, New Transport
  Developments, New Energy Philosophies, "Pipeline and Gas Journal,
  January 1971, pp. 113-115.
- Kamper, R. A. et al. "Some Applications of the Josephson Effect,"

  NBS Technical Note 81, U. S. Department of Commerce [Washington:
  U. S. Government Printing Office], October 1969.
- Kolm, H. H. and A. J. Freeman. "Intense Magnetic Fields," Scientific American, April 1965, pp. 66-76.

- Kunzler, J. E., and Tanenbaum. "Superconducting Magnets," Scientific American, June 1962, pp. 60-67
- , E. Buehler, F. X. L. Hsu, and J. H. Wernick. "Superconductivity in Nb<sub>3</sub>Sn at High Current Density in a Magnetic Field of 88 K gauss," Physics Review Letters, 6 (February 1, 1961), pp. 89-91.
- Langenberg, D. N. et al. "The Josephson Effects," Scientific American, May 1966, 30-39.
- Laurence, James C. <u>High-Field Electromagnets at NASA Lewis Research</u>
  <u>Center.</u> Washington, D.C.: National Aeronautics and Space
  Administration, 1968.
- . Superconductive Magnets. Washington, D.C.: National Aeronautics and Space Administration, 1967.
- D.C.: National Aeronautics and Space Administration, 1967.
- Aeronautics and Space Administration, 1965.
- Lewis Research Center. Aerospace Related Technology Conference for Industry and Commerce. Cleveland, Ohio: Lewis Research Center, 1967
- . Conference on Selected Technology for the Petroleum Industry,

  December 8-9, 1965. Washington, D.C.: National Aeronautics and

  Space Administration, 1966.
- . Hydrogen Safety Manual. Washington, D.C.: National Aeronautics and Space Administration, 1968.
- . A Technique for Joining and Sealing Dissimilar Materials.

  Washington, D.C.: National Aeronautics and Space Administration,
  1965
- Little, Arthur D., Inc. Advanced Studies on Multi-Layer Insulation

  Systems Final Report. Washington, D.C.: U. S. Government Printing

  Office, 1966
- "LNG: Bright New Star on the Energy Scene," Pipeline and Gas Journal, 197, (June 1970), pp. 39-43.
- "LNG Potential in the Seventies," <u>Petroleum Press Service</u>, December 1970, pp. 451-454.

- Lofstrom, J. "Uses of LNG for the Future," <u>Pipeline Industry</u>, 29 (October 1969).
- McCarty, R. D. Thermodynamic Properties of Liquid-Vapor Parahydrogen and Liquid-Vapor Oxygen. Washington, D.C.: U.S. Government Printing Office, 1965.
- Marshall Space Flight Center, Research Achievements Review. Vol. II.

  Report #7. Huntsville, Alabama: Marshall Space Flight Center,
  1967.
- Research Achievements Review. Volume 2. Cryogenic Technology
  Research at Marshall Space Flight Center. Washington, D.C.: U.S.
  Government Printing Office, 1967.
- Mathes, K. N. and S. H. Minnich. Study Made of Dielectric Properties of Promising Materials for Cryogenic Capacitors. Huntsville, Alabama: Marshall Space Flight Center, 1967.
- Matthias, B. T. et al. "Superconductivity of Nb<sub>3</sub>Sn," <u>Physical Review</u> September 15, 1954, p. 1435.
- Meissner, W., and R. Ochsenfeld. "Ein Neuer Efekt bei Eintritl der Supraleitfahigkeit," Naturwiss enschaften, 21 (1933), pp. 787-788.
- Minnich, S. H., and G. R. Fox "Cryogenic Power Transmission," Cryogenics, June 1969, pp. 165-176.
- Moeller, C. Eugene. NASA Contributions to Development of Special-Purpose Thermocouples. A Survey. Washington, D.C.: U. S. Government Printing Office, 1968.
- NASA Contributions to Significant Developments in Cryogenics; A Survey of NASA Literature. Albuquerque, New Mexico: University of New Mexico, Technology Application Center, 1971.
- Onnes, H. Kamerlingh. "Disappearance of the Electrical Resistance of Mercury at Helium Temperatures," Proc. K. Akad. Wetenschappen, 19 June 23, 1911, pp. 113-115.
- . "Experiments with Liquid Helium. The Electric Resistance.

  VIII. The Sudden Disappearance of the Ordinary Resistance of Tin and Super-Conductive State of Lead," Proc. K. Akad. Wetenschappen, 19, (1914), pp. 673-688
- . "Further Experiments with Liquid Helium, I. The Hall Effect and the Change of Resistance in a Magnetic Field at Low Temperatures. XI. Occurrence of Galvanic Resistance in Super-Conductors, When Brought into a Magnetic Field, at a Threshold Value of the Field," Verslag Akad. Wetenschappen, 22, 1914, pp. 1027-1033.

- Perkins, Porter J., R. P. Dengler, L. R. Niendorf, and G.E. Nies. Self
  Evacuation Multilayer Insulation of Lightweight Prefabricated
  Panels for Cryogenic Storage Tanks. Washington, D.C.: U. S.
  Government Printing Office, 1968.
- Plasmas and Magnetic Fields in Propulsion and Power Research: Conference Proceedings, October 16, 1969. Washington, D.C.: National Aeronautics and Space Administration, 1970.
- Sampson, W. B. et al. "Advances in Superconducting Magnets," Scientific American, March 1967, pp. 115-123
- Scott, R. B. Cryogenic Engineering. New York: D. Van Nostrand Company Inc., 1959.
- Smith, R. V. "Current Developments in Cryo-Medicine," Cryogenics, April 1969, p. 242.
- Sparks, L. L. et al. Cryogenic Thermocouple Calibration Tables. Cleveland, Ohio: Space Nuclear Propulsion Office, 1970.
- Stekly, Z.J., R. Rhome, E. Lucas, B. P. Strauss, and F. DiSalvo

  Advanced Superconducting Magnets Investigation. Washington, D.C.:

  U. S. Government Printing Office, 1968.

egg.

- "Superconducting Power Cables in 12 Years," <u>Industrial Research</u>, August 1969, pp. 19-20.
- Timmerhaus, K. D., ed. Advances in Cryogenic Engineering Vol. 1-15.

  New York: Plenum Press, 1967.
- , R. F. Kamm, and J. D. Bays. "Wither Cryogenics," in <u>International Advances in Cryogenic Engineering</u>. New York: Plenum Press, 1965.
- U. S. Industrial Outlook 1971. Washington, D.C.: U. S. Department of Commerce, Bureau of Domestic Commerce, 1971.
- Vance, R. W. and W. H. Duke, eds. Applied Cryogenic Engineering. New York: John Wiley & Sons, Inc. 1962.
- Wipf, S. L. and C. A. Guderjahn. A Study of Inherently Stable High-Field Superconductors. Summary Report, 25 June, 1968 - 31 July 1970. Washington, D.C.: U. S. Government Printing Office, 1970.

# ELECTROCHEMICAL ENERGY CONVERSION AND STORAGE

- Annual Survey of Manufacturers 1969. Washington, D.C.: U. S. Department of Commerce, Bureau of Domestic Commerce, 1970.
- Austin, L. G. Fuel Cells: A Review of Government Sponsored Research, 1950-1964. Washington, D.C.: National Aeronautics and Space Administration, 1967.
- "Batteries: Decade of Change," Chemical and Engineering News, 48 (December 14, 1970), p. 17.
- Bauer, Paul. <u>Batteries for Space Power Systems</u>. Washington, D.C.: National Aeronautics and Space Administration, 1968.
- "Developments to Watch," <u>Product Engineering</u>, 41 (August 31, 1970), p. 6.
- \_\_\_\_\_, Product Engineering, 42 (January 4, 1971), p.5.
- , Product Engineering, 42 (February 1, 1971), p. 5.
- Product Engineering, 42 (February 15, 1971) p. 5.
- , Product Engineering, 42 (March 15, 1971) p.6.
- Ford, Floyd E. "Use of the Third Electrode and the Recombination Electrode in Spacecraft Batteries," Science and Technology Accomplishments, 1969. Greenbelt, Maryland: Goddard Space Flight Center, 1970.
- \_\_\_\_\_\_, and T. Hennigan. Space Benefits Capsule Summary.

  Greenbelt, Maryland: Goddard Space Flight Center, 1971.
- Greene, A. M. "What's Happening to Fuel Cells?" <u>Iron Age</u>, March 19, 1970.
- Heise, George W., and N. Corey Cahoon. The Primary Battery. Vol. I. New York: John Wiley and Sons, Inc., 1971.
- Howard, John. "The Sleepy Battery Business Wakes Up," New Scientist and Science Journal, 50 (May 27, 1971), pp. 528-530.
- Kordesch, K. U. Hydrogen Air/Lead Battery Hybrid System. Atlantic City, New Jersey: Electrochemical Society, October 1970.
- Mantell, C. L. <u>Batteries and Energy Systems</u>. New York: McGraw-Hill, 1970.

- "Marketing Perspectives," Product Engineering, 42 (February 1, 1971), p. 9.
- NASA Contributions to Significant Developments in Electrochemical

  Energy Conversion and Storage; A Survey of NASA Literature.

  Albuquerque, New Mexico: University of New Mexico, Technology
  Application Center, 1971.
- Specification for Aerospace Nickel-Cadium Storage Cells. Greenbelt, Maryland: Goddard Space Flight Center, March 1971.
- U. S. Army Signal REsearch and Development Laboratory, Power Sources
  Division. Proceedings of the 14th Annual Power Sources
  Conference, 1960. Fort Monmouth, New Jersey: U. S. Army Signal Research and Development Laboratory, 1960.
  - Proceedings of the 15th Annual Power Sources Conference, 1961.

    Proceedings of the 16th Annual Power Sources Conference, 1962.

    Proceedings of the 17th Annual Power Sources Conference, 1963.

    Proceedings of the 18th Annual Power Sources Conference, 1964.

    Proceedings of the 19th Annual Power Sources Conference, 1965.

    Proceedings of the 20th Annual Power Sources Conference, 1966.

    Proceedings of the 21st Annual Power Sources Conference, 1967.

    Proceedings of the 22nd Annual Power Sources Conference, 1968.
  - U. S. Industrial Outlook 1971. Washington, D.C.: U. S. Department of Commerce, Bureau of Domestic Commerce, 1971.

`.`\$

Vielstich, Wolf. Fuel Cells Modern Processes for the Electrochemical Production of Energy. London: John Wiley and Sons, Ltd., 1965.

## HIGH TEMPERATURE CERAMICS

- Clauss, F. J. Engineer's Guide to High-Temperature Materials. Reading, Masachusetts: Addison-Wesley, 1969.
- Coble, R. L. and J. E. Burke. "Sintering in Ceramics." Progress in Ceramic Science, Vol. 3. New York: MacMillan, 1963, pp. 197-249
- Emrich, B. R., and S. D. Brown. "10 New Ways to Fabricate Ceramic Parts," Materials Engineering, 67 (May 1968), pp. 72-77.
- "Engineer's Guide to Structural Ceramics," <u>Materials Engineering</u>, 72 (November 1970), pp. 38-45.
- Fulrath, R. M. "A Critical Compelation of Ceramic Forming Methods, III Hot Forming Processes," American Ceramic Society Bulletin, 43 (1964), pp. 880-885.
- Gilman J. J. "The Nature of Ceramics," <u>Scientific American</u>, 217 (September 1967), p. 113.
- Grimm, N. "See-through Ceramics," Industrial Research, 13 (May 1971), pp. 40-43.
- Kalish, D., and E. V. Clougherty. "High Pressure Hot Pressing Refractory Materials," American Ceramic Society Bulletin, 48 (1969), pp. 570-578.
- Kuxler, D. J. Impact of Changing Technology on Refractories
  Consumption. Washington, D.C.: U.S. Department of Interior,
  Bureau of Mines, 1970
- NASA Contributions to Significant Developments in High-Temperature

  Ceramics; A Survey of NASA Literature. Albuquerque, New
  Mexico: University of New Mexico, Technology Application
  Center, 1970.
- National Academy of Sciences, National Research Council, Division of Engineering, Materials Advisory Board, Committee on Ceramic Processing. Ceramic Processing. Washington, D.C., 1968.
- "Revolution in Magnet Materials Brings New Design Advantages," Product Engineering, 42 (February 15, 1972), pp. 30-33.
- Sanders, W. A. and S. J. Grisaffe. "Hot Pressing of Hafnium Carbide (Melting Point 7,030 F.)," NASA Technical Note D-303.

  Washington, D.C.: National Aeronautics and Space Administration, 1960.

- Tymczak, R. W., C. H. McMurtry, and K. H. Sandmeyer. "High Temperature Ceramics," <u>Industrial Research</u>, 13 (December 1971), pp. 53-55.
- Vasilos, T., and R. M. Spriggs. "Pressure Sintering of Ceramics,"

  <u>Progress in Ceramic Science</u>, Vol. 4, J. E. Burke, ed. New York:

  <u>Pergamon</u>, 1966.
- Wachtman, J. B., Jr. "Mechanical Properties of Ceramics: An Introductory Survey," American Ceramic Society Bulletin, 46 (1967), pp. 756-774.

## HIGH TEMPERATURE METALS

- Aerospace Structural Materials. Washington, D.C.: National Aeronautics and Space Administration, 1969.
- Ault, M. G. "Engineering Mechanics and Materials," Selected Technology for the Electric Power Industry. Washington, D.C.: National Aeronautics and Space Administration, 1968.
- Barth, J. D., and H. O. McIntire. <u>Tungsten Powder Metallurgy</u>.

  Washington, D.C.: National Aeronautics and Space Administration, 1965.
- Brown, W. F. Review of Development in Plane Strain Fracture Toughness

  Testing. Philadelphia: American Society for Testing Materials,
  1970.
- High Strength Metallic Materials. Philadelphia: American Society for Testing Materials, 1967.
- Clauss, F. J. Engineer's Guide to High-Temperature Materials.

  Reading, Massachusetts: Addison-Wesley, 1969.
- Freche, J. C., and R. W. Hall. "NASA Programs for Development of High-Temperature Alloys for Advanced Engines," <u>Journal of Aircraft</u>, <u>6</u> (Septmeber-October 1969), pp. 424-431.
- Hebeisen, M. C., et al. Vacuum Melting and Casting of Superalloys. Washington, D.C.: National Aeronautics and Space Administration, 1971.
- Khol, R. "New Moves in Metallurgy," Machine Design, 42 (February 5, 1970), pp. 97-103.
- Klopp, W. D., P. L. Raffo and W. R. Witzke. "Strengthening of Molybdenum and Tungsten Alloys with HFC," <u>Journal of Metals</u>, 23 (June 1971), pp. 27-38.
- Machlin, I., R. T. Begley, and E. D. Weisert. Refractory Metal Alloys, Metallurgy and Technology. New York: Plenum Press, 1968.
- Manson, S.S. "Design Considerations for Long Life at Elevated Temperatures," Proceedings: Institution of Mechanical Engineering, 178, Part 3-A (1963-64), pp. 1-27.
- washington, D.C.: National Aeronautics and Space Administration, 1964, pp. 33-63.

- Manson, S.S., and G. Halford. "A Method of Estimating High Temperature Low Cycle Fatique Behavior of Materials," presented at the Symposium on Thermal and High Strain Fatique, Sponsored by the Institute of Metals, June 1967, London.
- \_\_\_\_\_\_, G. Halford, and D. A. Spera. "The Role of Creep in High-Temperature Low-Cycle Fatique." Preprint submitted for publication in the A.E. Johnson Memorial Volume, January 1969.
- NASA Contributions to Significant Developments in High-Temperature

  Metals; A Survey of NASA Literature. Albuquerque, New Mexico:
  University of New Mexico, Technology Application Center, 1971.
- Piearcey, B. J., and F. L. Ver Snyder. "New Casting Technique Strengthens Turbine Components," SAE Journal, 74 (June 1966), pp. 84-87.
- "Powder Metals Emerge from Black Magic to Become Hot Technology," Product Engineering, 42 (June 1971), pp. 50-54.
- Powdered Steel: A Tough Customer," <u>Business Week</u>, 2157 (January 2, 1971), p. 370.
- Recent Advances in Refractory Alloys for Space Power Systems.

  Washington, D.C.: National Aeronautics and Space Administration, 1970.
- Ridal, K. "Steel From Powder," New Scientist, 48 (November 12, 1970), pp. 325-327.
- Scrawley, J. E., and W. F. Brown, Jr. "Fracture Toughness Testing Methods," Fracture Toughness Testing and Its Applications. Philadelphia: American Society for Testing Materials, 1965, pp. 133-198.
- Sullivan, O. P., M. J. Donachies, Jr., and F. R. Morral. Cobalt-Base Superalloys--1970. Columbus, Ohio: Cobalt Information Center, 1970.
- "Technology Forecast," Metal Progress, 99 (January 1971), pp. 57-84.
- "Technology Forecast: Surveying the 70's," Metal Progress, 97 (January 1970), pp. 17-32.
- Vaccari, J. A. "Five Years of Progress in Aerospace," <u>Materials</u> Engineering, 71 (August 1970), pp. 28-37.
- . "Today's Superalloys: Good; Tomorrow's: Even Better,"

  Materials Engineering, 69 (May 1969), pp. 21-31.

- Ver Snyder, F. L., et al. "Directional Solidification in the Precision Casting of Gas Turbine Parts," Modern Casting, 52 (December 1967), pp. 68-75.
- \_\_\_\_\_, and R. W. Guard. "Directional Gram Structures for High Temperature Strength," <u>Transaction ASM</u>, <u>52</u> (1960), pp. 485-493.
- Waters, W. J., and J. C. Freche. "A High Strength Nickel-Base Alloy with Improved Oxidation Resistance Up to 2200 Degrees F.,"

  Journal of Engineering for Power, Transaction of the ASME, 90
  (January 1968), pp. 1-10.

### INTEGRATED CIRCUITS

- Aerospace Electronic System Technology. Washington, D.C.: National Aeronautics and Space Administration, May 1967.
- "The Application of Electron/Ion Beam Technology to Microelectronics," <u>IEEE Spectrum</u>, 8 (January 1971), pp. 23-37.
- Cline, James E., et al. "Scanning Electron Mirror Microscopy and Scanning Electron Microscopy of Integrated Circuits," <u>IEEE</u>

  <u>Transactions on Electron Devices</u>, <u>ED-16</u>. (April 1969), pp. 371-375.
- Curtis, David A. The Feasibility of Automated Visual Inspection of of Micro-Circuits. Washington, D.C.: National Aeronautics and Space Administration, July 1970.
- Gaertner Research, Inc. Coding Instructions for NASAP 69/I. Washington, D.C.: National Academy of Sciences, 1970.
- Hartin, Sam S. Large Scale Integration of Communications Circuits.
  Washington, D.C.: National Aeronautics and Space Administration,
  February 1971.
- Heffner, William A. Automatic Test Equipment for Electronic

  Components. Washington, D.C.: National Aeronautics and Space
  Administration, October 1969.
- IBM. Investigation of Refractory Dielectric for Integrated Circuits. Washington, D.C.: National Academy of Sciences, July 1969.
- "Ion Implantation; the Sock-it-to-'em Method to Dope Semiconductors,"

  The Electronic Engineer, 28 (January 1969)
- NASA Contributions to Significant Developments in Integrated

  Circuits; A Survey of NASA Literature. Albuquerque, New

  Mexico: University of New Mexico, Technology Application
  Center, 1971.
- Research Electronics Ames Program Review May 1970. Washington, D.C.:
  National Aeronautics and Space Administration, 1970.
- Review of Research Activities January 1, 1969 to December 31, 1969.

  Cambridge, Massachusetts: Research Directorate Electronics
  Research Center, 1970.

- Sarkesian, Ed. "When is a Component a Component," END Magazine, September 2, 1968.
- Schlegel, Earl. Study of Failure Modes of Multilevel Large Scale
  Integrated Circuits. Washington, D.C.: National Aeronautics
  and Space Administration, 1970.
- "Special Issue on Educational Aspects of Circuit Design by Computer-I,"

  IEEE Transactions on Education, E-12 (September 1969).
- Taylor, C. J. Semiconductor Wafer Improvements through Photoengraving. Washington, D.C.: U.S. Government Printing Office, July 1965.
- Triangle Research Institute. <u>Microelectronics in Space Research</u>.
  Washington, D.C.: National Aeronautics and Space Administration,
  August 1965.
- . Survey Study of Programming Languages for Automatic Testing of Electronic Components. Washington, D.C.: U.S. Government Printing Office, September 1969.
- U.S. Industrial Outlook 1971. Washington, D.C.: U.S. Department of Commerce, Bureau of Domestic Commerce, 1971.
- U.S. Markets 1971 Forecast," Electronics, 44 (January 4, 1971).

### INTERNAL GAS DYNAMICS

- American Society of Mechanical Engineers. <u>Journal of Heat Transfer</u>, Series C, No. 1. 81(February 1959), pp. 79-85.
- American Society for Metals. Fiber Composite Materials. ASM Seminar October 17-18, 1964. Metals Park, Ohio: 1964.
- American Society for Testing & Materials. Hot Corrosion Problems
  Associated with Gas Turbines. ASTM Special Publications, No. 421.
  Philadelphia: [n.d.].
- Broutman, L. J., and Krock, R. H., Modern Composite Materials, New York: Adison-Wesley, 1967.
- Gostelow, Paul, Dr. "Big-engine Aerodynamics," New Scientist and Science Journal. January 21, 1971. pp. 115-117.
- Harris, C. M. (ed). <u>Handbook of Noise Control</u>. New York: McGraw-Hill, 1957.
- "Jet Turbine Makers Build Sales Power," Business Week, April 3, 1971, pp. 22-24.
- Langely Research Center. Progress of NASA Research Relating to Noise

  Alleviation of Large Subsonic Jet Aircraft. Proceedings of a
  conference held at Langely Research Center, Hampton, Virginia,
  October 8-10, 1968. Washington, D.C.: National Aeronautics and
  Space Administration, Office of Technology Utilization,
  Scientific & Technical Information Division, 1968.
- Lewis Research Center. Aircraft Propulsion. Proceedings of a conference held at NASA Lewis Research Center, Cleveland, Ohio, November 18-19, 1970. Washington, D.C.: National Aeronautics and Space Administration, Scientific & Technical Information Office, 1971.
- Livingood, J. N., Ellerbrock, H. H. and Kaufman, A. A., <u>Turbine Cooling</u>
  Research Status Report. Washington, D.C.: National Aeronautics
  and Space Administration, 1971.
- NASA Contributions to Significant Developments in Internal Gas

  Dynamics; A Survey of NASA Literature. Albuquerque, New Mexico;
  University of New Mexico, Technology Application Center, 1971.
- New Generation Engines The Engine Manufacturer's Outlook. Society of Automotive Engineers, Air transportation Meeting, April 29 May 9, 1970, New York City.

- Oppenheimer & Co., Institutional Research Department. <u>Gas Turbines</u> The Coming Revolution in Industrial Power. New York, 1971.
- "Paper No. 60-WA-77," Annual Winter Meeting 1960. New York: American Society of Mechanical Engineers, 1970.
- Pratt & Whitney Aircraft. JT9D Technical Symposium. (February 18-20, 1969). East Hartford, Connecticut; 1969.
- Schwartz, Ira R., (ed). Basic Aerodynamic Noise Research. Proceedings of a conference held at NASA Headquarters, Washington, D.C., July 14-15, 1969. Washington, D.C.: National Aeronautics and Space Administration, Office of Technology Utilization, Scientific and Technical Information Division. 1969.
- United Aircraft Corporation, Pratt & Whitney Aircraft, Turbo-Power and Marine Department. Marine Power "Down the Hatch". Bulletin No. S-2240. East Hartford, Connecticut: 1969.
- U. S. Department of Commerce, Bureau of Domestic Commerce, <u>U. S.</u>
  Industrial Outlook 1971. Washington, D.C.: 1971.
- Yaffee, Mike. "FAA Noise Certification Seen Inevitable," Aviation Week & Space Technology, November 25, 1968 and December 2, 1968. pp. 39-50.
- , "Small Aircraft Turbofans Studies," Aviation Week & Space Technology, February 9, 1970, pp. 45-48.

### MATERIALS JOINING

- Alm, G. V. "Guide to Diffusion Bonding," <u>Materials Engineering</u>, May 1970, pp. 24-32.
- Aluminum Weld Development Complex Conference; Minutes. Huntsville, Alabama: Marshall Space Flight Center, January 19, 1966.
- Aluminum Weld Symposium Minutes. Huntsville, Alabama: Marshall Space Flight Center, July 1964.
- Bibliography on Welding Methods, with Indices. Washington, D.C.:
  National Aeronautics and Space Administration, February 1966.
- Brazing and Brazing Alloys A Bibliography. Washington, D.C.: National Aeronautics and Space Administration, 1966.
- Burghard, H. C., et al. <u>Development of Welding Techniques and Filler</u>

  Metals for High Strength Aluminum Alloys. San Antonio, Texas:

  Southwest Research Institute, October 11, 1965.
- Cahill, J. B. <u>Investigation of Brazed and Welded Connections</u>. Huntsville, Alabama: Marshall Space Flight Center, September 1969.
- Cheever, D. L., P. A. Kammer, R. E. Monroe and D. C. Martin. Welding-Base Metal Investigation. Columbus, Ohio: Battelle Memorial Institute, July 28, 1965.

7

- Cole, D. O. <u>Development of Controls of Time-Temperature Characteristics</u>
  <u>in Aluminum Weldments</u>. Torrance, California: Harvey Aluminum,
  October 1966.
- Conference on Technology Status and Trends. Washington, D.C.: National Aeronautics and Space Administration, 1964.
- Fish, R. E. Effects of High Frequency Current in Welding Aluminum Alloy 6061. Huntsville, Alabama: Marshall Space Flight Center, 1968.
- Haynes, J. L., C. G. Wages, and H. S. Haralson. <u>Development of Ultra-sonic Scanning System for In-Place Inspection of Brazed Tube Joints</u>. Huntsville, Alabama: Marshall Space Flight Center, September 14, 1970.
- Keith, R. E., M. D. Randall, and D.C. Martin. Adhesive Bonding of Stainless Steels Including Precipitation-Hardening Stainless Steels. Huntsville, Alabama: Marshall Space Flight Center.
- Koehler, M. L., A B. Kilgore, J. W. Metzler, and S. R. Sturges. <u>Welding</u>, <u>Brazing and Soldering Handbook</u>. Huntsville, Alabama: Marshall Space Flight Center, 1969.

- Laughner, V. H., ed. <u>Handbook of Fastening and Joining of Metal Parts</u>. New York: McGraw-Hill, 1956.
- Limiurk, N. K. High Voltage Electron Beam Welding of Aluminum Alloys.

  Canoga Park, California: North American Aviation, May 14, 1964.
- Masubuchi, K. Analysis of Thermal Stresses and Metal Movement During Welding. Columbus, Ohio: Battelle Memorial Institute, January 1969.
- . Integration of NASA-Sponsored Studies on Aluminum Welding. Columbus, Ohio: Battelle Memorial Institute, September 1967.
- , and R. E. Keith. Fundamentals of Selected Aspects of Deformation Characteristics of Adhesive-Bonded Joints and Metal-Adhesive Interfaces. Columbus, Ohio: Battelle Memorial Institute, January 1967.
- NASA Contributions to Significant Developments in Materials Joining;

  A Survey of NASA Literature. Albuquerque, New Mexico: University of New Mexico, Technology Application Center, 1971
- Pattee, H. E. "Brazing and Brazing Alloys," Machine Design, September 11, 1969, pp. 104-118.
- , R. M. Evans, and R. E. Monroe. <u>Joining Ceramics and Graphite</u> to other Materials. Washington, D.C.: National Aeronautics and Space Administration, 1968.
- Pigue, P. E. <u>Ultra Light Honeycomb Development for S-II Stage Improvement</u>. Tulsa, Oklahoma: AVCO Corporation, October 19, 1965.
- Pullock, D. D., W.V. Mixom, and W. W. Reinhardt. Mechanisms of Porosity Formation in Aluminum Welds. Huntington Beach, California: Douglas Aircraft Company, June 1966.
- Reicheneclur, W. J., and J. Heuschkel. <u>NASA Contributions to Metals</u>
  <u>Joining</u>. Washington, D.C.: National Aeronautics and Space Administration, 1967.
- Research Achievements Review. Vol. I. series 1-22. Huntsville, Alabama:

  Marshall Space Flight Center 1967.
- Research Achievements Review. Vol. II, series 1-12. Huntsville,
  Alabama: Marshall Space Flight Center, 1968.
- Rudy, J. F. "Welding and Welding Alloys," <u>Machine Design</u>, September 11, 1969, pp. 104-118.

- Schumacher, B. W., and F. D. Seaman. Qualitative and Quantitative

  Evaluation of Factors Producing High Efficiency Welds with an Outof-Vacuum Electron Beam Welder. Huntsville, Alabama: Marshall
  Space Flight Center, April 1969.
- Seaman, F. D. Relationship Between Weld Quality and Non-Vacuum Electron
  Beam Welding Procedures. Washington, D.C.: Natinal Aeronautics
  and Space Administration, 1966.
- Selected Technology for the Electric Power Industry. A Technology Utilization Conference held at Lewis Research Center, September 11-12, 1968. Washington, D.C.: National Aeronautics and Space Administration, 1968.
- Smith, J. F., and D. M. Borcinn. "Soldering and Soldering Alloys," Machine Design, September 11, 1969, pp. 104-118.
- Steen, M. V. Aluminum Alloy 2319 Weld Wire Investigation. Palo Alto, California: Lockheed Missiles and Space Corporation, 1964.
- Stenmann, L. G. Out-of-Vacuum Electron Beam Welding. Washington, D.C.: National Aeronautics and Space Administration, 1967.
- Strobelt, W. E. <u>Inert Gas Weldment Effects Study</u>. Seattle, Washington: Boeing Company, June 27, 1966.
- A Study of Optimium Methods for Preparing Aluminum Surfaces for Welding. Chicago, Illinois: Illinois Institute of Technology Research Institute, July 10, 1969.
- Symposium on Technology Status and Trends, April 21-23, 1965. Washington, D.C.: National Aeronautics and Space Administration, 1966.
- Targenson, R. T., et al. State of the Art Survey of Dissimilar Metal

  Joining by Solid State Welding. Seattle, Washington: Boeing

  Company, August 27, 1965.
- Vagi, S. J., R. E. Monroe, and R. M. Evans. <u>Joining of Nickel and Nickel-Base Alloys</u>. Washington, D.C.: National Aeronautics and Space Administration
- Van Leuven, A. C. <u>Pre-Stain Weld Development Program</u>. Canoga Park, California: North American Aviation, Inc., April 23, 1965.
- Welding Progress Summary. Huntsville, Alabama: Marshall Space Flight Center, 1965.

## MICROWAVE SYSTEMS

- Blume, Hans-Juergen C. Error Analysis of Input-Noise-Temperature

  Measurements of Low-Noise Amplifiers. Washington, D.C.: U. S.
  Government Printing Office, January 1969.
- \_\_\_\_\_. "Input impedance Display of One Part, Reflection-Type Amplifiers," Microwave Journal, 10 (November 1967), pp. 65-69.
- Clar, Philip L. "Microwave Integrated Circuit (MIC) Technology," Electro-Technology, October 1969.
- Epstein, Arnold S. "Semiconductor Lamp Prospects Glow Brighter and Brighter," Electro-Technology, July 1969, pp. 35-41.
- Flagiello, Steven C. A Guide to the Literature on Carbon Dioxide

  Lasers, 1 July 1968 30 June 1969. Washington, D.C.: U.S.

  Government Printing Office, 1970.
- Gilbert, George J. "Microwave Transistors: A Look to the Future," Microwaves, July 1970.
- Gunn, J. B., et al. "Solid State Microwave Devices: Domain and LSA Modes," Electro-Technology, August 1967, pp. 67-79
- Lasers and Masers, A Continuing Bibliography. Washington, D.C.:

  National Aeronautics and Space Administration, June 1968.
- McAloy, Nelson. 10.6 Micron Communication System! Washington, D.C.: National Aeronautics and Space Administration, November 1965.
- for ATS-F and ATS-G. Washington D.C.: National Aeronautics and Space Administration, May 1968.
- McDay, John B. et al. A Guide to the Literature on Mercury Cadmium

  Telluride Photodetectors, 1 January 1959 30 June 1969.

  Washington, d.C.: National Aeronautics and Space Administration,

  December 1969.
- McElroy, John. <u>DC-Biased Photoconductive Detection of Wideband</u>
  Carbon Dioxide Laser Signals. Washington, D.C.: National
  Aeronautics and Space Administration, February 1968.
- , et al. A Guide to the Literature of Carbon Dioxide Lasers,

  1 January 1964 30 June 1964. Washington, D.C.: National
  Aeronautics and Space Administration, November 1968.

- NASA Contributions to Significant Developments in Microwave Systems;

  A Survey of NASA Literature. Albuquerque, New Mexico:
  University of New Mexico, Technology Application Center, 1971.
- Optical Space Communications. MIT-NASA Workshop, August 4-17, 1968.
  Washington, D.C.: National Aeronautics and Space Administration, 1968.
- "Putting Power Where It Counts Economically," <u>Microwaves</u>, September 1970.
- "Solid State Technology Invades Microwave Frequencies," Aviation Week, 92 (June 22, 1970), p. 206.
- Thompson, Michael W. "Cornucopia for Implanted Ions," New Scientist, 48 (December 10, 1970), pp. 429-430.
- U. S. Industrial Outlook 1971. Washington, D.C.: U.S. Department of Commerce, Bureau of Domestic Commerce, 1971.
- "U.S. Markets 1971 Forecast," Electronics, 44 (January 4, 1971)
- Vapor Phase Growth System for Growing III/V Compounds and Epitaxial

  Layers. Washington, D.C.: National Aeronautics and Space
  Administration, March 1968.

## NONDESTRUCTIVE TESTING

- Aluminum Weld Development Complex Conference. Conference held at Marshall Space Flight Center, January 19, 1966. Huntsville, Alabama: Marshall Space Flight Center, 1966.
- Berger, H. "Newtron Radiography," <u>Scientific American</u>, November 1962, pp. 107-119.
- Clotfelter, W. N., et al. The Nondestructive Evaluation of Stress-Corrosion Induced Property Changes in Aluminum. Huntsville, Alabama: Marshall Space Flight Center, 1968.
- Cohen, S. E. The Applications of Liquid Crystals for Thermographic Testing of Bonded Structures. Huntsville, Alabama: Marshall Space Flight Center, 1966.
- Development of Nondestructive Methods for Determining Risidual

  Stress and Fatique Damage in Metals. Nashville, Tennessee:

  R.D. Benson and Associates, Inc. 1968.
- Duren, P. C., and E. R. Risch. Radiographic Interpretation Guide

  for Aluminum Alloy Welds. Huntsville, Alabama: Marshall Space
  Flight Center. 1970.
- Feinstein, L., and R. Hardby. <u>Surface-Crack Detection by Microwave</u>
  Methods. Moffett Field, California: Ames Research Center, 1967.
- Gabor, D., et al. "Holography," Science, July 2, 1971, pp. 11-23.
- General Dynamics Corporation, Convair Division. Eddy Current. Vol.I, of Basic Principles. Washington, D.C.: National Aeronautics and Space Administration, 1967.
- . Eddy Current, Vol. II, Equipment, Methods and Applications. Washington, p.C.: National Aeronautics and Space Administration, 1967.
- . Handbooks for Nondestructive Testing Using Ultrasonics.
  Washington, D.C.: National Aeronautics and Space Administration, 1969.
- . Instruction Manuals for Radiographic Nondestructive

  Testing. Washington, D.C.: National Aeronautics and Space
  Administration, 1971.
- . Introduction to Nondestructive Testing. Washington, D.C.:
  National Aeronautics and Space Administration, 1967.

- General Dynamics Corporation, Convair Division. Liquid Penetrant Testing. Washington, D.C.: National Aeronautics and Space Administration, 1967.
- \_\_\_\_\_. Magnetic Particle Testing. Washington, D.C.: National Aeronautics and Space Administration, 1967.
- . Radiography, Vol. I, Origin and Nature of Radiation.

  Washington, D.C.: National Aeronautics and Space Administration, 1967.
- D.C.: National Aeronautics and Space Administration, 1967.
- . Radiography. Vol. IV, Making a Radiograph. Washington, D.C.: National Aeronautics and Space Administration, 1967.
- Gershon-Cohen, J. C., and D. K. Benson. <u>The Emission of Exo-Electrons</u>
  <u>from Aluminum during Fatique</u>. Kansas City, Missouri: Midwest
  Research Institute, 1963.
- Hannah, K. J., et al. Development of the Ultrasonic Delta Technique for Aluminum Welds and Materials. Huntsville, Alabama: Marshall Space Flight Center, 1968.
- Masubuchi, K. Nondestructive Measurement of Residual stresses in Metals and Metal Structures. Columbus, Ohio: Battelle Memorial Institute, 1965.
- Moler, R. B. <u>Development of a Continuous Scanning Laminograph</u>.

  Chicago: Illinois Institute of Technology Research Institute, 1968.
- NASA Contributions to Significant Developments in Nondestructive

  Testing; A Survey of NASA Literature. Albuquerque, New Mexico:
  University of New Mexico: Technology Application Center, 1971.
- National Materials Advisory Board. <u>Nondestructive Evaluation</u>. Washington, D.C.: 1969.
- Nondestructive Testing: Trends and Techniques. Proceedings of the Second Technology Status and Trends Symposium, Marshall Space Flight Center, October 26,27, 1966. National Aeronautics and Space Administration, 1967.
- Pless, W. M., et al. Development, Fabrication, Testing and Delivery of Advanced Filamentary Composite Nondestructive Test Standards.

  Huntsville, Alabama: Marshall Space Flight Center, 1970.

- Ragent, B., and R. M. Brown (eds.). <u>Holographic Instrumentation Applications Conference</u>. Washington, D.C.: National Aeronautics and Space Administration, 1970.
- Research Achievements Review. Vol. III, Report #10. Huntsville, Alabama: Marshall Space Flight Center, 1969.
- Selzer, R. H. Digital Computer Processing of X-Ray Photographs.

  Pasadena, California: California Institute of Technology, Jet Propulsion Laboratories. 1966.
- Sharpe, R. S. Research Techniques in Nondestructive Testing.

  New York: Academic Press, 1970.
- Shulman, A. R. Principles of Optical Data Processing for Engineers.
  Greenbelt, Maryland: Goddard Space Flight Center, 1966.
- Szepesi, Z. Solid State Radiographic Image Amplifiers. Pittsburgh, Pennsylvania: Westinghouse Electric Corporation, 1968.
- Thompson, D. O. <u>Nondestructive Determination of Cohesive Strength of Adhesive Bonded Composites</u>. Huntsville, Alabama: Marshall Space Flight Center, 1969.

### SIMULATION

- Abt, Clark C. Serious Games. New York: Viking Press, 1970.
- Auerbach Corporation. Visual Information Display Systems. Washington, D.C.: National Aeronautics and Space Administration, 1968.
- Baker, Richard F., ed. Goddard Research and Engineering Management

  Exercise (GREMEX). Washington, D.C.: National Aeronautics and

  Space Administration, 1971.
- Bekey, George A., ed. <u>Mathematical Models of Public Systems</u>, Vol. 1, No. 1 of Simulation Councils Proceedings Series. La Jolla, California: Simulation Councils, Inc.,
- \_\_\_\_\_, and Walter J. Karplus. <u>Hybrid Computation</u>. New York: Wiley Press, Inc., 1968.
- , and Robert B. McGhee. Computing Methods in Optimization Problems: Gradient Methods for the Optimization of Dynamic System Parameters by Hybrid Computation. Los Angeles, California: University of Southern California, 1963.
- Brender, Karen D., and Charles P. Llewellyn. "Development and Utilization of a Space Station Mission Simulation Model," presented at Association of Computing Machinery 1967.

  National Conference. Washington, D.C.: National Aeronautics and Space Administration, 1967.
- Davidson, Jess, S. W. and Esten H. Baker. <u>Centrifuge Mounted Motion</u> Simulator. U.S. Patent 3,196,557, 1965.
- Donaghey, Charles E., Jr. A Generalized Material Handling Simulation System. Pittsburgh: University of Pittsburgh, 1968.
- General Electric Company. Study of Aerospace Structured Manufacturing Concepts. Vols. 1-3. Daytona Beach, Florida: 1971
- General Motors Corporation. <u>Interplanetary Guidance Systems Requirements Study</u>. Vol. 2, <u>Computer Program Descriptions</u>. El Segundo, <u>California</u>: 1966.
- GREMEX: The R & D Management Simulation Exercise. Greenbelt,
  Maryland: Goddard Space Flight Center, 1966.
- Grumman Aircraft Engineering Corporation. Man-System Locomotion and

  Display Criteria for Extra-Terrestrial Vehicles. Bethpage,

  New York: Grumman Aircraft Engineering Corporation, 1965.

- Harrell, J. W., and M. J. Argoud. <u>The 25-ft Space Simulator at the Jet Propulsion Laboratory</u>. Pasadena, California: California Institute of Technology, Jet Propulsion Laboratory, 1969.
- Hurt, George J., Jr. et al. An Evaluation of Two Guidance Schemes for a Manned Lunar Landing. Washington, D.C.: National Aeronautics and Space Administration, 1970.
- Hatfield, Jack J. <u>Integrated Time Shared Instrumentation Display</u>: U.S. Patent 3,537,096, 1970.
- Hewes, Donald E., and Amos A. Spady. Evaluation of a Gravity-Simulation Technique for Studies of Man's Self Locomotion in Lunar Environment. Washington, D.C.: National Aeronautics and Space Administration, 1964.
- Keefer, John M. Phonocradiogram Simulator. U.S. Patent 3,508,347, 1970.
- Korn, G.A. <u>Progress of Analog-Hybrid Computation</u>. Tucson, Arizona: Arizona University, 1966.
- Kucher, N.R. Studies of the Pulmonary Circulation. Philadelphia: General Electric Company, 1970.
- Lackner, Helmut G. Method and Apparatus of Simulating Zero Gravity Conditions. U.S. Patent 3,196,557, 1970.
- Levin, Kenneth L., and John G. Allen, Jr. <u>Lunar Landing Flight</u>
  Research Vehicle. U. S. Patent 3,191,316, 1965.
- Liebowitz, B. H., et al. Procedures for Management Control of Computer Programming in Apollo. Washington, D.C.: Bellcomm, Inc., 1966.
- NASA Contributions to Significant Developments in Simulation; A Survey of NASA Literature. Albuquerque, New Mexico: University of New Mexico, Technology Application Center, 1971.
- McCracken, M. C. "Simulation in Economics," Simulation, July 1967, pp. 49-50.
- McLeod, John. "Computer Simulation of the Hydrodynamics of the Cardio-vascular System," Simulation, Fall 1963, pp.7-9
- . "PHYSBE... A Physiological Simulation," <u>Simulation</u>, December 1966, pp. 324-329.
- \_\_\_\_\_. The Simulation of Difficult Systems, Simulation, April 1970, pp. 172-173.

- Mitler, G. Kamball, Jr., and Gene W. Sparrow. <u>Fixed-Based Visual-Simulation STudy of Manually Controlled Operation of a Lunar Flying Vehicle</u>. Washington, D. C.: National Aeronautics and Space Administration, 1970.
- Mitroff, Jan. A Study of Simulation Aided Engineering Design.

  Berkeley, California: University of California Space Science
  Laboratory, 1967.
- University of California, Institute of Ecology, Environmental Systems Group. A Model of Society. 1969.
- Morgenthaler, George W. "On the Selection of Unmanned Probes and Launch Vehicles for Exploration of the Solar System," Operations Research, 15 (July August 1967).
- , "Operations Research Applied to Problems in Logistics and Economics of Space Transportation," presented to the 19th Annual Meeting of the Operations Research Society of America, Chicago, May 26, 1961. Baltimore, Maryland: Martin Marietta, 1961
- Progress in Operations Research. Vol. 1, Russell L. Ackoff, ed. New York: Wiley Press, Inc., 1961, pp. 363-419.
- NASA Contributions to Significate Developments in Simulation: A

  Survey of NASA Literature. Albuquerque, New Mexico: University
  of New Mexico, Technology Application Center, 1971
- National Aeronautics and Space Administration. Forecasts and Appraisals for Management Evaluation. Vol. 1. Washington, D.C.: 1965.
- . Man-Machine Interactive System Simplifies Computer-Aided Circuit Design. Washington, D.C.: 1970
- Orlando, J. A., and A. J. Pennington. "BUILD A Community Development Simulation Game," presented at the 36th National Meeting of the Operations Research Society of America, Miami Beach, Florida, November 10-12, 1969.
- Pennington, A. J., et al. Research and Education in Management of

  Large-Scale Technical Programs. Appendices A-E. Philadelphia:

  Drexel Institute of Technology, 1969.
- Philbrick, G. A. "Analogs Yesterday, Today and Tomorrow," Simulation, Fall 1963, pp. 11-17

- Phillips, C.L. Simulation of High-Order Hybrid Control Systems. Auburn, Alabama: Auburn University, 1968.
- Ragazzini, John R., et al. "Analysis of Problems in Dynamics by Electronic Circuits," Simulation, September 1964, pp. 54-64.
- Rea, Robert H., et al. Applications of Systems Analysis Models: A Survey. Washington, D.C.: National Aeronautics and Space Administration, 1968.
- . "NASA Contributions to Mathematical Modeling and Simulation with Applications Outside of the Aerospace Industry," in Proceedings, Sixth Space Congress, Cocoa Beach, Florida, March 17-19, 1964. Vol II, Lloyd E. Jones, III, ed. Cocoa Beach, Florida: Canaveral Council of TEchnical Societies, 1969, pp. 18-19.
- Reinfields, Jaris, et al. AMTRAN Hardware -- An Electronic Interface to Simplify and Speed Up Man Machine Communications. Huntsville, Alabama: Marshall Space Flight Center, 1966.
- Roberts, Edward B. The Design of Research and Development Policy. Cambridge, Massachusetts: Massachusetts Institute of Technology, 1963.
- Ross, L. W. "Simulation of Air and Water Pollution Dynamics: A Survey." Simulation, April 1970, pp. 172-173.
- Sasser, W. Earl, and Thomas H. Naylor. "Computer Simulation of Economic Systems... An Example Model," <u>Simulation</u>, January 1967, pp. 21-32.
- Simulation. [Special Apollo Issue], July 1970.
- Snyder, M. F., and V.C. Rideout. <u>Computer Simulation Studies of the Venous Circulation</u>. Madison, Wisconsin. University of Wisconsin, 1968.
- A Study on the Use of Fighter Aircraft to Provide a Zero g Environment in Support of Space Manufacturing Experiments. Huntsville, Alabama: Marshall Space Flight Center, 1969.
- Watt, Kenneth, et al. " A Model of Society," Simulation, April 1970, pp. 153-164.
- Wayne State University. A Model of and Problems Encountered in a Metropolitan Police Department. Detroit, Michigan: 1969.

### TELEMETRY

- Brigham, Morrow. "Monograph on Processing Experimental Data by Fast Fourier Transforms," LTV/IEEE Spectrum, December 1967.
- Computer Science Corporation. Advanced Applications of Ople Concepts. Washington, D.C.: National Aeronautics and Space Administration, 1970.
- Cole, C.E. The Interrogation, Recording, and Location Systems

  Experiment. Washington, D.C.: National Aeronautics and Space
  Administration, 1969.
- Frank, Ronald A. "Bell Digital Network Predicted," <u>Computer World</u>, September 9, 1970.
- Gruenberg, Elliot L. (ed) <u>Handbook of Telemetry and Remote Control</u>. New York: McGraw-Hill, 1967.
- Lasers and Masers. Washington, D.C.: National Aeronautics and Space Administration, June 1968.
- Lumb, Dale. "A Study of Codes for Deep Space Telemetry," in <u>Proceedings</u> of the IEEE International Convention, New York City, March 20-27, 1967.
- , and Larry B. Hofman. An Efficient Coding System for Deep Space Probes with Specific Application to Pioneer Missions. Washington, D.C.: National Aeronautics and Space Administration, 1967
- McAvoy, N., et al. 10.6 Micron Laser Communications System Experiment for ATS-F and ATS-G Washington, D.C.: National Aeronautics and Space Administration, May 1968.
- McElroy, J. H. "Carbon Dioxide Laser Systems for Space Communications," in <u>International Conference on Communications</u>, San Francisco, California; June 6-8, 1970.
- NASA Contributions to Significant Developments in Telemetry; A

  Survey of NASA Literature. Albuquerque, New Mexico: University
  of New Mexico, Technology Application Center, 1971.
- OART-Research Electronics Ames Programs Review. Washington, D.C.:
  National Aeronautics and Space Administration, May 1970.

- Rudin, Harry, Jr. "Data Transmission: A Direction for Future Development," <u>IEEE Spectrum</u>, <u>1</u> (February 1970), pp. 79-85.
- Slunder, C. J., A F. Hoenie, and A. M. Hall. <u>Thermal and Mechanical</u>
  <u>Treatment for Precipitation-Hardening Stainless Steels</u>. Huntsville, Alabama: Marshall Space Flight Center, 1968.
- Smith, Gene. "A.T.&T. Planning and Digital Network," New York Times, September 1, 1970.
- Stattel, R. J. and J. E. Pownell. <u>Airborn Transistorized Telemeter</u>
  System Model SST-1. Washington, D.C.: National Aeronautics
  and Space Administration, April 1965.
- Tausworthe, Robert C. Theory and Practical Design of Phase-Locked Receivers. Vol. I. Washington, D.C.: National Aeronautics and Space Administration, 1966.
- M. F. Easterling, and A. J. Spear, "A High-Rate Telemetry System for the Mariner Mars Mission 1969," in Proceedings of the International Telemetering Conference, Los Angeles, October 8-11, 1968.
- Vermillion, Charles H. Constructing Inexpensive Automatic Picture-Transmission Ground Stations; A Report. Washington, D.C.: National Aeronautics and Space Administration, 1968.