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MARCH 15 – 16, 1966

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STRUCTURAL ADHESIVE BONDING CONFERENCE

MARCH 15 - 16, 1966

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DESIGN CRITERIA FOR BONDED STRUCTURES

9:30 - 10:10 A. M.

TUESDAY - MARCH 15, 1966

N66 34127

SOME DESIGN CONSIDERATIONS FOR ADHESIVE BONDING

by

E. C. Burkart
Chief, Research and Development
Rohr Corporation, Chula Vista, California

ABSTRACT

This paper covers some of the design details and materials selection criteria which are important considerations for the designer of bonded structures. The advantages and limitations of bonded structures are discussed, along with design suggestions for better utilization of the bonded concept. A comparison between adhesive bonding and mechanical fastening is made in several standard types of construction. The basic concepts of a bonded sandwich are discussed relative to optimizing from both functional and manufacturing standpoints. Suggestions are given for insuring the compatibility of the various elements of a sandwich structure through the proper selection of materials and design configuration. Also included is a brief description of a program presently in existence which investigates the feasibility of substituting adhesive bonding for mechanical fasteners on large rocket thrust structures.

SOME DESIGN CONSIDERATIONS
FOR ADHESIVE BONDING

by

Edward C. Burkart

ROHR CORPORATION

January 1966

SOME DESIGN CONSIDERATIONS FOR ADHESIVE BONDING

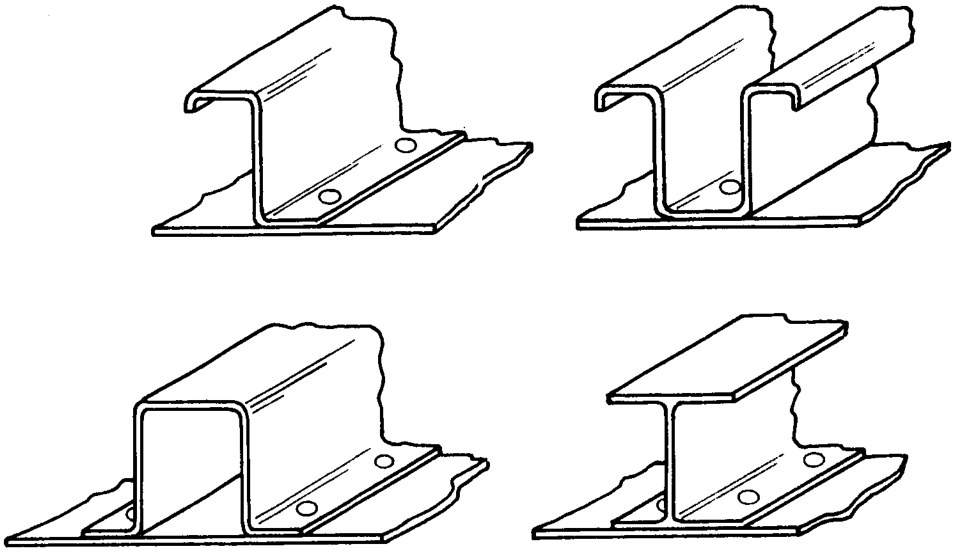
Adhesive bonding is simply another way of joining materials together, and the basic engineering principles which govern good structural design are also of course the foundation for bonded structures design. Bonding, like welding, provides a continuous means of attachment, as opposed to the intermittent type of joint which results when rivets or bolts are used. As such, bonding has many advantages, and some disadvantages, but in any event its continuous nature carries with it the possibility of 100% joint efficiency. The other dominant characteristic of adhesive bonded construction is the visco-elastic damping properties of the adhesive itself, and where fatigue resistance and crack propagation resistance are serious problems, this property has great importance.

Adhesive bonding engineering techniques are not in principle different from other kinds of engineering, and this discussion will not attempt to review these basic ideas. But there are some differences in bonded design which because they are more obscure than the details of mechanical fastener joining, often discourage the designer from employing bonding. This discussion will cover several of these considerations.

For convenience, we have divided bonded structures design into two categories: (1) metal-to-metal, and (2) sandwich.

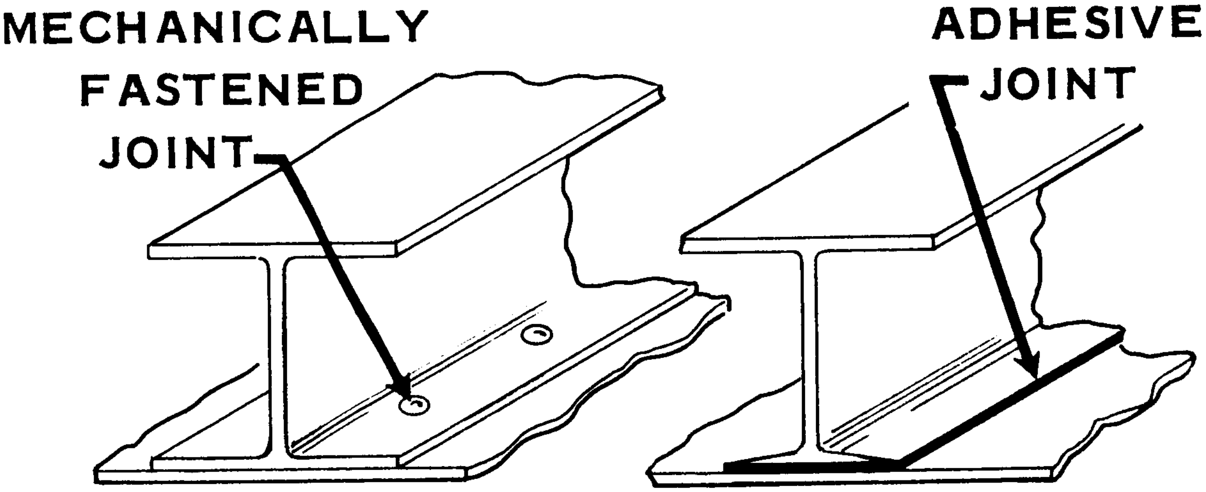
METAL-TO-METAL ADHESIVE BONDING

Figure 1 illustrates the time-honored stringer and skin configuration for aerospace structures, where loads are distributed in various proportions between stringers and skins, and where the stringers function as skin stiffening



STANDARD SKIN & STRINGER DESIGN

FIG. 1



COMPARISON OF BONDED VERSUS MECHANICALLY FASTENED STIFFENER CONFIGURATIONS

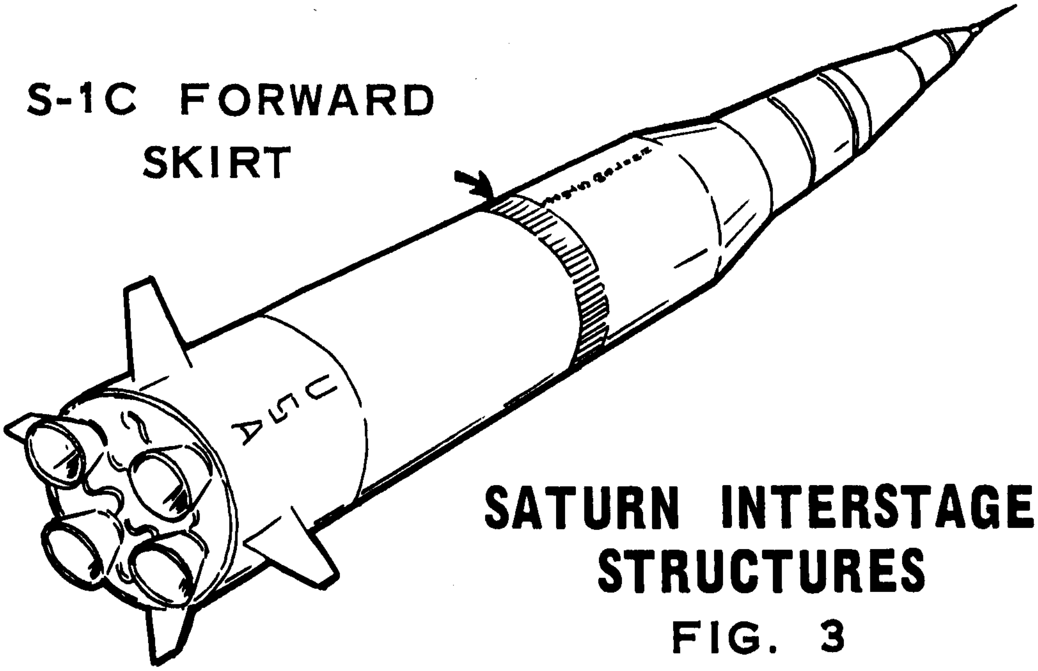
FIG. 2

members. Common examples of this type of design are cylindrical rocket interstructures and airplane fuselage and lifting structures.

It is possible to realize certain structural efficiencies from the use of adhesive bonding with this basic configuration, substituting adhesive for the fasteners. A comparison of the riveted version with the bonded version is shown in Figure 2. The basic difference between the two is that the skin flange of the bonded stringer is less than two thirds as wide as the riveted stringer, and is tapered. The narrower tapered flange on the bonded stringer is possible because the joint has higher unit shear and tensile strengths, and because it is unencumbered by fastener edge margin considerations, or section loss from fastener holes. Furthermore, the elimination of an abrupt lateral section change at the edges of the stiffening member gives a more uniform load distribution through the section. Where the joint is loaded in tension, the tapered skin flange has a physical profile which more nearly approximates the bending moment profile across the section. Where fatigue strength and crack propagation resistance are major considerations, the bonded design affords a large weight saving. Where static strength is the basic consideration, the bonded version still provides a very respectable advantage.

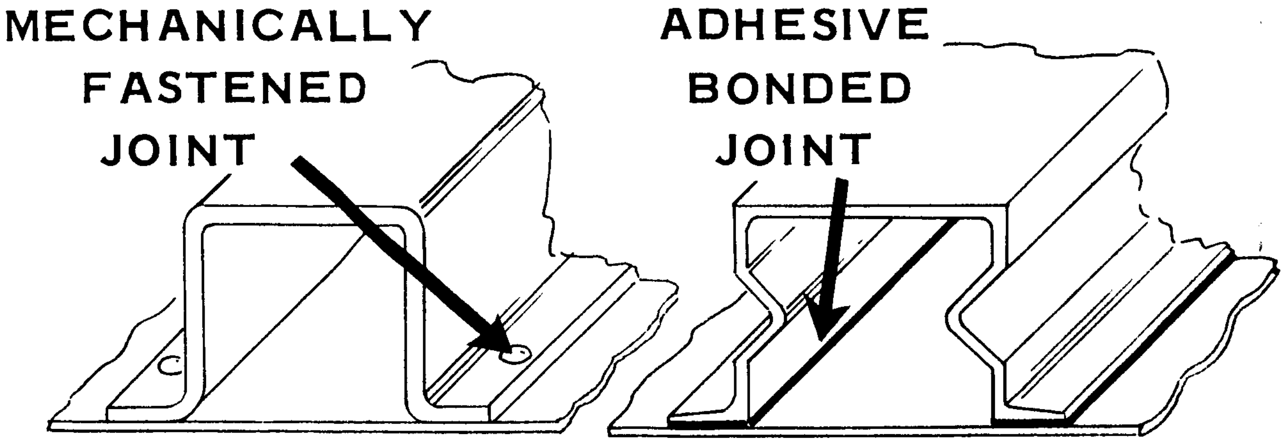
An example of the latter would be the Saturn S-1-C forward skirt as shown in Figure 3.

This structure is presently made of rolled sheet hat sections riveted and bolted to a cylindrical skin and circumferential rings. Under a program sponsored by Marshall Space Flight Center, an investigation is being made into the feasibility of replacing the mechanical fasteners with adhesive bonding.



SATURN INTERSTAGE STRUCTURES

FIG. 3



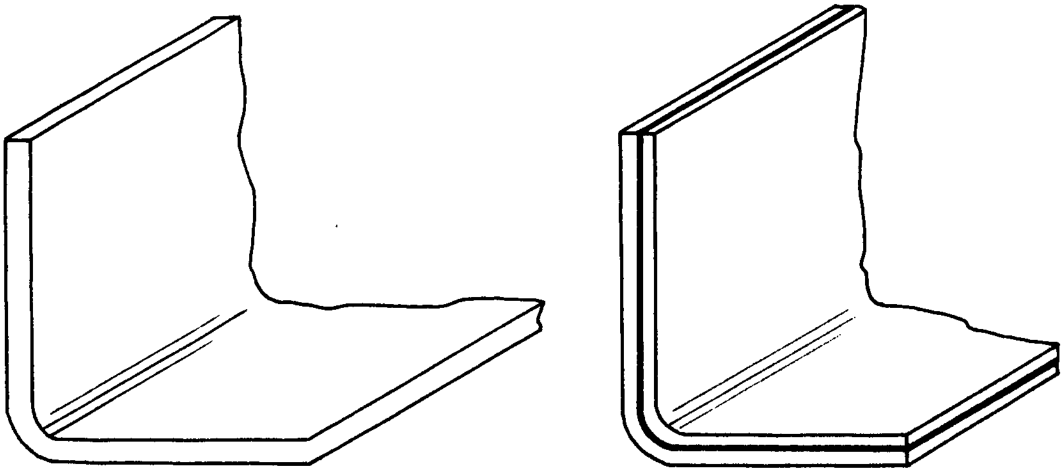
S-1C INTERSTAGE STRUCTURE

FIG. 4

A comparison between the existing mechanically fastened hat section and the adhesive bonded one is shown in Figure 4. Please note the narrower tapered skin flanges. Preliminary test segments of this cylinder show excellent strengths, and a very worthwhile weight reduction. In addition, a change of this kind has the important advantage of being compatible with the existing envelope and tooling, and can therefore be readily introduced if desired into an existing program.

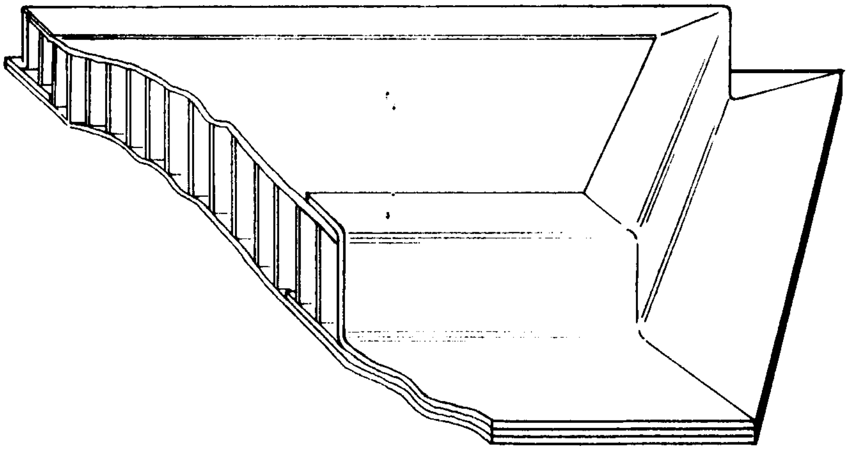
Another type of innovation in metal-to-metal design is the laminated sheet concept, wherein detail parts are made from thin sheet laminates. As shown in Figure 5, in this kind of construction standard single sheets of metallic or non-metallic materials are replaced by a combination of thinner sheets adhesively bonded together. The purpose of laminates such as this is to reduce weight and provide added fatigue resistance. These sheets may be used as skins or sub-structure, in an otherwise mechanically fastened design, or as face sheets on a sandwich.

To illustrate the use of this kind of design, consider an application where local skin stability is critical. In this case it is usually advantageous to replace a single sheet of material with two thinner sheets bonded together. The laminated sheet can be sized to provide similar rigidity, with a lower overall weight. An example would be the replacing of a single .040 sheet with two thinner sheets bonded together such that the overall thickness of the laminate is slightly greater than the solid .040 sheet. The laminate is lighter in weight because the density of the adhesive is only a fraction of that of the sheet. Where fatigue resistance is the critical item, still greater weight savings can be obtained.



SINGLE SHEET **LAMINATED SHEET**

FIG. 5



TYPICAL BONDED SANDWICH

FIG. 6

In effect, laminated sheet provides a sandwich consisting of two load carrying face sheets separated by a low density core, which in this case is the adhesive itself. The laminated sheet materials for skins, webs, miscellaneous sheet metal bracketry, etc., are usually formed after bonding; hence the bonding operation is simple and inexpensive. For this kind of fabrication, flat sheet stock in mill sizes is bonded in various thickness combinations and stored as raw material. Subsequent forming operations such as stretching, braking, or drawing, can be readily performed, provided the designer allows somewhat greater-than-normal bend radii.

ADHESIVE BONDED SANDWICH DESIGN

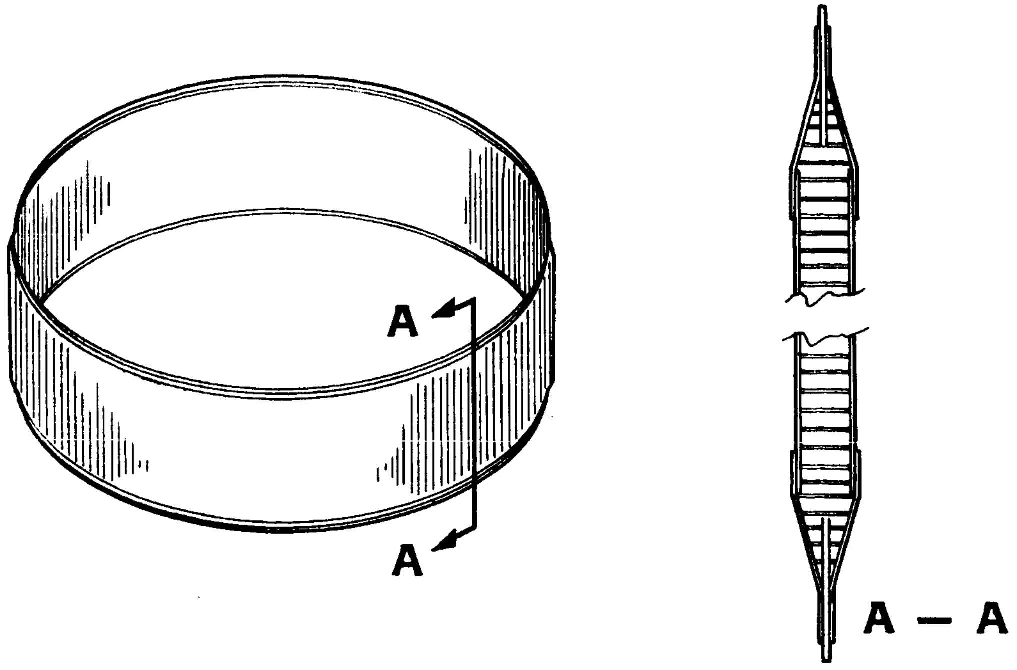
Figure 6 illustrates the second basic type of bonded design, the familiar sandwich structure which uses two load carrying face sheets bonded to a low density core material. This configuration usually provides such advantages as higher strength-to-weight ratio, higher rigidity-to-weight, extra smoothness, and superior fatigue resistance. Following is a discussion of several design details which should be useful in working with sandwich construction.

Facing Materials - The most common facing materials for aerospace structures are metals such as aluminum, titanium, beryllium, and steel. The selection of a facing material depends upon the usual properties such as strength, elastic modulus, thermal conductivity, etc. Although aluminum has been the basic metal facing in use in sandwich construction for many years, the high strength titanium alloys are starting to become widely used. These alloys have higher strength-to-weight ratios than aluminum, and the face sheet stabilization furnished by the core permits effective utilization of this added strength. In certain applications beryllium sheet provides even further weight savings because of its higher elastic modulus.

Where structures have well ordered load patterns, sandwich face sheets can effectively use materials with oriented load carrying characteristics. A rocket interstage structure, as shown in Figure 7, where the loads are essentially longitudinal, is a good example of such a load pattern. A material with suitable load carrying characteristics for possible use in this application would be an oriented filamentary composite. Such a composite would use a resin matrix, along with a reinforcing material consisting of filamentary glass, steel, boron, etc., depending upon the application. Uni-directional fibrous cloth provides another form of oriented fiber face sheet. A honeycomb sandwich compression cylinder would probably use face sheets made of a combination of filament wound longitudinal fibers, plus a few girth oriented fibers.

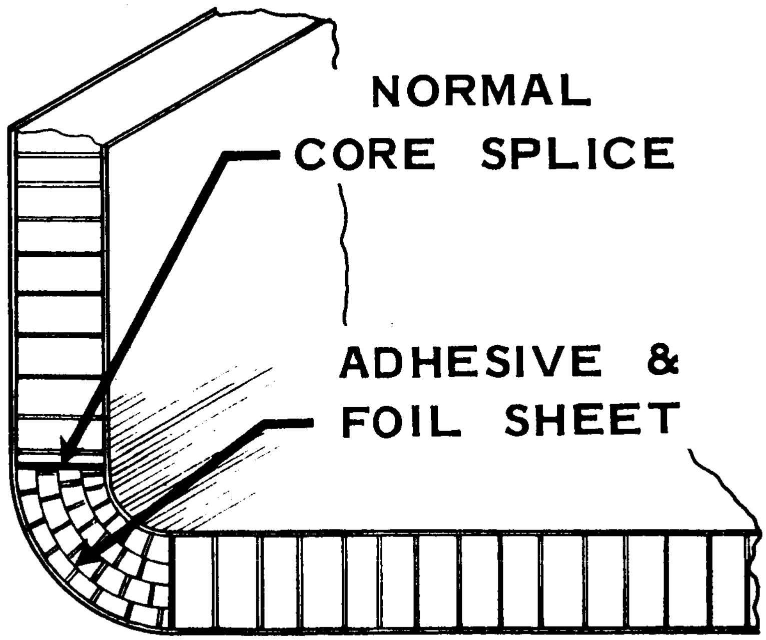
Core - The most widely used core is the cellular honeycomb type. When operating temperatures permit, aluminum core is still generally the most efficient structural core available. However, functional requirements such as elevated temperature resistance or specific thermal conductivity properties, oftentimes dictate the use of steel, titanium, or plastic core. A point to remember here is that core and face sheets need not be made from the same kind of material. For example, aluminum core may be used with fiberglass reinforced plastic or titanium face sheets. In order to resist moisture penetration into the interior of the structure, the core should of course be non-perforated.

Core node strength is an important consideration which is sometimes overlooked. Except for steel or titanium core, a special type of adhesive bonding is used in making core node connections. Unless the node bond strength is relatively high at the bonding temperature, the finished product is likely to include failed node bonds, especially if the core-to-face sheet adhesive has a high



ADHESIVE BONDED SANDWICH CYLINDER USING ORIENTED FIBER FACE SHEETS

FIG. 7



LAMINATED CORE APPLICATION

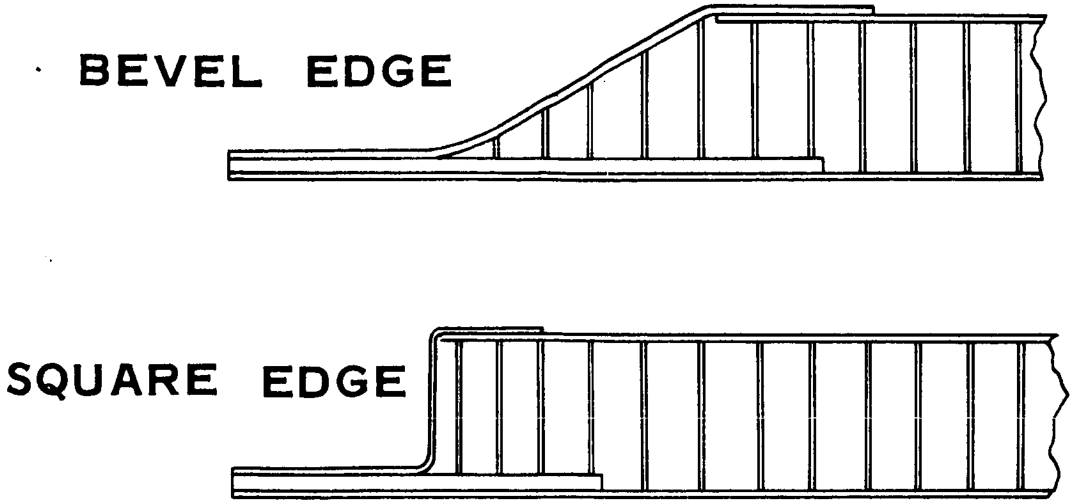
FIG. 8

volatile content. Node bond strengths in core vary rather widely. Resistance welded core gives very good node strengths but has the disadvantage of not being fluid tight at the node connection. Such core may not be satisfactory when water entry and water migration in the sandwich is a critical item, because it has basically the same characteristics in this regard as perforated core.

We anticipate the use of filamentary materials for honeycomb core. Just as in the case of the sandwich face sheets, oriented fiber cells can provide weight savings when properly applied. At the same time, filament wound core, for example, may ultimately be the cheapest core available.

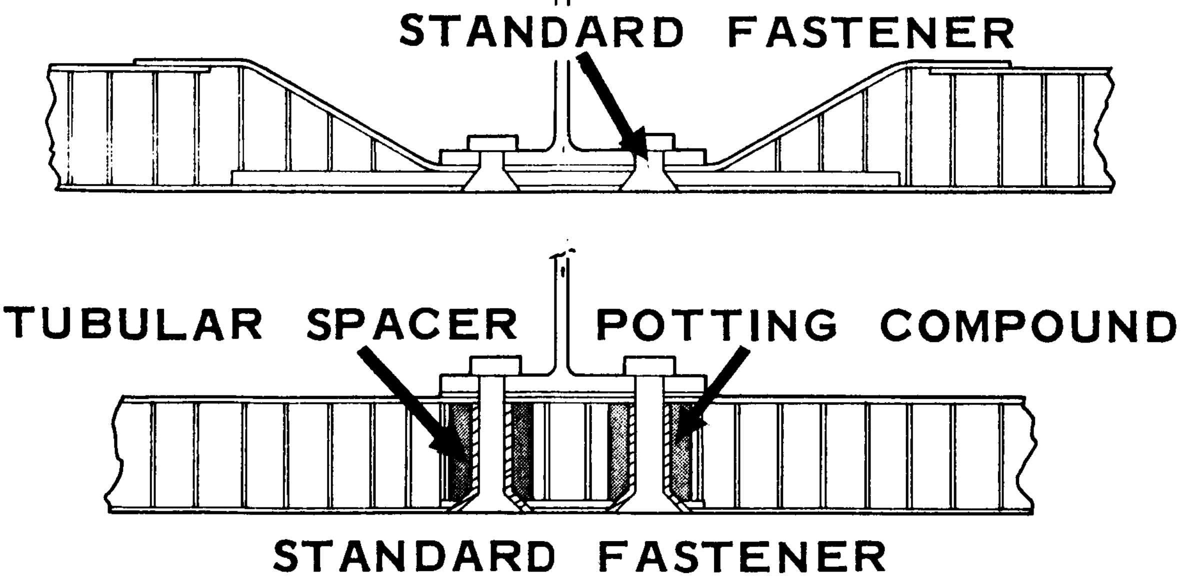
One core problem which faces the designer of thicker structures is that of forming thick core sections around rather sharp radii. One of the better design solutions to this problem is to laminate a series of core layers as shown in Figure 8. This technique consists of using layers of core separated by, and bonded to, interleaved sheets of foil. This procedure permits forming about a relatively small radius while still maintaining the structural integrity of the core. Another area in which the interleaving technique can be used is where added lateral core rigidity is needed in order to prevent lateral collapsing of cells during cure.

Edge Members - The selection of edging for any sandwich is as much dependent on manufacturing limitations as upon the functional requirements of the part. When speaking of edge members we include peripheral shear doublers and edge closures as pictured in Figure 9. By and large, the edge closure is a non-structural part used principally as an edge seal and a device for protecting the exposed core edge. As such it should be designed of as thin a material as



SANDWICH EDGE MEMBER DESIGN

FIG. 9



ALTERNATE METHODS OF MECHANICAL ATTACHMENT TO SUBSTRUCTURE

FIG. 10

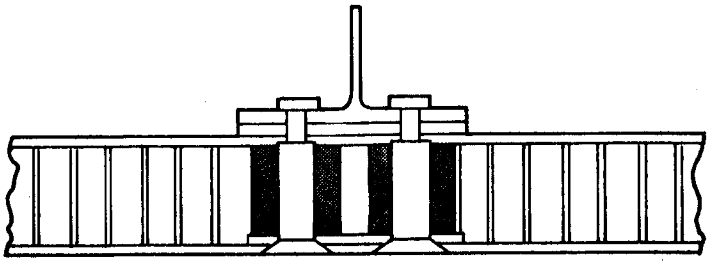
possible so long as it will stand up by itself in its operational environment. In some cases it may be appropriate to omit the edge closure entirely. The peripheral shear doubler should be heavy enough to carry the perpendicular panel shear loads, plus edgewise shear and bearing loads. These basic objectives can be attained by any one of several design configurations. However, the design which will usually be lightest and easiest to manufacture is the square edge where there is no tapered core transition. This configuration permits a narrower shear doubler and a more reliable bond between the shear doubler and outer skins and between the shear doubler and core. One of the more difficult manufacturing problems on tapered core transitions is that of obtaining sufficient bond pressure in this area without moving the core laterally. With square edging, full bonding pressure is applied to all areas of the sandwich, including the entire shear doubler area.

On assemblies which require metal edge members and metal skins, the question arises as to whether edge doublers should be bonded into the assembly or chem-milled integrally into the skins. Various functional and economic factors determine which course of action is taken. From a functional standpoint, the chem-milled doublers are probably lighter and more reliable because they do not have the additional glue line between the doublers and the face sheets. However, when bonded doublers are used the added glue line gives added fatigue resistance, and more readily permits the use of tapered doublers if desired. From an economics point of view, the bonded doubler is usually cheaper when only one doubler is used and the doubler is relatively uncomplicated. When doublers are multi-layered and are otherwise complicated by cutouts, etc., it is usually cheaper to chem-mill.

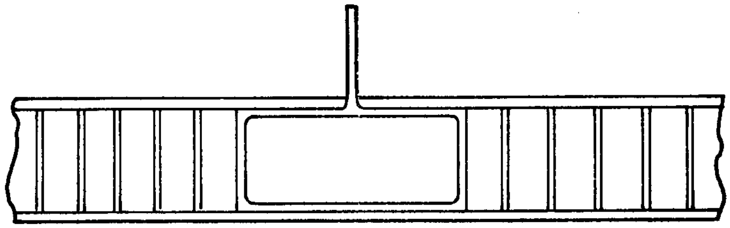
Fasteners - When a sandwich has a laminated metal-to-metal edge band, standard fasteners are used for attachment to sub-structure as shown in Figure 10. Where it is desirable to attach the sandwich to the sub-structure through the full depth of the assembly the fastener problem becomes more complicated. In this instance the usual practice is to densify the core in the fastener area by inserting a potting compound or by installing a tubular spacer as shown in Figure 10. These methods attach the fastener to the sandwich face sheets and core while at the same time reacting the fastener installation forces. One method which we are presently experimenting with is a stepped fastener, pictured in Figure 11, wherein the tubular spacer and the fastener are combined into one. The stepped fastener is similar to any other rivet or bolt, including blind rivets and bolts, except that it incorporates a shoulder which permits the fastener to accept its own driving forces, rather than depend upon a separate spacer or potted area. The stepped fastener may be potted during installation, and driven wet.

Alternate means of attaching a sandwich panel to sub-structure is by burying an extrusion in the sandwich when it is originally manufactured. This method, also shown in Figure 11, is probably more effective than the preceding methods because it provides a continuous attachment to the bonded sandwich and eliminates the problems of water entry and appearance which are associated with installing fasteners through the sandwich face.

Adhesives - Although there are hundreds of structural adhesive systems available on the market, there is usually one material best suited for each application. Aside from mechanical properties requirements, consideration must be given to a variety of characteristics in order to make the adhesive compatible with manufacturing limitations and its ultimate functional environment.



STEPPED FASTENERS



INTEGRAL STRIP INSERT

ALTERNATE METHODS OF
ATTACHMENT TO SUBSTRUCTURE

FIG. 11

In the area of manufacturing compatibility, the designer must recognize that the material must have shelf life and tackiness characteristics which will enable the shop to handle it in a practical manner. The volatile content of the selected adhesive must be low enough to avoid excessive gas pressure build-up during bonding. Where complex contours and heavy material gages are bonded, the adhesive must be sufficiently insensitive to pressure variations to accept a certain amount of manufacturing tolerances.

From a functional standpoint, the most important properties to look for in the adhesive are those of strength retention under the extremes of temperature and moisture environment which the bonded assembly will experience in service. For rocket applications, the designer would usually look for an adhesive which has a very flat strength curve through the extremes of temperature in which it will be expected to operate. Since the duration of exposure on rocket applications is oftentimes relatively short, the testing involved in selecting such an adhesive is usually quite simple. Where an adhesive is selected for a long duration application, the selection of an adhesive becomes more complicated, because qualifying tests for the adhesive must be run for extended periods of time. Since we have not as yet established a satisfactory method of extrapolating short duration test data over long time exposures, the selection of an adhesive to operate at elevated temperature, for example for 50,000 hours, can involve a fairly formidable testing program.

Another adhesive property which is very important is cure temperature. The ideal cure temperature for an adhesive would probably be about 140°F. This material would permit handling at room temperature during layup, and then curing at a slightly elevated temperature. For the present, we have a number of good 250°F curing adhesives which represent a substantial improvement over the old 350°F systems. The lower curing temperatures are advantageous because they do not affect the metallurgical properties of the adherend, are less demanding of tools, reduce thermal distortion during bonding, and require less expensive bond cycles and facilities.

When selecting adhesives it is necessary to select a whole family of materials, not simply a core-to-face sheet adhesive. This family of materials should also include a core splicing adhesive, a core-to-edge-member adhesive, and a potting compound.

The core splicing adhesive should have an expansion ratio during cure of approximately 2, while at the same time having a low volatile content. For ease of manufacture it should be in the form of a tape which is slightly tacky at room temperature. With this combination of properties the core splicing adhesive will normally provide a good tight core fit during layup and a solid core joint after cure. The core-to-edge-member bond would normally utilize the same adhesive as used for the core splice. Potting compound should have high compressive strength, low density, and good impact resistance. In addition, this material must be compatible with the other bonding operations used on the assembly.

When is adhesive bonding the best choice? Bonding presents a more efficient design in almost all structural applications where the thermal and chemical operating environments are compatible with the adhesives, and where the required part envelope will accept a bonded configuration. Certainly the majority of aerospace structures fall in this category. The reasons for the better efficiency of bonded structures are clear. First, bonding offers superior fatigue resistance because of the absence of stress concentrations and because the natural self-damping properties of bonded structures are high compared with mechanically fastened structures. Secondly, bonded joints are more efficient than mechanically fastened joints, and more nearly approach the strength of the parent material because they form a continuous attachment, free of the interruptions associated with intermittent fastening. Thirdly, bonded sandwich structures make a more economical use of structural material in compression, resulting in a strength-to-weight ratio which is superior to conventional sheet and stiffener construction. Local compressive buckling strength is greatly increased because individual skin panel dimensions are those of a single core cell. A sandwich panel, properly designed in bending, for instance, may be stressed in its compressive face to the yield strength of the material, while a conventional frame and stiffener panel will fail in buckling at the compressive stress considerably below yield. This same increased utilization of material is also true in shear panels made of sandwich construction. Shear stress values approaching the ultimate shear strength of the facing material may be used in sandwich structures, whereas conventionally stiffened panels will buckle at critical buckling stresses well below the ultimate shear strength of the material. As improvements in materials technology produce higher strength-to-weight ratio materials such as 8-1-1 titanium, material gages become thinner and material buckling problems become more severe. Consequently, in order to take full advantage of these new high strength materials, it is necessary to design structural components which are continuously stabilized in a manner such as is found in sandwich design.

SESSION I - PAPER 2.

DESIGN OF HEAVY-GAGE BONDED HONEYCOMB SANDWICH

10:20 - 11:00 A. M.

TUESDAY - MARCH 15, 1966

N66 34128

DESIGN OF HEAVY-GAGE BONDED HONEYCOMB SANDWICH

by

Charles F. Herndon
Advanced Design Project Engineer
General Dynamics, Fort Worth

ABSTRACT

We are now at the threshold of an era when sandwich in extremely heavy forms is becoming extremely attractive. This is true for the future generations of large size boosters and particularly for the skirt components.

The main attraction of heavy sandwich is its potential weight savings for the compression-load-critical components. In these components, a sandwich construction can efficiently provide resistance to instability failures and at the same time accommodate the other attendant loading conditions. However, since the payoff for weight savings is relatively low for the heavier booster areas, other factors must be carefully scrutinized before sandwich can be selected. These factors include relative costs, reliability, and confidence in sandwich technology.

The confidence and knowledge which exists in heavy-gage sandwich technology is the major theme of this paper. It can be shown that, with caution and discrimination at various design points, heavy-gage sandwich can be approached in a way to avoid encountering major problems. Some of the key potential problem areas are associated with adhesive selection and the definition of joint concepts. Even though the majority of the bonded surface area is over honeycomb core, the decisions regarding the designs and adhesives at the edgemembers are so important that they may determine the success of a heavy-gage sandwich design.

DESIGN OF HEAVY-GAGE BONDED HONEYCOMB SANDWICH

BY

CHARLES F. HERNDON
PROJECT DESIGN ENGINEER
GENERAL DYNAMICS
FORT WORTH DIVISION

FOR

PRESENTATION AT THE CONFERENCE ON

STRUCTURAL ADHESIVE BONDING

AT

MARSHALL SPACE FLIGHT CENTER
HUNTSVILLE, ALABAMA

ON

MARCH 15-16, 1966

DESIGN OF HEAVY-GAGE BONDED HONEYCOMB SANDWICH

Introduction

Heavy-gage sandwich applications are rare. Seldom have skin gages over one-tenth of an inch been required. However, we are now at the threshold of an era when sandwich in extremely heavy forms is becoming attractive. This is true for the future generations of large size, heavily loaded boosters and particularly for the skirt and intertank structures.

The main attraction of heavy sandwich lies in its potential weight savings for the compression-load-critical components. In these components a sandwich construction can efficiently provide resistance to instability failures as well as accommodate the other attendant loading conditions. But as we all know, merely offering a nominal potential weight saving of 10 to 30 percent in the lower booster structure may be insufficient reason to select sandwich.

What are some of the other considerations? They include such items as, costs (tooling and manufacturing), reliability, and possibly the most important of all - the level of confidence in sandwich technology.

This latter item constitutes the major theme of this presentation. For, I believe that a greater knowledge of the characteristics of sandwich construction will lead to a more objective comparison with other structures. In other words, greater confidence in sandwich will lead to a more realistic prediction of costs, weights, and realiability. For this confidence in sandwich to be fully realized, it must be supported by the carefully coordinated efforts of the Engineering Design, Manufacturing, and Quality Control Departments.

Adhesive Selection

No decision is more important to the design of heavy-gage honeycomb than the selection of an adhesive. This decision directly affects design concepts, tool configurations, manufacturing methods, and quality control techniques. Many excellent adhesive systems do exist but the number which are acceptable can be reduced to just a few when examining the many and divergent requisites of a heavy-gage honeycomb design. Whereas the selection of materials for fittings, skins, and core may clearly be decisions for the Materials and Stress engineers, the designer should serve as a focal point in picking the adhesive. The designer must provide an insight into a number of subtle potential problem areas. He must relate the adhesive characteristics to his design concepts, and then in turn to such areas as bond tool design, assembly operations, and eventually to damage vulnerability and structural repairability.

In contrast to thin skin sandwich, possibly the most important adhesive characteristic for heavy-gage sandwich is a high shear strength. In light sandwich the overriding need may be to resist damage from handling and post-bond fabrication operations and therefore demands adhesive toughness and resistance to edge peel tendencies. However, in the heavy-gages, the designer sees the need to develop the strengths of thick skins without resorting to extremely long overlaps at the critical edgemembers. As I will show later, long overlaps introduce weight penalties, quality variables, and severe manufacturing problems, and the latter two items may not be detected early enough to avoid serious production schedule and cost overruns. For example, an overlap length of six inches is required to develop a .125-inch-thick skin of aluminum (7075T6) when using an allowable adhesive strength of 1400 psi. That length can be reduced to two inches with an adhesive allowable of 4200 psi. If that difference is applied to a circumferential sandwich boundary member for a large cylinder, the comparisons in weight and fabrication are obvious.

Certainly there is much more than shear strength to be considered in adhesive selection. Properties such as shear modules, (G), and "Tau Max" (maximum adhesive shear strength) are important parameters. However, the most convenient adhesive design tool still is the L/T curve. This is an empirically derived curve which relates the average shear strength of an adhesive to the ratio of bond overlap length divided by the skin thickness. And it is the data for this curve which can be made to apply directly to the design requirements.

There are some other major considerations for the designer. For example, large heavy-gage honeycomb bond assemblies present a problem in merely bringing the adhesive to the required curing temperature. All adhesives are somewhat sensitive to the time-temperature bond cycle. The thick skin panels, which probably will include a number of thick internal members, present the opportunity for large variations in heat-up rates. Also, it is likely that considerable time span will be required to perform the cycle. Whereas, common practice with the usual thin skin panels is to raise the glue line temperature to the cure point (normally 350°F) in less than 30 minutes, the heavy sandwich may require well over one hour. That places a double burden on the adhesive; it must produce consistent strengths with both slow and fast heat-up rates, and at the same time it must produce good quality over a large range of heating time durations.

Closely associated with the above adhesive cure considerations is one of using a single adhesive system for both the core and metal to metal areas. Certainly, one system is more easily controlled than two. But also, the use of two requires considerable continuous additional effort to maintain dual standards for cleanliness, processing, and inspection.

Another factor to carefully consider is the basic chemistry of the adhesive. Regardless of apparent advantages, an adhesive which produces volatiles during cure is hazardous. It necessitates bond tool bleed provisions which are difficult to provide for large complex panels and an internal sandwich venting system (perforated honeycomb core) which is undesirable. Perforated core is objectionable because any contaminating fluid (fuel, moisture, hydraulic fluids, etc.) which enters the completed panel is free to circulate and may lead to internal corrosion, excess weight, or deterioration of strength.

With heavy-gage sandwich structures it is difficult to make the mating parts conform adequately with only light bonding pressures. It is highly desirable, then, to use an adhesive system which achieves a high shear strength without the application of high bonding pressures. This is always true, but becomes nearly mandatory in heavy-gages. The reason is that the heavy skins possess sufficient inherent bending stiffness so that normal minor waviness conditions and slight mismatches do not disappear with nominally low bond pressures. It may take 50-100 psi merely to assure that the adhesive feels at least 25 psi over regions of improper contour matching.

Needless to say, the general approach to heavy sandwich must include the consideration of extreme temperature environments. Although an immediate subject application may require no temperature extreme, a good precaution is to select an adhesive which is relatively versatile. We have all seen examples where the initial environmental criteria has changed significantly during the structural design phase. In fact, this almost seems to be the rule.

Structural Design

Regarding structural design, I first will review the conditions which can create the need for heavy-gage sandwich. Generally, sandwich is suggested by the requirement to transmit extremely high compression loads through a plate or shell, since sandwich excells in instability prone structures. An added load condition which further suggests the selection of honeycomb sandwich is the existance of high forces which are transverse to the direction of primary compression loads. These lateral forces may be generated by internal pressures, thermal gradients, or concentrated load introductions. Whatever their source, these complex forces suggest the need for a structure which has a character more isotropic than orthogonal. It also suggests the capability to effectively distribute load carrying material without reducing its buckling efficiency. For example, in sandwich, the core thickness is the major buckling parameter. If high local loading requires additional material to keep the skin stresses within reason, the skin thickness can be increased without an important influence on buckling strength. Likewise, added internal reinforcement can readily be provided. Sandwich then, is apparently a good candidate for booster skirt structures, booster fuel tanks, and the compression surface of bending components (wings, fins, stabilizers).

Using a hypothetical idealized booster skirt structure as a model, I would like to develop some specific requirements, and then heavy-gage sandwich solutions.

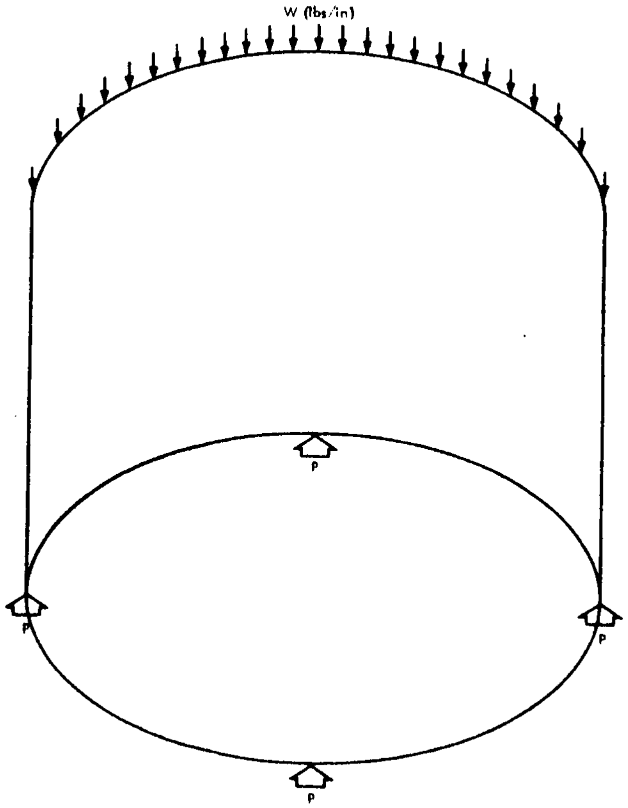


Figure 1 HYPOTHETICAL BOOSTER SKIRT

In this example, Figure 1, it is necessary to transmit high concentrated forces through a shell so that at the upper boundary the loads are distributed somewhat uniformly. Analyses can be made to show that the forces resulting from the applied loads can be forced to shear out laterally into the shell by the addition of material to stiffen the lateral regions. This can readily be provided, as required, by increasing the skin thickness in the region between load application points. Another consideration is the diminishing force level along the longitudinal lines of force application. This need can be provided for effectively by tailoring the skin gages along those lines on the basis of the shear strength capacity of the skin material.

Combining the above two considerations can produce a stepped skin arrangement as shown schematically in Figure 2. Here, zone F represents a maximum skin gage with thicknesses stepping down to that designated as Zone A.

The following sketch reveals a basic sandwich shell which includes several skin gages. For convenience I have assigned numerical values. If the applied forces (P) are approximately 2,000,000 pounds each, the skin thickness can vary from .375 at the lower end to .100-inch in the region designated as zone A. Also, note that a slight thickness increase can be provided around the upper boundary to nullify any locally induced forces. The panel core thickness is one and one-half inches as determined by a general instability analysis.

The above details exemplify the type of information which may be input to the structural designer. He has the criteria, loads, general geometry, and general analytical solutions. His problem is to assemble these inputs into a rational design which is producible and reliable.

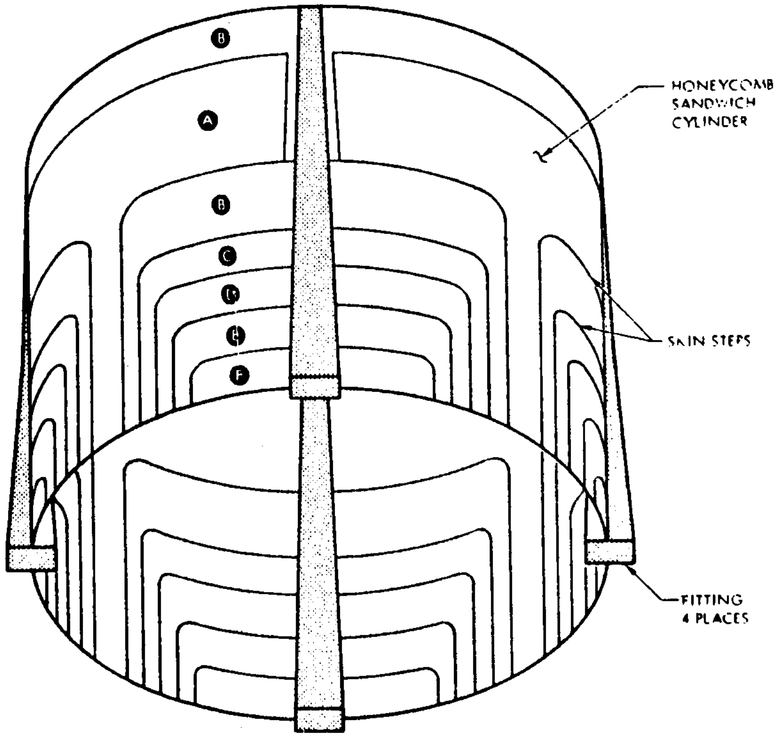


Figure 2 SKIN GAGE VARIATION ADJACENT LINE OF LOAD FEED-OUT

One decision to be made immediately is to maintain a constant core thickness to eliminate the need to match blind skin steps (or tapers). In lighter sandwich applications skin steps are normally placed against the core. This approach yields a uniform panel thickness which is aerodynamically clean and which readily mates with adjacent structural members. In heavy-gage sandwich internal steps can cause serious difficulties inasmuch as the thicker skins are also stiffer. Slight mismatching of the skin and core steps will result in sizable voids or very thick (and weak) glue lines. This is normally not a problem in thin skin sandwich because the bond pressures force the skins to conform to the core. The condition of bridging is illustrated in Figure 4.

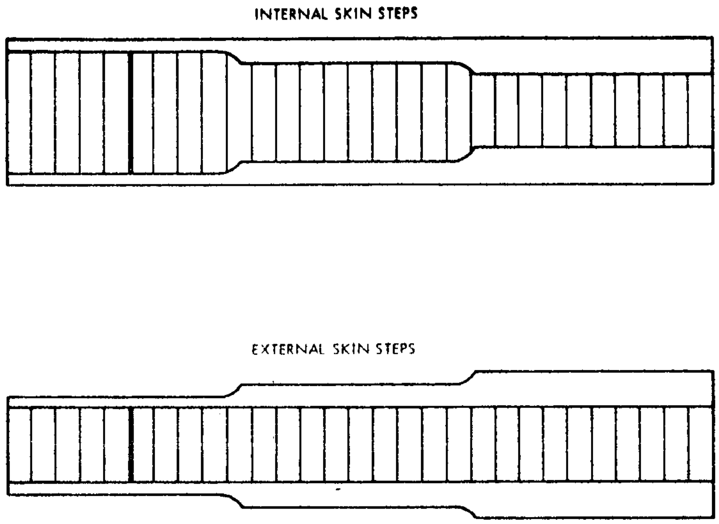


Figure 3 INTERNAL vs EXTERNAL SKIN STEPS

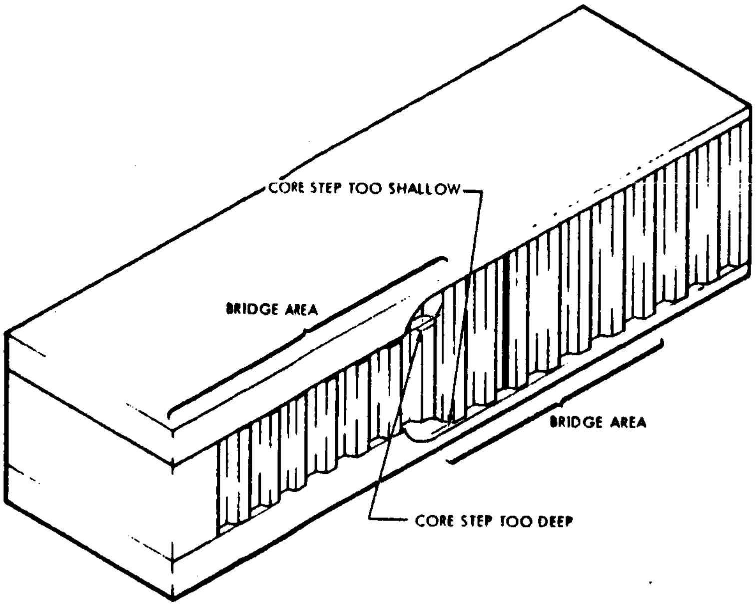


Figure 4 SKIN STEP TO CORE STEP MISMATCH

Joints and Splices

The major effort in honeycomb sandwich design is generally devoted to the definition of joints and splices. And, although a large sandwich shell includes a relatively small number of joints, they will constitute the majority of the design and manufacturing problems. If, in light sandwich, edgemember design is important, in heavy-gages it is critical. As a result a substantially greater effort must be made to assure that the edgemember bond integrity can be maintained. The penalty may be significantly higher initial tooling and fabrication costs, and in some cases it may involve edgemember weight increases.

One precaution that can be taken to assure bond joint integrity is to establish tooling and fabrication procedures that are readily controlled. An example that is common to cylindrical structures is the forming requirement of curved sandwich edgemembers. A rule here is to require, if at all practicable, the use of the stretch forming process as opposed to an incremental brake forming. The reason is that stretch forming inherently produces bond surfaces which have a more uniform curvature and smoothness than does brake forming. The local waviness of the bond surface can nearly be avoided completely. The disadvantage of local bond surface waviness is that it allows the cured adhesive thickness to vary considerably from its normal .006 to .007-inch thickness. Experience has shown that thickness variations of only a few thousandths result in substantial strength variations. This can primarily be attributed to the fact that the valley areas of the adhesive have not received sufficient bonding pressure.

At this point I want to focus your attention on a design tool which I alluded to earlier. This is the L/T curve. It represents one way to relate the calculated average shear stress to the adhesive overlap length and skin gage. It is empirically derived by testing a family of specimens which include the assumed design skin thickness and a range of overlap lengths for each thickness.

For an L/T curve to be valid, all test points must be made with one adhesive, one test temperature, and one face material.

Recently, at the Fort Worth Division, we discovered that an additional factor is significant which we had not previously recognized; that is, the direction of loading (compression or tension). Conventional L/T testing generally is based on the double overlap tension specimen (Figure 5). This configuration is relatively convenient to prepare and is easy to test. And in the lighter skin gages, it has proven to be generally reliable and relatable to design applications. However, we have found that it may not always be sufficiently accurate, particularly with heavy

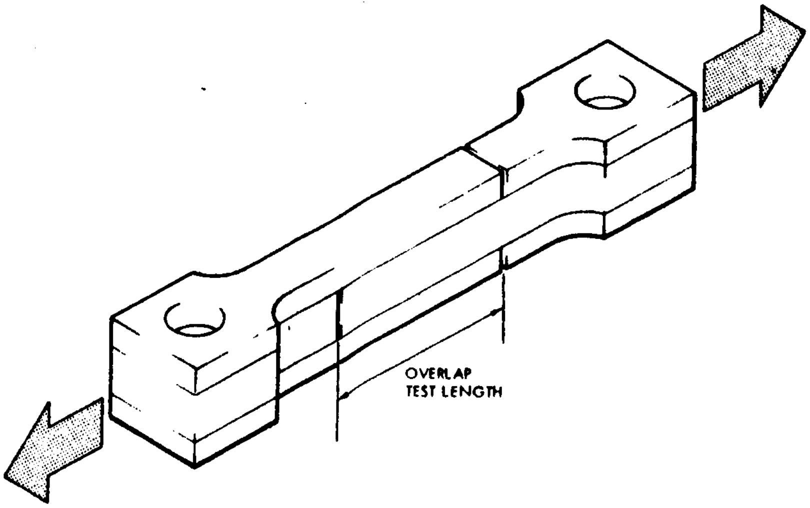


Figure 5 DOUBLE OVERLAP TENSION TEST SPECIMEN

gages. Our work has shown that compression overlap L/T specimens (Figure 6) produce higher values.

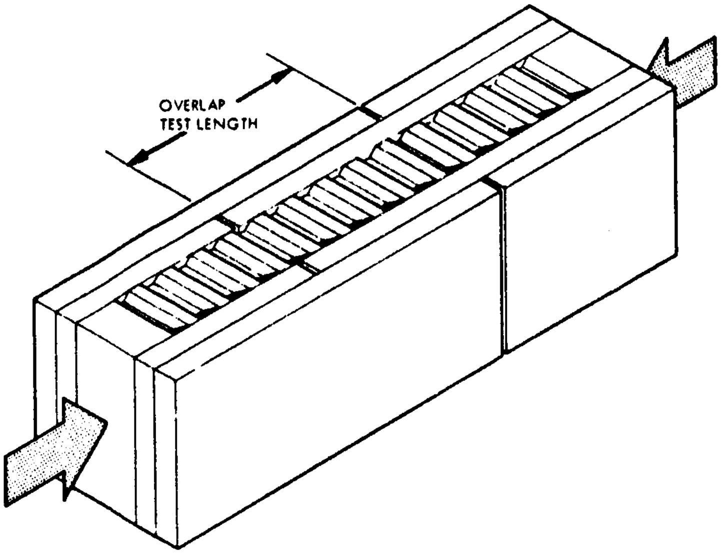


Figure 6 DOUBLE OVERLAP COMPRESSION TEST SPECIMEN

Because sandwich is generally selected to react high compression loads, this finding is extremely significant. Figure 7 summarizes L/T data for both tension and compression overlap shear tests.

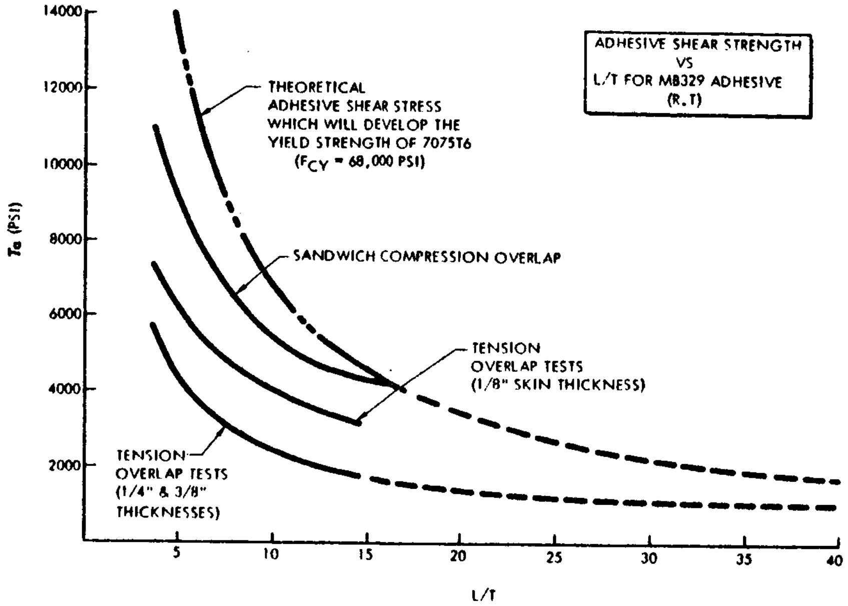
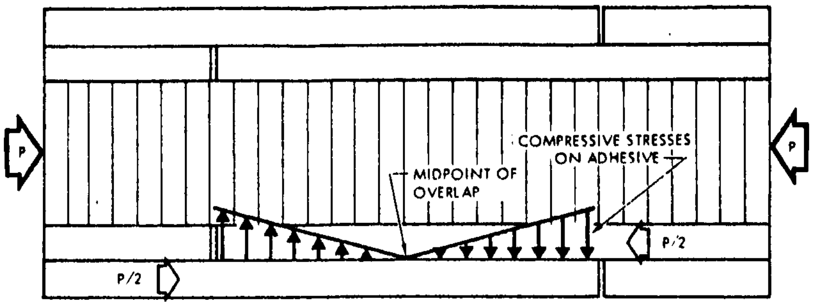
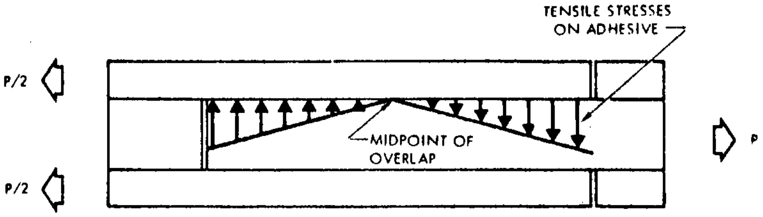


Figure 7 HEAVY-GAGE L/T CURVES

All of the data acquired for the curves involved skin gages from .100 to .500. It is also noted that the extremely heavy gages ($\geq$ .25) fall into a separate curve for the tension tests, but not in the compression tests. We have observed this phenomenon with each of two adhesive systems, although this data was plotted for Narmco's Metlbond 329. The apparent difference between the tension and compression results is that the tension test produces a local normal tensile force on the adhesive at the end of the overlap in addition to the in-plane shear stress. On the other hand, the compression overlap loading produces a normal compression force on the adhesive at the end of its overlap (Figure 8).



SANDWICH COMPRESSION OVERLAP



DOUBLE OVERLAP TENSION

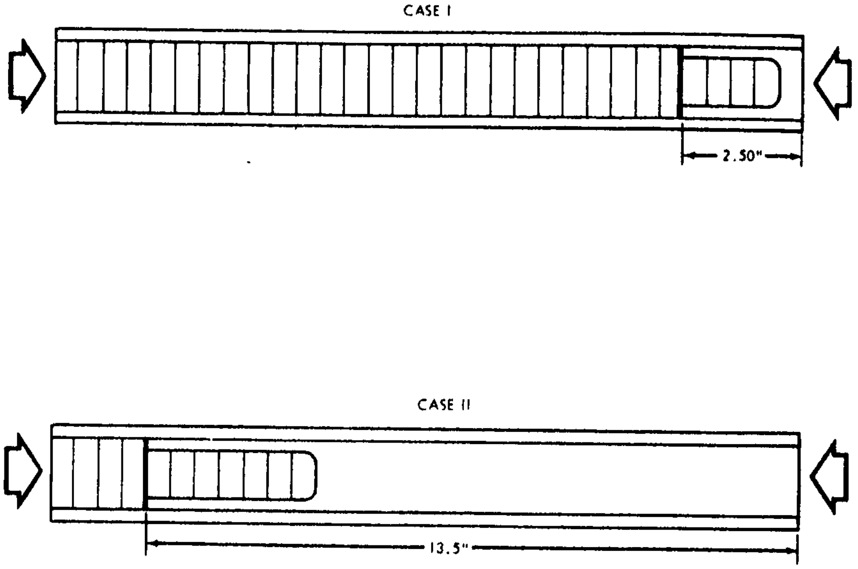
Figure 8 OVERLAP SPECIMENS SHOWING NORMAL FORCES ACTING ON THE ADHESIVE

Since the adhesive is weaker in tension than it is in compression, the computed average stress for the tension loading will be the lower of the two. Likewise, the separation of the heavier gage data from the .125 data for the tension overlap curve, results from relatively higher end tensile stresses on the heavy gages.

It becomes apparent after examining this kind of data that careful attention must be given to the basic functions required of a sandwich joint. Then, too, it must be appreciated that empirical methods are essential in determining the joint overlap length.

Referring again to Figure 7, we can measure the significance of tension versus compression loading on a .25-inch-thick skin at an edgemember by a simple example. If we assume a design at an L/T = 10, we obtain an adhesive shear stress average of 5500 psi or a bond joint strength of 13,750 lbs/in.per skin. This also would coincide with a bond line length of 2.50 inches. However, if we were to use the tension data, we would find with the same L/T and bond length that the adhesive shear stress average would be 2300 psi. This would then provide only a 5750 lbs/in. capacity. Another way to see this comparison is to look at the effect on a compression edgemember which is designed to use .250-inch skins and to carry a total load of 27,500 pounds per inch of joint width (Figure 9).

Although the illustration of Figure 9 may be extreme, it serves to point out not only a significant weight penalty, but also a very important difference in producibility. Obviously, the long overlap shown is completely impractical because of the machining, forming, and bonding difficulties. By this I mainly wish to bring out that this L/T phenomenon is of primary importance in selecting the heavy-gage joint and fabrication concepts. Once the basic concepts are determined, the detail effort will consist of the typical design task of providing the required balance between ease of fabrication and total structural integrity.



Case I

Skin T = .250 in.
Skin Load = $\frac{27000}{2}$ = 13,500 lbs/in.

at L/T = 10; τ_a = 5500 psi (See Figure 7 Compression Curve)
then L = 2.50 inches.

Case II

Skin T = .250 in.
Skin Load = 13,500 lbs/in.

τ_a = 1000 psi (See Figure 7 Tension Curve Extrapolated)
L = 13.5 inches

Figure 9 OVERLAP LENGTH COMPARISON FOR A CYLINDER EDGEMEMBER

A factor which has often impeded the utilization of sandwich is that of uncertainty in bond quality. Because the bond line is hidden, it is difficult to inspect, especially in a quantitative way. This is a valid problem and becomes more critical with the increase in required adhesive load capability associated with heavy-gage sandwich. Aside from the usual ultrasonics methods, little progress has been made towards easing this problem. It holds without saying that void detection, and a certain amount of destructive test sampling are mandatory. One other known approach is the use of a portable adhesive shear test instrument developed at the Fort Worth Division. This instrument is identified as the Portashear and is currently being examined for its potential value on heavy-gage applications. The principle of the Portashear is to apply a force to a small button specimen machined on a metal to metal bond surface (Figures 10 & 11). The average shear stress on the adhesive of the test button surface can be computed by measuring the force and dividing it by the exact specimen area. This stress for any specific button tested can then be related to the design ultimate stress for the corresponding region of the sandwich panel.

The main reason for closing with mention of the Portashear is to show how closely related are the functions of sandwich design and quality control. Because of the apparent uncertainties of maintaining absolute control of bond quality, the foreknowledge of the availability of post bond adhesive strength data will provide assurance to the design engineer. This is assurance that he needs to establish

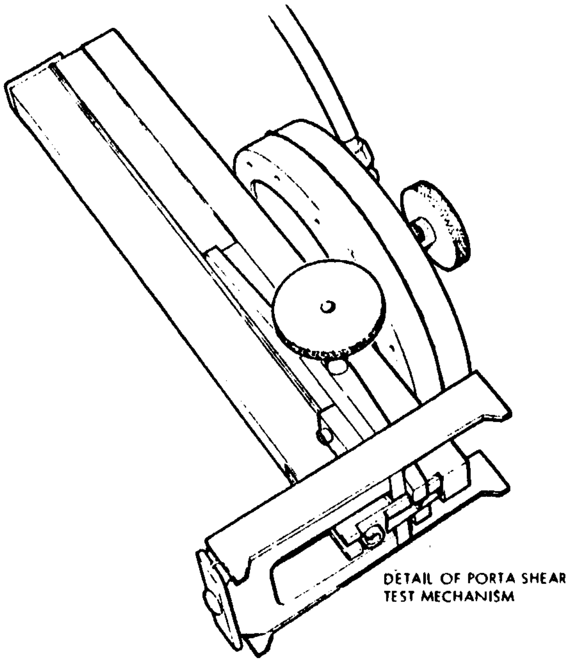
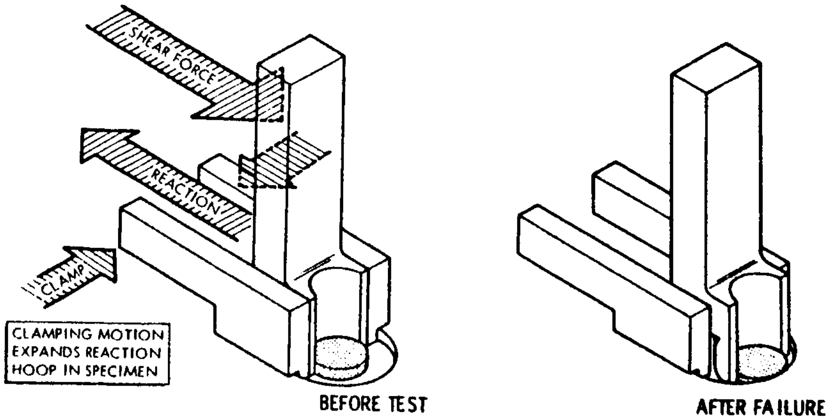


Figure 10 DRAWING OF PORTASHEAR ADHESIVE TESTER



BEFORE TEST

AFTER FAILURE

Figure 11 SCHEMATIC DRAWING OF PORTASHEAR SHEAR MECHANISM realistic design objectives and allowable strengths at the inception of the design phase.

Conclusion

Heavy-gage bonded honeycomb sandwich offers beneficial payoffs for large highly loaded boosters. It is a versatile structure which through an efficient utilization of available material can result in substantial weight savings. Also, it inherently possesses some of the usually recognized attributes of sandwich, such as, fatigue resistance, vibration dampening, and relative ease of assembly and installation.

Although the primary feature of sandwich is its resistance to compression buckling forces, it can readily be tailored to accommodate other major loading conditions without jeopardizing its primary function. Additional material can readily be provided to accept concentrated loads and equipment mounting supports.

There are design and fabrication problems peculiar to heavy-gage sandwich. However, these are similar in magnitude to any advanced structural concept and they can be resolved with careful attention to certain key areas. These areas principally concern the selection of a suitable adhesive, the control of the fits and mismatches of panel details, and the definition of a positive plan of quality control and inspection. Also, it is imperative in the design of sandwich edgemembers that the experimentally derived adhesive joint data be carefully correlated with the actual design load conditions.

SESSION I - PAPER 3.

DEEP HONEYCOMB CORE

11:00 - 11:40 A. M.

TUESDAY - MARCH 15, 1966

N66 34129

DESIGN OF THICK HONEYCOMB CORE STRUCTURES

C. A. Haudenchild, Manager
Bondolite Engineering (Sandwich Structures)
Goodyear Aerospace Corporation
Akron, Ohio

ABSTRACT

The demand for weight reduction in flight vehicles suggests the use of lightweight sandwich structures. Several factors must be considered to obtain minimum-weight sandwich constructions. High-strength thin facings combined with lightweight thick core can offer a highly efficient structure when careful consideration is given to practical proportions between facings and core, edge design details, and manufacturing techniques. This paper discusses design approaches to relatively thick-core sandwich constructions. A large cylindrical structure subjected to axial compression loading and panel-type structures requiring low deflection under transverse loads are briefly analyzed. The selection of structural adhesives for thick-core structures and manufacturing considerations also are discussed.

INTRODUCTION

When designing structures subjected to bending or pressure loadings, relatively thick honeycomb sandwich configurations may be considered to achieve minimum weight. Sandwich beams with core thicknesses ranging from 4 to 12 in. deep were compared with known data of thin configurations for actual strengths. Some loss in shear strength must be considered when the sandwich increases in depth. The rigidity of the thick sandwich beams is predictable with standard bending equations.

Certain types of space hardware require rigid panel structures with relatively light loading. Solar collectors and solar-cell supporting platforms fall in this category. Sandwich structures with facings of aluminum alloys 0.003 in. thick have been evaluated and used in this type application. Although proved sandwich equations can be used to guide the designer, physical tests often are desirable when dealing with very lightweight constructions.

Sandwich cylinders using high-strength facings with low-density honeycomb core may offer substantial weight savings when compared with monoque or sheet-stringer constructions. Determining the overall buckling of the sandwich wall or general instability is the major design consideration for this type of structure. The effects of local buckling in sandwich faces also must be considered. Complex end connections may require detail physical tests to verify analysis and to determine effects of manufacturing variables.

CHARACTERISTICS OF THICK HONEYCOMB SANDWICH SUBJECTED TO BENDING AND SHEAR LOADS

Honeycomb sandwich panel structures, when subjected to bending loads, can be analyzed. Using a simply supported beam with a center-point loading as an example,

Maximum core shear stress - $f_s = \frac{P}{t + t_c}$,

Maximum facing stress - $f_c = \frac{Pa}{4t_c t_f}$,

and

Maximum panel deflection - $\delta = \frac{Pa^3}{48D} + \frac{Pa}{4t_c G_c}$,

where

f_s = core shear stress (psi),
f_c = compression stress in face (psi),
δ = maximum deflection (inches),
P = concentrated line load (pounds per inch),
a = span of loaded beam (inches),
t = total thickness of panel ($t_c + 2t_f$),
t_c = core thickness,
t_f = face sheet thickness,
D = bending stiffness of panel, and
G_c = modulus of rigidity.

To determine the validity of the above equations for a thick honeycomb sandwich, several beam panels were constructed and physically tested.

These panels incorporated honeycomb core 4-, 6-, and 12-in. thick. The core was 5052 aluminum alloy, 0.25-in. cell of 0.003-in. foil. The faces were 2024-T3 aluminum alloy, 0.125-in. thick.

The panels were bonded with a vinyl-phenolic film adhesive. The adhesive conformed to the requirements of MIL-A-5090D (Type I)[a] specification. Tests were conducted on short beams (see Table I) to obtain the allowable shear stresses for thick honeycomb core. The length of the flexural shear specimens was such that the core would fail prior to face buckling.

Long beam specimens were used to obtain face stress and deflection data for the 4-, 6-, and 12-in. core panels (see Table II). The bending specimens were made long enough to develop face stresses above the yield point of the material. Figure 1 illustrates the flexural loading of a thick sandwich test beam.

TABLE I - SHORT-BEAM FLEXURE TESTS

Specimen	Core thickness (in.)	Span (in.)	Core ribbon orientation	Shear stress (psi)
A	4	24	Longitudinal	500
B	4	24	Longitudinal	537
C	4	24	Transverse	292
D	4	24	Transverse	300
E	6	28	Longitudinal	435
F	6	28	Longitudinal	425
G	6	28	Transverse	293
H	6	28	Transverse	298
I	12	32	Longitudinal	272
J	12	32	Longitudinal	286
K	12	32	Transverse	217
L	12	32	Transverse	196

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[a] Adhesives, Heat Resistant, Airframe Structural, Metal to Metal. Washington, D.C., Department of Defense.

TABLE II - LONG-BEAM FLEXURE TEST RESULTS

(CORE RIBBON IN LONGITUDINAL DIRECTION)

Specimen	Core thickness (in.)	Span (in.)	**Shear stress (psi)**
M	4	84	55,900
N	6	84	54,800
O	6	84	53,600
P	6	84	45,900
Q	12	84	48,900

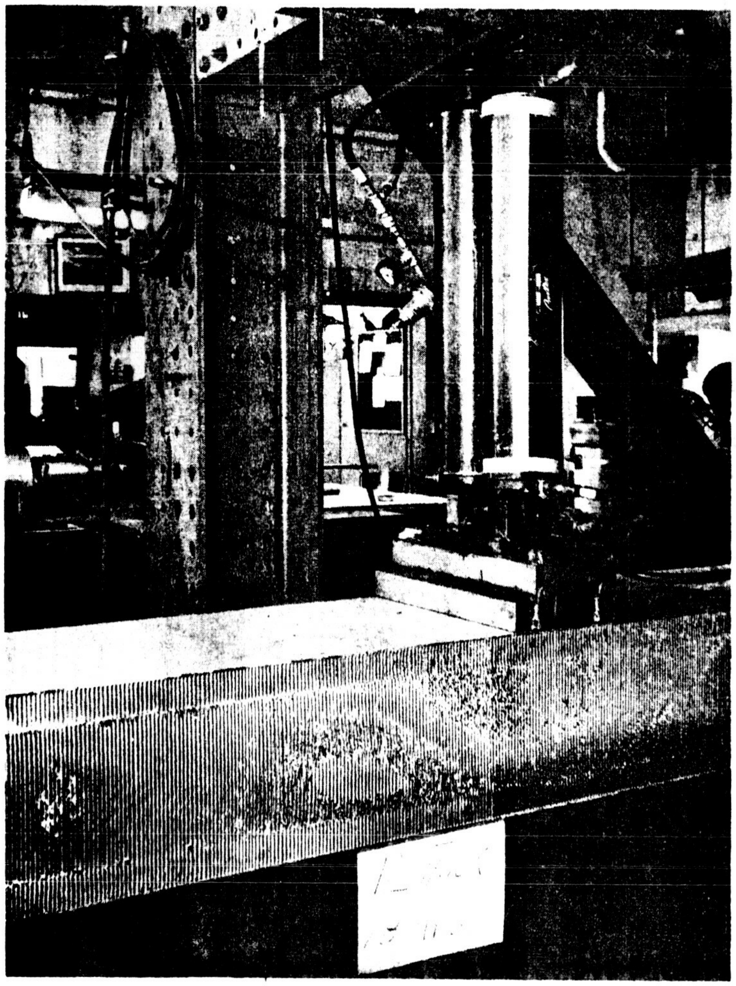

Figure 1 - Bending Test for 12-In.-Thick Panel

Results of these tests are plotted in Figure 2 to compare core shear strength with core thickness. The curves are extended to include published typical core shear strength for the more conventional thickness range.

During the long-beam flexure tests, deflection readings were recorded to provide a comparision between theoretical deflection calculations and actuals. These data are shown in Figure 3. The shear strength of thick honeycomb core is somewhat lowered when the thickness is increased. The decrease is somewhat greater in the longitudinal core ribbon direction.

In the long-beam bending specimens, the stress level of the faces on all beam tests exceeded the yield stress of the material tested. The average face stress was 51,820 psi, compared with yield stress of 2024T-3 aluminum of 40,000 psi.

In designing payload hardware for space applications, relatively large rigid panel structures with minimum weight frequently are required. Examples of this type of structure are solar collectors and solar cell platforms for producing electrical energy in outer space.

During an investigation of candidate materials for a 40-ft diameter solar collector planned for the turboelectric Astec program, several types of ultralightweight honeycomb sandwich configurations were investigated. Forty-eight folding pedals form the reflecting surface illustrated in Figure 4. One of the more promising sandwich configurations developed for these folding pedals is a honeycomb sandwich utilizing a core-thickness to face-thickness ratio of approximately 100 to 1.

Listed below and in Table III is a description and results of beam flexure tests conducted on a group of specimens.

1. Structural Components

    Faces - 0.003 - 5052H25 aluminum foil
    0.002 - 5052H25 aluminum foil

    Core - 1/8 - 0.007 - 5052H39 aluminum honeycomb

    Adhesive system - FM-1000 film, 0.025 psf

    Specimen size - width: 3 in., length: 14 in., nominal thickness: 0.30 in.

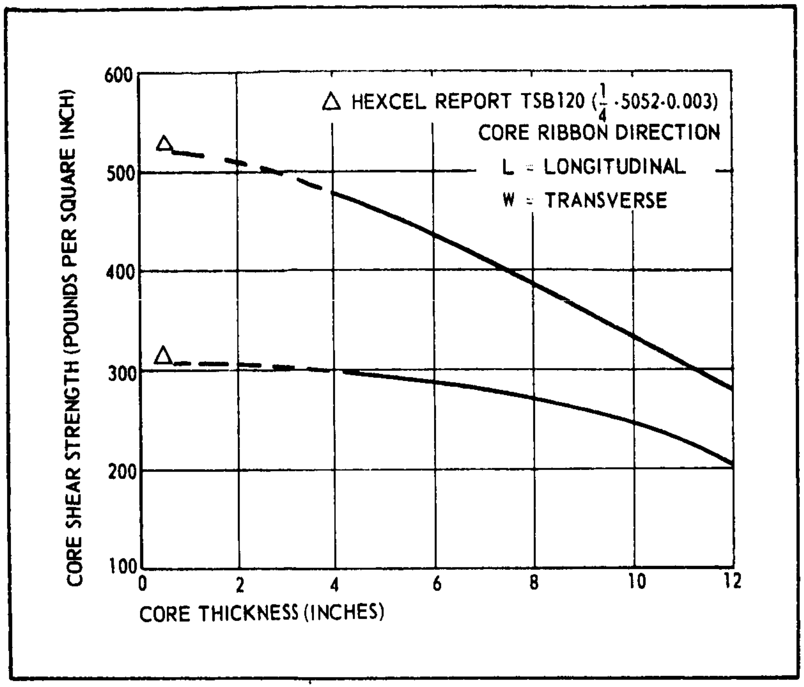

Figure 2 - Effect of Core Depth on Shear Strength

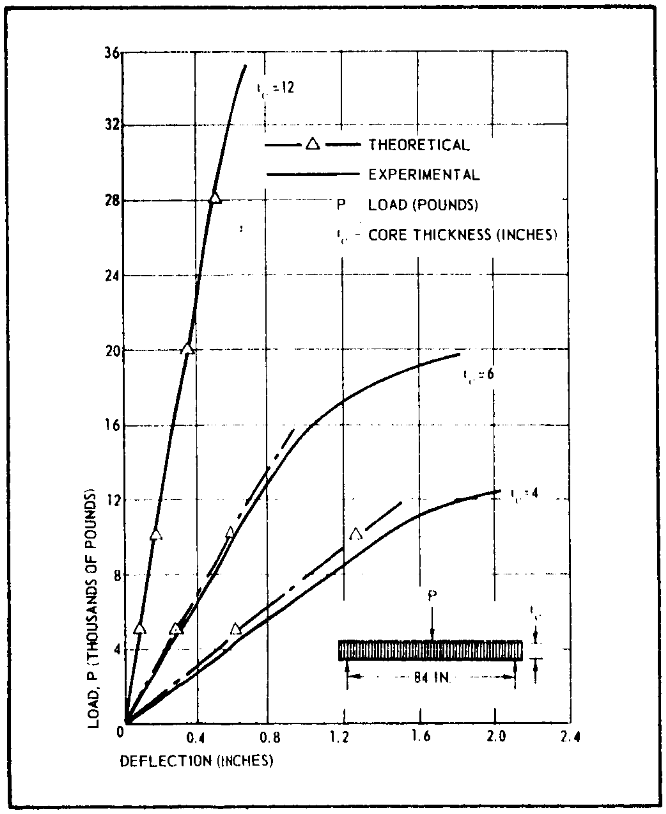

Figure 3 - Beam Deflection of Thick Honeycomb Sandwich Construction

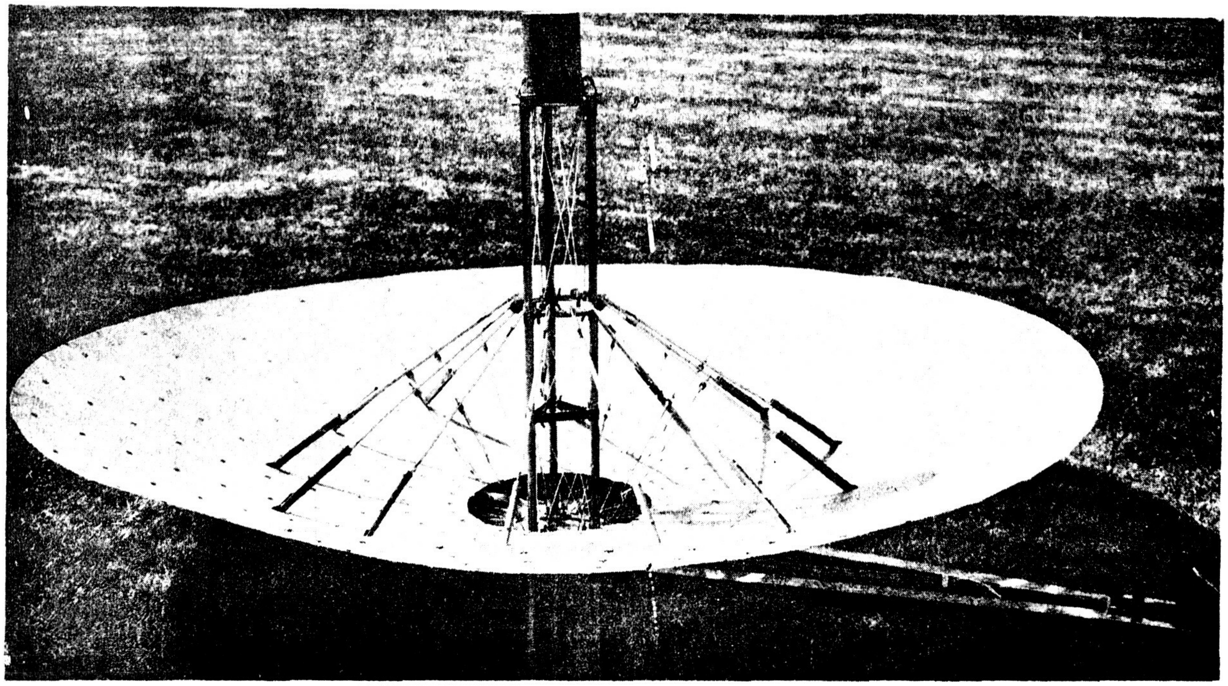

Figure 4 - Sandwich Solar Collector (Core-Thickness to Face-Thickness Ratio of 100 to 1)

TABLE III - FLEXURE TESTS ON SPECIMEN GROUP

Specimen no.	Specimen thickness (in.)	Flexural stress (psi)	Typical mode of failure
1	0.302	31,164	Combination buckling of face and core cells
2	0.297	30,530	Combination buckling of face and core cells
3	0.302	29,309	Combination buckling of face and core cells
4	0.302	29,680	Combination buckling of face and core cells
5	0.302	30,893	Combination buckling of face and core cells
6	0.302	33,339	Combination buckling of face and core cells
7	0.302	34,821	Combination buckling of face and core cells
8	0.302	35,191	Combination buckling of face and core cells
9	0.302	33,710	Combination buckling of face and core cells
10	0.299	32,603	Combination buckling of face and core cells
11	0.297	31,692	Combination buckling of face and core cells
12	0.300	32,866	Combination buckling of face and core cells
Average		32,150	

2. **Test conditions**

Longitudinal core ribbon direction - 8-in. span, 1/4 span, 2-point loading

Loading rate - develop failure in 3 to 6 min

Test temperature - +76 F

The above results produced an average face stress of 32,150 psi, which approaches the yield strength of the facing material. The test failures were a combination of face buckling and core compression, which indicated that a balanced design had been achieved for this aluminum honeycomb sandwich.

To achieve minimum weight in this type of construction, the amount of structural adhesive must be reduced to a minimum. The bonding adhesive system used weighed approximately 0.05 psf of sandwich. The overall weight of 0.2 psf of the complete sandwich is approximately the same weight as single sheet thickness of 0.014 in. aluminum. The stiffness characteristics of this composite far exceed those of the aluminum sheet. The honeycomb sandwich construction appears to approach practical minimum weight. Considerable amount of strength data, including creep characteristics and strength over a wide range of temperatures, have been developed for this type construction.

SANDWICH CYLINDERS SUBJECTED TO AXIAL COMPRESSION LOADS

1. GENERAL

Cylindrical structures subjected to axial compression loads often are important elements in space structures. Relatively thick honeycomb sandwich shell designs may offer promising weight savings.

The main factor to be considered in designing a sandwich cylinder subjected to axial compression loading is the overall buckling of the sandwich wall. This factor assumes the faces are capable of preventing local failures due to wrinkling or dimpling.

2. LOCAL DIMPLING OR MONOCELL BUCKLING

Facings can buckle or dimple into spaces between core cell walls. The stress at which dimpling of the sandwich facing will occur is given by

$$F = 2\frac{E'}{\lambda}\left(\frac{t}{s}\right)^2 ,$$

where

E' = effective compressive modulus of elasticity of the facing at stress F_c

$\lambda = 1 - \mu^2$ and μ is Poisson's ratio of facings

t = facing thickness

s = core cell size.

3. FACE SHEET WRINKLING DUE TO CORE FAILURE

When the honeycomb cell size is small enough to prevent local dimpling, the faces can be considered as supported by an elastic medium - that is, the core. Failure of the faces can occur independent of the dimensions of the structure if the core cannot stabilize the faces. This is known as face sheet wrinkling. Since this failure is localized, curvature can be neglected, and the flat plate criteria can be used in the analysis.

Mr. S. Yusuff developed equations for face sheet wrinkling. For perfectly smooth faces, the wrinkling stress is given by

$$F_{cr} = 0.96 \sqrt[3]{\eta E_f E_c G_c} ,$$

where

$$\eta = \frac{2\left(\frac{E_t}{E_f}\right)}{1 + \left(\frac{E_t}{E_f}\right)}$$

and E_t is the tangent modulus at F_{cr}.

4. BUCKLING OF SANDWICH WALL

The facing stress at which buckling of a sandwich cylinder wall will occur is given by

$$F_c = \frac{2}{5} K \frac{E' h}{r\sqrt{\lambda}} \sqrt{1 + R_f} ,$$

as referenced in MIL-HDBK-23, Part III.[a]

The constant, K, is a theoretical coefficient depending on sandwich bending and shear rigidities related by the parameter V in equation

$$V = \frac{2E' t t_c}{3\lambda d r G_c} ,$$

and R_f is a ratio of the facing stiffness to the sandwich stiffness. Generally, this ratio is a small number and can be neglected.

5. SANDWICH CYLINDERS SUBJECTED TO AXIAL COMPRESSION LOADS - INTERTANK STRUCTURE

a. General

Goodyear Aerospace designed a large cylindrical structure for the George C. Marshall Space Flight Center under Contract No. NAS8-11553. This structure is part of a program that includes the design, development, and testing of a honeycomb sandwich structure approximately 33 ft in diameter and 22 ft in length. In the initial course of the program, various comparisons were made among pure sandwich, combination of sandwich, and other structural materials. Early in the program, the pure metal honeycomb sandwich composite appeared to offer the lowest overall weight. Figure 5 shows the relationship of the intertank structure to the Saturn V vehicle. Details at the top illustrate the sandwich segment panels that were dictated by manufacturing and handling considerations. The intertank structure is located between the liquid oxygen (LOX) tank and the fuel tank. This paper does not have the space to describe in detail the complete design criteria, analysis, and other investigations associated with this program. The discussion that follows outlines both the highlights and the design conclusions reached.

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[a] MIL-HDBK-23, Part III: Design Handbook for Composite Construction for Flight Vehicles. Washington, D. C., Department of Defense, October 1962.

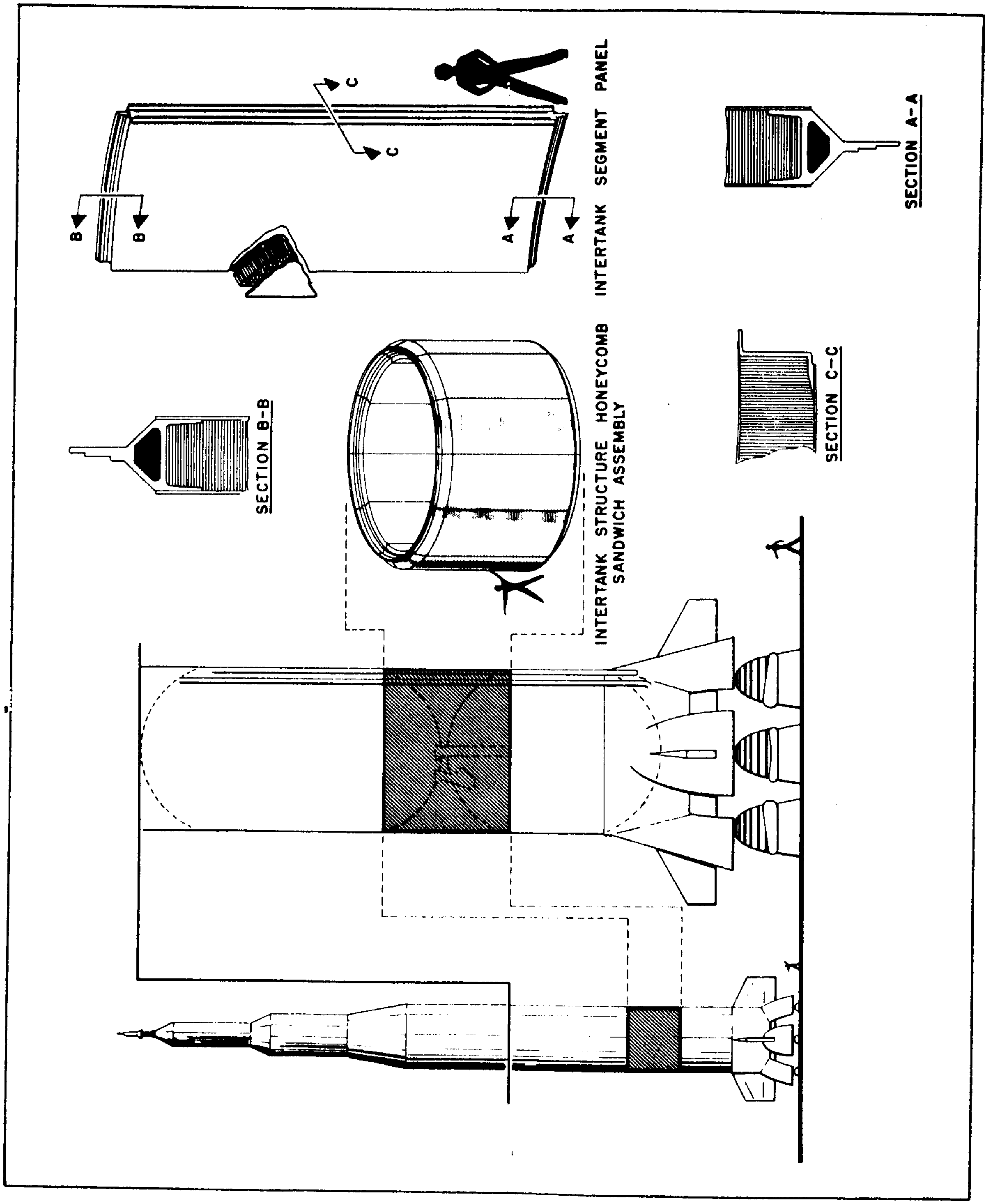


Figure 5 - Bonded Sandwich Intertank Structure for Saturn V

b. Intertank Design Criteria

The cylindrical section is subjected to axial thrust loads, shears, and bending moments at various temperature conditions. The applied loads and temperatures vary during the flight cycle. The maximum loading occurs on the structure at station 912.006. The axial load due to the bending moment is added to the axial thrust load. These combined loads are considered to act uniformly around the circumference of the cylinder and are constant throughout the length. This load is 9867 lb per linear inch (ultimate compression).

This maximum shell compression load is developed from the following conditions:

Moment = $M = 250 \times 10^6$ in.-lb (limit)
Load due to M = $P_M = -2032$ lb per inch (limit)
Axial load = $P_A = -5016$ lb per inch (limit)
N_c = ultimate axial load = $(P_M + P_A) \times 1.4 =$ 9867 lb per inch
Shear = S = 296,800 lb (ultimate)
Differential pressure = q = ±4.2 psi (ultimate)
Temperature range = -70 F to +143 F

c. Buckling Analysis of the Sandwich Wall

The basic intertank honeycomb sandwich cylinder is designed to prevent buckling instability under axial compression loads. The design is based on the analysis of cylinders as presented in Forest Products Laboratory Report No. 1830 and in MIL-HDBK-23, Part III.

In analyzing the cylinder, the following physical properties are used:

1. Aluminum honeycomb core:
    3/8 - 5052 - 0.002
    $F_c = 285$ psi
    $G_c = 39,000$ psi
2. Face skin material:

. 7178-T6 aluminum bare alloy

$F_{t_y} = 74,000$ psi

$F_{c_y} = 74,000$ psi

$E = 10,500,000$ psi

The allowable compressive buckling stress is determined from MIL-HDBK-23, which uses a reduced modulus of elasticity (E_t = tangent modulus) obtained from the stress-strain curve for 7178-T6 aluminum.

The maximum compressive load is N_c ultimate = 9867 lb per inch. By selecting a face thickness (t) of 0.08 in.,

$$f_c = \frac{N_c(\text{ultimate})}{2t}$$

$$= \frac{9867}{2(0.080)}$$

$$= 61,600 \text{ psi.}$$

At this stress level, the tangent modulus, E_t, is approximately

$$E_t = 8,900,000 \text{ psi.}$$

With a cylinder diameter equal to 33 ft, the mean radius is

$$r = \frac{396}{2}$$

$$= 198 \text{ in.}$$

Based on the selected face sheet and honeycomb core physical properties, the total sandwich thickness can be derived from the equations given in MIL-HDBK-23 for the critical buckling stress of a sandwich cylinder. A 3.50-in. sandwich thickness, d, was determined, with a honeycomb core thickness of 3.50-0.16 = 3.34 in.

The brief analysis shown below verifies the critical buckling stress in the faces using the above parameters:

$$F_{cr} = \frac{2KE'h}{5r\sqrt{\lambda}}$$

$$= 63,000 \text{ psi,}$$

where

$K = 0.978$ (from Figures 4 through 7, MIL-HDBK-23, and $V = 0.03$)

$E' = 8.9 \times 10^6$

$h = d - t = 3.50 - 0.08 = 3.42$ in.

$r = 198$ in.

$\sqrt{\lambda} = 1 - \mu^2$

This sandwich configuration yields a basic weight of 3.33 psf.

Table IV shows a summary of face sheet thicknesses of 0.07 and 0.09 and of corresponding sandwich thicknesses, critical stress levels, and the basic sandwich weight for each:

TABLE IV - COMPARISON OF FACE SHEET THICKNESSES

Item	0.07-in. thick	0.09-in. thick
Tangent modulus (E_t)	6.25×10^6	10^7
Stress level (f_c)	70,500 psi	54,800 psi
Sandwich thickness (d)	5.57 in.	2.70 in.
Critical stress (F_{cr})	70,800 psi	55,500 psi
Cylinder weight	3.543 psf	3.425 psf

Figure 6 shows a curve of weight versus sandwich thickness, where the 3.50-in.-thick sandwich develops the minimum weight for this configuration. The weights are calculated with the same core and adhesive material for each sandwich.

d. Investigation of Thermal Stress on Buckling

Thermal stresses affect the buckling strength of the structure. The presence of a temperature difference in the face sheets of a sandwich cylinder produces a biaxial stress condition in each face sheet. When the outer skin is at the higher temperature, hoop and axial stress in the outer skin is in compression, while the inner skin is in biaxial tension. In the elastic range, both modulii of elasticity and stresses

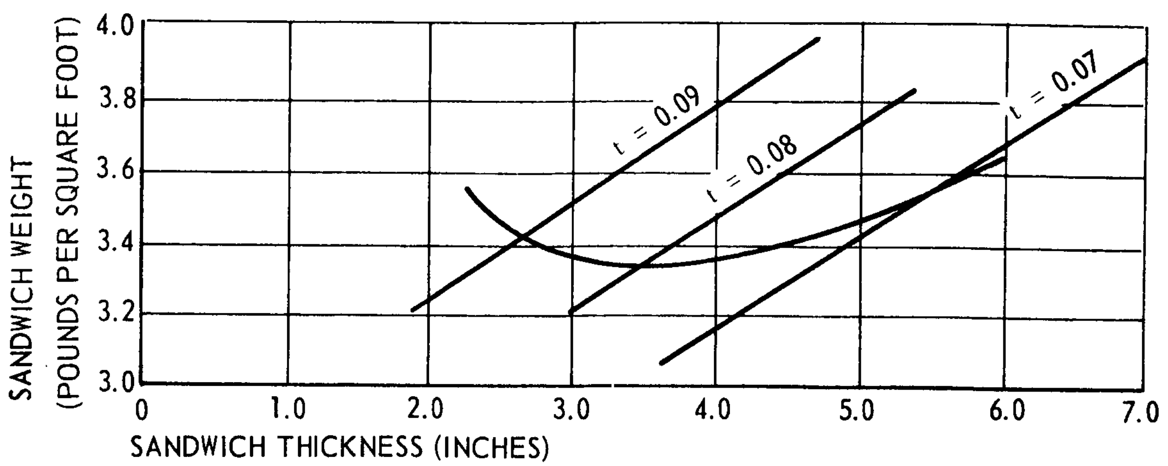


Figure 6 - Determination of Minimum Weight for Sandwich Intertank Structure

are equal, with the stresses being opposite. These stresses can be added directly to the axial loads, provided the maximum total stress is within the elastic range of the material.

If the maximum total stress exceeds the proportional limit of the material, the face stresses cannot be added directly and must be determined by using the stress-strain diagram in combination with equations of equilibrium and strain compatibility. With these parameters and the buckling equations for a sandwich with dissimilar faces (outer face beyond proportional limit and the inner face below), the critical buckling stress in the outer face is calculated to be 66,500 pounds per square inch.

Since the actual stress level varies with combinations of axial stresses from the axial load and temperature difference in the face sheets, the margin of safety also will vary accordingly. At maximum design load of 9867 lb per inch load, the temperature gradient across the sandwich is 90 F. The actual stress level in the outer face sheet increases from 58,000 to 66,500 psi.

The maximum temperature gradient occurs at engine cutoff, when the axial load is 8242 lb per inch (48,650 psi). From the thermal analysis, the temperature difference between the inner and outer face sheets is 144 F. With the above axial load and temperature gradient, the actual stress in the outer face sheet becomes 62,500 psi, which is below the allowable critical stress of 66,500 psi.

e. Design Detail

The design of the intertank structure consists of 12 honeycomb sandwich panels that are mechanically fastened to form a cylinder. An individual panel is shown in Figure 7. The 12 panels are composed of adhesive-bonded honeycomb with aluminum faces and core. Around the edge of each panel is an extruded aluminum edge. These extrusions connect into the Y rings of the LOX and fuel tanks. Longitudinal extrusions at each edge of the honeycomb sandwich panels join the panel segments.

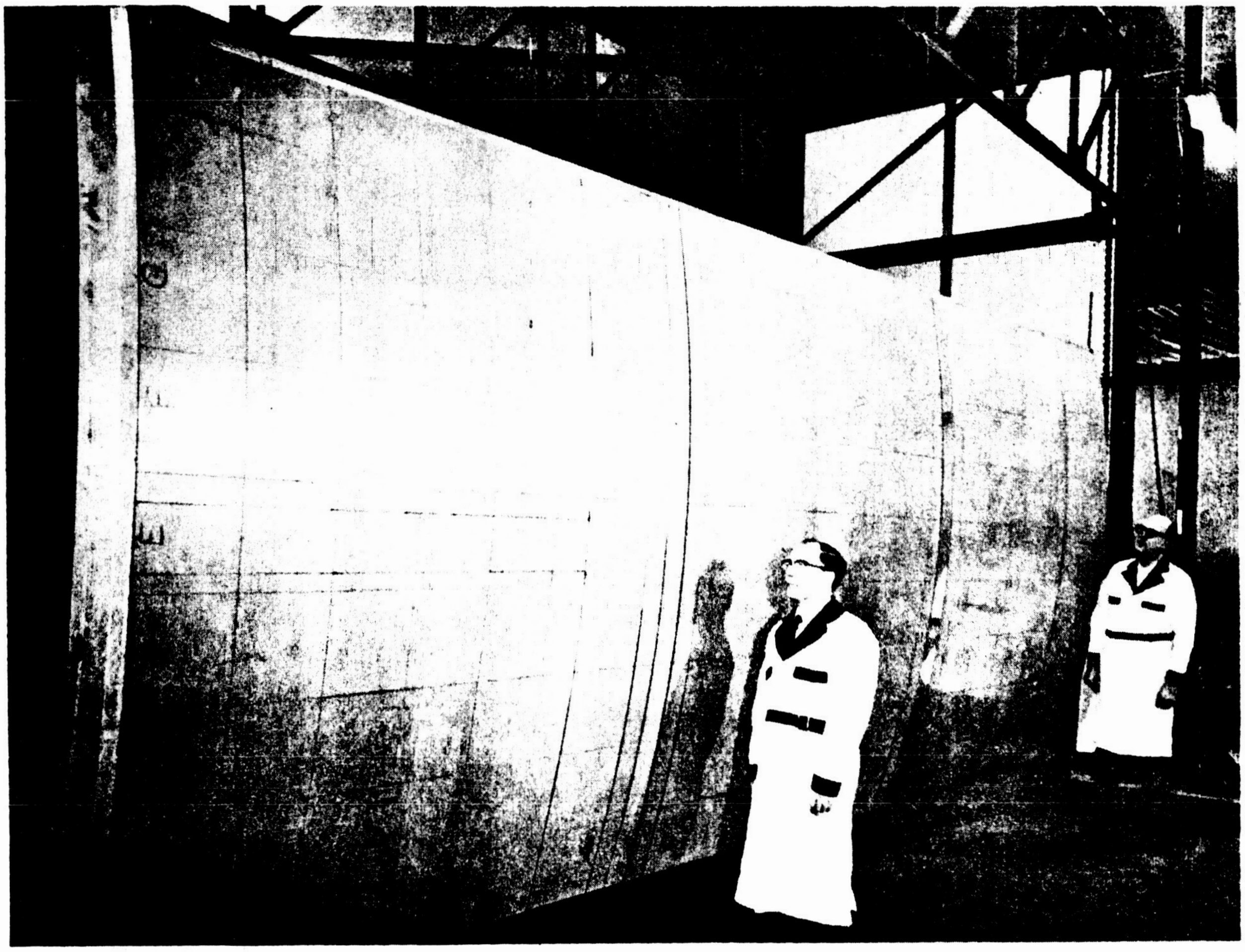


Figure 7 - Intertank Segment Panel

f. Selection of Bonding Adhesive

Several factors had to be considered before selecting adhesives. Among the most important factors was consideration of the operating temperature range and weight. To maintain minimum weight, careful consideration was necessary in selecting the adhesive for bonding the facings to the core, since this step represented a large area (approximately 4400 sq ft of bond line). The problem of obtaining face skins large enough to eliminate splices within the panel is always a factor in designing large honeycomb structures. Due to manufacturing limitations, facings of the individual panels had to be spliced. These spliced joints, as well as the metal-to-metal joints at the extremities of the panels, required special design considerations. Due to the extremely high loads being transmitted at the splice joint and the end connection, an extensive test program was established to determine the overall efficiency of the design and various candidate adhesives. In Figure 8, column compression test specimens are shown incorporating face splice joints. This type of test proved that the splice joint strength exceeded the buckling strength of the facings.

Figure 9 shows specimens of the panel end connections that were used to evaluate adhesive end design details. Manufacturing variations, effective tolerances and metal facings, adhesive glue lines, and several other factors were studied in this phase of the program. The final design resulted in average strengths of 13,174 lb per linear inch.

Two structural adhesives were selected for bonding the structure. A combination type using nitrile-phenolic and modified epoxy bonds the facing to the honeycomb core.

A polyamide epoxy type developing very high shear strengths was selected for the metal-to-metal bonds. These adhesives are compatible from a chemical standpoint.

As this paper is being written, preparations are under way to conduct a static test on the complete intertank structure.

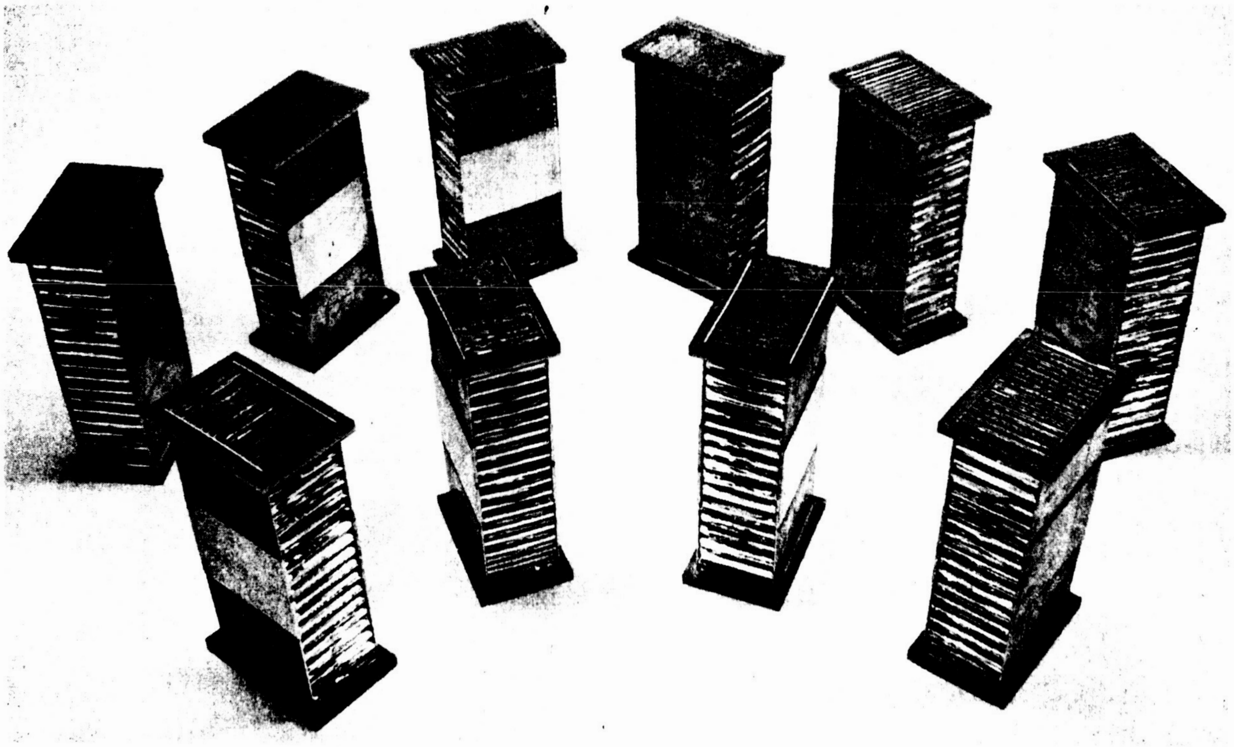


Figure 8 - Compression Test Specimens

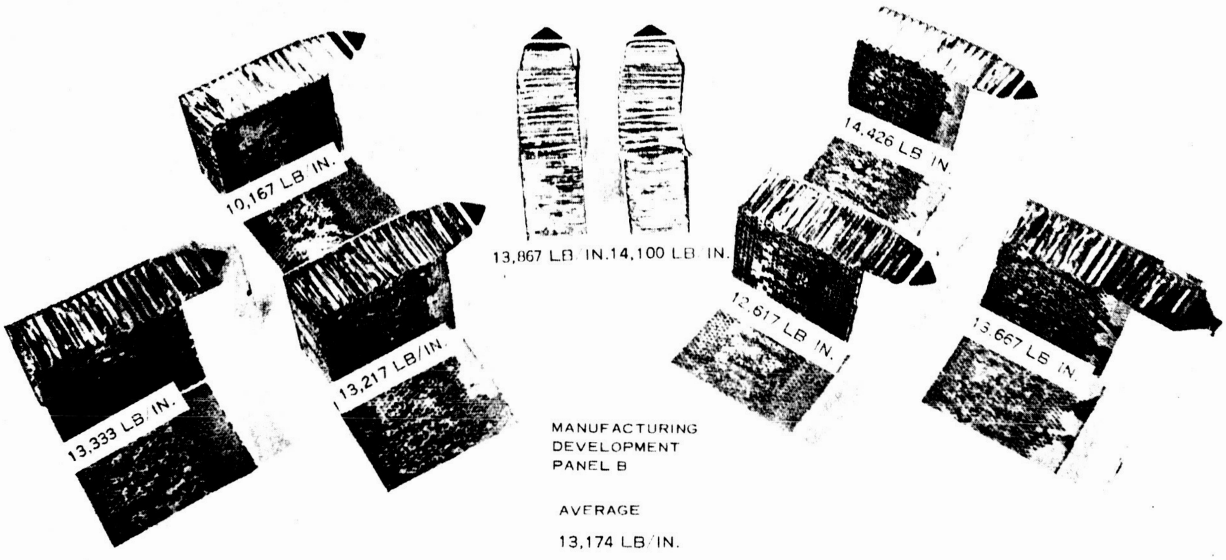


Figure 9 - End Connection Compression Tests

Figure 10 shows the structure in final stages of assembly, with loading tension rods partially installed. The test loads will be applied by 144 hydraulic actuators operating from a common hydraulic source. This test facility can develop a combined axial load of 14,000,000 lb.

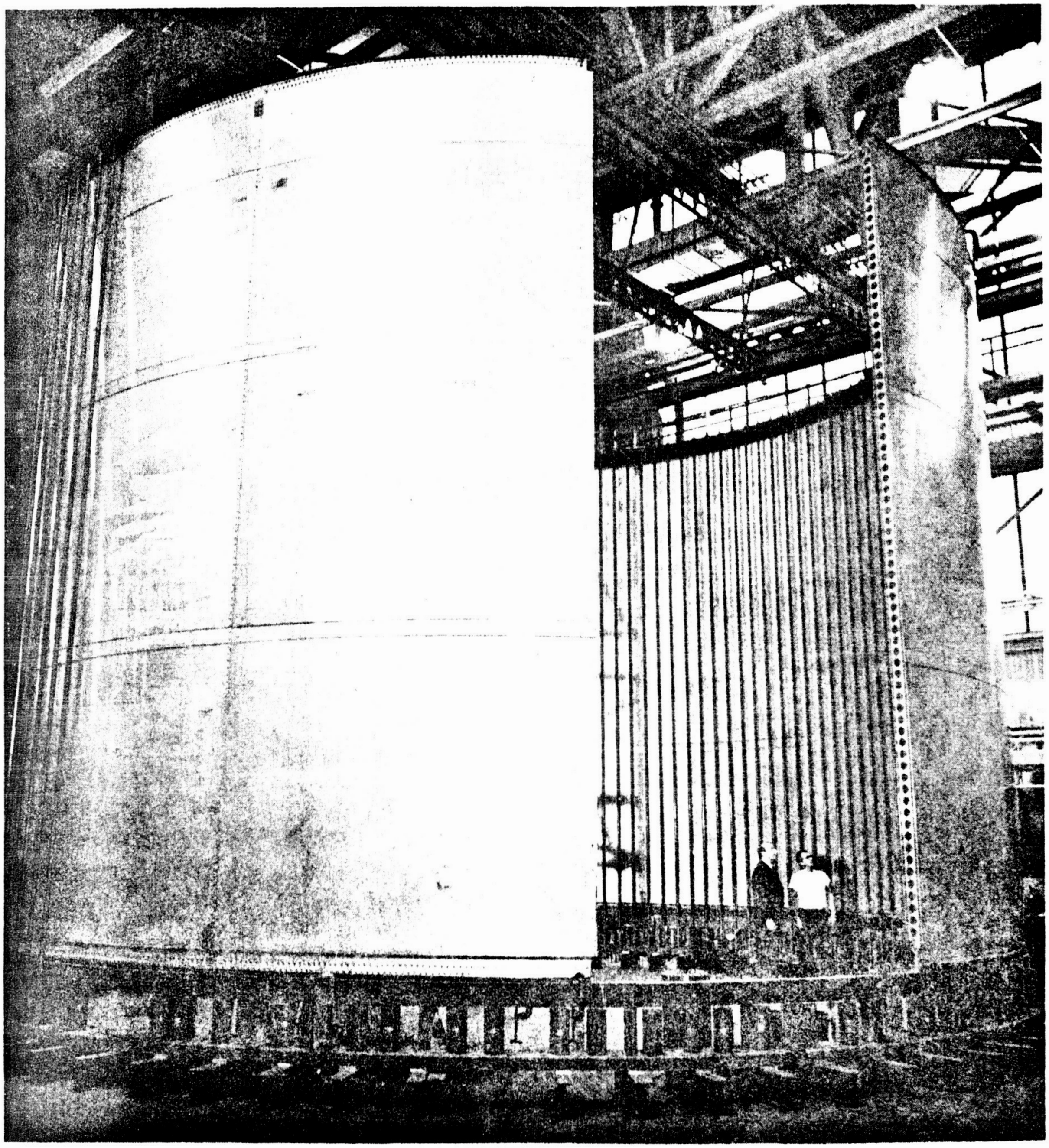


Figure 10 - Intertank Structure Partially Erected in Test Facility

N66 34130

SESSION II - PAPER 1.

MATERIALS FOR SPACE CABIN ENVIRONMENTS

1:30 - 2:10 P. M.

TUESDAY - MARCH 15, 1966

SPACE CABIN MATERIALS

by

Robert Holden
Grumman Aircraft Engineering Corporation

ABSTRACT

The outgassing characteristics of a number of non-metallics exposed to space craft cabin environment is reported. Included is a general description of analytical methods employed involving primarily gas chromatography and infra-red spectroscopy. Behavior of materials in oxygen and vacuum is reflected in their percentage weight losses.

Space Cabin Materials

by

Robert Holden
Grumman Aircraft Engineering Corporation

The selection of non-metallics for space craft construction has introduced certain problems which were not considered to be of significance in the manufacture of air craft.

Functional and structural integrity of non-metallics must be maintained when exposed to the extremes of space environment; in addition, their successful employment in a space vehicle crew compartment area is dependent on outgassing potential and stability to a selected habital atmosphere.

During space flight an astronaut must be equipped with his own environmental control system. This can be a space suit or a supply of survival gases for "shirt sleeve" conditions in a space vehicle cabin.

Due to weight problems, present space vehicles are equipped with minimum atmospheric purification systems depending on small activated carbon filters, lithium hydroxide beds, and most importantly, on a careful selection of materials for construction.

I will discuss this latter problem of material selection methods with you, and how we at Grumman are employing such for the Lunar Excursion Module.

The basic environmental exposure equipment employed for evaluating non-metallics and their outgassing products is represented by Slide 1. Essentially, the main reaction chamber is a two liter resin flank fitted with a Teflon Viton rubber gasket and with greaseless Viton "O" ring ball socket joints. The center joint is fitted with two thermocouples which serve as a sample holding device. Both thermocouple junctions are in contact with the sample. Heat is supplied by means of a mantle and regulated through a time-proportioning control instrument of the pulsing type and thermocouples attached to the mantle element. The two thermocouples are used to indicate sample temperature on a portable potentiometer and a circular chart recorder. The resin flank is combined with a series of traps and inlet tubes to permit evacuation, pressure readings, oxygen introduction, helium flow for trappings purposes, and sample withdrawal for quantification of volatile outgassing components. Samples of non-metallics are exposed for 72 hours at 200°F and 5 psia oxygen; weights are about 7 to 10 grams and the area is approximately 16 square inches. The upper operating temperature of LEM is 160°F and the crew compartment will be maintained at 5 psia 100% oxygen. A 40°F increase in environmental exposure temperature is added for a safety factor.

Basically, the analytical technique consists of exposing a designated sample under the stipulated test condition, after which a gas sample is withdrawn from the resin flank into a 250 ml. gas bottle. The remaining volatile gases are entrained in helium and collected in a liquid nitrogen trap. The trap is fixed to the gas inlet valve of an F & M 720 chromatograph and the components fractionated and identified by infrared and mass spectra. The gas bottle containing the outgassed mixture sample with its major components now identified through the previous procedure of concentration and chromatographic fractionation is fixed to an F & M 609 flame ionization chromatograph. Fractionation is accomplished using helium as a carrier gas and the components quantitated by comparing areas against previously prepared standards. Carbon monoxide is determined by employing the F & M 720 chromatograph and a 6 foot 1/4" stainless steel 5A molecular sieve column.

For a specific example, I have included a non-metallic which was analyzed for potential atmospheric contamination production using the gas chromatography procedure previously outlined. The material is a two-part epoxy conformal coating, - Dennis 1160 - which had been cured at 160°F for 8 hours. Film thickness was approximately 10 mls and the exposed surface area was 32 sq. inches within the reaction flaks. Slide 2 is a chromatograph representing the separation of the cryogenically trapped volatiles. Peak "C, D, E" is a composite consisting of an early elution of methyisobutyl ketone, a mixture of toluene and the ketone followed by a predominant cut of toluene. Slides 3A, 3B, & 3C show the I.R. spectra of the two compounds taken at intervals as indicated on the chromatograph. A return to slide 2 shows the water peak which, when trapped, was contaminated with xylenes. All other compounds isolated were shown to pure.

Slide 4 represents a chromatogram produced from a gas sample taken from the resin flask. The compounds were quantitated using this chromatogram against pure standards of known concentration. Concluding, the epoxy outgassed the following detectable compounds after exposure to the test conditions:

1 - Acetaldehyde

2 - Acetone

3 - Methyl isobutyl ketone

4 - Toluene

5 - Water

6 - N Butonol

7 - Ethylene glycol monobutylether

8 - Xylenes

9 - Total Organics - expressed as pentane per 100g of material outgassed - 1000 micro moles per 100g.

The weight loss of materials in 100% oxygen at 200°F and at 10^{-6} mm. Hg. along with the amount and nature of any toxic products envolved is evaluated bearing in mind the quantity and location of the material used within the LEM crew compartment. Where two or more similar materials fulfil design requirements equally well, preference will be given to the use of the material suffering the least environmental degradation or producing the least toxic outgas product. Reference is made to a joint Air Force Document "Threshold Limit Values for Toxic Chemicals" APF-161-2-1; TB Med 265 dated 8 Feb. 1962; current literature and NASA information. The weight limitations of materials are controlled so that the reference maximum acceptable contaminant levels are not exceeded. A major contaminant of an outgassed mixture is one which exceeds 50 micromoles/100g of material tested expressed as pentane equivalents. Failure to identify an outgassing component above this concentration level shall constitute a materials rejection.

Throughout the past year and a half the Grumman Engineering Laboratory has evaluated over 100 materials, isolating and identifying some 40 odd outgassing compounds. Slide 5 shows a comparison of those compounds isolated throughout our material evaluation program and those stripped from a Mercury space craft carbon filter following an actual orbital flight Slide 6.

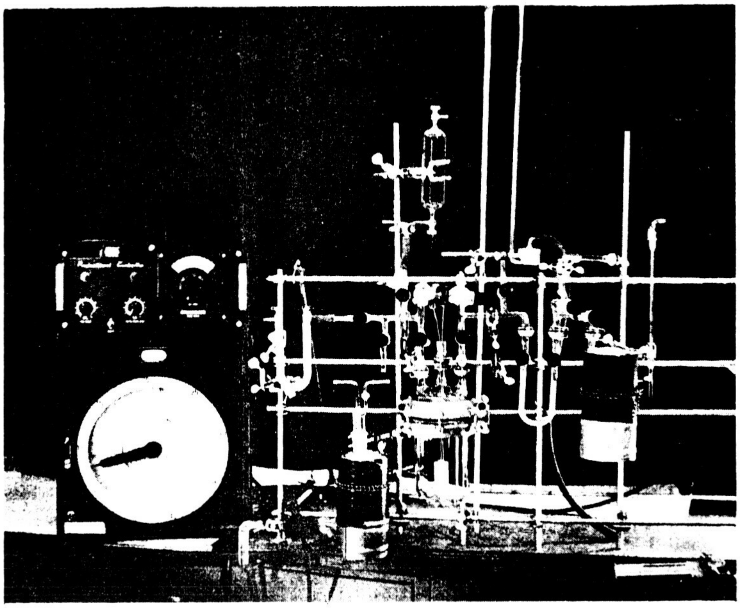
In summary, I would like to present data on some of our findings in particular for those materials evaluated for use within the LEM Crew Compartment.

Slide 7 lists findings of a partial list of approximately 20 materials accepted for use along with the most pertinent test results.

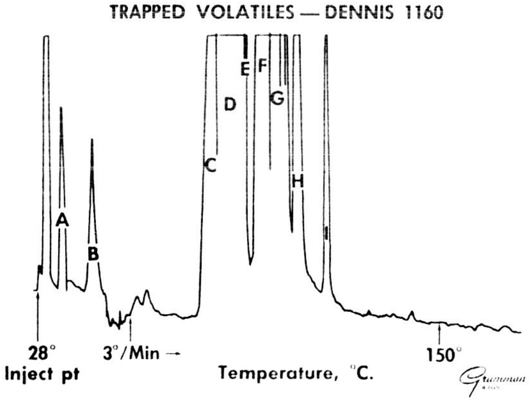
Slide 8 is of interest since it points out the problem of material interaction with oxygen and the difficulty of fixing a per cent weight loss as an acceptance criteria. In most cases, if a material seriously interacts with oxygen, although it shows a low weight loss or a possible weight gain, the outgassing products will be extremely high, thus forbidding its use within the crew compartment.

Lastly Slide 9 list a number of material along with the most important reasons for their rejection.

At the present time, the Grumman Materials Laboratory is continuing to search out more rapid analytical testing procedures for evaluating non-metallics for space craft applications.



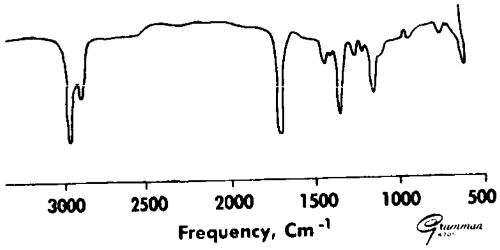
Slide 1



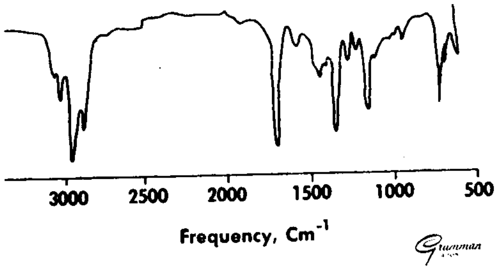
A - Acetaldehyde
B - Acetone
C - MIBK
D - MIBK > Toluene
E - Toluene > MIBK
F - Water
G - Xylene
H - n-Butanol
I - Ethylene glycol monobutyl ether

Slide 2

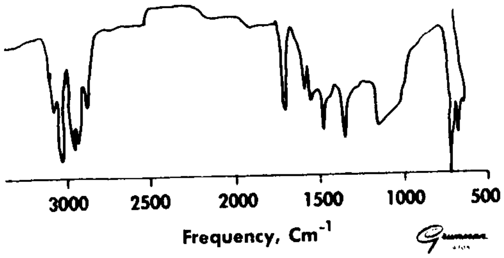
PEAK C, IR SPECTRUM, MIBK



PEAK D, IR SPECTRUM, MIBK > TOLUENE

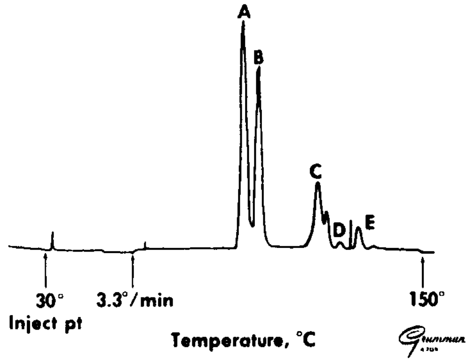


PEAK E, IR SPECTRUM, TOLUENE > MIBK



Slide 3

FLAME IONIZATION — VOLATILES, DENNIS 1160



A - Methyl isobutul ketone
B - Toluene
C - Xylene
D - n-Butanol
E - Ethylene glycol monobutyl ether

Slide 4

Slide 5

COMPONENTS ISOLATED FROM NON-METALLICS UNDER SIMULATED LEM ENVIRONMENTAL EXPOSURE CONDITIONS

water
ammonia
carbon monoxide
carbon diodide
hydrogen cyanide

aliphatic siloxanes
aromatic siloxanes
boron trioxide
mineral oil
dicydiamide

ethylene
methyl methacrylate
styrene
benzene
toluene
xylene
n-propyl benzene
chloroform
methane
aliphatic hydrocarbons (C_8 & Above)

2-propanol
n-butanol
cyclohexanol
tri-methyl silanol
acetone
methyl ethyl ketone
methyl isobutyl ketone
acetaldehyde
butyraldehyde
valeraldehyde
2-methyl butanal
furfural
acrolein
ethylene glycol monethyl ether
ethylene glycol monobutyl ether
ethylene glycol monomethyl ether acetate
n-propyl acetate
acetic acid

Slide 6

FOUND IN MERCURY SPACECRAFT

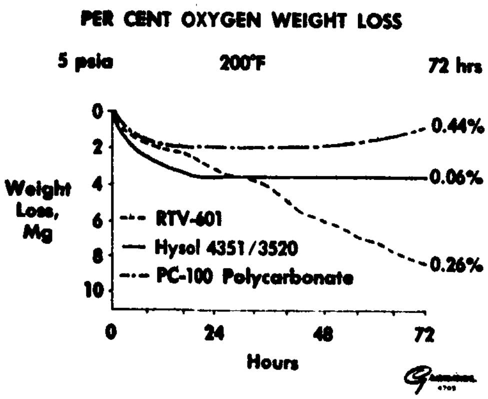
freon 11
freon 12
freon 114
freon 22
freon 23
ethylene dichloride
toluene
n-butanol
vinyl chloride
ethanol
m-xylene
o-xylene
vinylidene chloride
methylene chloride
bensene

trichloreothylene
acetone
methyl ethyl ketone
methyl isopropyl ketone
n-propanol
acetaldehyde
ethyl acetate
methanol
1-4-dioxane
cyclohexane
formaldehyde
hexamethylcyclotrisilane
methyl chloroform
ethylene

Reference: Saunders - Contamination Pattern in the Enclosed Atmosphere of Mercury Spacecraft. Page 709-723 T&A Proceedings of the first Space Vehicle Thermal And Atmospheric Control Symposium.

Slide 7

Product	Type	Supplier	% vacuum wt. loss 10^{-6}, 200^{o}F, 48 hrs.	% Oxygen wt loss 200^{o}F-72 hrs. 5 psia	Total Organics Micro moles/ 100g expressed as pentane	Comments
Elastomers						
Silastic 675-24-480	Silicone	Dow Corning	0.09		16	
Silastic 6526-4300	Silicone	Dow Corning	0.56	0.29	14	
Viton A	Fluorocarbon	Du Pont	0.12	--	8	
IDSA 9250	Urethane	Disogrin Ind.	0.30	0.14	15	
Plastics						
Plex 55 Stretch	Methyl methacrylate	Swedlow & Goodyear	0.46	0.49	25	Slight warpage of sample in 0_2 & vac.
Plex II UVA	Methyl methacrylate	Goodyear	0.03	--	15	Slight warpage in 0_2 & vac.
Merlon	Polycarbonate	Mobay	0.03	0.15	16	
Kynar PVF_2	Polyvinylidene fluoride	Raychen	0.11	0.15	41	
Adhesives						
Metalbond 329	Epoxy	Narmco	0.06	--	17	
FM-1000	Nylon-epoxy	Bloomingdale	0.67	0.56	75	High C0 Toluene
RTV 731	Silicone	Dow Corning	0.95	0.40	7	Acetic acid low
Epon 919	Epoxy	Shell	1.0	0.16	40	MIBK & toluene solid outgassing identified as cellosolve; C0
Potting Compounds						
RTV-601	Silicone	Dow Corning	0.50	0.26	69	Methane outgassing
Stycast 1090	Epoxy	Emerson & Cuming	--	--	--	
EC 1663	Silicone	3M	0.90	0.49	152	Ethanol; Vacuum solid outgassing - siloxanes
DC 325	Silicone	Dow Corning	1.0	0.25	112	Solid outgassing, siloxanes
Coatings						
400 Series Gray	Poly ester	3M	1.46	1.17	129	Toluene xylene cellosolve (outgassing) (0_2 wt. gain)
N 350A	M_oS_2 Phenolic Binder	Lubeco Corp.	1.0	--	123	Thin film (high temp. cure)
Greases						
FS-1281	Fluorocarbon	Dow Corning	1.9	0.04	35	
PR-240 AC	Fluorocarbon	Du Pont	3.9	0.07	25	



PER CENT OXYGEN WEIGHT LOSS

5 psia 200°F 72 hrs

Slide 8

Slide 9

Product	Type	Supplier	% vacuum wt. loss 10^{-6}, 200°F, 48 hr.	% Oxygen wt loss 200°F-72 hrs. 5 psia	Total Organics Micro moles/ 100g expressed as pentane	Comments
Elastomers						
S-604-7	Silicone	Parker Seal	0.69	--	--	Heavy opaque solid outgassing siloxanes
Plastics						
Vespl SP-1	Polyimide	Du pont	0.21	--	17	High C0, failed odor
A. B. S.	A. B. S.	U. S. Rubber	--	wt. gain	48	failed odor softens in 0_2
Adhesives						
Epon 923	Epoxy	Shell	1.1	0.19	230	High total organics, C0
Tadcal	Polyester	Fasson	2.7	wt. gain	3000	Oxygen interaction (high C0, acetaldehyde)
Bondmaster M648/ Ch 16	Epoxy	Rubber & Asbestos	2.4	--	421	High total organics (cyclohexanol)
Potting compounds						
Sylgard 184	Silicone	Dow Corning	1.2	0.17	93	Heavy solid outgassing in vacuum (siloxanes)
Coatings						
Emralon 320	Fluoro Carbon dispension	Acheson Colloids	1.5	1.7	2400	High total organics C0 (R. T. cure)
		Andrew Brown	2.2	2.2	4600	High Total organics (benzene)
Brolite XA-193						
FX-705	Fluorocarbon	3M	6.3	4.7	520	High wt. loss (solid outgassing (acidic)
Ucarsil R-101	Silicone	Union Carbide	5.7	--	19,900	Ethanol high vacuum wt. loss
Greases						
Braycoat 617	Fluorocarbon	Bray Oil	86	--	--	High Vacuum wt. loss
Whitekote 505	Hydrocarbon	Alpha Molykote	25	--	63	High Vacuum wt. loss
Misc.						
Stur D Lace 18 D96	Buna-N on dacron	Gudebrod	0.33	--	128	HCN

SESSION II - PAPER 2

CRYOGENIC ADHESIVE APPLICATION

2:10 - 2:50 P. M.

TUESDAY - MARCH 15, 1966

N66 34131

CRYOGENIC ADHESIVE APPLICATION

RICHARD L. LONG
MISSILE AND SPACE SYSTEMS DIVISION
DOUGLAS AIRCRAFT COMPANY, INC.

PRESENTED TO
STRUCTURAL BONDING SYMPOSIUM
MARSHALL SPACE FLIGHT CENTER

HUNTSVILLE, ALABAMA

15 – 16 MARCH 1966

DOUGLAS

DOUGLAS MISSILE & SPACE SYSTEMS DIVISION

CRYOGENIC ADHESIVE APPLICATION

R. L. Long
Section Chief, Adhesives & Plastic Development
Non-Metallics Branch
Materials Research & Production
Methods Department

ABSTRACT

Thermosetting and catalyzed adhesives are used extensively on the Saturn S-IV and S-IVB stages in elevated and cryogenic environments. The history of the selection of the adhesives and the evolution from the original choices to the materials currently used at Douglas for cryogenic environments are discussed in this paper.

Only the low temperature cure bonding materials are carried forward, and the general presentation concentrates on polyurethane adhesives. The discussion on polyurethane adhesives includes a limited evaluation of the advantages and disadvantages of this material both with respect to properties and the processes of application.

Special tests and more useable methods of data presentation have evolved as a by product of the development of the polyurethane adhesives and new applications are possible.

Recommendations are made to obtain a more versatile low temperature curing cryogenic adhesive, and suggestions are made on the economical application of new research and development techniques to obtain this new material.

INTRODUCTION

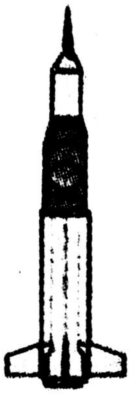
This presentation is intended only as a superficial introduction to the subject of adhesives and to the problems connected with their use in cryogenic environments. The subject will be confined to the structural bonds on the Saturn S-IV and S-IVB stages and will be presented in a form to indicate the general types of work done on adhesives.

To best handle this subject, the discussion will be presented in four parts: (1) some typical applications and the corresponding requirements, (2) a brief history of the selection of the adhesives both hot and cold setting, (3) some typical problems and developments as applied to low temperature curing polyurethane adhesives, and (4) recommendations on future work to improve cold setting cryogenic adhesives.

APPLICATIONS

The Saturn S-IV was the first Douglas-manufactured stage to use liquid hydrogen as a propellant (Figure 1). This stage had structurally bonded sandwich interstages, common bulkhead, clips to hot and cold locations, internal insulation and clips bonded to the inside wall of the liquid hydrogen tank plus numerious miscellaneous fabrics, instruments and wire bundles bonded in

DOUGLAS SIV



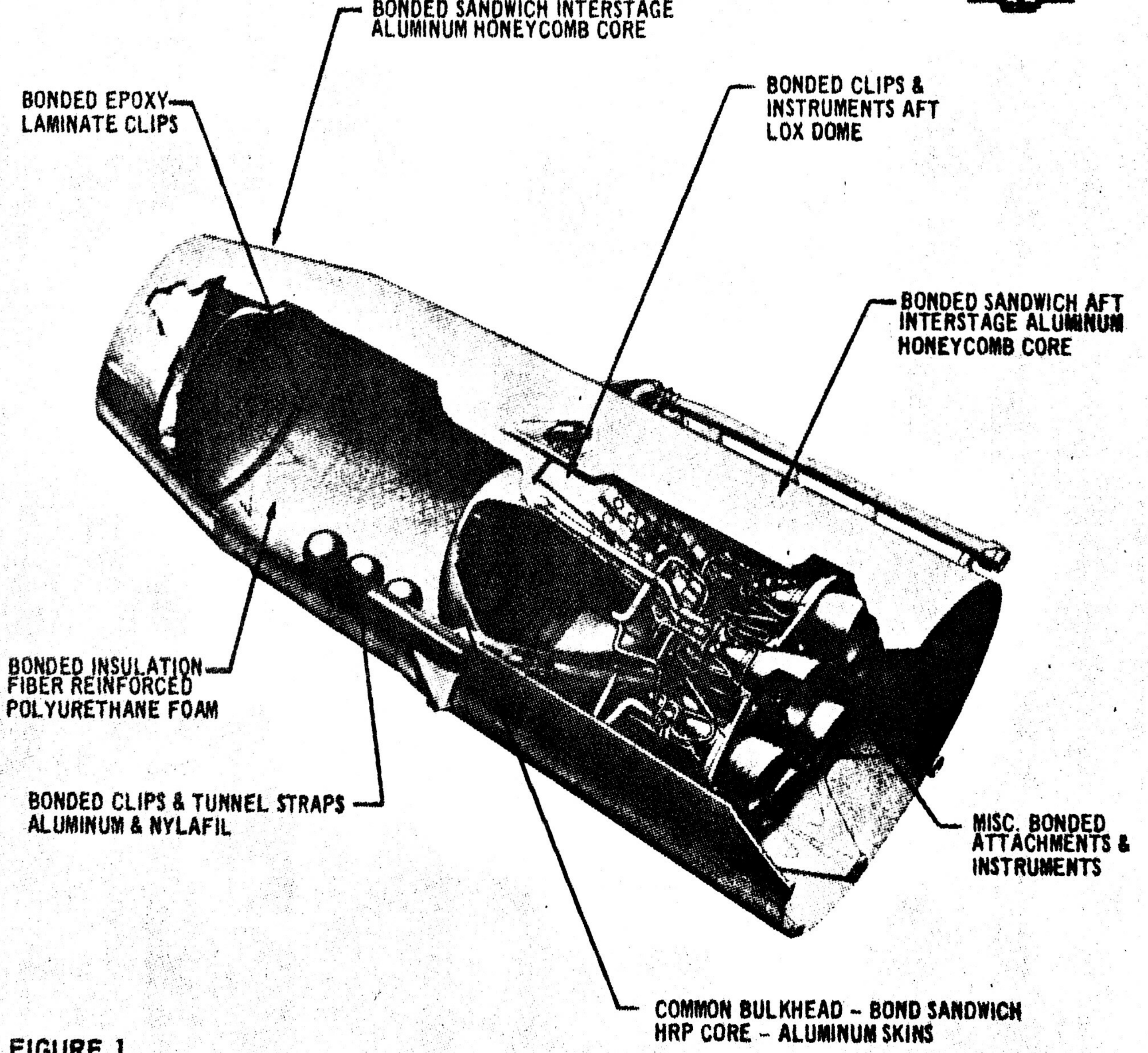


FIGURE 1

position. Each joint had its characteristic environmental envelope which influenced the requirements of the adhesive.

The next generation stage, the Saturn S-IVB, has essentially the same applications of bonding as the S-IV except the interstages and skirt are skin and stringer construction instead of bonded sandwich (Figure 2).

The requirements for the temperature resistance of the different adhesives range from -423.7°F to +250°F with some individual adhesives exposed to a change in temperature of 500°F (Figure 3). The loads on the adhesives are usually low enough to provide a large margin of safety. As a shear joint in the bulkhead, the HT 424 adhesive uses 20-25% of its potential strength. This estimate results from the core being a limiting shear resistant structure good for approximately 420 psi while the adhesive will resist 2400 psi in the same environment. Clips are bonded with a material good for 4,000 to 6,000 psi shear and are by design loaded to less than 500 psi when the bond line is at its working temperature of -100 to -150°F.

ADHESIVE APPLICATIONS

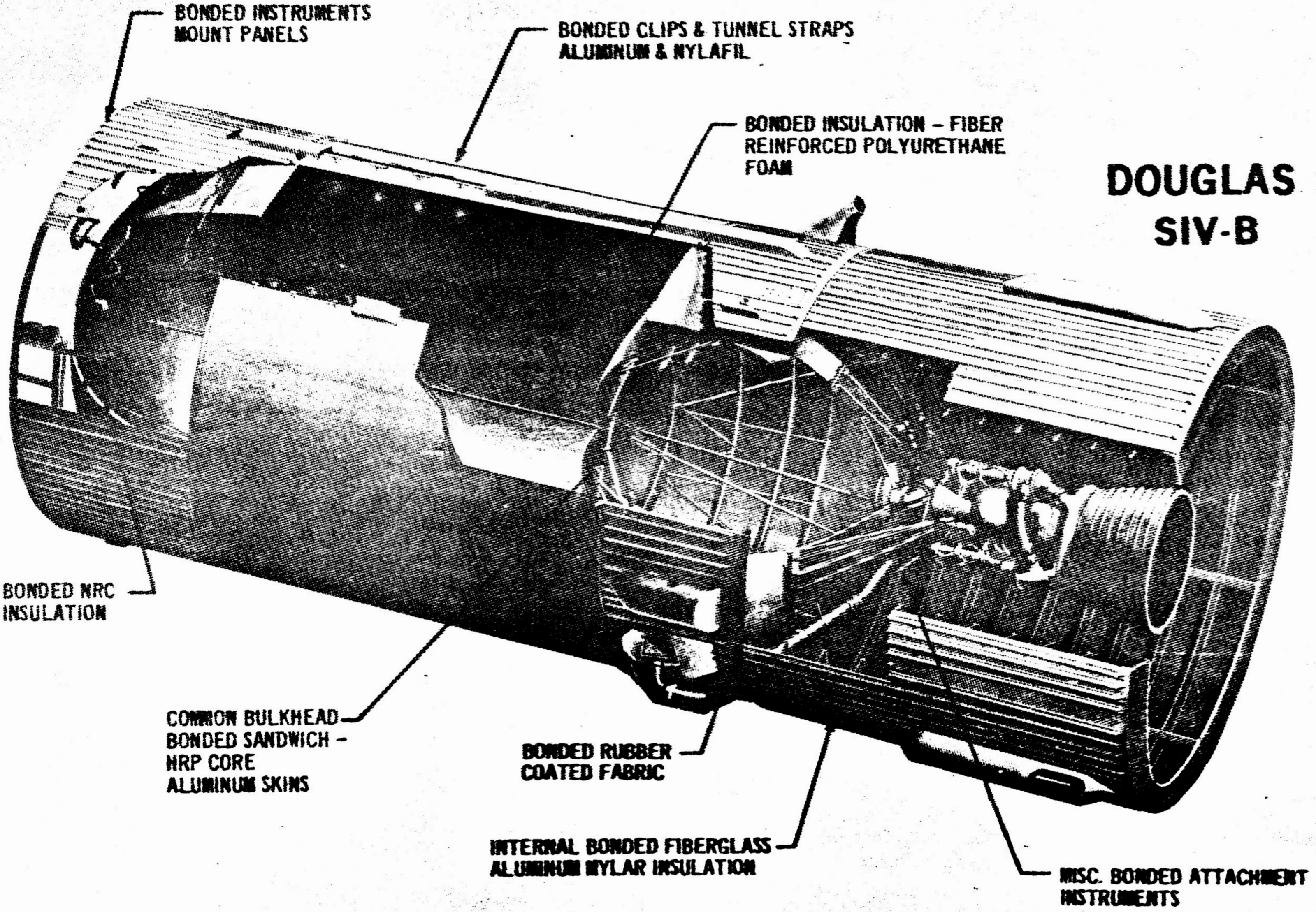


FIGURE 2

ADHESIVE TEMPERATURE ENVIRONMENT

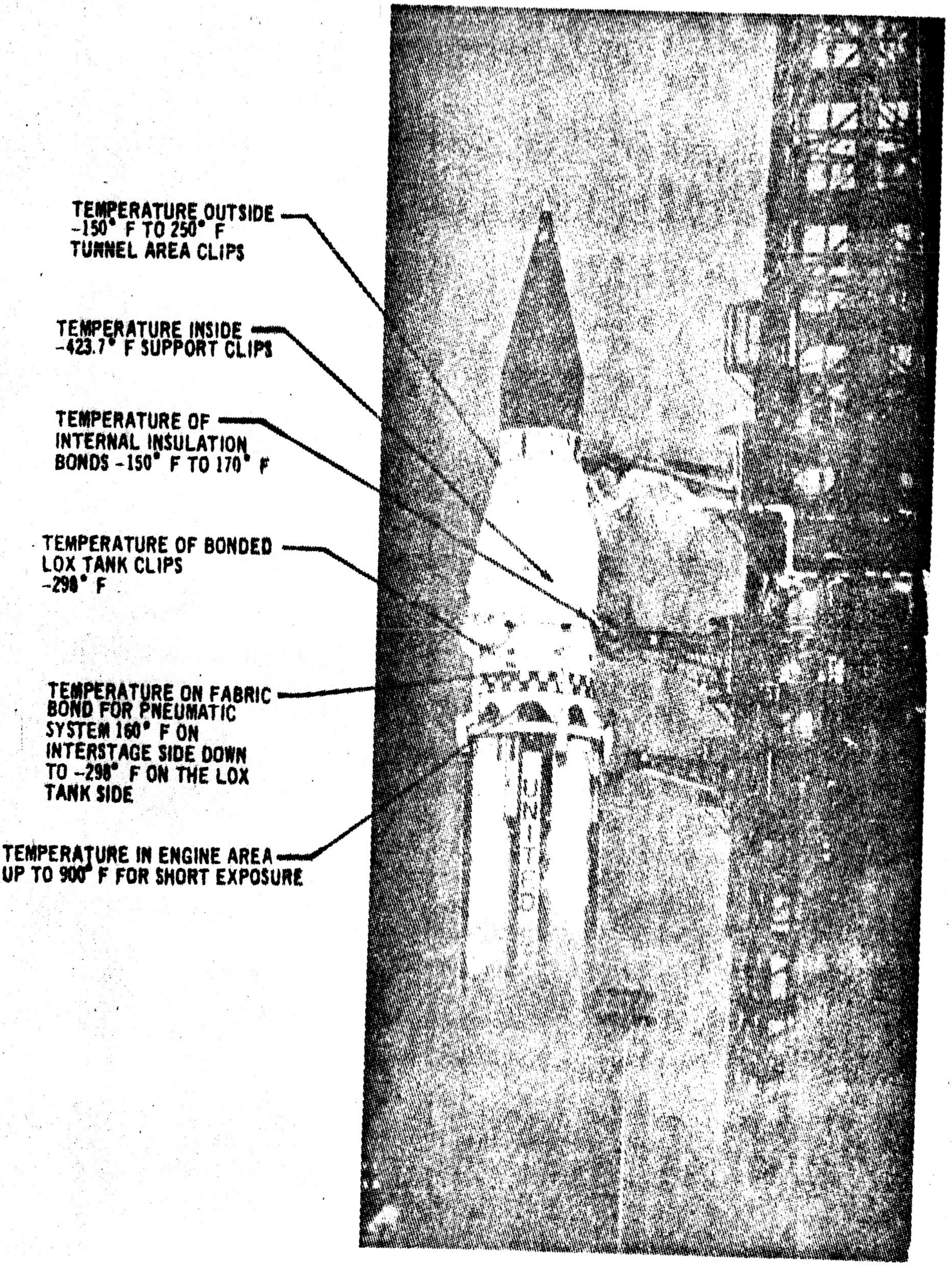


FIGURE 3

HISTORY

Thermosetting Adhesives

Most of the large scale structures were bonded with thermosetting adhesives to take advantage of the latitude of processing and to obtain uniformity in the bond line and properties (Figure 4). The initial literature search during the proposal stage of the Saturn S-IV revealed a National Bureau of Standards Report of some work done by DR Frost in the Denver Cryogenic laboratory. This publication showed the filled epoxy phenolic adhesive 422J produced by Adhesives Engineering to be the best of the few thermosetting adhesives tested at that time. The filled epoxy phenolic did not have the highest strength at all test temperatures but it had reasonable strength that was useable and unlike its competitors was the most uniform over the entire temperature range. A hot-melt version of this solvent dispersion system came on the market from Bloomingdale Rubber under the trade number of HT 424. Since the solvents employed to impregnate the carrier cloth to make this filled epoxy phenolic adhesive film degraded its strength, it was decided to use the solvent-free hot-melt system.

Figure 4 shows the uniformity of performance of the HT 424 in lap shear coupons. Figure 5 shows a superior performance of the

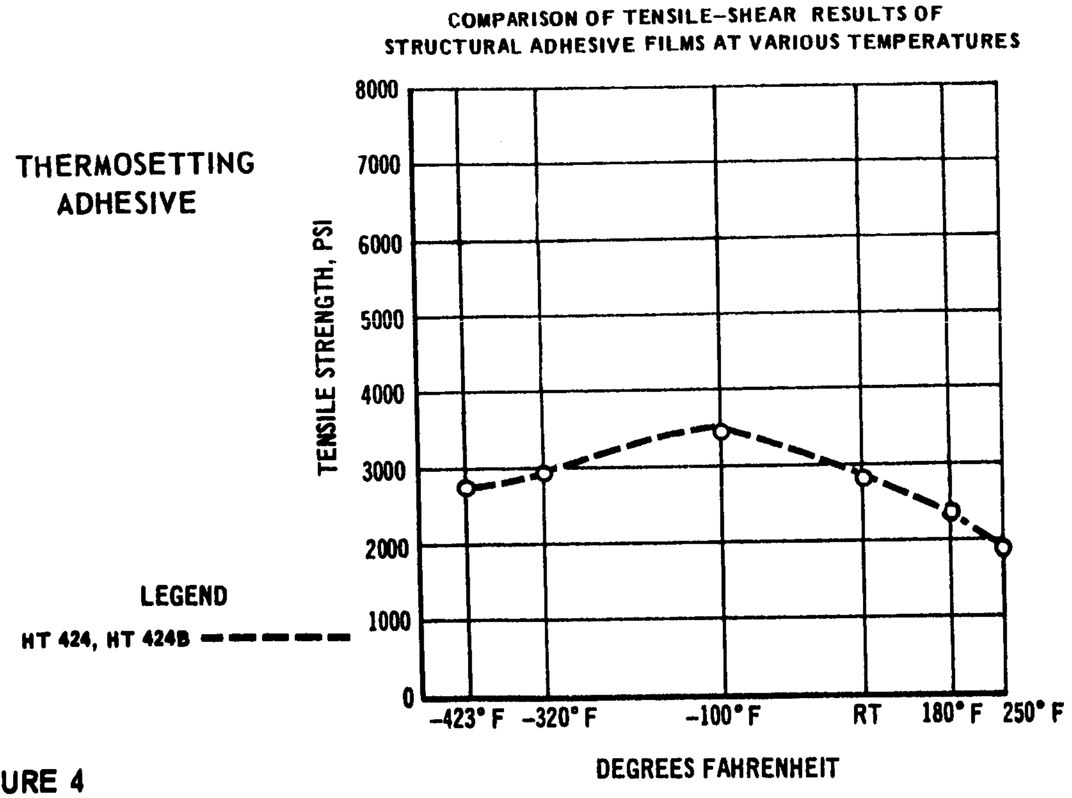


FIGURE 4

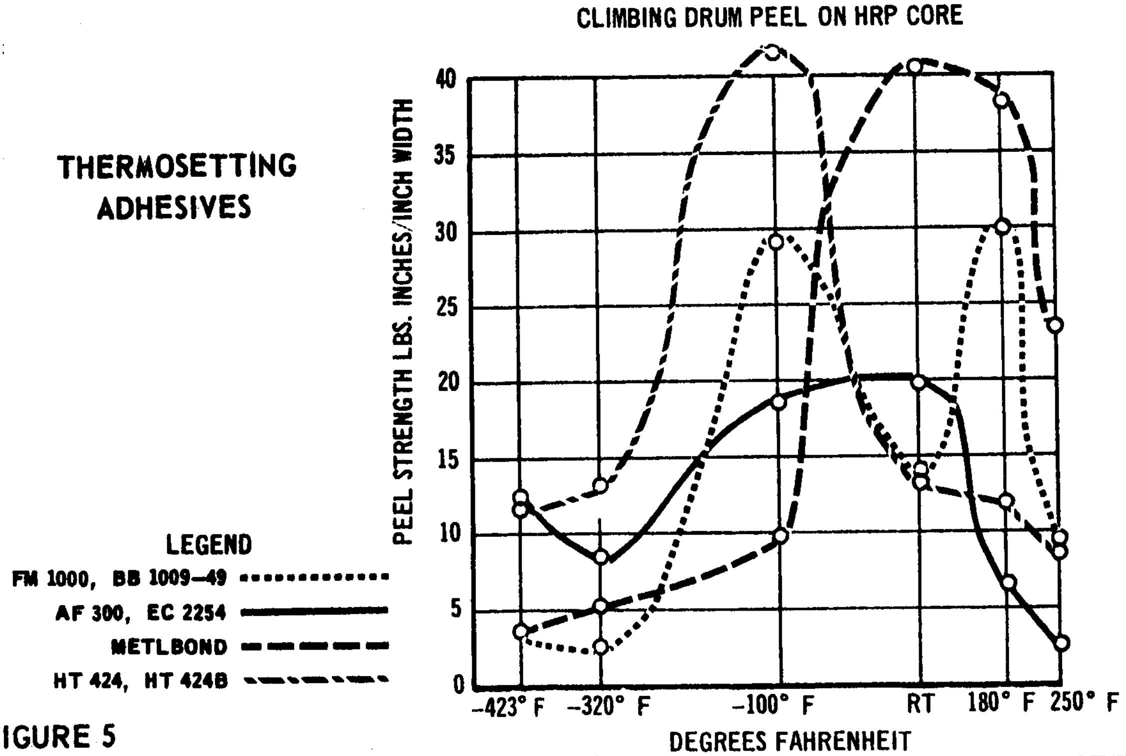


FIGURE 5

brittle filled epoxy phenolic at colder temperatures. The peel values show all of the materials tested subject to material imbrittlement at decreasing temperatures; therefore, differences in shear performance with temperature probably result from the differential coefficients of contraction of the adhesive with respect to the adherends. Since the filled epoxy phenolic is specifically formulated to minimize this difference in thermal contraction, its strength, though not the highest, is all available to take external loads.

No real competitors have arisen in the field of thermosetting adhesives to justify any change in this initial choice of material. Future applications requiring tighter weight restrictions or different processing temperatures may necessitate more than the usual market survey.

Low Temperature Curing Bonds

A literature search at the outset of the Saturn program for data on room temperature curing catalyzed adhesive systems turned up no data of value.

An initial screening of room temperature curing materials was conducted on a variety of materials such as straight epoxies, conventional elastomers, and filled epoxies. The best initial performer was a filled epoxy Lefkoweld LF-109 produced by the Leffingwell Corporation in California. Its thixotropic nature also made it a good candidate for joining foam blocks to the hydrogen tank wall for insulation because it did not soak into foam and leave a starved bond line (Figure 6). This material is still used today for this purpose.

The room temperature bonding of clips and doublers was not as successful as desired using this filled epoxy because the materials inherent brittleness resulted in low peel strength. In addition, the high viscosity of the material produced a bond line so thick that, unlike HT 424, would destroy itself in differential contraction. This led to the search for a better material that turned out to be the more elastomeric polyether urethane shown in Figure 6.

POLYURETHANES

The remainder of this presentation will cover the evaluation of this tempermental material as an adhesive and will consider future replacement materials.

ROOM TEMPERATURE SETTING ADHESIVES

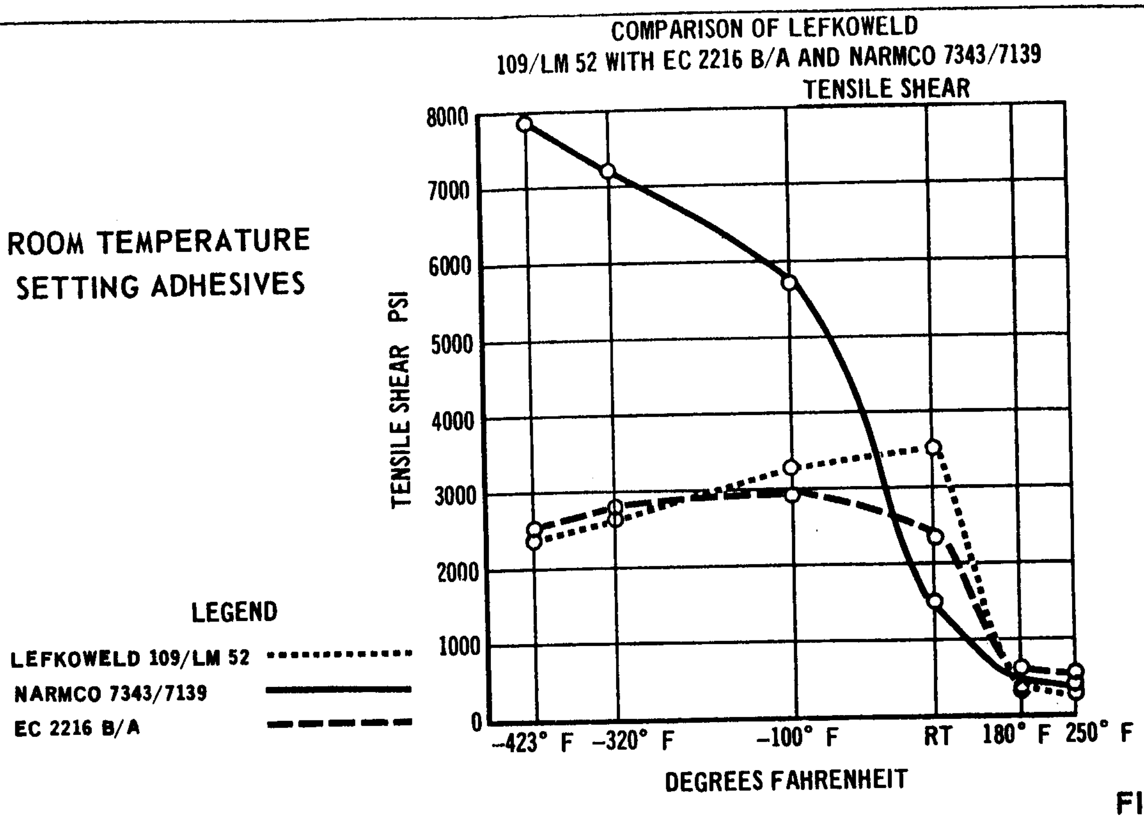


FIGURE 6

CHEMICAL DEFICIENCIES IN POLYURETHANE ADHESIVES

- WATER REACTING
- AUTOCATALYTIC REACTING
- SENSITIVE TO STOICHIOMETRY
- NO USABLE HIGH TEMPERATURE PROPERTIES
- CREEP AT ROOM TEMPERATURE
- RAPID AGING

FIGURE 7

Chemical Deficiencies

Polyurethane has certain inherent chemical deficiencies; it reacts with water including moisture from the humidity in the air (Figure 7); it sometimes undergoes an autocatalytic reaction when stored for a long time; its properties vary widely with small differences in stoichiometry; it has poor to no useable elevated temperature strength; it suffers from an excessive room temperature creep; and it shows rapid changes in properties on aging some of which are not desireable.

Processing Problems

These shortcomings lead to serious processing problems (Figure 8). A few of the more critical problems are control of both the intentional and unintentional reactive groups in the raw materials. A storage must be maintained that will inhibit fractional crystallization, in fact, any crystallization and water accrual in the raw stock. This material requires hot-melt mixing to diffuse the MOCA into the polyurethane resin. The ratio of MOCA to resin is normally 11 to a 100 parts by weight showing that slight errors in weighing have excessively large influences on the stoichiometry of the mix. The equivalent weight difference between the MOCA and water is about 1 to 3 with the result that a much smaller quantity of water will completely react the resin. Access of water to the

PROCESSING PROBLEMS WITH POLYURETHANE ADHESIVES

- CONTROL OF RAW MATERIALS STOICHIOMETRY
- STORAGE
- SOLID-LIQUID MIXING
- STOICHIOMETRY RESULTING FROM MIXING
- HUMIDITY CONTROL DURING ASSEMBLY
- TEMPERATURE CONTROL ALL OPERATIONS
- AGING EFFECT
- CORROSION CONTROL

FIGURE 8

INVESTIGATIONS TO FIND AN IMPROVEMENT IN ADHESIVE CHEMISTRY

- OTHER POLYETHER POLYURETHANES
- POLYESTER POLYURETHANES
- POLYOL POLYURETHANES
- EPOXY POLYURETHANE ADDUCTS
- REACTIVE SOLVENTS AND POLYURETHANES
- PRIMERS
    - SILANES
    - DV-1180
- INTERLEAVED FILMS

FIGURE 9

unmixed and mixed polyurethane resin has to be carefully regulated. This includes the time and temperature the resin is exposed to controlled humidities. Notice, it is not suggested that water be completely excluded since it is felt a little water improves adhesion of the polyurethanes to the metal.

The temperature at which the polyurethane is cured affects its properties. The time that test specimens are aged after the elevated portion of its cure influences the test results obtained. A practical effective wait is approximately 5 days. In excessively corrosive environments, corrosion will creep under a urethane bond line at the metal-to-urethane interface if the bond line edge is exposed. This is not currently a major problem because most of the bond lines are never exposed and the few that could be are not subject to the excessively corrosive environments seen by corrosion test specimens. In fact, aging specimens in other than the Douglas El Segundo test facility show little or no corrosion effects.

CORRECTIVE ACTION

Chemical

Several attempts were made to find equivalent polymers that had fewer deficiencies (Figure 9). Polyether urethanes with greater and lesser reactive isocyanate groups were tried. Polyester urethanes were tried. Polyols and mixtures of polyether urethanes with caster oil were tried, none of which produced even equivalent properties. Epoxy adducts for the polyether urethane were tried as were reactive solvents for the MOCA catalysts. These systems were intended to permit a liquid adhesive mix, but again the results were not very promising. Several primers for the polyether urethane were tried ranging from epoxies to urethanes with several hybrids, the most successful were the silanes. Silane primers did show a real improvement both in the consistency of the resulting peel strength and as an added corrosion inhibitor (Figure 10).

Attempts were also made to change the performance of bonded joints by interleaving different film materials such as polyvinyl chloride, Mylar*, polyethylene etc. Normally, these material tend to perform as parting agents but when tested at cryogenic temperatures showed no deterioration of the bond and in some case improved a little. The improvement did not justify the added processing efforts that would be required to use this type of joint but it was an interesting curiosity that developed in this program.

Physical

The greatest effort to obtain consistant results from polyurethane adhesives was put into the evaluation and control of its physical environment (Figure 11).

POLYURETHANE ADHESIVE WITH REACTIVE SOLVENTS AND PRIMERS

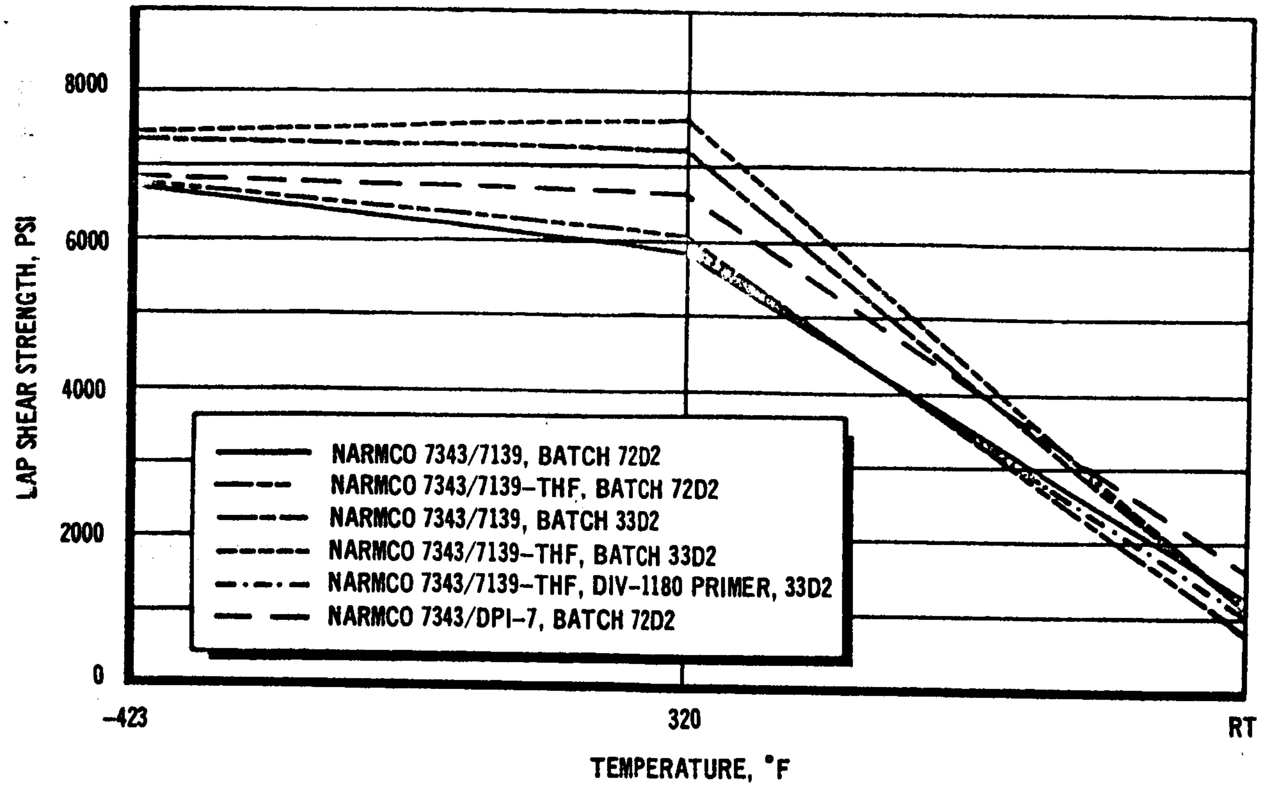


FIGURE 10

INVESTIGATION TO IMPROVE POLYURETHANE ADHESIVES USING PHYSICAL CHANGES

- PACKAGING
- RECEIVING INSPECTION
- STORAGE
- PREPROCESSING
- PERSONNEL TRAINING
- PROCESSING

FIGURE 11

The choice of packaging was of concern. A small container when opened would allow all the material to be used easily, or any excess could be discarded with a minimum cost penalty. This would prevent contamination of unused material. Conversely, the receiving inspection of small containers could be prohibitively expensive when can-to-can variations within a batch appeared, and this did occur all too frequently. The containers had to be gas tight to prevent moist air from breathing in to them and, thereby, reacting the free isocyanate. Large containers such as 55 gallon drums minimized receiving inspection costs but required special desiccated breathers or dry nitrogen purges to permit removal of the resin from the container. Subsequent shipment of material to work locations required more conviently sized containers, so the small package and the problems of packaging under a dry nitrogen purge were not eliminated. This procedure permitted the packaging in small containers of only that material destined to be consumed within a short period of time.

Receiving inspection was established on every batch of resin and lot of MOCA. In the case of the 55 gallon quantities, every drum was tested. The tests conducted included the determination of free -NCO in the resin and free NH_2 in the MOCA as 4,4'-methylene-bis-(2 chloroaniline). Originally, even water content determinations were taken on the MOCA but, as will be seen later, this proved to be unnecessary. Room temperature tested lap shear specimens and -320°F tested T-peel specimens were evaluated on each incoming shipment as the final proof of performance.

Each shipment of drum size was preprocessed to remove the effects of stratification and fractional or complete crystallization should it exist. This was done by heating the drum in an oven set at 120°F for 24 hours then rolling the drum for an additional 24 hours. This procedure effectively remelts any crystals and produces a homogeneous material. Once this was done, the containers were stored under controlled temperature conditions to preclude any recrystallization.

All personnel scheduled to do any adhesive bonding with this material were sent to a Douglas operated training course to learn a consistent method of using polyurethane adhesives.

The processing and the control of the processing exercised in the use of polyurethane adhesives is extensive and exacting. A typical example of a controlled process used in the evaluation program for polyurethane adhesives is shown in Figure 12. Most of the items and the reason for the control is self-evident. Some of the more crucial steps and the purpose will be discussed.

SPECIMEN PREPARATION PROCEDURE D

VARIABLE	
COUPON ENVIRONMENT	OVEN DRY 20 MIN AT 160° F
MIX ENVIRONMENT	R.T.
PREOUTGAS	YES
PREOUTGAS TEMPERATURE	200° F
PREOUTGAS TIME	20 MIN AT TEMPERATURE
MOCA MELT TEMPERATURE	250° F
MOCA MELT TIME	60 MIN.
L–100 MIX TEMPERATURE	120° F
MIXING TECHNIQUE	HAND STIR
MIX TIME	1 – 3 MIN.
POST OUTGAS	120° F
POST OUTGAS TIME	3 – 5 MIN.
CENTRIFUGE TIME	3 – 5 MIN.
ASSEMBLY ENVIRONMENT	R.T.
ADHESIVE APPLICATION	SEALANT GUN
BOND LINE CONTROL	SHIMS
SIZING BOND LINE	PRESS TO STOPS
TIME MIX TO ASSEMBLY	~ 30 MIN.
VACUUM	YES
PRESSURE	15"
FLUCTUATION	9" – 17"
TIME UNDER PRESSURE	24 HOURS
TIME NO PRESSURE	– –
TIME AT TEMPERATURE	24 HOURS
TEMPERATURE OF CURE	160° F
TIME, MIX TO TEST	9 DAYS
SPECIMEN TYPE	PLATE
PRESSURE MODE	
PRESSURE PARTING AGENT	TEFLON

FIGURE 12

The drying time on cleaned surfaces is controlled (Figure 13) to not more than four hours to prevent contamination but not less than two hours to permit the water on the active aluminum surface to come into equilibrium with the humidity of the surrounding air. All of the absorbed water is assumed able to combine with free isocyanate in the adhesive and this is compensated for by reducing the amount of MOCA theoretically required. It is also important to note that water pickup on an active aluminum surface at equilibruim appears independent of the humidity of the environment. The curve of moisture pickup on the resins definitely shows the open time or exposure of the unmixed and mixed adhesive should be kept to a minimum.

If the melt time at 250°F on the MOCA is permitted to extend to at least an hour, and if the resulting weight loss is water the curve in Figure 14 shows that most of the water entrained in the MOCA is removed. Post analysis on melted MOCA shows this description to be a reasonably accurate representation of the phenomenon. Excessive melt times running over eight hours deteriorate the MOCA to such an extent that it becomes unusable.

Increasing the mix temperature does not improve the performance of the polyurethane adhesive (Figure 15), whether it is a high or low storchiometric ratio of ingredients.

POLYURETHANE ADHESIVE

EFFECT OF TIME AND RELATIVE HUMIDITY ON WATER PRESENT IN T-PEEL SPECIMEN

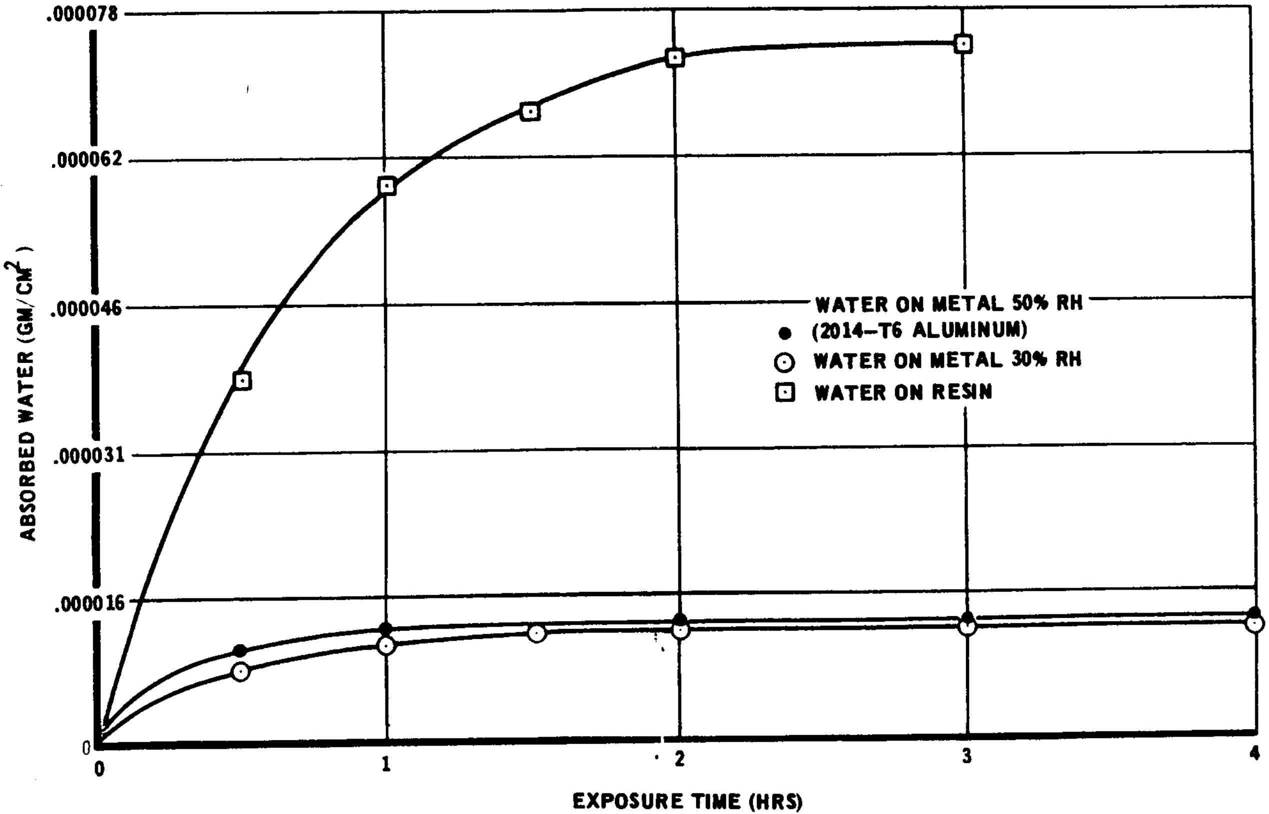


FIGURE 13

PERCENT WEIGHT LOSS VS. HEATING TIME AT 250° F. FOR DU PONT MOCA

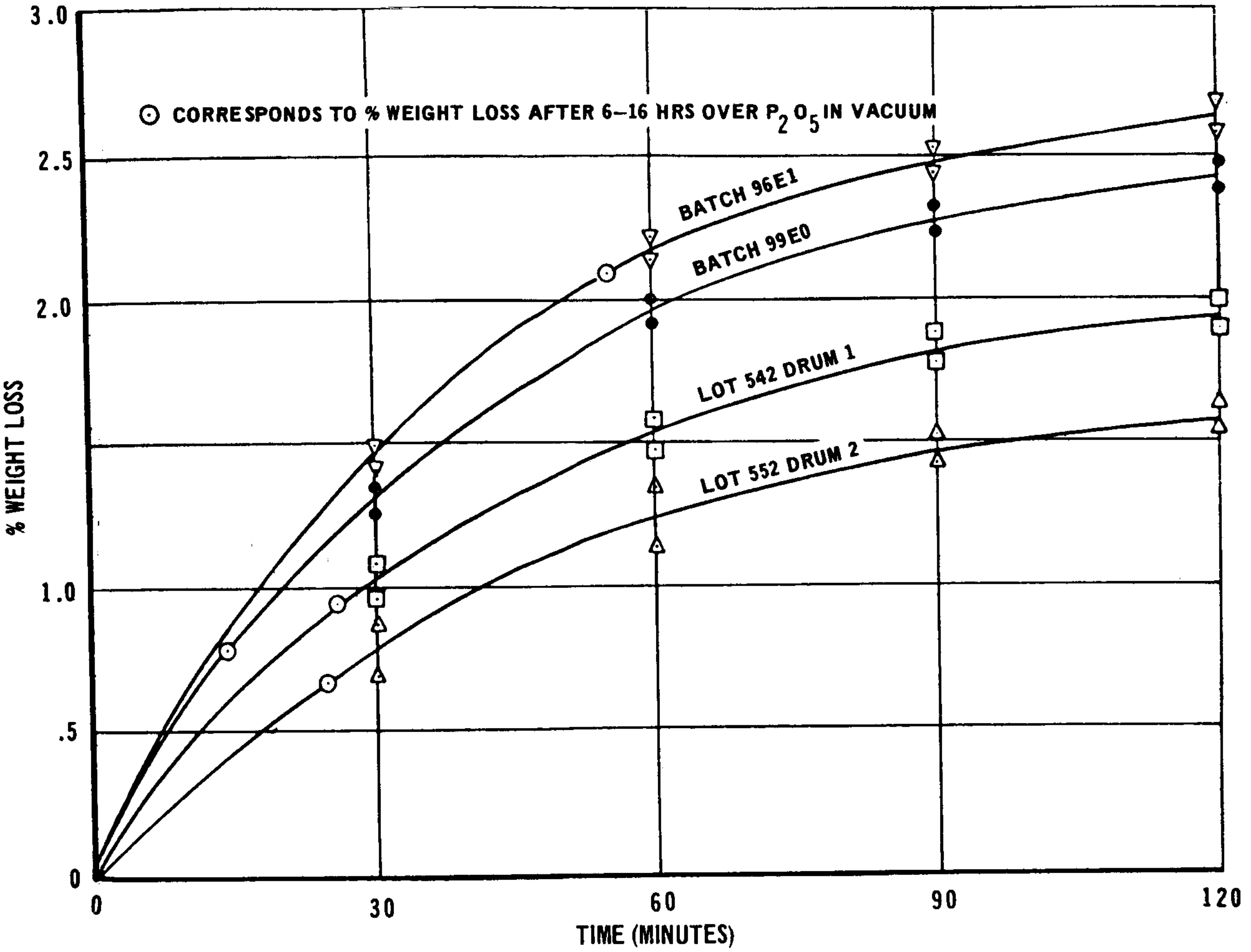


FIGURE 14

POLYURETHANE ADHESIVE

EFFECT OF RESIN MIX TEMPERATURE ON T-PEEL STRENGTH
(ADIPRENE L-1.0, LOT 890 AND MOCA, LOT 542)

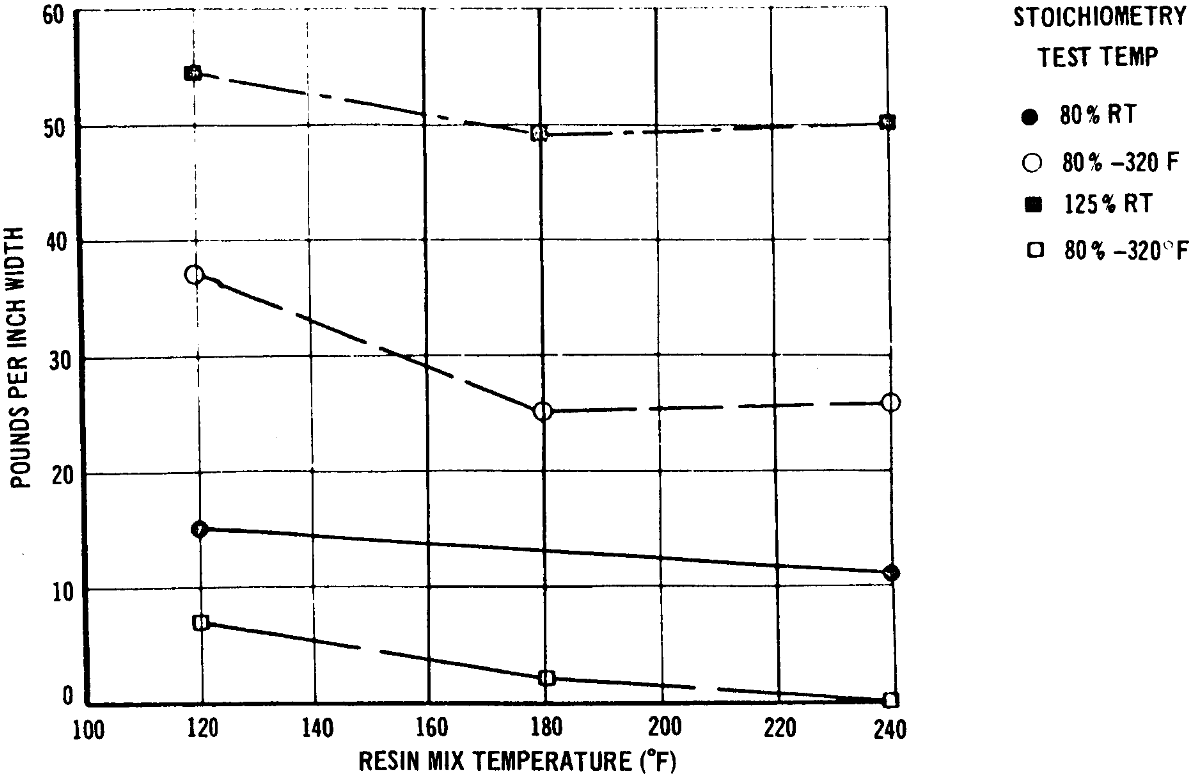


FIGURE 15

EFFECT OF WATER ON RESIN/HARDENER STOICHIOMETRY

SOURCE OF WATER	EXPOSURE TIMES	EFFECT ON RESIN/ HARDENER STOICHIOMETRY
1. 12.3 GMS. HARDENER MIXED WITH 100 GMS. RESIN	2 – 3 MINUTES	92.4%
2. WATER ABSORBED ON $Na_2\ Cr_2\ 0_7/H_2\ S0_4$	2 – 4 HOURS	20.61%
3. WATER ABSORBED ON SPREAD FILM OF RESIN/ HARDENER MIXTURE	40 MINUTES	98.94%
4. WATER ABSORBED ON 1/8" BEAD OF RESIN/ HARDENER MIXTURE	40 MINUTES	4.73%
TOTAL OF 1, 2 AND 3		211.95%
TOTAL OF 1, 2 AND 4		117.7%

RESIN – 4.200% ACTIVE – NCO

SPECIMENS EXPOSED TO 50% RELATIVE HUMIDITY AT 72° F

ASSEMED GLUE LINE – .002 INCH

FIGURE 16

The effect of water accrual in the bond line of a T-peel specimen is a function of the assembly method and time. Figure 16 shows that a spread film exposed for approximately 40 minutes in a 50% relative humidity environment will cure without MOCA. Unfortunately, the strengths obtained from water-cured urethanes are not competitive with the MOCA-cured varieties. Further inspection of the numbers show why an 80 to 82% starting stoichiometric ratio is used at Douglas on T-peel coupons and also why installation times for bonded parts on a stage are kept to a minimum of open-face time.

Figure 17 shows there is no significant effect on the T-peel values of specimens bonded under various pulsing and nonpulsing vacuum pressures. Most partial vacuum pressuring systems work with a pump full on or off principle, thereby, subjecting a specimen undergoing cure to a fluctuating pressure proportional to the sensitivity of the control switches and the leak rate of the system. Figure 18 graphically illustrates what this pulsing pressure can do to the reproducibility of values within a set of T-peel specimens.

The time a polyurethane adhesive is exposed to a cure temperature of 160°F controls the rate at which subsequent aging will bring the material to its full properties (Figure 19). Since it is desirable to reach the full properties of the adhesive as rapidly

EFFECT OF PRESSURE ON T-PEEL STRENGTH AVERAGE OF 5 SPECIMENS

(ADIPRENE L-100, LOT 890)

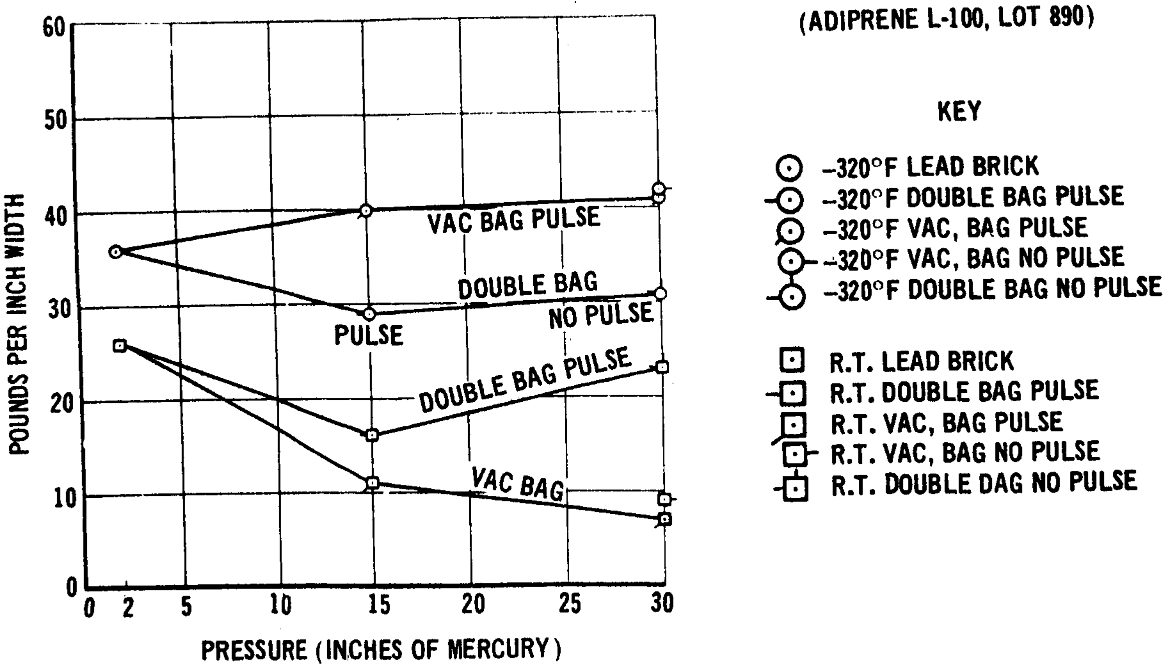
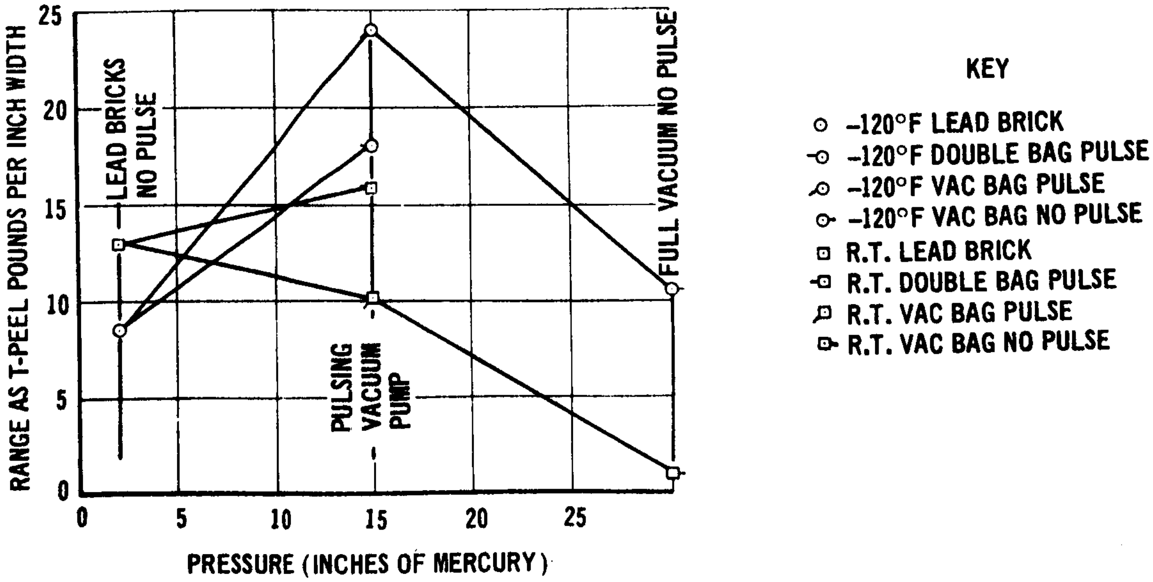


FIGURE 17

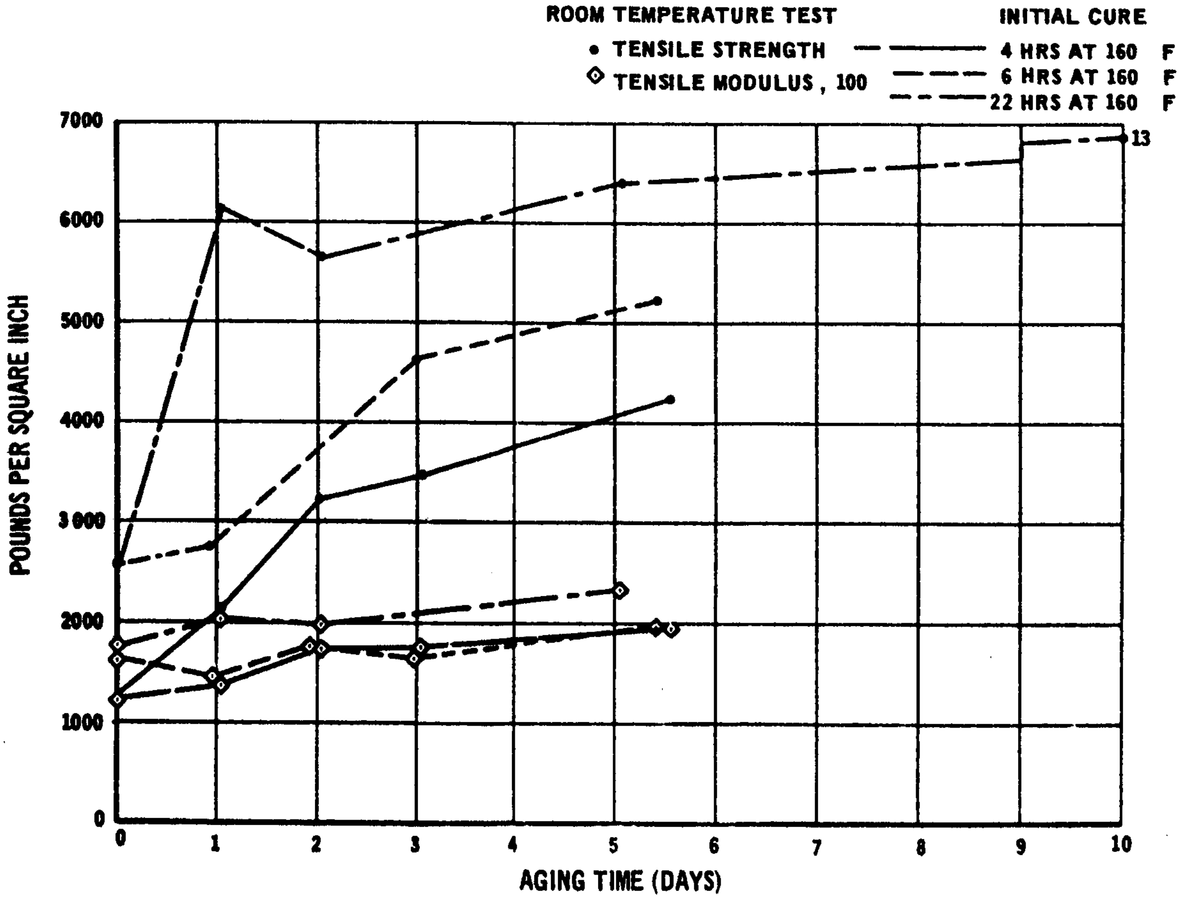
T-PEEL VARIANCE VS. PRESSURE

(ADIPRENE L-100, LOT 890)



PULSE VS. NO PULSE, AVG. VALUES

FIGURE 18



TENSILE STRENGTH AND MODULUS VS
RT AGING TIME 160 CURE TEMPERATURE

FIGURE 19

as possible in production, a 24-hour cure time at 160°F was chosen.

Another, rarely considered peculiarity of this adhesive is its rapid change of properties on aging after cure is supposedly complete. Infrared analysis will show the complete disappearance of free isocyanate long before the cure period of 24 hours at 160°F is over. Yet, as seen in Figure 20, the -320°F T-peel properties do not begin to approach equilibrium until the material is about ten days old. The examination of room temperature T-peel values (Figure 21) also confirm this condition. This behavior would suggest the polymerization of a polyurethane adhesive is not completed with the first isocyanate reaction. Observations of physical parameters such as change in hardness and the solid solution of early crystals tend to support the idea of the existence of a slower, more thermodynamically stable, secondary reaction. It even has an effect on the room temperature lap shear values as shown in Figure 22.

As expected, bond line thickness has a critical effect on the performance of a polyurethane joint. As a general rule, thicker bond lines up to 20-30 thousandths of an inch have an improved room temperature, T-peel strength increasing in direct proportion to thickness. If cryogenic temperatures are imposed on a peel specimen, the peel strength increases for bond line up to 1.8 - 2.2 thousandths of an inch where it peaks out at a maximum then

EFFECT OF ROOM TEMPERATURE AGING ON –320 F T–PEEL STRENGTH

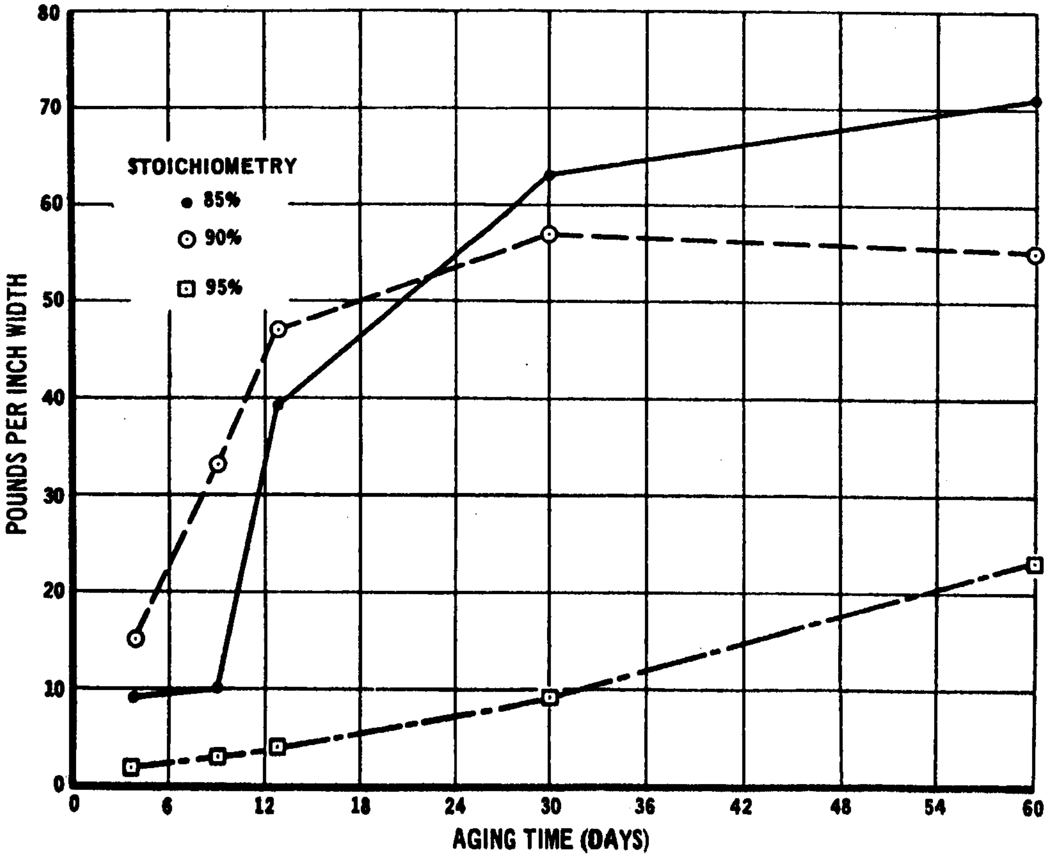


FIGURE 20

EFFECT OF ROOM TEMPERATURE AGING ON ROOM TEMPERATURE T-PEEL STRENGTH

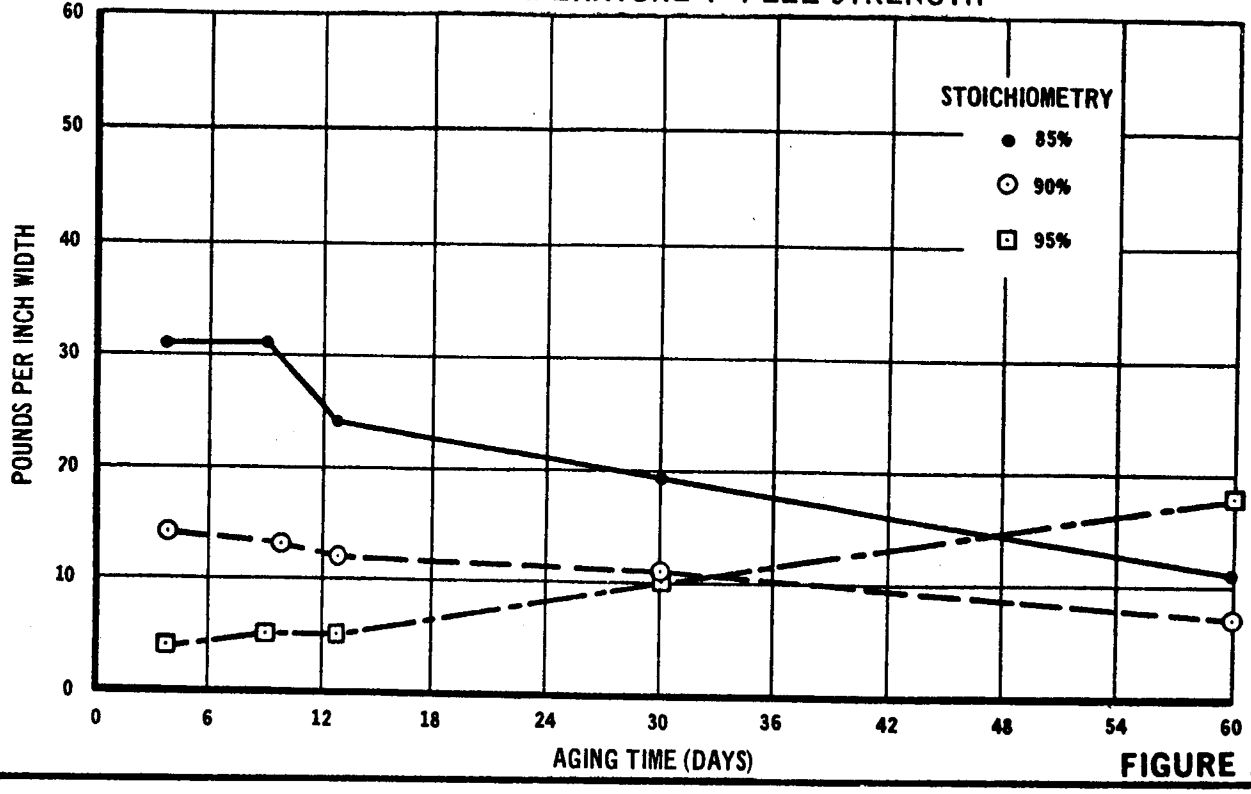


FIGURE 21

EFFECT OF ROOM TEMPERATURE AGING ON SHEAR STRENGTH

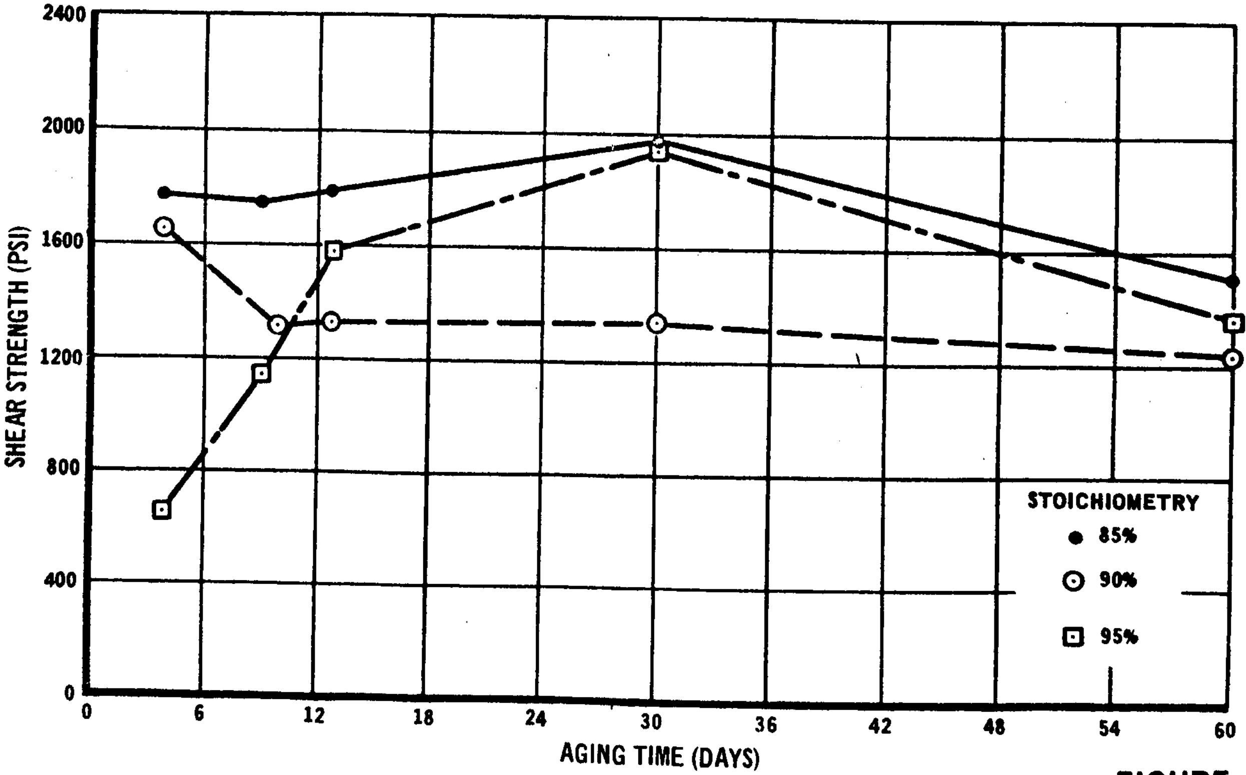


FIGURE 22

falls off for any further increase in thickness.

Another characteristic of bond lines that has proven troublesome and is independent of test temperature is the thinning of the bond line at the periphery of a thin bonded plate which occurs when the plate is held in place during bonding by a fluid pressure such as a vacuum bag or by localized high pressure generated by defective pressure-multiplier, vacuum tools. This peripheral area is usually subjected to the highest shear loads and has the least amount of low modulus material to absorb the strain. This explains why the tips of the fingers on a scalloped doubler always appear to fail first.

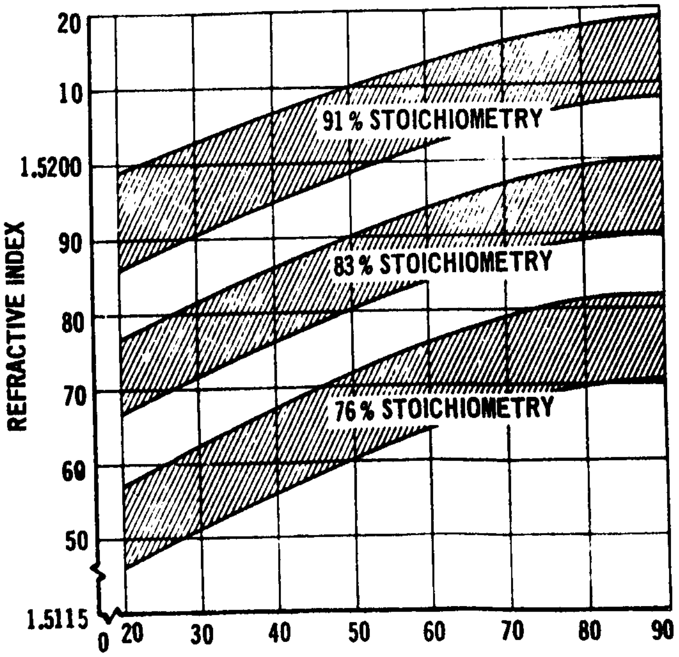
A remedy for these problems is to establish a method of obtaining a uniform bond line thickness or even a slightly thicker bond line at the edge of a bonded joint (Figure 23). In the case of the polyurethane adhesive, there has been no good way to make it into a more conventional film adhesive with any useable storage life. The next best approach that has been tried is to introduce low coefficient of linear expansion fibers and fabrics into the bond line. In illustration, a glass fabric forces the bond line into a more uniform thickness eliminating thin bond lines at the periphery of doublers. In addition, the low thermal contracting glass reduces the thermal stressing of the joint caused by high

CONTROLLED BOND LINE THICKNESS

- FILM
- FABRIC
- TIME-VISCOSITY CONTROL

FIGURE 23

POLYURETHANCE ADHESIVE MIX



PERFRACTIVE INDEX AS A FUNCTION OF TIME

FIGURE 24

shrinking adhesive joined to low shrinking adherends.

Tests of dumbbell specimens with overlay doublers showed an increase of 10% in the shear strength of the joint when the fabric was added. This increase was obtained at both room and cryogenic temperatures. The short pot life of the adhesive and the continued effort required to perfect a fabric carrier adhesive system suitable for production has forestalled its application to current hardware.

The system presently used to control bond line thickness is close process time and temperature control. This permits the adhesive to be applied within a reasonable viscosity range for each application. The method is workable in production and supplies fairly consistent results within 85% of that possible with fabric film thickness control.

PROCESS CONTROL TESTS FOR SPECIAL APPLICATIONS

Apart from the usual bonding process control tests which range from water-break-free tests on cleaned metals to coupon testing, several tests were generated and conducted to gain specialized knowledge of the polyurethane's performance in some of the unusual environments where it was used.

The first significant test devised was a general one designed to obtain a measure of the accuracy of the mixing of the material prior to use on a production part. This meant a method had to be developed to measure adequacy of mix prior to the gel of the adhesive. The method tried for this purpose was to determine the refractive index of the mixture at a fixed time after mixing (Figure 24).

As can be seen from Figure 24, a distinct and delineating relationship exists between the refractive index of a mixture of polyurethane adhesive for each stoichiometric relation as a function of time.

Figure 25 shows the relation of refractive index to stoichiometry at exactly 20 minutes after mix and at a temperature of 67°F. This time corresponds closely to the time normally required by production to commence the application of mixed adhesive on parts if the mix is acceptable. There is only one flaw in this system, but it effectively nullifies its usefulness; that is the range of detection of stoichiometry corresponding to a given refractive index reading is too large to guarantee the mix falls into an acceptable range. The control range sought on stoichiometry is approximately 4% and this procedure gives a value somewhere within a 10% range.

POLYURETHANS ADHESIVE MIX

REFRACTIVE INDEX OF NARMCO 7343/7139 AND ADIPREN L-100/MOCA.
TWENTY MINUTES AFTER MIX

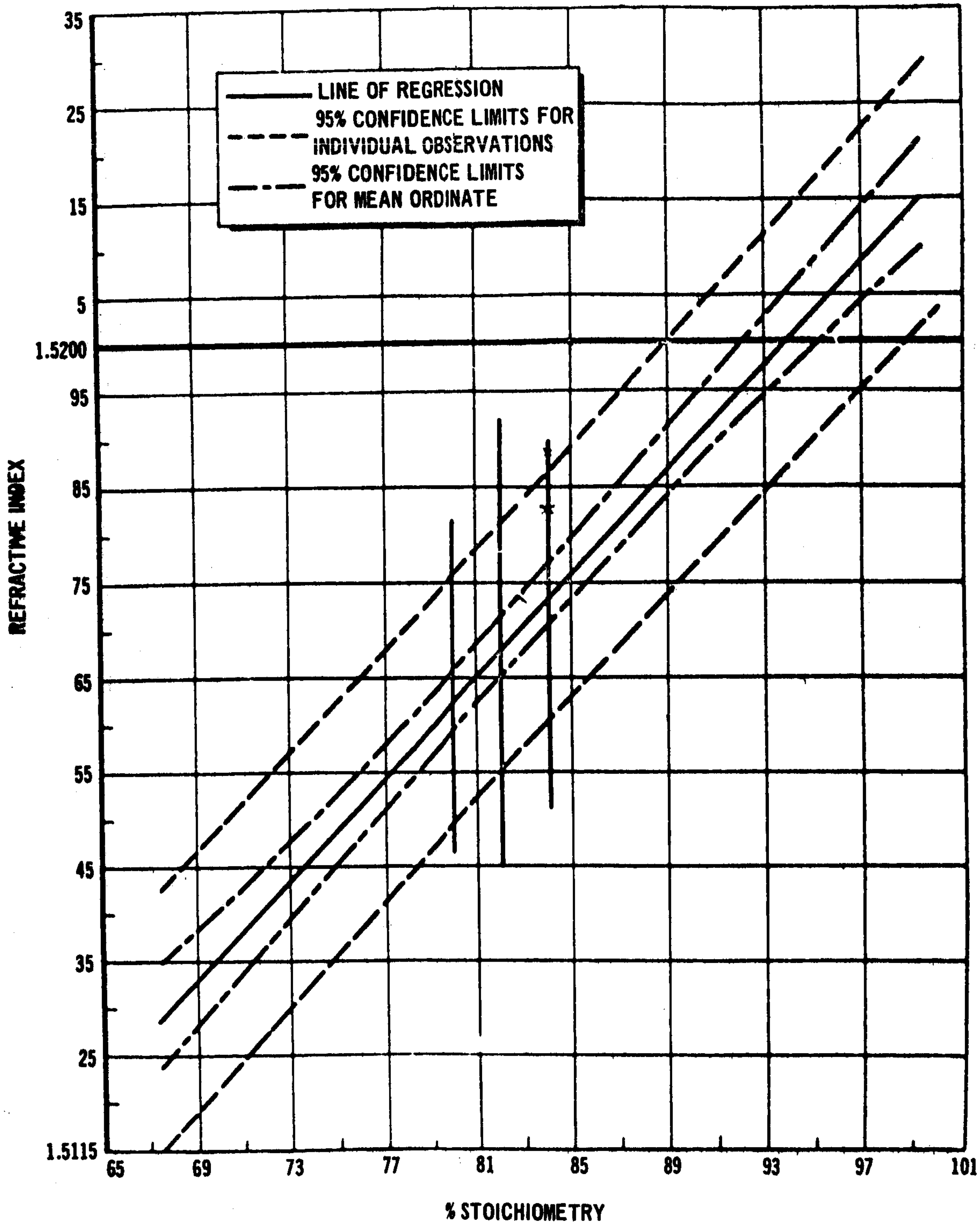
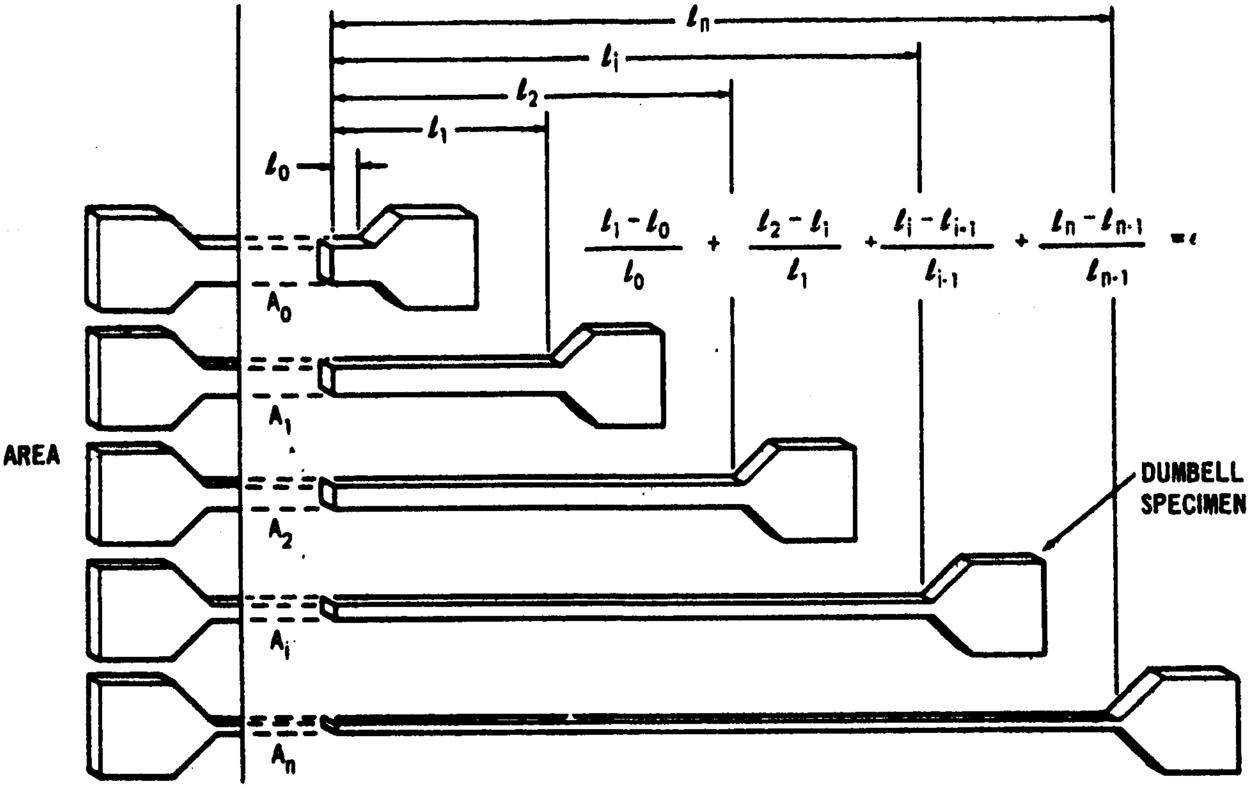


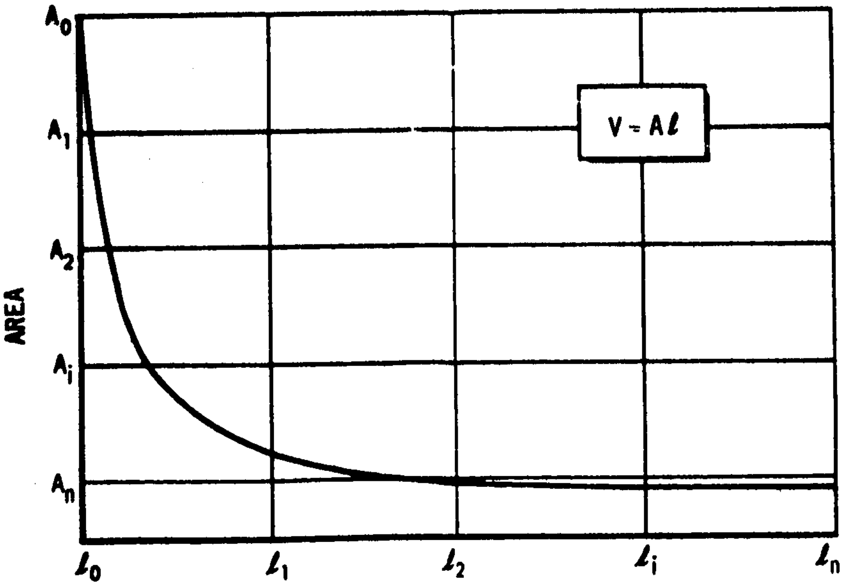
FIGURE 25

The next significant test or treatment of test data developed was the derivation of the "energy modulus". As can be seen in Figure 26, polyurethane as a sheet material behaves like an elastomer. At room temperature it has elongations that have exceeded 500%. From a practical point of view this makes the determination of a useable engineering modulus difficult. Since the polyurethane approximates a rubber, neither its strain nor its cross-sectional area at high load simulate the small strains of metal closely enough to arrive at a simple Young's modulus that is accurate. Figure 26 illustrates these two departures. The top illustration shows the strain can be reduced to a natural strain (a natural log function of stretch) and the bottom illustration shows the cross-sectional area changes along a hyperboloid as a function of stretch. If the volume of the original parallelopiped between the gauge lengths of the specimen remain constant and the test speed is set to minimize viscoelastic effects, the energy modulus shown in Figure 27 can be derived. Estimates on the above assumptions show an error of 3 - 5% which is as precise as the data reading but yields results far more accurate than other methods of computation. This derivation can be found in the appendix of this printed paper. The interesting feature of the energy modulus is that it not only is a version of Young's tensile modulus it is also the work per unit volume required to cause a given strain. This feature has

MODULUS



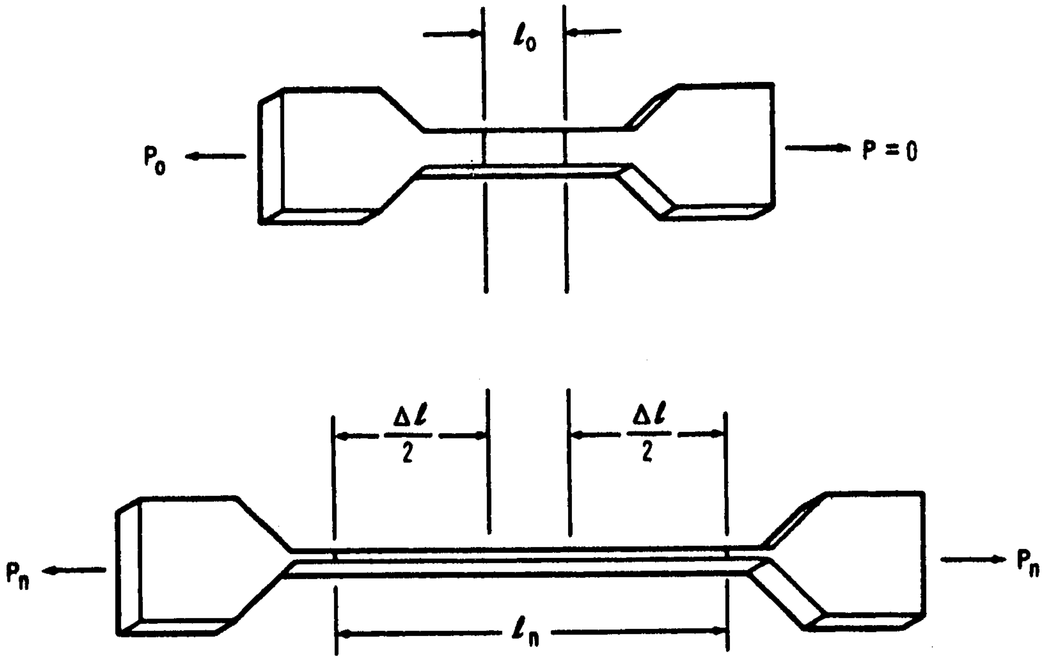
NATURAL STRAIN ACCUMULATION



RELATIONSHIP OF CROSS-SECTIONAL AREA TO STRAIN

FIGURE 26

MODULUS



DUMBBELL TENSILE SPECIMENS UNRESTRAINED AND IN TENSION

RUBBER INDUSTRY "MODULUS" (M)

$$M = \frac{P_n}{A_o}$$

YOUNG'S MODULUS (E)

$$E = (P/A_o)(l_o / l_f - l_o)$$

ENERGY MODULUS (E) EQUATION

$$E' = -\frac{\cdot n}{V}(l_n - l_o)$$

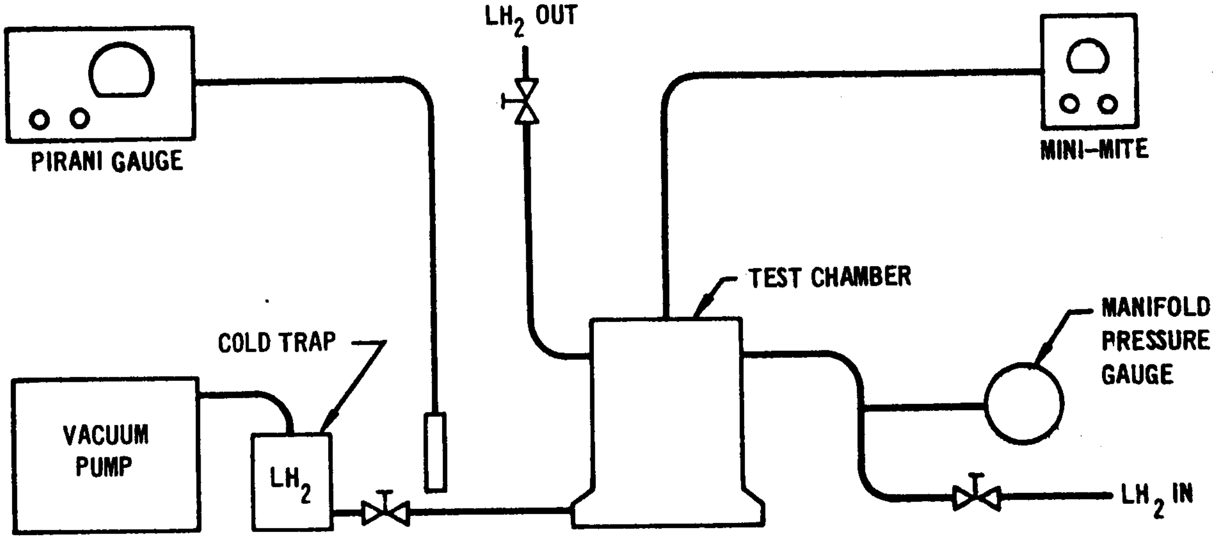
FIGURE 27

considerable potential in evaluating polymers under various environmental test conditions that have not been fully explored.

Figure 27 also shows the conventional Young's modulus as used by design engineers and the industry peculiar modulus used as a quality and property control test for rubber. This last modulus is actually a tensile stress at an arbitrarily established strain or at rupture and is not a design useable number.

Polyurethane adhesives have been applied in areas where it was to serve a multiple function such as a bonding agent and a seal to propellant liquids and gases. As a result, special tests were devised to measure the permeability of propellents through bond lines. The most common test apparatus for this purpose used at Douglas is shown schematically in Figure 28. As can be seen, the test propellent is liquid hydrogen which generates one of the worst diffusion or leak problems both because of its small molecular size and its extreme cold causing microcracks in most bond lines that result in leak paths. The latter phenomenon is the most probable problem area. The technique consists of applying liquid hydrogen to one face of a bonded patch over an open hole and drawing a vacuum on the cavity under the hole (Figure 29). The gas buildup in the cavity that occurs on standing is measured by a

SCHEMATIC DIAGRAM OF LH_2 PERMEABILITY TEST

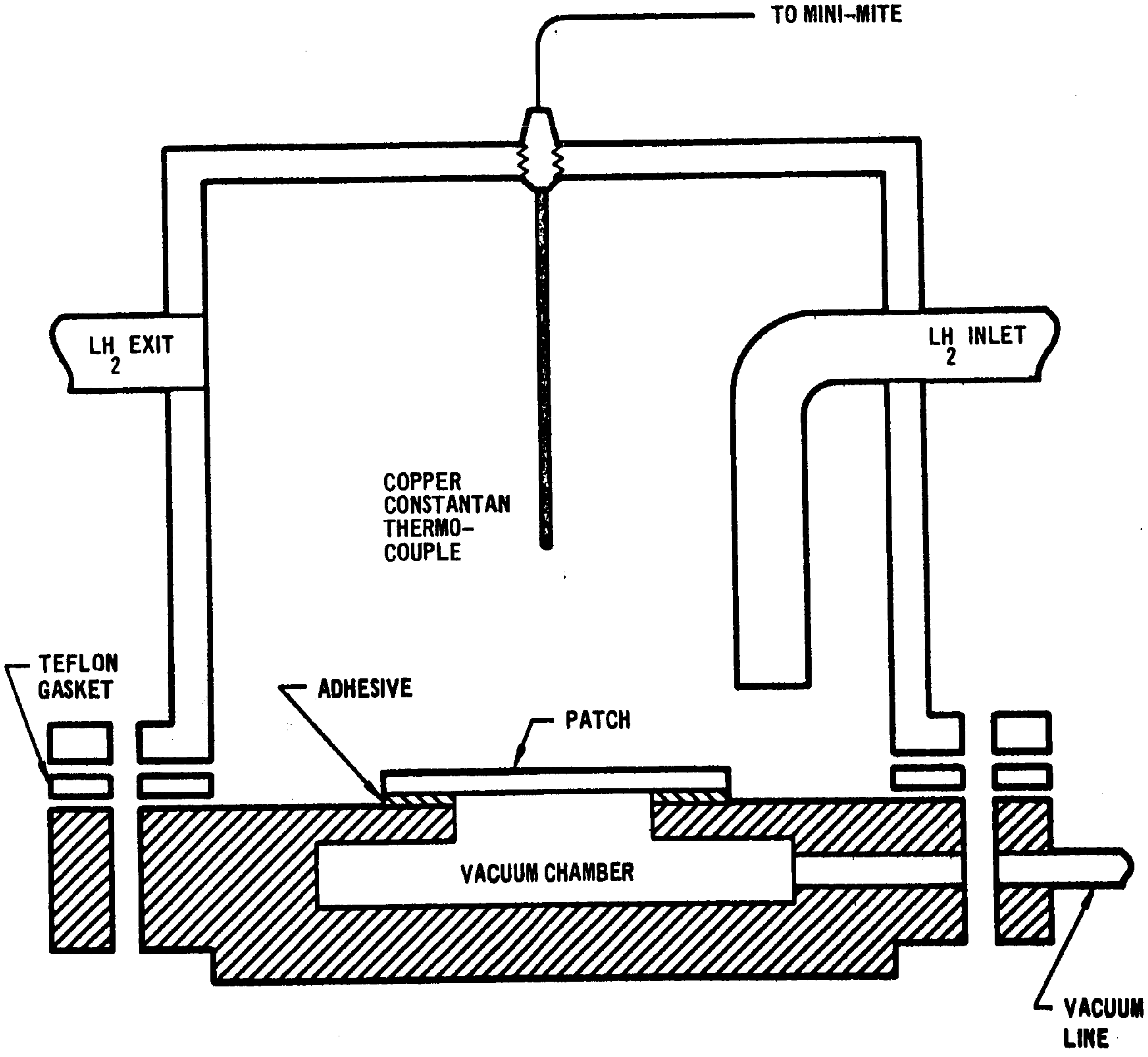


NOTES:

1. PIRANI GAUGE MEASURES VACUUM PRESSURE IN THE VACUUM CHAMBER
2. MINI-MITE MEASURES TEST CHAMBER TEMPERATURE
3. MANIFOLD PRESSURE GAUGE MEASURES TEST CHAMBER PRESSURE
4. SEE FIGURE 2.

FIGURE 28

FULL SECTION VIEW OF LH_2 PERMEABILITY TEST CHAMBER



NOTES:

1. PATCH SIZES VARIABLE: 4" AND 5" DIAMETER.
2. VACUUM PORT: CONSTANT 2" DIAMETER
3. BASE AND PATCH: 2014-T6 ALUMINUM.
4. ADHESIVE: NARMCO 7343/7139.
5. A STIFFENER, CONSISTING OF A 2" X 2" AND 1" THICK HONEYCOMB BLOCK (ALUMINUM FACED) AND CEMENTED TO THE UPPER SIDE OF THE PATCH, HAS BEEN OMITTED FOR CLARITY.

FIGURE 29

pressure buildup or a mass spectrometer. Polyurethanes have a record of performing very well in this test as hydrogen gas barrier mediums. Unlike simple films and barring the formation of microcrack leak paths, the bonded joint forces the hydrogen to diffuse through the long distance of the faying surface bond.

In addition to leak resistance of a bond line, bonded configurations as dynamically similar to actual applications as practicable were fabricated (Figure 30) and tested under a simulated environment. Figure 30 shows a typical specimen used to evaluate bonded doublers as a means of repairing a weld leak on one of the Saturn IV common bulkheads. This type of repair proved effective in test and in performance for stages developing minor leak problems in the field.

In the case of leaks on the liquid oxygen (LOX) side of the common bulkhead, an additional type of testing had to be introduced. The finished bonded repair in a LOX tank had to be LOX impact resistant and had to remain so after several cycles of stressing. Again polyurethane adhesives were used to make the bonded repair but the edges of the bonded joint had to be sealed with a material that was also impact resistant. Figure 31 shows typical specimens used for the cyclic stressing of this type of bonded joint. The object is to determine if the protective coating of Dynatherm 4327

SPECIMEN FOR LIQUID HYDROGEN WELD LEAK REPAIRS SATURN COMMON BULKHEAD

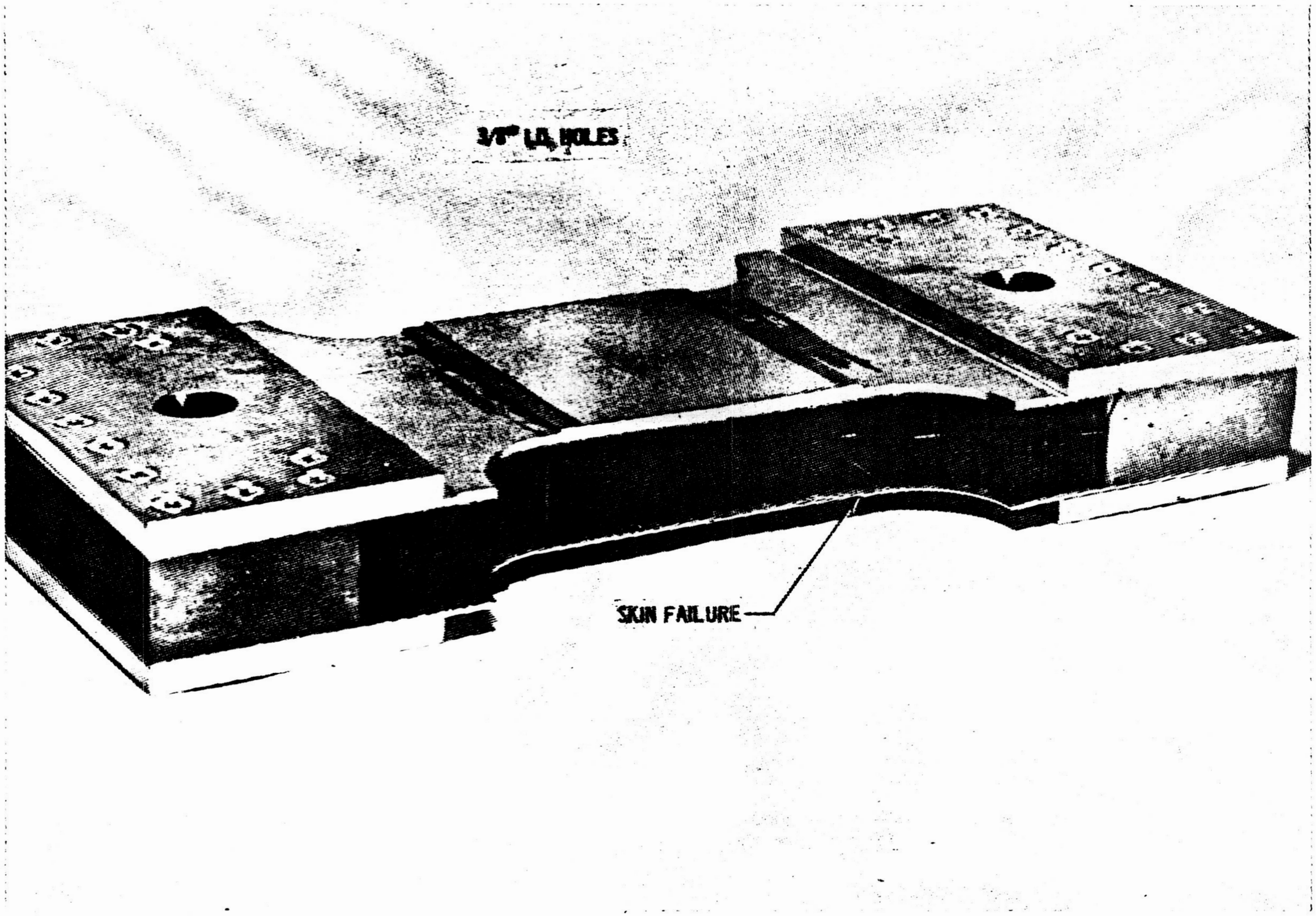


FIGURE 30

SPECIMENS FOR LOX TANK WELD LEAK REPAIRS USING BONDED DOUBLERS
WITH PROTECTED BOND LINES SATURN COMMON BULKHEAD

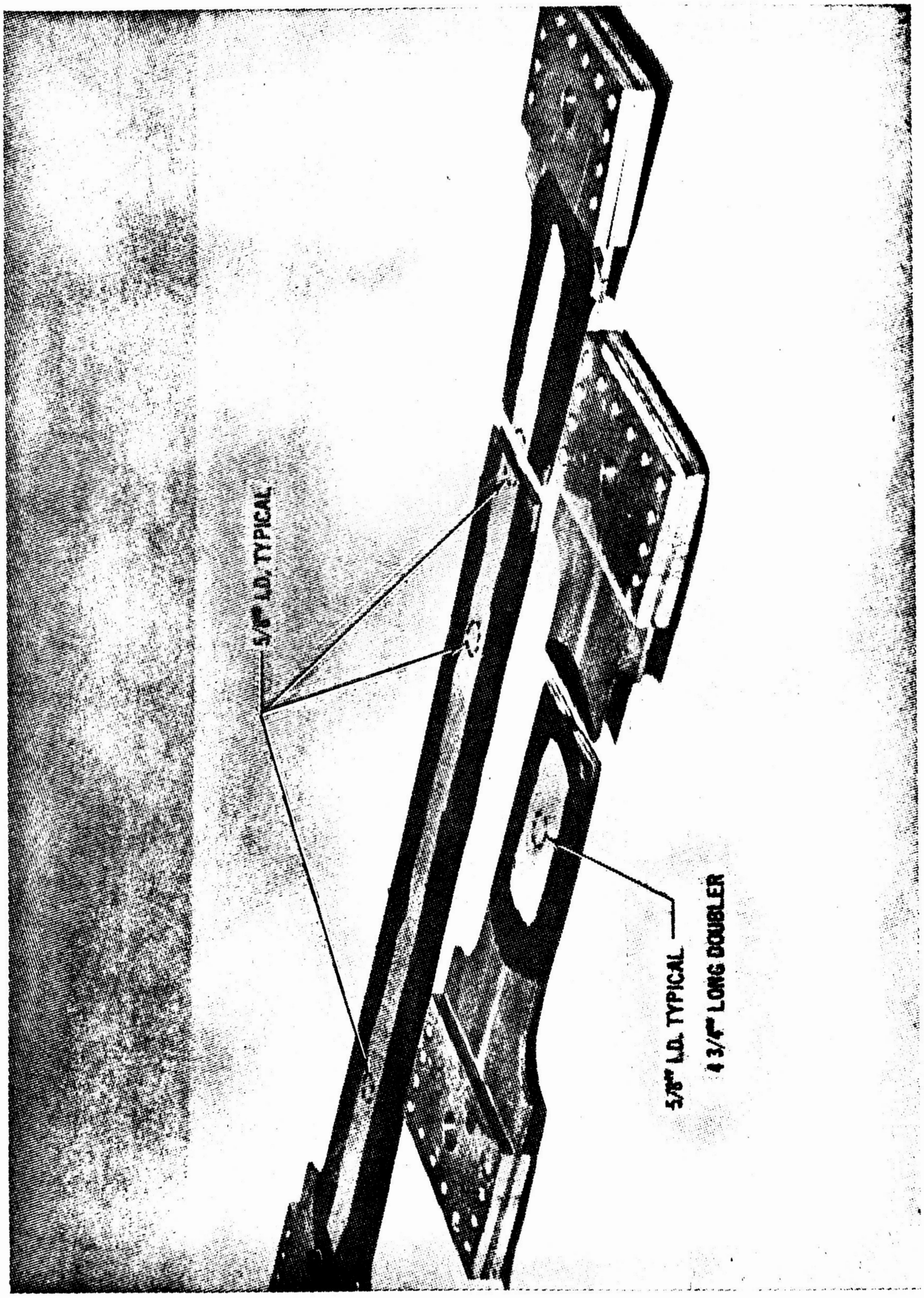


FIGURE 31

(polyvinylidene fluoride with a binder) will crack and split causing an exposure of the underlying bond line.

Impact tests at the 72 ft-lb energy level delivered to a 1/2-inch diameter sample by the ABMA type LOX impact tester were conducted on all the individual materials used in this type of joint. In addition, 1/2-inch diameter polyurethane bonded aluminum laminates were made with and without the Dynatherm edge protection, and then were tested. These tests showed that the joints properly protected were about as LOX impact sensitive as the test cup containers. This level of testing exceeds by a wide margin of safety any impact energy level anticipated for the locations where this type of bonded joint is used.

All bonded joints used on the Saturn stage were thoroughly tested both in the laboratory and in full scale field tests. This includes everything from the chemical analysis of the polymers to the dynamic testing of components while subject to cryogenic temperatures. To illustrate the large scale testing by one typical example, the polyurethane-bonded aluminum bracket and Nylafil wire bundle standoff tests conducted using the 8-foot tank will be discussed. A series of internal insulation tests, which required an 8-foot diameter dynamically similar tank to the Saturn S-IV stage, were being conducted at the Douglas Sacramento liquid hydrogen test site. The external portion of the tank was unused. To maximize the data output from these expensive test runs, it was decided to add tests to the external surface for bonding, sealant, and coating materials in configurations and under mechanical loads anticipated for the full scale stage. The intention was to determine if the small scale laboratory tests truly indicated the material performance in the environment and under the aging and cycling conditions the full scale stage would undergo.

In the case of the polyurethane adhesive joints, several configurations of two basic types of parts were tested: the aluminum bracket or clips and the inverted Nylafil golf tee (a wire bundle stand-off). Figure 32 shows one arrangement of these attachments on the tank wall and the method of applying mechanical loads at design loading angles to simulate the anticipated combined loads of shear and tensile on the bond line.

Figure 33 is a photo of one of the arrangements of the test clips on the external wall of the 8-foot tank as it appears prior to installation of the clip loading fixtures.

Figure 34 shows the type of loading applied to a tunnel strap undergoing test.

EIGHT FOOT TANK TESTS

LOADING 1B04041 WIRE BUNDLE SUPPORT CLIPS

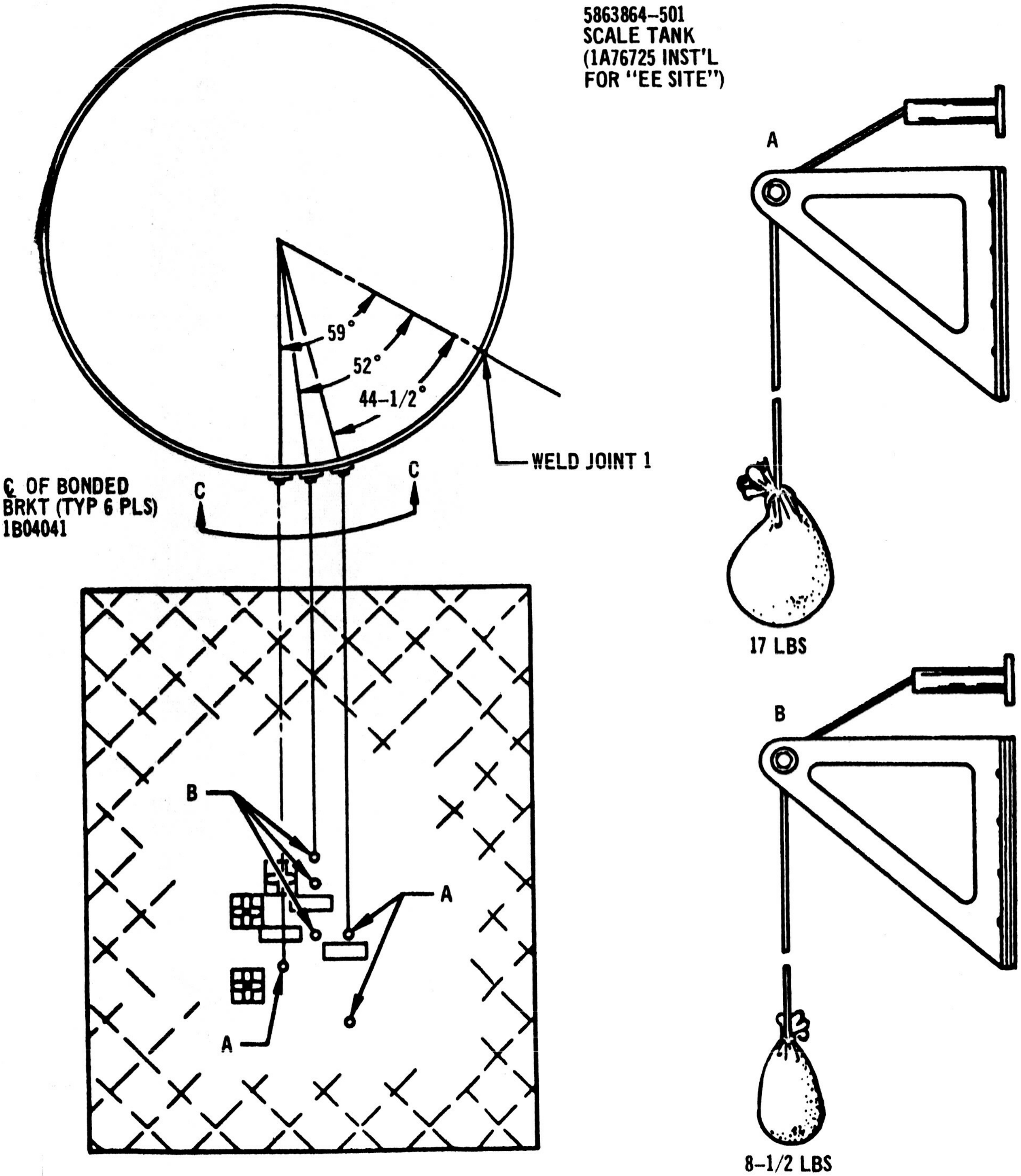


FIGURE 32

CLOSEUP OF 1B31142, 1B04041 AND 1B28892 ATTACHMENTS BONDED TO 8' TANK WITH POLYURETHANE ADHESIVES

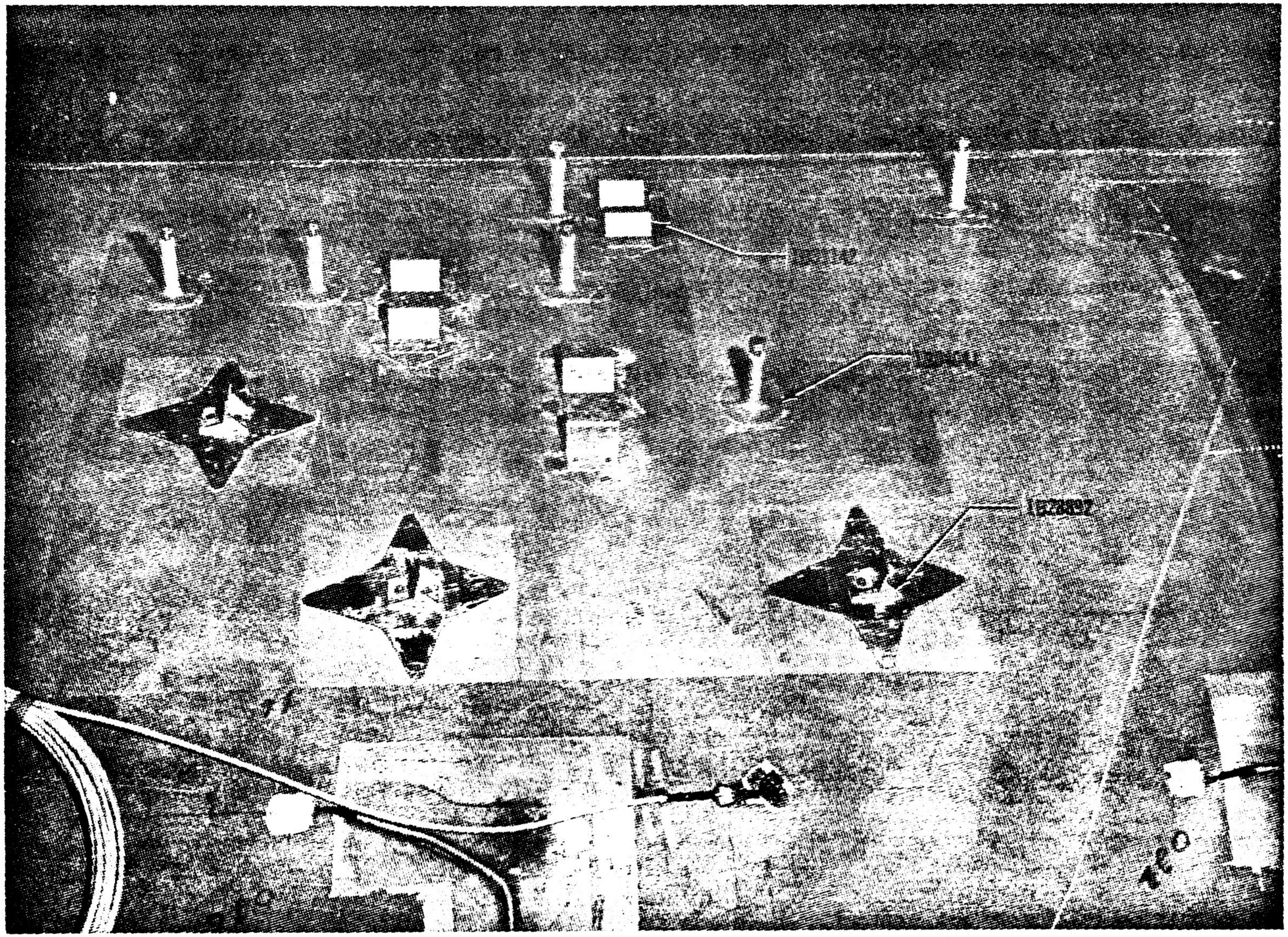


FIGURE 33

FIGURE 34. EIGHT FOOT TANK TESTS

LOADING 1B31142 TUNNEL STRAPS

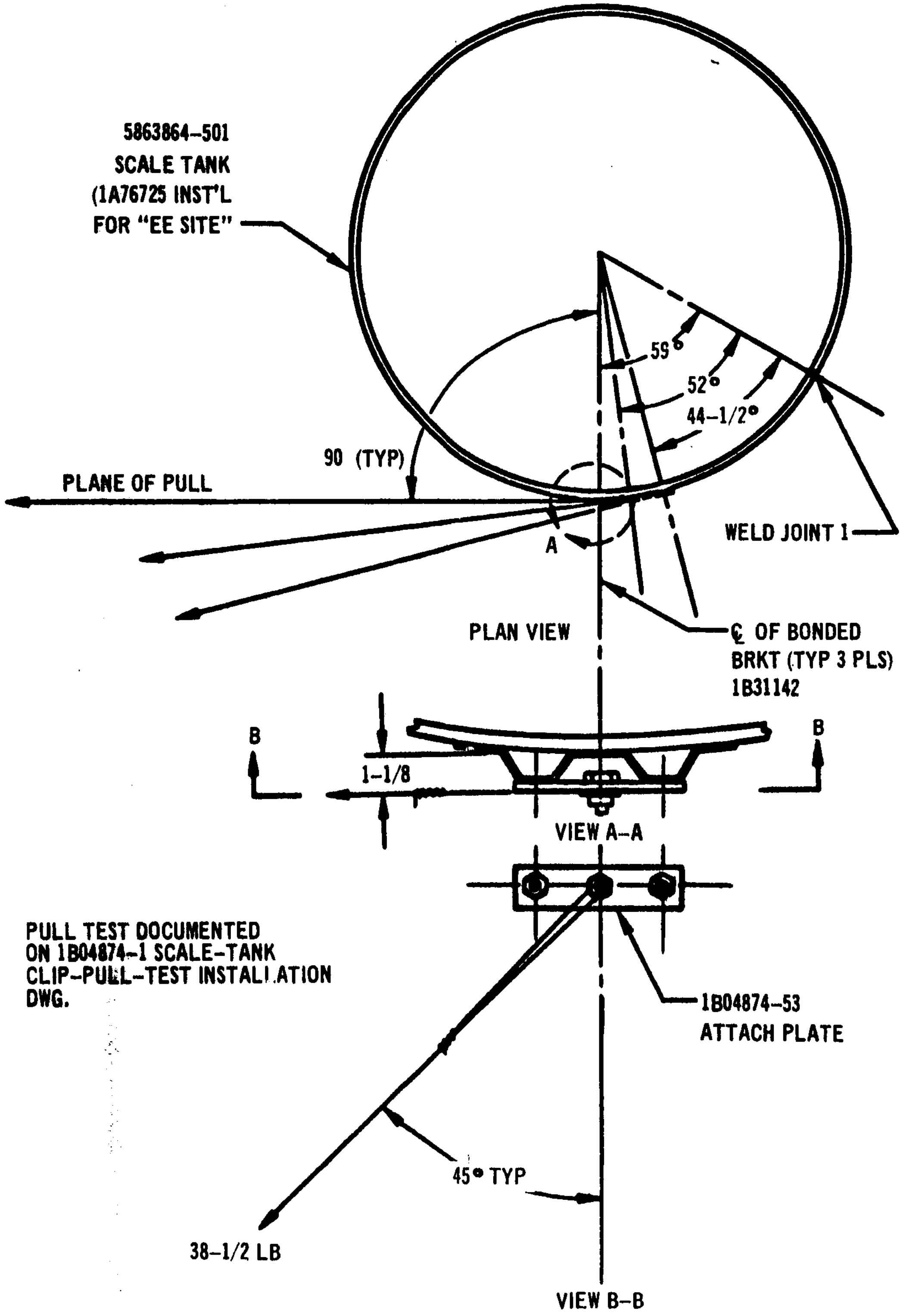


FIGURE 34

Figure 35 is a photo that shows the loading fixtures used to test the Nylafil wire bundle stand-offs used on the S-IVB stage of the Saturn vehicle.

Figure 36 is a photo of the 8-foot tank on the test stand with all the bonded attachments under load. The results of seven cycles of this testing showed that properly applied attachments using materials that were acceptable when tested on a laboratory scale were more than adequate for the design.

CONCLUSIONS (Figure 37)

1. Polyether urethanes reacted with MOCA still produce the best low temperature curing adhesive for cryogenic applications available.

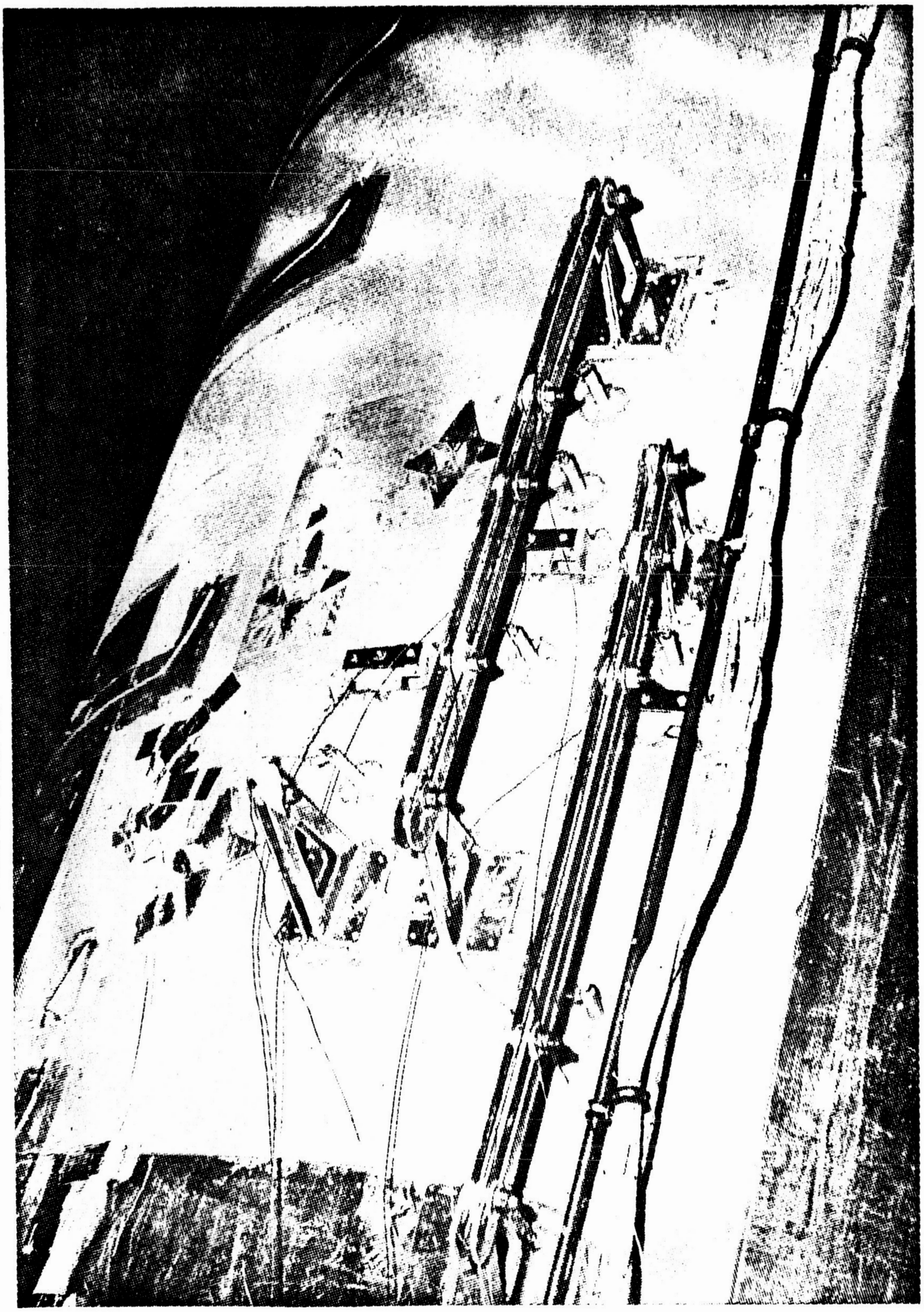
2. Polyurethane adhesives still possess the following troublesome deficiencies:

    a. Water sensitivity

    b. Poor room temperature creep

    c. Poor elevated temperature strength

    d. Requires solid-liquid blending to mix.



CLOSEUP VIEW OF LOAD DIRECTING ATTACHMENTS IN RELATION TO NYLAFIL WIRE BUNDLE SUPPORT CLIPS

FIGURE 35.

VIEW OF 8FT TANK ON TEST STAND WITH ALL BONDED ATTACHMENTS UNDER MECHANICAL LOAD

FIGURE 36

CONCLUSIONS ABOUT POLYURETHANE ADHESIVES RELATED WITH MOCA

- POLYURETHANES ARE THE BEST LOW TEMPERATURE CURING CRYOGENIC ADHESIVES CURRENTLY AVAILABLE.
- POLYURETHANES STILL POSSES TROUBLESOME DEFICIENCIES:
  a. WATER SENSITIVITY.
  b. POOR ROOM TEMPERATURE CREEP.
  c. POOR ELEVATED TEMPERATURE STRENGTH.
  d. REQUIRES SOLID–LIQUID BLENDING TO MIX.

FIGURE 37.

RECOMMENDATIONS

MAKE A NEW ADHESIVE BY MAKING A:

- NEW BACKBONE POLYMER.
- NEW POLYMER ALLOYS.
- NEW COMPOUNDS.
- SAME COMBINATION OF ABOVE.

FIGURE 38.

RECOMMENDATIONS

It is recommended that a new adhesive not possessing the inherent chemical dificiencies of a polyurethane be developed. This can be done by synthesizing a new backbone polymer or making new polymer alloys by compounding new material mixes or some combination of these (Figure 38).

The purposes of the new adhesive would be as follows (Figure 39).

1. Eliminate water sensitivity
2. Obtain true room temperature cure
3. Increase high temperature cure
4. Obtain liquid-liquid mixing
5. Reduce room temperature creep
6. Improve aging stability
7. Ease storage requirements

There is a practical problem connected with the synthesizing of a new polymer and that is in the evaluation of the small quantities normally produced in a laboratory batch. This is an area where Douglas is doing some significant work.

PURPOSE OF A NEW ADHESIVE

- ELIMINATE WATER SENSITIVITY.
- OBTAIN TRUE ROOM TEMPERATURE CURE.
- INCREASE HIGH TEMPERATURE PERFORMANCE.
- OBTAIN LIQUID – LIQUID MIXING.
- REDUCE ROOM TEMPERATURE CREEP
- IMPROVE AGING STABILITY.
- EASE STORAGE REQUIREMENTS.

FIGURE 39.

It started when the adhesives application personnel were told the quantity to be supplied for tests from these batches was less than their normal spillage in mixing. It turned out that this average quantity would be approximately 15 grams total which is actually less than normal waste in mixing.

The applications personnel accepted the challenge. Figure 40 shows the type of tests that can be applied to this quantity of material and the data that can be obtained. To do this required the development of semimicro-sized specimens and special test techniques. Figure 40 shows that the vibrating reed test apparatus will supply a dynamic modulus and also the rate of cure at various temperatures if required. The penetrometer will supply hardness numbers, softening temperature, glass transition temperature and linear coefficient of expansion. Accounting for material to chemically characterize a new polymer approximately 5 grams have been consumed for this much information on the base polymer. This leaves 10 grams for conventional adhesive tests. To get any significant quantity of tests results, the coupons were reduced to the semimicro size shown in Figure 41. Comparison with the full size specimens will show a reduction to approximately quarter scale. Using this sizing on the specimens it is possible to get the number of individual test coupons derived as shown in Figure 42. In fact there is even a little margin for waste.

EXPERIMENTAL ADHESIVE EVALUATION (15 GRAM QUANTITY)

TYPE OF TEST	DATA OBTAINED
VIBRATING REED	DYNAMIC MODULUS RATE OF CURE
PENETROMETER	HARDNESS SOFTENING TEMPERATURE GLASS TRANSITION TEMPERATURE LINEAR COEFFICIENT OF EXPANSION
LAP SHEAR	SHEAR STRENGTH
T-PEEL	PEEL STRENGTH
DUMBBELL TENSION	TENSILE STRENGTH STATIC MODULUS ELONGATION

FIGURE 40

SEMIMICRO TEST SPECIMENS

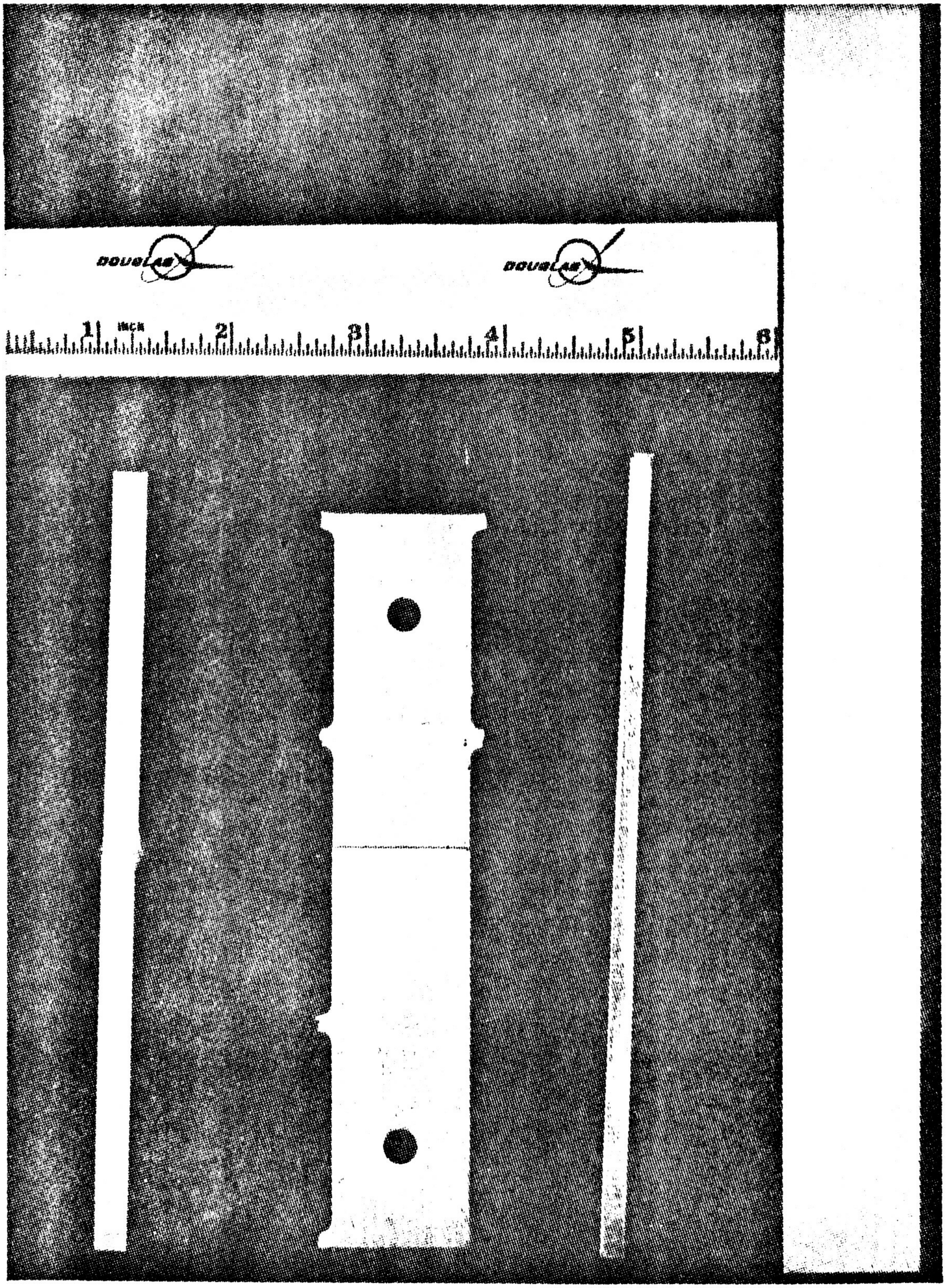


FIGURE 41

SEMIMICRO SPECIMENS MADE WITH 10 GRAMS OF ADHESIVE

ESTIMATED AVERAGE DENSITY	$\rho = 1.2$
QUANTITY AVAILABLE	10 GRAMS
VOLUME	$\left(\frac{10}{1.2}\right)(.061023) = .5125932\ IN^{-3}$
VOLUME PER LAP SHEAR:	$(.125)\ (.25)\ (.01) = .000313\ IN^{-3}$
VOLUME PER T PEEL:	$(.125)\ (6)\ (.01) = .0075\ IN^{-3}$
DUMBBELLS:	$(.0313)\ (3.5) = .04695\ IN^{-3}$

NO. OF SPECIMENS

LAP SHEARS	30	$.00939\ IN^{-3}$
T-PEELS	15	$.11250\ IN^{-3}$
DUMBBELLS	8	$.37560\ IN^{-3}$
		$.49749\ IN^{-3}$

SHEET SIZE (.0313 X 3 X 4) .49749

WASTE $.5125932 - .49749 = .0151\ IN^{-3}$ OR 2%

FIGURE 42

CORRELATION OF SEMIMICRO AND STANDARD T-PEEL ROOM TEMPERATURE TEST

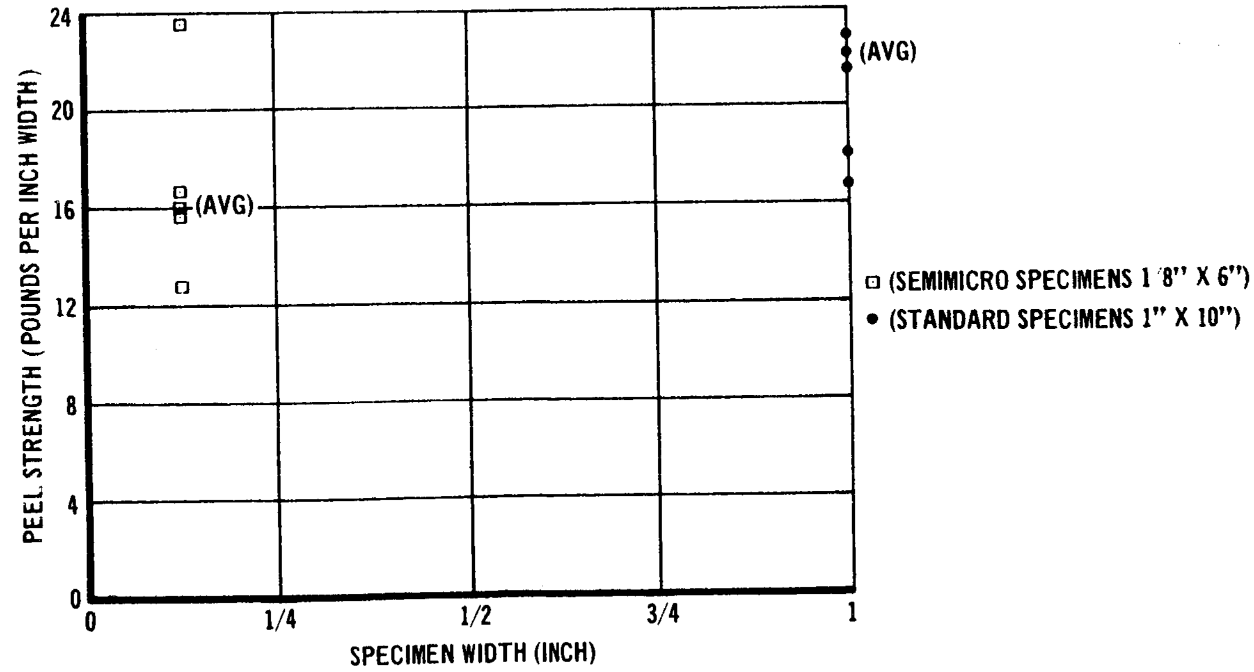


FIGURE 43

This semimicro system to obtain test results was tried on one of the conventional polyurethane adhesives to permit the correlation of the test results to full size specimens. There was ample material for the tests; in fact there was some material left over in a cured sheet form. Figure 43 shows a correlation of T-peel test results of full size specimens to its minature couterpart. Figure 44 shows the comparative lap shear results, and Figure 45 shows the comparative tensile dumbbell results. These latter tests supply information on base polymer tensile strength, static modulus which can be presented in several forms, as explained before, and elongation. All the semimcro tests were conducted on an Instron test machine.

This just shows there need be little, if any, sacrifice of data for small quantity testing for this new room temperature curing cryogenic adhesive.

CORRELATION OF SEMIMICRO AND STANDARD LAP SHEAR SPECIMENS
ROOM TEMPERATURE TEST

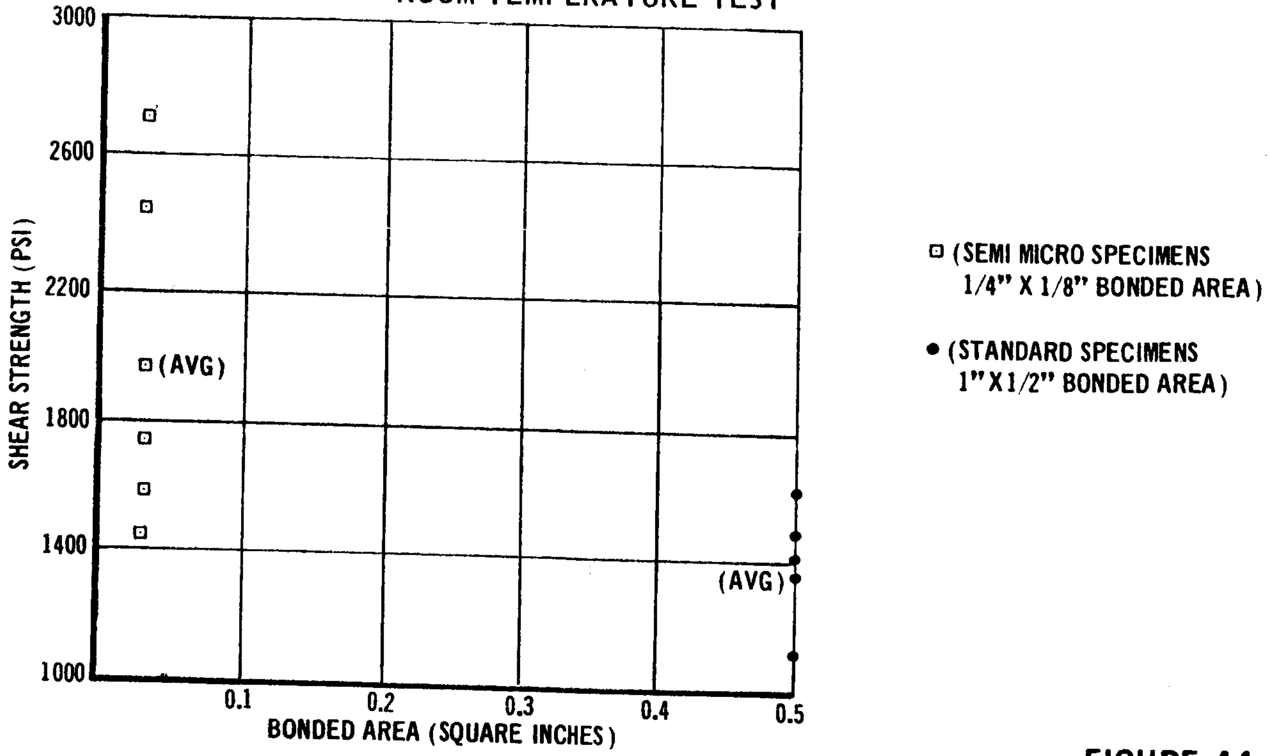


FIGURE 44

CORRELATION OF SEMIMICRO AND STANDARD DUMBBELL TENSION SPECIMENS
ROOM TEMPERATURE TEST

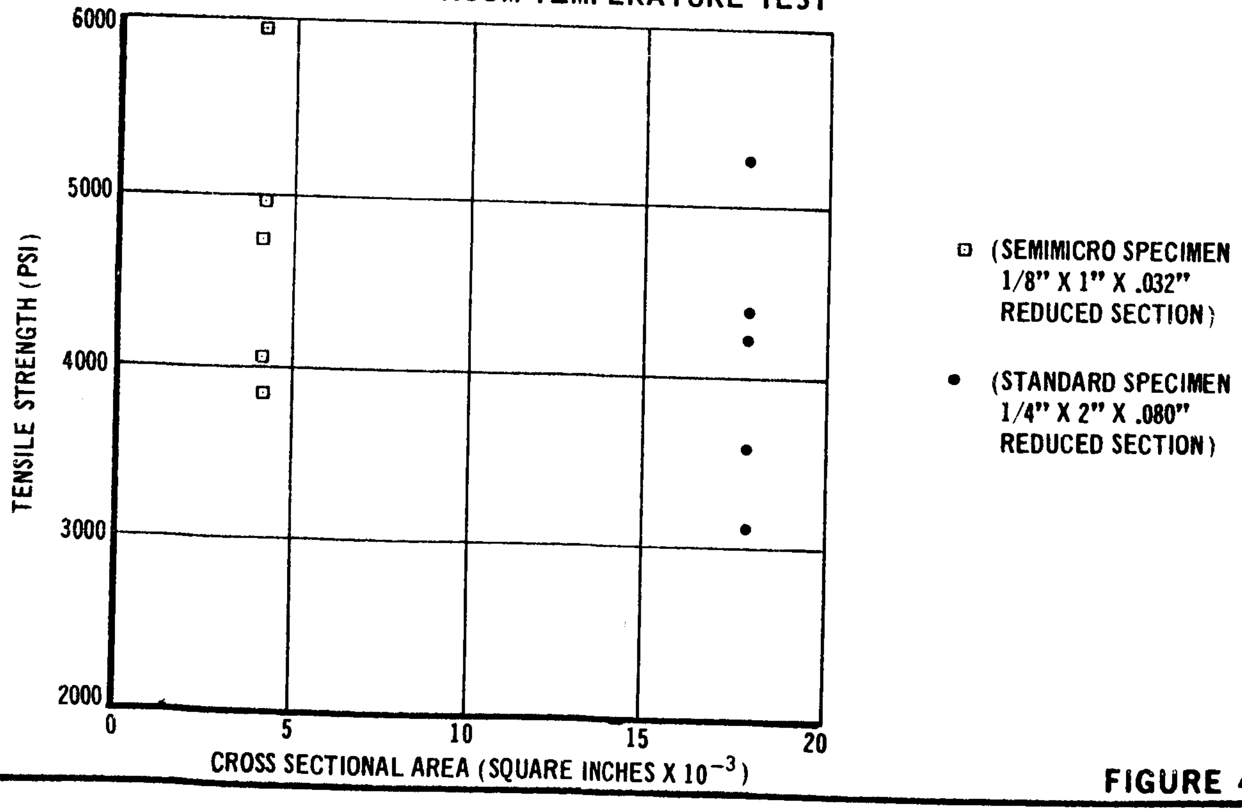


FIGURE 45

APPENDIX I

EFFECT OF WATER ON RESIN STOICHIOCMETRY

SAMPLE CALCULATIONS

Calculations were made to determine the percentage change in stoichiometry of hardener/resin mixtures caused by absorbed water on cleaned metal faying surfaces and absorbed water in the resin spread over the faying surfaces. To accomplish these calculations certain assumptions had to be made and the calculations performed in chronological order.

Information For Calculations

Molecular Weights

Water (H_2O)	18.016
Isocyanate (-NCO)	42.018
MOCA ($R\text{-}2NH_2$)	267.156

Conversion Factor

Change gm/cm^{-3} to gm/in^{-3} multiply by 16.387.

Data

Specific gravity of Adiprene L-100	1.2 gms/cm^{-3}

Assumptions

All weight increase is due to water.

All water reacts with the isocyanate in the resin.

Datum

Bondline thickness is .002 inch.

Resin Mix Stoichiometry

Computation of the hardener/resin stoichiometry in the resin mixes.

Resin Chemistry

Three separate batches of material were used and the respective isocyanate contents by weight percent were determined analytically by the wet chemical procedure in Appendix I. These values are as follows:

Batch #1	4.249 wt% -NCO
Batch #2	4.200 wt% -NCO
Batch #3	3.883 wt% -NCO

Adhesive Mix Ratio

The three batches were mixed in the following ratios with MOCA considered to be a 100% of the bifunctional amine:

Batch #1	11.6	gms MOCA/100 gms resin
Batch #2	12.3	gms MOCA/100 gms resin
Batch #3	12.99	gms MOCA/100 gms resin

General Equation Derivation

The derivation of the general solution is as follows:

Using a batch size of 100 grams of resin, the weight percent of active -NCO may be converted to grams of -NCO as follows:

$$\frac{(\text{wt\% -NCO})\ (100\ \text{grams})}{(100\%)} = \text{grams NCO}$$

Therefore, the wt% value and the grams of active -NCO per 100 grams of resin is the same number.

grams -NCO = wt% -NCO; for 100 grams of resin

The number of chemical equivalents of active -NCO is determined as follows:

$$\frac{\text{grams -NCO}}{\text{molecular wt of -NCO expressed in grams per gram mole}}$$

$= \text{g moles of active -NCO}$

Since the molecular weight of only one functional group (-NCO) is used in this calculation, the resulting number is also the number of chemical equivalents of active -NCO.

therefore:

$$\frac{\text{grams -NCO}}{\text{molecular wt of -NCO expressed in grams per gram mole}}$$

$= \text{g equivalents of active -NCO}$

Substituting the molecular wt of -NCO:

$$\frac{\text{grams -NCO}}{\text{42.018 grams/g mole}} = \text{No. g equivalents -NCO}$$

Conducting the computations to convert the grams of MOCA to g equivalents.

$$\frac{\text{molecular wt of MOCA expressed in grams per gram mole}}{\text{number of gram equivalents per gram mole}}$$

$$= \frac{\text{grams of MOCA}}{\text{g equivalent of MOCA}}$$

Since MOCA has two active ($-NH_2$) groups per molecule, the number of chemical equivalents per mole is two.

Substituting values:

equation (1)

$$\frac{267.156 \text{ grams/g mole}}{2 \text{ g equivalents/g mole}} = 133.385 \frac{\text{grams}}{\text{g equivalent}} \quad \text{(MOCA)}$$

To have one active amine for each active isocyanate group the number of equivalents of the amine groups as MOCA must equal the number of equivalents of isocyanate groups ($-NCO$). This relationship is considered to be a 100% stoichiometric mix of hardener/resin.

therefore:

No. g equivalents ($-NCO$) = No. g equivalents (MOCA)

Since the information really sought is the number of grams of MOCA to produce a one to one ratio on g equivalents, a substitution of the grams of MOCA per g equivalent of $-NCO$ can be obtained by multiplying:

$$\frac{(\text{No. g equivalents } -NCO)(133.385 \text{ grams MOCA})}{\text{g equivalent MOCA}}$$

= grams of MOCA @ 100% stoichiometry

To determine the actual percent stoichiometry, it is necessary to ratio the actual number of grams of MOCA to that required for a theoretical 100% stoichiometric mix.

$$\frac{(\text{actual grams of MOCA})(100)}{(\text{theoretical grams of MOCA})} = \text{actual percent stoichiometry}$$

Substituting back for the number of grams of MOCA theoretically required:

$$\frac{(\text{grams of MOCA})\quad(\text{g equivalents MOCA})\quad(100)}{(\text{No. g equivalents -NCO})(133.385\ \text{grams MOCA})}$$

$$= \text{actual percent stoichiometry}$$

Substituting for the number of g equivalents of -NCO:

<u>equation</u> (2)

$$\frac{(\text{grams of MOCA})(42.018\ \text{grams/g mole})(\text{g equivalents MOCA})(100)}{(\text{grams of -NCO})(133.385\ \text{grams MOCA})}$$

$$= \text{actual percent stoichiometry}$$

Simplifying and substituting wt% -NCO for grams of -NCO:

<u>equation</u> (3)

$$\frac{(31.5)(\text{grams of MOCA})}{(\text{wt\% -NCO})} = \text{percent stoichiometry}$$

Equation (3) is the general solution that requires only the substitution of the number of grams of MOCA used per 100 grams of resin and the resins corresponding weight percent of active -NCO.

Resin Stoichiometry Calculations

The particular solution for the percent stoichiometry using Batch #1 with its corresponding mix ratio is as follows:

Using equation (3) on Batch #1

$$\frac{(31.5)(11.6)}{(4.249)} = 86.2\% \text{ stoic.}$$

Batch #1	86.2% stoic.
Batch #2	92.4% stoic.
Batch #3	105.3% stoic.

Faying Surface Water Effect On Resin Stoichiometry

Determination of the quantity of water on the metal faying surface that will contact the adhesive and its effect on the resin stoichiometry.

Test Data

Metal strips (1" x 10" x .032" thick bare 2014-T6 aluminum alloy) were used to determine water pickup on a chemically active surface. Surfaces were chemically etched in a sodium dichromate/sulfuric acid solution, oven dried, then placed in the environments as noted.

	DRY BOX (9% RH, 72 ± 2°F)	CONSTANT TEMP. ROOM (50% RH, 72 ± 2°F)
Batch	Weight Gain	Weight Gain
#1	.0010 grams	.0015 grams
#2	.0007 grams	.0013 grams
#3	.0012 grams	.0013 grams

Weight gains are based on 20 square inches of surface.

The batch numbers correspond to the metal coupons used ultimately with the batches of mixed resins noted.

The weights are average increases from several measurements taken between 2 to 4 hours of exposure to the respective environments.

General Equation Derivation

Using the assumptions that all the weight gain is water and that all the water participates in a chemical reaction to tie up free isocyanate, it is possible to derive a general equation that will express the weight of water per unit area in terms of percent stoichiometry per unit thickness of bondline. This derivation is as follows:

$$\frac{\text{(Weight of water as grams/in.}^2}{\text{(Molecular weight of water as grams/g mole)}} = \text{g moles of active ater/in.}^2 = \text{g equivalents of active water/in.}^2$$

The second equality exists because water has only one functional group per molecule to participate in this reaction; therefore, the number of g moles of water equals the number of g equivalents of water.

Rearranging:

equation (4)

$$\frac{\text{(Weight of water as grams/in.}^2}{\text{(Molecular weight of water as grams/g mole)(g equivalents of active water)}} = 1$$

Rearranging equation (1) to the same form:

equation (5)

$$\frac{(267.156 \text{ grams/g mole})}{(2 \text{ g equivalents/g mole})(133.385 \text{ grams/g equivalent})} = 1$$

Which means equation (4) equals equation (5). Therefore, by substracting equation (5) from equation (2) and adding equation (4) to equation (2) its value has not changed but it is now in terms of water instead of MOCA. This manipulation produces equation (6) as follows:

equation (6)

$$\frac{\left(\begin{array}{l}\text{Weight of water as}\\ \text{grams/in.}^2\end{array}\right)\left(\begin{array}{l}\text{42.018 grams}\\ \text{/g mole}\end{array}\right)\left(\begin{array}{l}\text{g equivalent}\\ \text{active water}\end{array}\right)(100)}{(\text{grams of -NCO})(\text{18.016 grams/g mole of active water})}$$

$$= \text{Actual percent stoichiometry}$$

Simplifying and substituting wt% -NCO for grams of -NCO from equation (3).

equation (7)

$$\frac{(233.23)(\text{grams of water/in.}^2)}{(\text{wt\% -NCO})} = \frac{\text{percent stoichiometry}}{\text{100 grams of resin}}$$

This equation gives the effect of one square inch of wetted metal surface on a 100 grams of resin.

To make this relationship more useful, it is put in terms of mils of bond line thickness per square inch of the two faying surfaces that make up the bonded joint.

To accomplish this requires the introduction of the density of the adhesive mix. It is assumed the resin and MOCA both have the same density (p). This assumption is accurate within reasonably close tolerances.

$$p \text{ (resin)} = p \text{ (MOCA)} = 1.2 \text{ grams/cm}^3$$

The volume (V) of the mix is:

$$V = \frac{(100 \text{ grams resin} + \text{number grams MOCA})}{(1.2 \text{ grams/cm}^3)(16.387 \text{ cm}^3/\text{in.}^3)}$$

<u>equation</u> (8)

$$V = \frac{(100 \text{ grams resin} + \text{number grams MOCA})(\text{in.}^3)}{19.6644 \text{ grams}}$$

Since the total adhesive mix including MOCA are included in the volume, the following substitution has to be made in equation (7):

(100 grams resin + no. grams MOCA) for (100 grams of resin)

Rearranging equation (7)

$$\frac{(233.23)\begin{pmatrix}\text{no. of grams}\\ \text{of water/in.}^2\end{pmatrix}(100\text{ grams resin + no. grams MOCA})}{(\text{wt\% -NCO})}$$

= Percent stoichiometry (water only)

Substituting the volume equivalent from equation (8)

$$\frac{(\text{in.}^3)(233.23)\begin{pmatrix}\text{no. of grams}\\ \text{of water/in.}^2\end{pmatrix}\begin{pmatrix}\text{100 grams resin +}\\ \text{no. grams MOCA}\end{pmatrix}}{(\text{wt\% -NCO})(19.6644)\text{ grams}}$$

= Percent stoichiometry (water only)

Putting the equation in terms of two metal faying surfaces contributing moisture and putting the thickness of bond line on a per mil basis the equation reduces to the following:

$$\frac{(2)(233.23)\begin{pmatrix}\text{No. of grams}\\ \text{of water/in.}^2\end{pmatrix}\begin{pmatrix}\text{100 grams resin +}\\ \text{no. grams MOCA}\end{pmatrix}(\text{in.}^3)}{(\text{No. .001 in.})(\text{wt\% -NCO})(19.664)(\text{grams})}$$

= Percent stoichiometry (water only)

Simplifying:

equation (9)

$$\frac{(23721.039)\begin{pmatrix}\text{No. of grams}\\ \text{of water/in.}^2\end{pmatrix}\begin{pmatrix}\text{100 grams resin +}\\ \text{no. grams MOCA}\end{pmatrix}}{(\text{No. mils bond thickness})(\text{wt\% -NCO})}$$

= Percent stoichiometry (water on metal only)

Faying Surface Water Stoichiometry Calculations

The particular solution for the effect on the stoichiometry caused by water on the metal based on the data is as follows:

Using equation (9) and Batch #1 and Dry Box value:

$$\frac{(23721.039)(.001/20)(100 + 11.6)}{(2)\quad(4.249)} = 15.58\% \text{ stoichiometry}$$

NOTE: A .002 inch thick bond line was assumed.

Effect of water on the metal as percent stoichiometry:

	DRY BOX (9% R.H., 72 ± 2°F)	CONSTANT TEMP. ROOM (50% R.H., 72 ± 2°F)
Batch	% Stoic.	% Stoic.
#1	15.58	20.94
#2	11.10	20.61
#3	20.71	22.43

Resin Absorbed Water Effect On Stoichiometry

This section covers the determination of adhesive stoichiometry caused by water being absorbed into the surface of the exposed adhesive.

Data was acquired by exposing dried low cylindrical walled, flat bottomed, glass Petri dishes containing a layer of resin and empty to a normal laboratory environment and determining the weight increase with respect to time:

Data

Petri Dish Dimensions

Dimensions of the dish:

Diameter (d) = 3.5 inches

Depth (h) = .375 inch

Weight Changes Of The Dish

TIME OF EXPOSURE	EMPTY DISH WEIGHT INCREASE	DISH & RESIN WEIGHT INCREASE
20 min.	.0004 gms	.0013 gms
40 min.	.0006 gms	.0064 gms
60 min.	.0012 gms	.0088 gms

Calculation Of Absorbed Water

Determination of the water absorbed into the surface of the adhesive as a function of time.

Surface of the dish:

$$\text{Flat surface} = \frac{\pi d^2}{1} = \frac{\pi(3.5)^2}{4} = 9.621 \text{ in.}^2$$

$$\text{Cylindical surface} = \pi dh = \pi(3.5)(.375) = 4.4835 \text{ in.}^2$$

Total dish surface area (A)

= 2 (flat surface) + 2 (cylindical surface)

$$A = 2(9.621) + 2(4.4835) = 28.209 \text{ in.}^2$$

When resin was placed in the dish in a thin layer the area of glass remaining to adsorb water is reduced by 9.621 square inches. The ratio of effective area to total area was calculated so the weight increase due to absorption of moisture in the resin could be determined.

$$\begin{pmatrix}\text{Total surface}\\ \text{area of dish}\end{pmatrix} - \begin{pmatrix}\text{resin covered}\\ \text{area}\end{pmatrix} = \text{exposed area of glass}$$

$$(28.209 \text{ in.}^2) - (9.621 \text{ in.}^2) = 18.588 \text{ in.}^2$$

$$\text{Ratio} = \frac{18.588 \text{ in.}^2}{28.209 \text{ in.}^2} = .636$$

If the weight increase of the empty dish is multiplied by the ratio of exposed glass surface, the weight increase caused by adsorption of water to the glass can be calculated.

Empty dish wt increase (.636) = Water adsorbed on exposed glass of resin containing dish

Separating the water adsorbed to the exposed glass from the total weight gain of the dish containing resin gives the water absorbed into the surface of the resin.

Example:

Basis 20 minutes exposure time:

Empty dish wt

(.0004)(.636) = .0002544 grams of water adsorbed on the exposed glass

Total weight gain of the dish with resin

.0013 grams in 20 minutes

Substracting the adsorbed water on the glass gives the absorbed water into the surface of the resin as follows:

.0013 - .0002544 = .0010456 grams adsorbed water in resin

DISH & RESIN (wt increase)	WATER ADSORBED ON EXPOSED GLASS	WATER ABSORBED BY THE RESIN
.0013 gms	.0003 gms	.0010 gms
.0064 gms	.0004 gms	.0060 gms
.0084 gms	.0008 gms	.0080 gms

The weight of water absorbed into the surface of the resin per square inch is the total weight change of the resin as a function of time divided by its exposed surface area.

Example:

Total water absorbed by the resin in 20 minutes:

$$\frac{(.0010 \text{ grams})}{(9.621 \text{ in.}^2)} = 1.04 \times 10^{-4} \text{ gm/in.}^2$$

TIME OF EXPOSURE	RESIN WEIGHT INCREASE (grams/in.^{2})
20 minutes	1.04×10^{-4}
40 minutes	6.24×10^{-4}
60 minutes	8.32×10^{-4}

General Equation Derivation (Spread Adhesive)

The effect of the water absorbed into the surface of the resin on the adhesive stoichiometry when the adhesive is spread with a spatula so that it uniformly covers the metal faying surface in a thin film is obtained as follows:

Using equation (9) and considering only one surface is exposed instead of two:

$$\frac{(23721.039)\begin{pmatrix}\text{No. of grams}\\ \text{of water/in.}^2\end{pmatrix}\begin{pmatrix}\text{100 grams resin +}\\ \text{no. grams MOCA}\end{pmatrix}}{(2)\ (\text{No. mils bond thickness})\ (\text{wt\% -NCO})}$$

$$= \text{Percent stoichiometry (water in resin only)}$$

Simplifying:

equation (10)

$$\frac{(11860.519)\begin{pmatrix}\text{No. of grams}\\ \text{of water/in.}^2\end{pmatrix}\begin{pmatrix}\text{100 grams resin +}\\ \text{no. grams MOCA}\end{pmatrix}}{(\text{No. mils bond thickness})\ (\text{wt\% -NCO})}$$

$$= \text{Percent stoichiometry (water in resin only)}$$

Resin Stoichiometry Calculations

Example:

Using Batch #1 adhesive and an exposure time of 20 minutes on the adhesive:

$$\frac{(11860.519)\ (1.04 \times 10^{-4})\ (100 + 11.6)}{(2)\ (4.249)} = 16.20\%$$

The bond line thickness was again assumed .002 inch thick.

BATCH #1

TIME OF EXPOSURE	EFFECT OF RESIN ABSORBED WATER ON PERCENT STOICHIOMETRY
20 minutes	16.20%
40 minutes	97.36%
60 minutes	129.60%

BATCH #2

TIME OF EXPOSURE	EFFECT OF RESIN ABSORBED WATER ON PERCENT STOICHIOMETRY
20 minutes	16.49%
40 minutes	98.94%
60 minutes	131.93%

BATCH #3

TIME OF EXPOSURE	EFFECT OF RESIN ABSORBED WATER ON PERCENT STOICHIOMETRY
20 minutes	17.95%
40 minutes	107.68%
60 minutes	143.57%

Examination of the effects of water absorption into the adhesive shows the spatula method of application requires a very rapid closure of the faying surfaces to be joined if any stoichiometric control is to be maintained.

General Equation Derivation (Bead Application)

The use of a sealant gun to lay a "bead" of adhesive on a test specimen permits the minimum of adhesive surface exposure to a maximum of resin deposited. The water absorbed by the adhesive using this technique can be calculated as follows:

> The weight of water absorbed on a bead of adhesive applied to a standard T-peel coupon was determined by calculating the surface area of the adhesive bead and multiplying by the amount of absorbed water per square inch.

Surface area of "bead" $(A_b) = \pi$ (bead diameter) (bead length)

$$A_b = \pi\ (.1675\ \text{in.})\ (10\ \text{in.}) = 5.26\ \text{in.}^2$$

Not all the water absorbed into the surface of a bead of adhesive finds its way into a bonded joint. The volume of the bead of adhesive exceeds the volume of the adhesive used to join the two halves of a T-peel coupon. This squeezed out adhesive takes with it the amount of water proportional to the volume.

The volume of the bead is as follows:

$$V_b = \pi\,(.08375)^2(10) = .220\ \text{in.}^3$$

The quantity of water absorbed is the surface area of the bead times the water absorbed per square inch.

<u>equation</u> (11)

$$\frac{(5.26\ \text{in.}^2)\ (\text{No. grams of water/in.}^2)}{(10\ \text{in.}^2)}$$

$$= (.526)\ (\text{No. of grams of water/in.}^2)$$

Substituting equation (11) into equation (10):

equation (12)

$$\frac{(11860.519)(.526)\begin{pmatrix}\text{No. of grams}\\ \text{of water/in.}^2\end{pmatrix}\begin{pmatrix}\text{100 grams of resin +}\\ \text{no. grams MOCA}\end{pmatrix}}{(\text{No. mils bond thickness})(\text{wt\% -NCO})}$$

$$= \text{Percent stoichiometry (water in bead only)}$$

To adjust for the adhesive squeeze out and its resultant reduction of water in the cured bond line, it is necessary to ratio the volume of adhesive in the cured bond line to the volume of the adhesive bead applied, both per square inch of specimen. This is done as follows:

Volume (V_b) of adhesive bead per square inch of specimen:

$$V_b = \frac{.220 \text{ in.}^3}{10 \text{ in.}^2 \text{ of specimen faying surface}}$$

$$V_b = .022 \text{ in.}^3/\text{in.}^2 \text{ of specimen}$$

Volume (V_l) of adhesive in cured bond line per square inch of specimen:

$$V_l = \frac{(\text{No. of mils bond thickness})(1 \text{ in.}^2)(\text{in.})/(\text{in.}^2)}{(1000 \text{ mils})}$$

Ratio of the volumes:

$$\frac{V_l}{V_b} = \frac{(\text{No. of mils bond thickness})(\text{in.}^2)(\text{in.})/(\text{in.}^2)}{(1000\text{ mils})\ (.022\text{ in.}^3)/\text{in.}^2}$$

Simplifying:

$$\frac{V_l}{V_b} = \frac{(\text{No. of mils bond thickness})}{22\text{ mils}}$$

Applying the ratio of squeeze out to equation (12):

$$\frac{\begin{pmatrix}\text{No. of mils}\\ \text{bond thickness}\end{pmatrix}(11860.519)(.526)\begin{pmatrix}\text{No. of grams}\\ \text{of water/in.}^2\end{pmatrix}}{(\text{No. mils bond thickness})\ (\text{wt\% -NCO})}$$

$$\frac{(100\text{ grams of resin + Number of grams MOCA})}{(22\text{ mils})}$$

= Percent stoichiometry (water in resin only).

Simplify:

$$\frac{(283.574)\begin{pmatrix}\text{No. of grams}\\ \text{of water/in.}^2\end{pmatrix}\begin{pmatrix}\text{100 grams of resin +}\\ \text{number of grams MOCA}\end{pmatrix}}{(\text{wt\% -NCO})}$$

= Percent stoichiometry (water in resin only)

Resin Stoichiometry Calculations

Example of a particular solution:

Using Batch #1 adhesive and an exposure time of 20 minutes on the bead of adhesive.

$$\frac{(283.574)\ (1.04 \times 10^{-4})\ (100 + 11.6)}{4.249} = .65\%$$

The bond line thickness does not enter into this equation.

BATCH #1

TIME OF EXPOSURE	EFFECT OF RESIN ABSORBED WATER ON PERCENT STOICHIOMETRY
20 minutes	.65%
40 minutes	3.91%
60 minutes	5.21%

BATCH #2

TIME OF EXPOSURE	EFFECT OF RESIN ABSORBED WATER ON PERCENT STOICHIOMETRY
20 minutes	.79%
40 minutes	4.73%
60 minutes	6.31%

BATCH #3

TIME OF EXPOSURE	EFFECT OF RESIN ABSORBED WATER ON PERCENT STOICHIOMETRY
20 minutes	.86%
40 minutes	5.15%
60 minutes	6.87%

Cumulative Effect Of Water On Stoichiometry

The total effect on the stoichiometry by both the water and MOCA is cumulative with all the water reacting preferencially to the slower reacting MOCA.

Example:

> Using Batch #1 adhesive on T-peel coupons that have stabilized in the constant temperature room for two hours and a bead of adhesive that has been left exposed for 20 minutes. Using a two mil bond line thickness the values alread computed my be added to give a cumulative stoichiometry.

BATCH #3

TIME OF EXPOSURE	EFFECT OF RESIN ABSORBED WATER ON PERCENT STOICHIOMETRY
20 minutes	.86%
40 minutes	5.15%
60 minutes	6.87%

Cumulative Effect Of Water On Stoichiometry

The total effect on the stoichiometry by both the water and MOCA is cumulative with all the water reacting preferencially to the slower reacting MOCA.

Example:

Using Batch #1 adhesive on T-peel coupons that have stabilized in the constant temperature room for two hours and a bead of adhesive that has been left exposed for 20 minutes. Using a two mil bond line thickness the values alread computed my be added to give a cumulative stoichiometry.

Stoichiometry of the Adhesive mix	86.2%
Stoichiometric contribution of water on the metal	23.37%
Stoichiometric contribution of absorbed water in the adhesive bead	.65%
TOTAL	110.22%

APPENDIX II

MODULUS DETERMINATION

All material in tension will stretch as the load increases. Up to a certain load, the material will resume its original shape on removal of the load. This phenomenon of returning to its original form is called elasticity. There is a load beyond which the material stays stretched and this value is defined as the elastic limit. A definite property known as the modulus of elasticity (E), may be determined if the stretch or stress is proportional to the load or strain.

Rubber and rubber like material's are commonly subjected to relatively large deformations and the strain is not proportional to the stress. The modulus of elasticity (E) is not constant and the conditions to which a given value applies must be stated.

There have been many methods derived for calculating modulus resulting in considerable spread in values and confusion as to what the values represent. It is for this reason, the equations used to derive the modulus in this report have been specified.

Rubber Industry "Modulus" (M)

The tensile modulus (M) is measured to get a direct comparison to vendor supplied data which is always presented in this form as an industry convention.

Tensile moduli (M) were calculated using the formula employed by rubber technologists, of load divided by area. These values compare favorably to those reported by the manufacturer. The calculations used to derive this modulus disregard the fact that since the volume remains almost constant during stretching and the length increases (as indicated by the percent elongation) the cross sectional area must change proportionally.

Young's Modulus (E)

The conventional modulus (E) is reported to supply a modulus in a form usually used for design. The modulus as used by metallurgists does consider changes in elongation. This is sufficient for materials having low elongation because the change in cross sectional area is negligible. Rubber like materials exhibit change in area. It is for this reason that the formula for the energy modulus was derived.

Energy Modulus (E')

The energy modulus (E') formula incorporates both the elongation and the change in area. This yields not only a modulus for a given stress but also the amount of work per unit volume required to produce such stress. Work and modulus happen to have the same numerical value when using this formula. The energy modulus and the tensile modulus at 100% elongation only are identical because $l_f - l_o$ is unity.

Shear Modulus (G) By Kinetic Theory

The formula for the shear modulus (G) is reported to supply a means for calculating Poisson's ratio in a laterally unrestrained stressed elastomer. Poisson's ratio is given by:

$$\mu = \frac{E}{2G} - 1$$

SPECIMEN PREPARATION

Die Cutting Dumbbels

Cured sheets of material may be cut into dumbbell tensile specimens using a 4-1/2" x 1" dumbbell cutting die.

Marking Gages

Ink bench marks are placed on the reduced section of each dumbbell specimen equidistant from its center, one inch apart, and perpendicular to its longitudinal axis. Three measurements should be read for the thickness, one at the center and one at each end of the reduced section of the specimen and the average of the three measurements used as the nominal thickness in calculating the cross-sectional area.

TESTING

Room Temperature Testing

The dumbbell specimens are tested for tensile strength and elongation per Federal Test Method Standard Number 406, Method 1013. The specimens tested at room temperature placed in the grips of a Tensile Testing Machine. The machine started and the distance between the two bench marks continuously monitored until failure. The ultimate elongation is calculated by letting ℓ_f equal the observed distance between bench marks on the stretched specimen at the time of rupture as follows:

$$\text{Elongation } \% = \frac{\ell_f - \ell_o}{\ell_o} \times 100$$

Where: ℓ_f = Observed distance between bench marks on the stretched specimen.

ℓ_o = Original distance between bench marks.

Low Temperature Testing

The elongation of specimens tested at temperatures other than room temperature may be determined by using the head travel of the Tensile Testing Machine. This was recorded on a load deflection graph and used as ℓ_f in the equation. This measurement gives the total strain in the entire specimen including that outside of the gauge lengths. Measurements should be made at room temperature to establish the ratio of total specimen strain to the strain within the gauge length. This would permit the separation of the gauge length strain from the total strain under conditions where the gauge length strain can not be measured directly.

Moduli Calculations

Four separate formulas for moduli are presented. Three of these were used for calculating the modulus. The fourth was presented to enable the reader to calculate the shear modulus and Poisson's ratio.

Rubber Industry "Modulus" (M)

The rubber industry, tensile "modulus" is as follows"

$$M = \frac{P_n}{A_o}$$

Where: M_n = "Modulus" at a given elongation.

P_n = Load at a given elongation.

A_o = Original cross-sectional area.

n = % Elongation: 100, 200, 300, etc.

This method of calculation reports, as "modulus", a load at a corresponding elongation divided by the original cross sectional area. In reality it yields tensile stress at a specified elongation which is a convention of the rubber industry.

Young's Modulus (E)

The method of reporting the conventional modulus is per the classical theory of stress per unit strain as follows:

$$E = (P/A_o)(\ell_o / \ell_f - \ell_o)$$

This calculation does not compensate for the change in cross sectional area in elastic materials.

Energy Modulus (E')

The following is the derivation of what shall be henceforth called the energy modulus (E'). Neither the rubber industry nor the classical Young's moduil adequately describes the performance of an elastomer under load.

Two features considered negligible in the computation of a Young's modulus are; first, the change of cross-sectional area induced on a material that is laterally unrestrained and; second, the natural strain which is actually the summation of the total strain that corresponds to each increment of stretch.

If these two features are incorporated into a modulus, the modulus would give an accurate measure of the energy to cause a uniaxial deformation. In other words it would be a measure of material toughness which is usually the most valuable property of an elastomer and is the main reason it is used in specified designs.

To evolve this new modulus will require a few statements to establish a datum.

Unrestrained Uniaxial

The elastomer is subjected to uniaxial stress and is unrestrained in the lateral directions. This closely approximates the case for a simple dumbbell specimen.

Constant Volume

An elastomer undergoes no volume change when stressed. This statement has been proven accurate within one percent. Therefore V is assumed constant and $V = A\ell$ which is the cross sectional area times the length. Then $A = V/\ell$ or the cross sectional area is given in terms volume divided by length. In a plot A versus ℓ the resultant curve is a hyperbola.

Natural Strain

The true strain; hereafter, called the natural strain is the summation of the total strain that corresponds to each increment of stretch.

Where the natural strain is:

$$\epsilon = \Sigma_{i=0}^{n}\left(\frac{\ell_1 - \ell_0}{\ell_0} + \frac{\ell_2 - \ell_1}{\ell_1} + \cdots + \frac{\ell_i - \ell_{i-1}}{\ell_{i-1}} + \cdots + \frac{\ell_n - \ell_{n-1}}{\ell_{n-1}}\right)$$

Where ℓ_0 is the unstressed initial length of the specimen gage length and ℓ_i is any greater length caused by an imposed load.

Putting the natural strain in the lineal form:

$$\epsilon = \Sigma_{i=0}^{n} \frac{\ell_{i+1} - \ell_i}{\ell_{i-1}}$$

This can be restated as:

$$\epsilon = \Sigma_{\ell_0}^{\ell_n} \frac{\Delta \ell}{\ell}$$

where the ℓ's are general variables of length.

Put in the differential form by letting $\Delta \ell$ approach 0:

$$\epsilon = \int_{\ell_0}^{\ell_n} \frac{d\ell}{\ell}$$

Which is the useable expression for the natural strain.

<u>Modulus As Modified Stress Over Modified Strain</u>

The energy modulus E' like Young's modulus will retain the concept of modulus equals stress over strain only with two added features. The natural strain will replace the rectilinear strain and the cross sectional area used to compute stress will be the true area at the corresponding stress level.

Starting with Young's modulus: $E' = \sigma / \epsilon$ which is modulus equals stress over strain.

Stress $\sigma = P/A$ or load over cross sectional area.

Substituting in the modulus equation:

$$E' = \frac{P/A}{\epsilon}$$

Since the cross sectional area equals the volume divided by the length.

$$A = V/\ell$$

Substituting for A_o in the modulus equation.

$$E' = \frac{P}{\frac{V}{\ell}\epsilon}$$

Replacing the rectilinear strain with the natural strain.

$$E' = \frac{P}{\frac{V}{\ell}\int_{\ell_0}^{\ell_n} \frac{d\ell}{\ell}}$$

Placing the like variables (ℓ) behind the integrand.

$$E' = \frac{P}{V\int_{\ell_0}^{\ell_n} \ell^{-2}\, d\ell}$$

Rearranging to obtain a more convienent form:

$$\frac{P}{E'} = V\int_{\ell_0}^{\ell_n} \ell^{-2}\, d\ell$$

Performing the indicated integration:

$$\frac{P}{E'} = -V\left[\ell^{-1}\right]_{\ell_0}^{\ell_n}$$

Substituting the limits:

$$\frac{P}{E'} = -V\left[\ell_n - \ell_0\right]^{-1}$$

Solving for E':

$$E' = -\frac{P}{V}\left(\ell_n - \ell_0\right)$$

This equation gives the energy modulus of a substance at any degree of specimen strain provided the corresponding load is used. To indicate this feature and to put the equation in a more general form the variable load (P) will have the same subscript (n) applied as its corresponding gage length (ℓ_n) at load (P_n). This subscript (n) shows these values are any corresponding measured dimensions.

CONCLUSIONS

The following conclusions may be made about polyurethane elastomers.

1. The energy modulus (E') or work per unit volume to effect rupture decreases with decreasing temperature.

2. The tensile strength increases when the temperature is decreased.

3. The ultimate elongation passes through a maximum with decreasing temperature.

4. The low tensile strength observed at -320°F and the sharp drop in elongation from -100 to -320°F indicates a phase change in the basic material may have occurred between these temperatures.

REFERENCES

1. Saunders, J. H. and Frisch, K. C., Polyurethanes, Chemistry and Technology, Part I, Chemistry, Interscience Publishers, New York, 1962, p. 268.

2. Flory, P. J., Principles Of Polymer Chemistry, Cornell University Press, Ithaca, New York, 1953, Chapters 11 and 13.

3. TR-01646 pp. 15-16; Test Report Case No. 54808

SESSION II - PAPER 3.

STATUS OF ADHESIVE RESEARCH (MSFC)

3:00 - 3:40 P. M.

TUESDAY - MARCH 15, 1966

STATUS OF ADHESIVES RESEARCH AT MARSHALL SPACE FLIGHT CENTER

by

Dr. W. Hill

Materials Division, Propulsion and Vehicle Engineering Laboratory
Marshall Space Flight Center, Huntsville, Alabama

Delivered March 15, 1966, at the Structural Bonding Symposium at NASA, Marshall Space Flight Center, Huntsville, Alabama.

ABSTRACT

Poly-2-oxazolidones, polyisocyanurates, polyurethanes, bisphenol epoxies, and epoxy esters were examined in a synthetic program for developing a cryogenic adhesive with an expanded temperature range of utility.

Accelerated aging of polyurethane bonded lapshear specimens in tests conducted to date have indicated bond deterioration even for primed specimens, although primed bondlines maintained higher strengths throughout. A primer screening study revealed certain silane derivatives to be good primers for aluminum adherends bonded with polyurethane adhesive systems.

A relationship between time of cure at a given temperature and Bell peel strengths at two test temperatures was used to derive an expression for time required for minimal cure of a polyurethane adhesive system at a given temperature.

Preparation of fluorinated monomers for fluorinated polymer systems was recently initiated.

STATUS OF ADHESIVES RESEARCH AT MARSHALL SPACE FLIGHT CENTER

INTRODUCTION

Adhesive research and development programs in the laboratories of MSFC and under contractural assignment include projects for development of new materials as well as projects for more effective process design and utilization of known materials. Individual programs range from those designed for immediate alleviation of a specific problem to those planned to yield more fundamental advances in the state-of-the-art.

Some of the adhesive applications with which we are most familiar are the fabrication and repair of the common bulkhead, fabrication of instrument panels, fabrication and attachment of cryogenic insulation, bonding of clips and straps to the face of the vehicle, attachment of fuel exclusion risers to the interior of the fuel tanks, and attachment of reinforcing composites to the skirt sections of the vehicle. In some of these applications, the bond line is potentially subjected to a temperature gradient of -423°F to +200°F, and this has been a basic problem.

EXPERIMENTAL FORMULATIONS FOR BROAD TEMPERATURE RANGE

In connection with a proposition for an adhesive system with a wider temperature range of utility, the Central Research Laboratory of Borden Chemical Company, under contract NAS8-11518, conducted an exploratory program which included synthesis and evaluation of the following classes of resins: poly-2-oxazolidones, polyisocyanurates, polyurethanes, bisphenol epoxies, and epoxy esters.

a. *Poly-2-oxazolidones* are copolymers of epoxy resins with polyurethanes. These adhesive systems demonstrated a wider and more useful temperature range than the epoxy or the polyurethane systems alone. A formulation of the diglycidyl ether of bisphenol A with Adiprene L-315 and diaminodiphenylmethane gave lapshear tensile values on aluminum of 3640 psi, 3750 psi, and 2580 psi at -320°F, 73°F, and 250°F, respectively.

b. *Polyisocyanurates* result from catalytic polytrimerization of isocyanate terminated polyurethane prepolymers. Lapshear tensile strengths above 1000 psi at 400°F were obtained; however, the cure temperature of 160°C was impractically high. Best results were obtained with a formulation of diphenylmethane diisocyanate with a polyurethane prepolymer catalyzed by calcium naphthenate. The following lapshear data are typical of this system: 1630 psi, 1530 psi, 1410 psi and 1410 psi at -320°F, 73°F, 250°F, and 400°F, respectively.

c. *Polyurethanes* which exhibited useful strengths over the temperature range of -423°F to +250°F included MOCA-cured Adiprene L-315 and several custom made isocyanate terminated prepolymers, also cured with MOCA. Isocyanate content was invariably high, however, and the pot lives very short. Typical Adiprene L-315/MOCA values for lapshear tensile were: 6500 psi, 4085 psi, and 1315 psi at -320°F, 73°F, and 250°F, respectively.

d. *Bisphenol epoxies* synthesized under this program gave results comparable with Epon 828. In general, it was found that the tensile shear strengths at -320°F increased with the epoxy content of the starting resin. The characteristic brittleness of epoxy resins at cryogenic temperatures resulted in low peel strengths. Diglycidyl ether of bisphenol A, when cured with methylenedianiline for 2 hours at 165°C, gave the following lapshear tensile values: 4340 psi, 3390 psi, 2790 psi at -320°F, 73°F, and 250°F, respectively.

e. Epoxy esters (polyglycidyl esters of polycarboxylic acids) were subjects of an extensive synthetic program designed to prepare pure materials. The best overall performance was observed with triglycidyl trimesoate cured with methylenedianiline: 2920 psi, 2700 psi, 2700 psi, 2410 psi, and 1700 psi at -423°F, -320°F, 73°F, 250°F, and 400°F, respectively. T-peel values were on the order of 1-2 lb/in; anything done to flexibilize these systems to meet T-peel requirements severely impaired high temperature tensile shear strengths.

Of these five polymer classes, the first two - polyoxazolidones and polyisocyanurates - appear from this data to be the most derserving of further investigation.

ACCELERATED AGING OF POLYURETHANE SYSTEMS

Accelerated aging of polyurethane bonded lapshear specimens has been accomplished under environmental conditions more severe than mornal ambient surroundings. Table I shows data obtained when lapshear specimens bonded with Narmco 7343/7139 adhesive systems were aged under conditions of 98-100°F at 98-100 percent humidity. Three series of adherends were used: (a) primed with 3M Company's XC-3901, a silane derivative; (b) primed with Goodyear's G-207; (c) unprimed. The specimens were pulled at room temperature, +180°F, and -300°F. Schematic representation of the data in Figure 1 emphasizes the following points:

1. Over a period of 35 days, drastic, progressive bond deterioration is evident in all three series in tests run at room temperature and +180°F. Tests run at -300°F do not strongly reflect the effects of aging.

LAPSHEAR STRENGTHS (PSI) AFTER AGING SPECIFIED

Primer	Test Temperature	Zero Time	1 Day	3 Days	7 Days	14 Days	21 Days	28 Days	35 Days
XC-3901	R.T.	1936	1902	1770	1612	1502	1352	1184	1100
	+180°F	1308	1622	1388	1206	1646	1034	1240	932
	-300°F	6740	6642	6802	7350	6658	6890	6616	6230
G-207	R.T.	2194	2020	1274	1230	914	898	1342	870
	+180°F	664	694	614	528	410	434	452	352
	-300°F	5740	5782	5816	5530	5210	5870	5562	5736
Unprimed	R.T.	1466	1064	900	528	414	634	488	670
	+180°F	486	368	398	302	316	264	237	212
	-300°F	-	-	-	-	-	-	-	-

TABLE I. Lapshear Specimens (Narmco 7343/7139) Aged at 98°-100°F, 98-100% Humidity

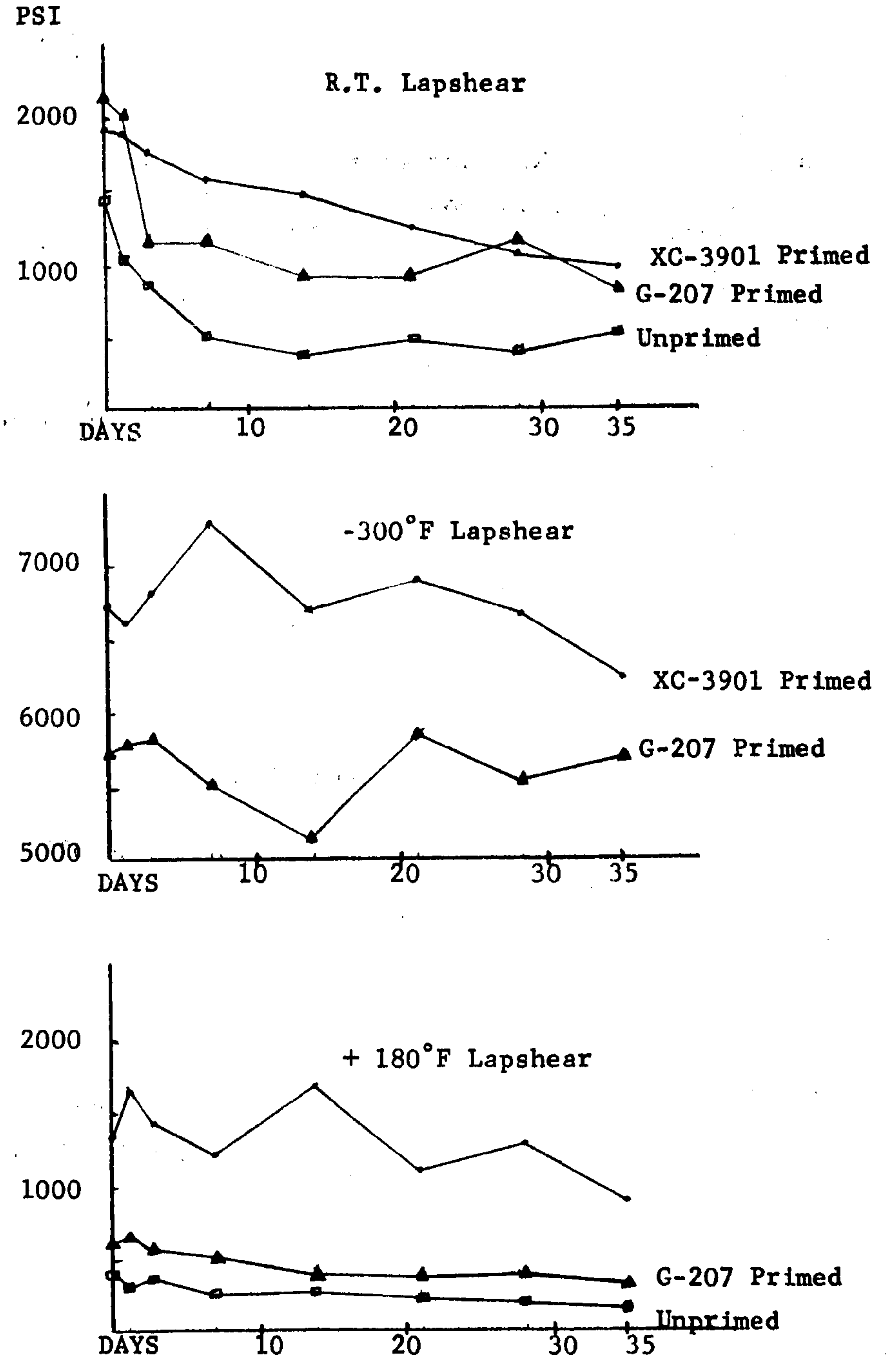


FIG. 1. LAPSHEAR SPECIMENS (NARMCO 7343/7139) AGED AT 100°F; 100% HUMIDITY

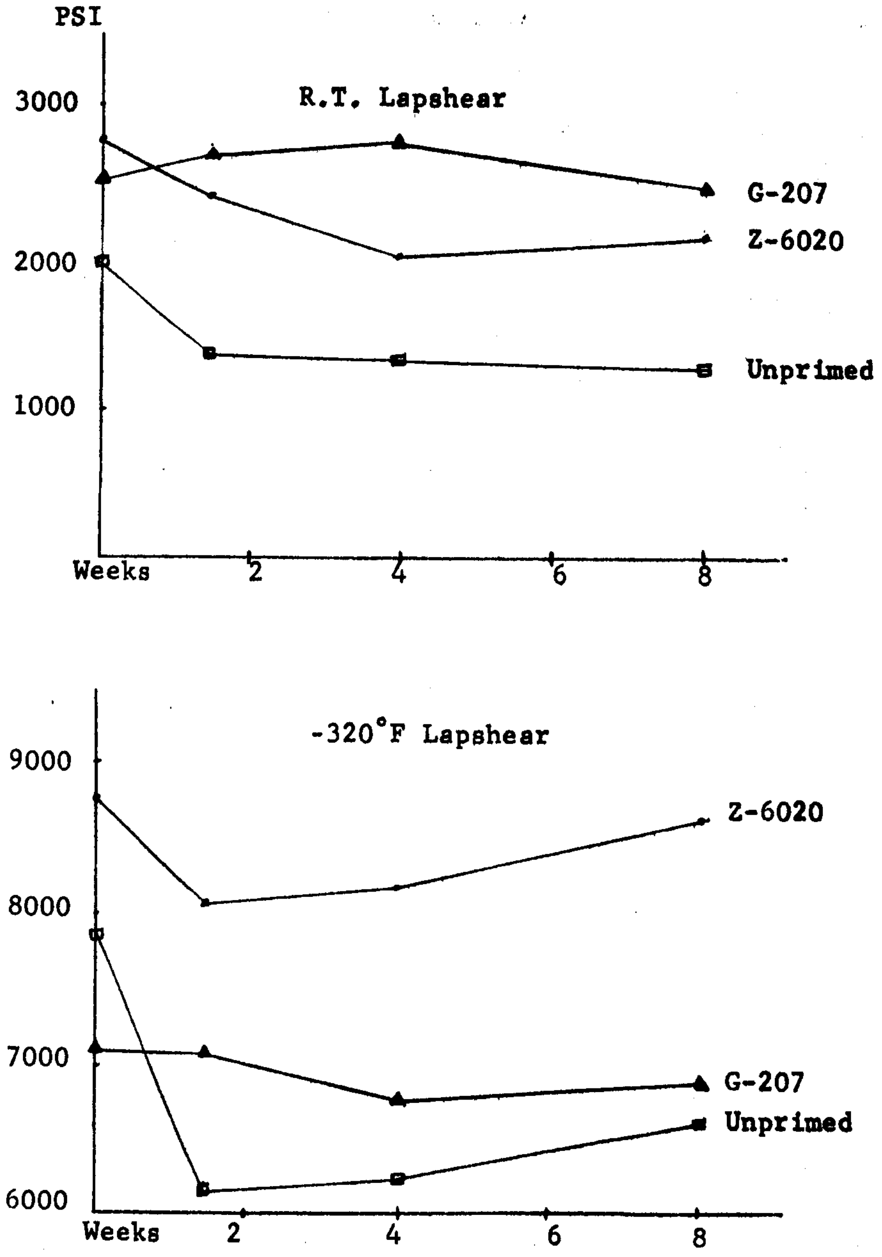


FIG. 2. LAPSHEAR SPECIMENS (NARMCO 7343/7139) AGED AT 70°F; 100% HUMIDITY

2. Initial strengths and, therefore, final strengths for primed specimens were much better than for the unprimed. The silane derivative, XC-3901, appears somewhat superior to G-207.

A similar experiment in accelerated aging was run by the Narmco Division of Whittaker Corporation under contract NAS8-11958. Conditions here were somewhat milder, 70°F at 100 percent humidity, and aging continued over a period of eight weeks with specimens being pulled at room temperature and at -320°F. Results are summarized in Figure 2 and indicate that:

1. The lower temperature (70°F versus 100°F) in this experiment gave less bond deterioration.

2. Primed specimens exhibited better strengths than unprimed specimens, with the silane-type primer (Dow Corning's Z-6020 in this case) having an edge over G-207.

PRIMER STUDIES

Other work under the program, NAS8-11958, "Optimization of the Performance of Polyurethane Adhesive System over the Temperature Range of -423°F to +200°F," has yielded additional interesting data on the beneficial effect of primers as used with the Narmco 7343/7139 systems. Although the room temperature tests (Fig. 3) for unprimed specimens showed good values (avg. 2121 psi), the strength of specimens primed with Dow-Corning Z-6020 primer was considerably improved, yielding an average of 3580 psi, and more important, a higher minimum value (2000 psi versus 1200 psi) for the series. Figure 4 illustrates improvement observed with the same primer for specimens pulled at -320°F, while Fig. 5 represents values from tests at 150°F. Similar improvements were observed at all temperatures with Bell specimens.

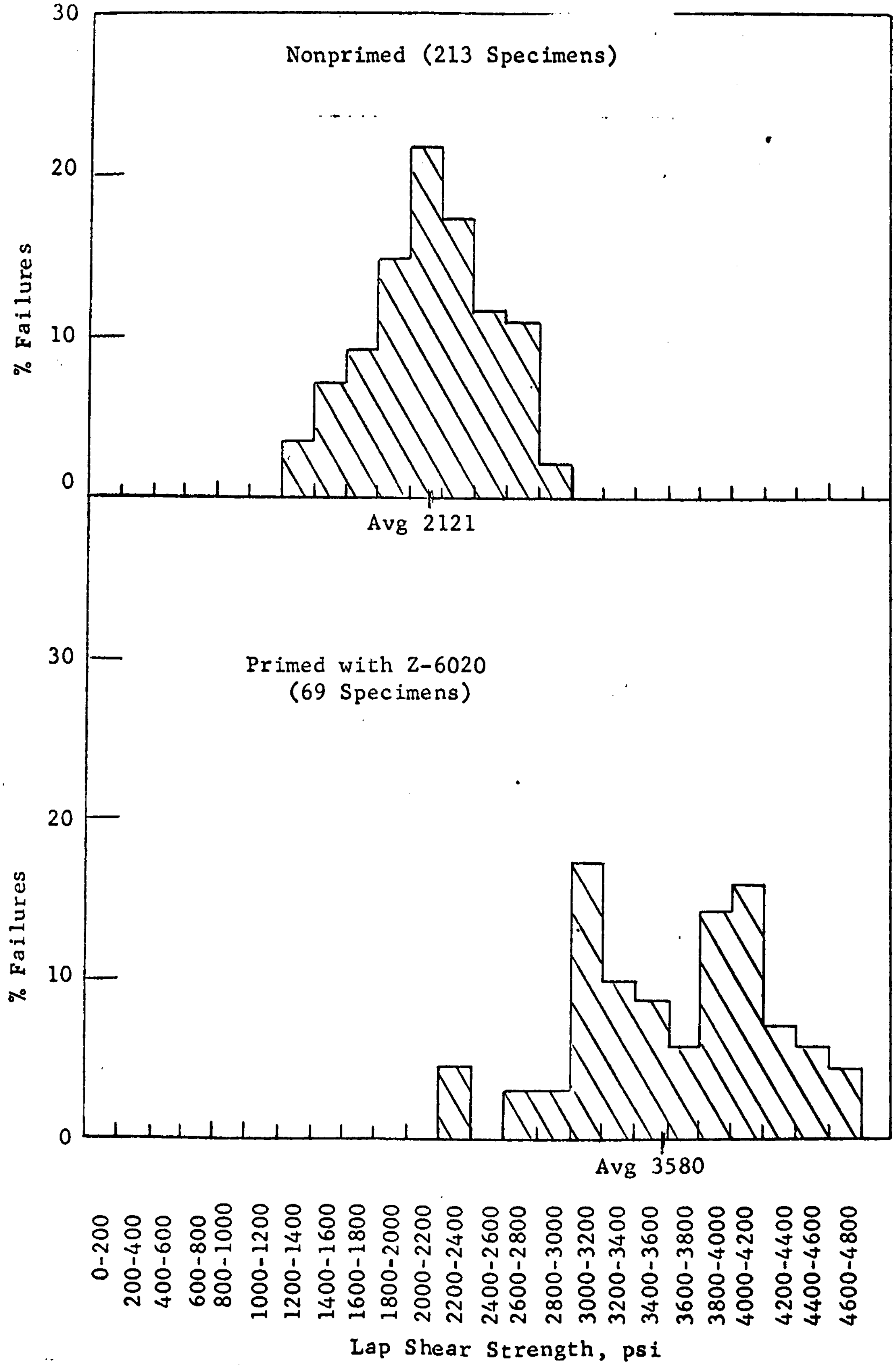


FIG. 3. DISTRIBUTION OF ROOM TEMPERATURE LAPSHEAR STRENGTHS (NARMCO 7343/7139)

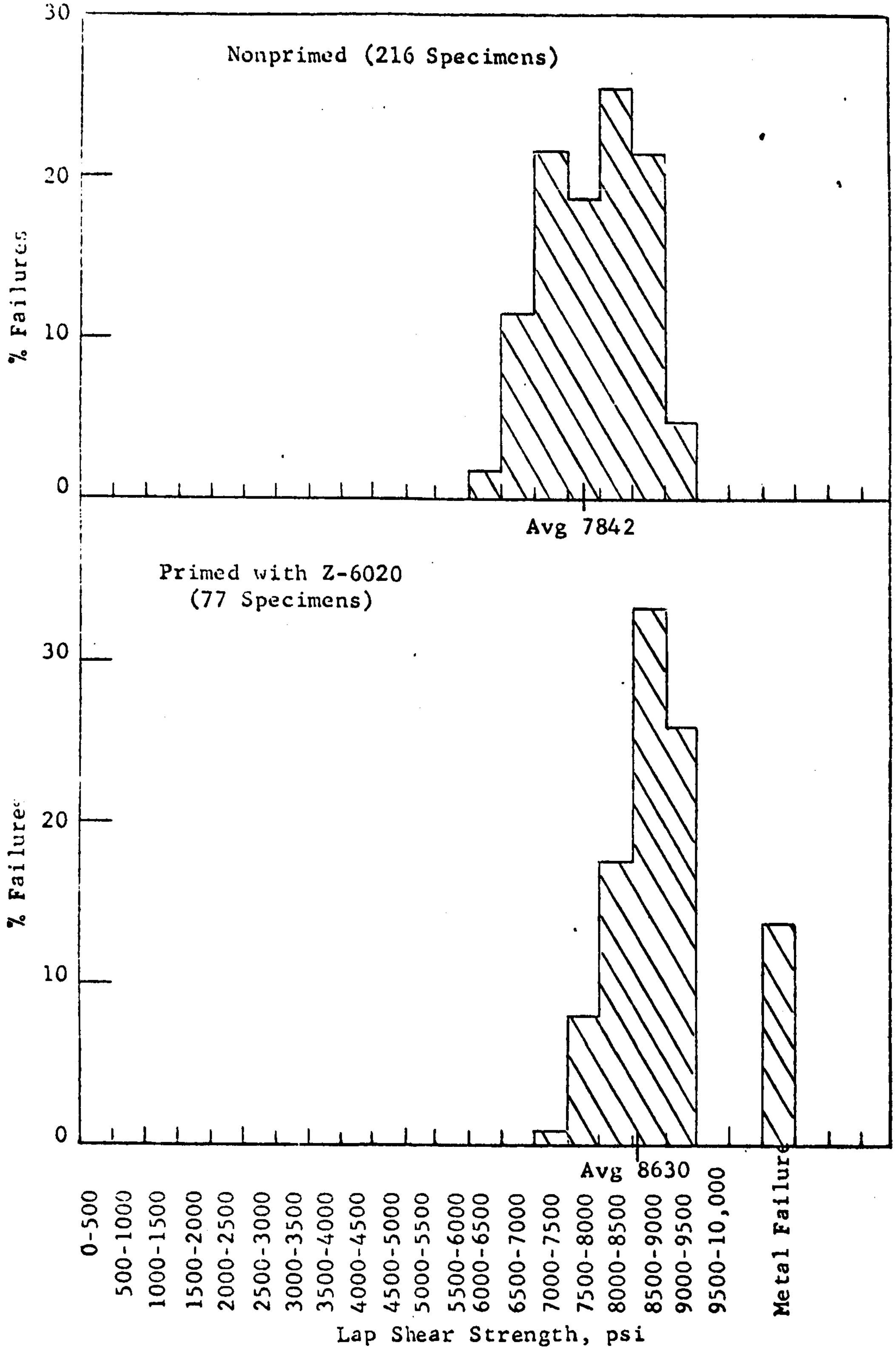


FIG. 4. DISTRIBUTION OF -300°F LAPSHEAR STRENGTHS (NARMCO 7343/7139)

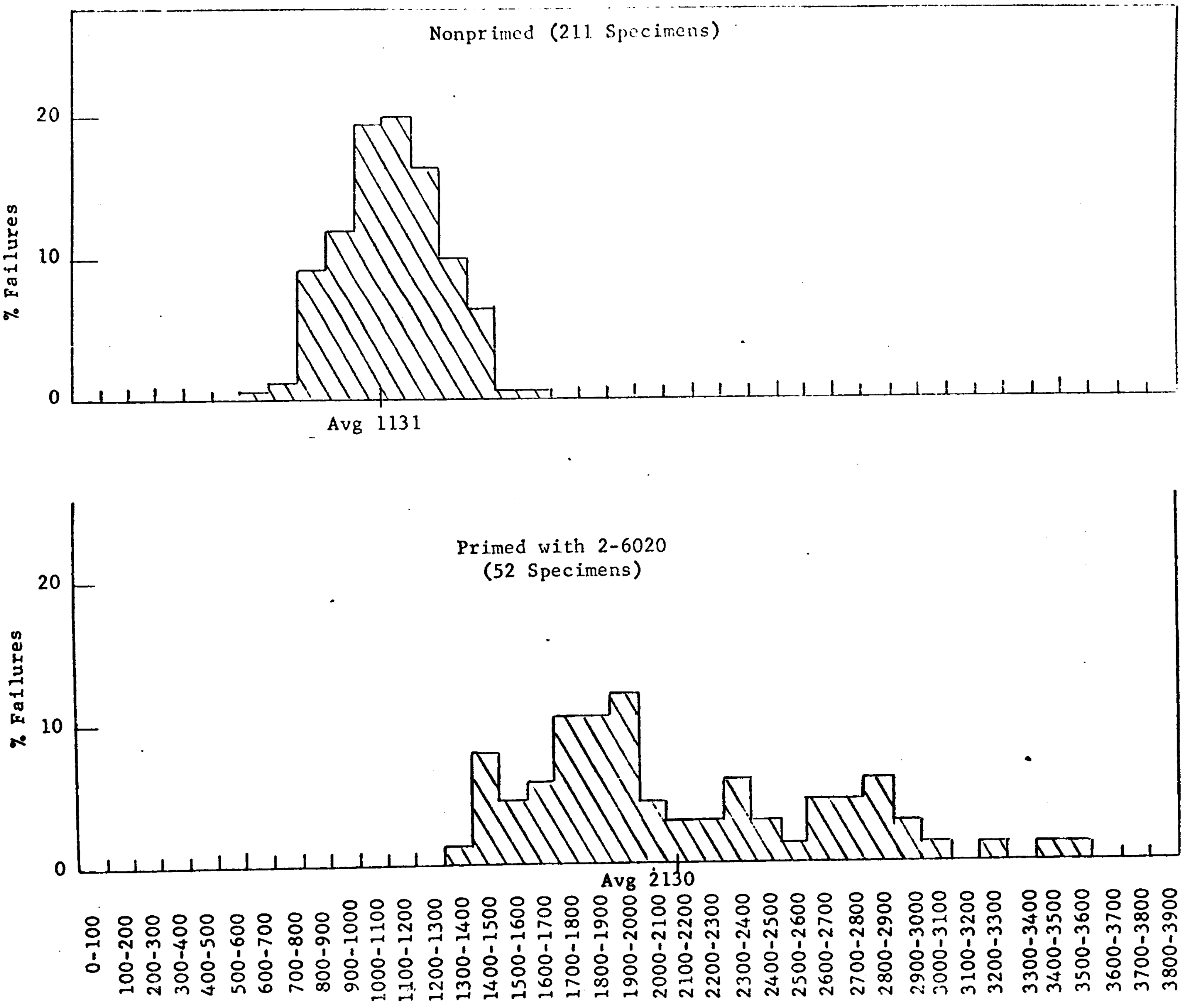


FIG. 5. DISTRIBUTION OF 150°F LAPSHEAR STRENGTHS (Narmco 7343/7139)

CURE CYCLE FOR POLYURETHANES

On the same program, NAS8-11958, it was reported that when Bell peel specimens fabricated from the Narmco 7343/7139 system were pulled at room temperature and at -320°F, the curves for peel strength versus time of cure coudd be summarized as shown in Fig. 6.

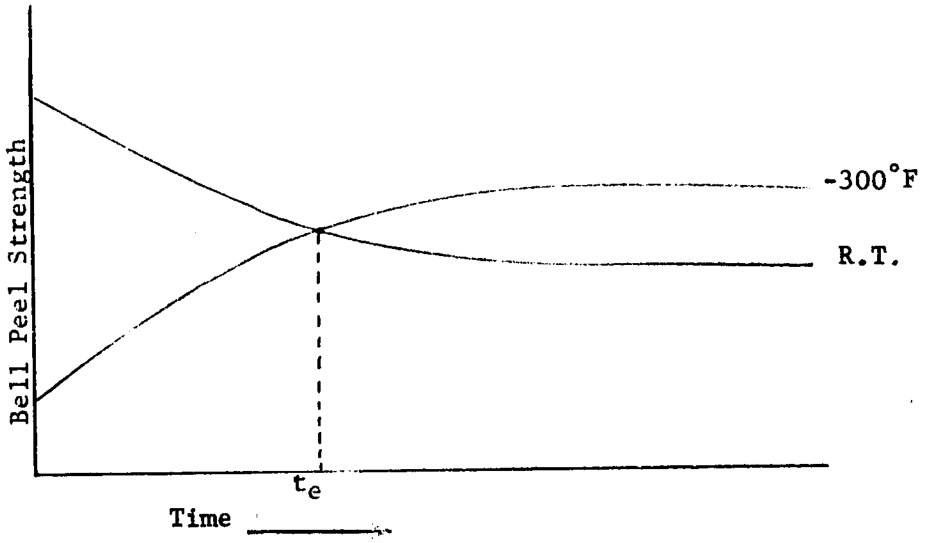


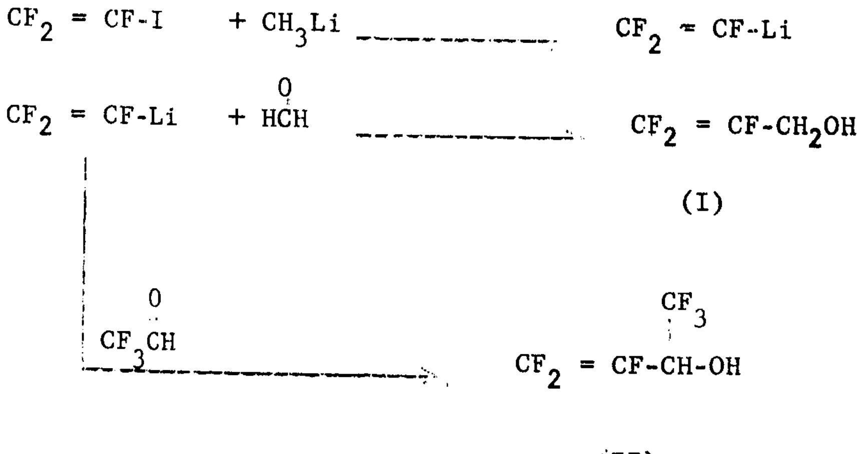
FIG. 6. BELL PEEL STRENGTH VERSUS CURE TIME (7343/7139)

The intersect, t_e, of the -320°F and R.T. curves can be considered a time of minimal cure at that particular temperature. Several cure temperatures were plotted in this manner and the shape of the curve was the same for each; only te was changed. Values observed were:
t_e = 270 hrs at R.T.; t_e = 9.5 hrs at 160°F; t_e = 3.3 hrs at 205°F; t_e = 1 hr at 250°F. An equation expressing the relationship is:
$\log t_e = 3.29 - 0.0137T$.

LOX COMPATIBLE ADHESIVES

NAS8-11068, conducted by the Whittaker Corporation, Narmco Division, is a polymer synthesis program designed to provide the basic materials for an adhesive formulation which will be insensitive to impact under liquid oxygen. Some very significant advances in synthesis of fluorinated monomers and polymers have been reported. Unfortanately, the contract is classified and cannot be discussed here.

A program for fluorinated polymer synthesis was initiated in the laboratories at MSFC only a few months ago At this time, two previously unknown alcohols have been prepared; 2,3,3-trifluoroallyl alcohol (I) and 2,3,3-trifluoro-1-trifluoromethylallyl alcohol (II).



$$CF_2 = CF\text{-}I + CH_3Li \longrightarrow CF_2 = CF\text{-}Li$$

$$CF_2 = CF\text{-}Li + \overset{O}{HCH} \longrightarrow CF_2 = CF\text{-}CH_2OH$$

(I)

$$CF_2 = CF\text{-}Li + CF_3\overset{O}{CH} \longrightarrow CF_2 = CF\text{-}\overset{CF_3}{CH}\text{-}OH$$

(II)

These materials are intermediates in routes to fluorinated epoxy monomers.

Some of these activities, as for example the one last described, probably will not meet with immediate success, but of the work summarized here enough progress has been made in each area to encourage continuation. Most of the problems in adhesive bonding we find do not yield one hundred percent to an intensive experimental and developmental approach, but solutions which are only marginal often become refined by experience until fully acceptable. Such marginal solutions could probably mature a great deal more rapidly if communication among adhesion practitioners in a given industry were more facile and unfettered.

SESSION II - PAPER 4.

THE FUNCTION OF THE PLASTICS TECHNICAL EVALUATION CENTER (PLASTEC)

3:40 - 4:10 P. M.

TUESDAY - MARCH 15, 1966

N66 34133

THE FUNCTION OF THE PLASTICS TECHNICAL EVALUATION CENTER (PLASTEC)

By

Harry E. Pebly, Jr., Director

ABSTRACT

PLASTEC is one of 22 specialized information analysis centers monitored by the Department of Defense. The center is located at Picatinny Arsenal, Dover, New Jersey. It provides a centralized source of information on properties and applications of plastics in defense, on project and contract activity and on personnel active in the field. Its output is two-fold: answers to inquiries and reports written by its staff on timely areas of materials, technology, processing and applications. Services are available to government agencies, their contractors and suppliers, libraries and universities. Information on plastics is also available by subscription to the Plastics Section of Engineering Index and through various services of the Commerce Department's Clearinghouse for Federal Scientific and Technical Information.

THE FUNCTION OF THE PLASTICS TECHNICAL EVALUATION CENTER (PLASTEC)

By

Harry E. Pebly Jr., Director

The purpose of this paper is to acquaint you with the function of the Plastics Technical Evaluation Center (acronym PLASTEC), to intersperse a few comments about the information problem and to mention briefly other sources of information in science and technology.

Today we have technical information sources of two basic types within the government. We have at the base the traditional library with its important role of providing documents and reference and bibliographic service. We have in recent years seen the development of a second type, the specialized subject information center, where a particular material, technology or application is the sole subject of consideration. The two types complement one another, each drawing upon the other for the specialties it has to offer. Whether it be small, informal and arranged on shelves, or large and mechanized, the specialized information center is one of the more effective solutions for providing the scientist and engineer with an orderly and timely flow from the large amount of information generated today in every branch of science and technology.

The Department of Defense has recognized 22 information analysis centers as authoritative sources in the field of their particular defense mission. These centers try to answer requests for specific information from anyone connected with the defense effort. Several of them publish reviews or state-of-the-art reports which are generally available for public sale. And they are usually well informed as to who is active in the field and what programs are underway. Through these centers the government attempts to provide for the most effective use of the results of its giant R&D programs.

PLASTEC

PLASTEC, established in 1960, is the information analysis center for plastics, the one place designated to collect, evaluate and disseminate technical information in the field. The Department of Defense assigned the Army the responsibility of operating PLASTEC and located it at Picatinny Arsenal in Dover, New Jersey. There PLASTEC is able to supplement its knowledge with the talents available in the large plastics laboratory of the arsenal. PLASTEC has concentrated in four areas: reinforced plastics; plastics in electrical and electronic applications; plastics in packaging and plastics in mechanical goods. The center aspires to coverage of the entire plastics and polymer field.

The remainder of the staff exists primarily to provide operational services for the specialists. These services are publications, library and administrative, a total of seven people and eleven total. The staff is supplemented by the consulting services of the Plastics & Packaging Laboratory, about 25 professionals, and the contract services of Franklin Institute, equal to two or three people. Franklin does the processing of all reports, particularly indexing and abstracting.

Output of PLASTEC

The output and the input reflect our end-item orientation. The original needs of the services were agreed to be in hardware rather than in research and we have bent our efforts accordingly. Our experience and records show a continuing demand for research information and we hope to enlarge our operation to cover applied research in plastics and polymers. Our services - the output - are available to the entire DOD community simply by asking for them. NASA and their contractors have received the same considerations and services as have DOD agencies.

The first service is providing answers to inquiries. A letter, a visit or a phone call will get us going. No red tape or costs are involved except for unusually large demands. A note should be made here on what we are not to do. We should not be asked to assume your responsibility in deciding on the final solution to your problem. We can and will provide carefully weighed information and our best opinion on the subject, from which you, the customer, can make the final decision. We try to enforce this particularly when a contractor comes to us for help.

Inquiries most often concern materials, properties or applications and we have set up our storage and retrieval system accordingly. Documents going into our library have these three items indexed as basic identification of the report contents. It has already been noted that our specialists were picked to cover particularly the areas of structural plastics applications, electrical and electronic uses, packaging and mechanical goods. We will, however, tackle any area in which the government has an interest. Our replies may be specific information or data, references to selected reports and literature, names of people or groups doing work in the field, active government contracts, or combinations of these. As a rule of thumb, we try to limit our effort to an inquiry to two or three days of work. Projects requiring weeks of work require separate funding.

PLASTEC tries not only to provide you with information, but an evaluation of its worth. There should be with every answer the consideration of how much work has been done in the field, how much more is underway, who is doing it, for whom is it being done and why, the meaningfulness of the test methods and last but not least, corroboration of the data from other sources. In other words, there is supplied a judgment factor.

To provide this evaluation there must be specialists on the subject at the center. It is these specialists, with years of experience in the field and well-regarded by their colleagues throughout the country, who distinguish the specialized centers of today. Ideally they should be close at hand to a laboratory in order to keep up with the field and maintain touch with the new problems and attempted solutions.

The second and more lasting output are our publications. They are in two series: Reports and Notes. The basic difference lies in the degree of preparation. Many of the reports are state-of-the-art, that is, the best available information on a subject. Some of the subjects thus covered are filament winding, structural plastics in aircraft, nondestructive testing, plastic tooling, properties of plastics at cryogenic temperatures and fluidized bed coatings. In publication are reports on weathering of reinforced plastics, compatibility of plastics and liquid propellants, and glass reinforced thermoplastics. The report series also includes data compilations (electrical properties), guides and directories. The Note series contains information on a particular phase of a subject and does not include narrative evaluation. A Note could be the forerunner of a state-of-the-art report if the larger need is justified. Annotated bibliographies are a large part of the Note series. The brochure includes the published reports.

Both in the preparation of answers to questions and of reports, the resources of the Picatinny Plastics and Packaging Laboratory are a valuable adjunct. The Adhesives Research Section is part of this Laboratory. Professionals in all aspects of polymer science and technology are consulted by the PLASTEC specialists, greatly multiplying the available talents that can be brought to bear on a problem. New materials, equipment and processing techniques can be observed and evaluated in this laboratory.

There is a third area of output, less formally defined, but generally categorized as "knowing what's going on". The inflow of over 1000 visitors and PLASTEC visits to 600 outside laboratories, private companies and technical meetings has contributed in great measure to knowing what, where and why. While this is not always information in the strictest technical sense, it has improved liaison between industry and the military to the highest point it has enjoyed.

Specific examples of how we can be helpful are these: providing background reports and reviews when a new project is contemplated or funded; providing names of persons or laboratories in the other services who are active in the same field or who have expressed interest in the field; providing contacts in the technical and trade societies in the field and a summary of relevant programs they may have underway; and providing an awareness of general trends affecting the particular project.

PLASTEC Reports of Wide General Interest

The best compilation and index of government specifications on plastics is a report of PLASTEC, Note 6. Here over 700 military specs are listed, with subdivision by materials and by applications. The extent of use if noted (that is, whether all three services or a single service) and the preparing agency is stated. Information on obtaining specifications is given. PLASTEC gets more calls on specs than on any other subject. In fact, that's what led to the compilation. A second compilation of specifications covers the field of reinforced plastics, designed PLASTEC Note 3.

Who in the government is knowledgeable and active in specific materials and applications? The best guide is another PLASTEC publication, Report 5A, "Directory in Plastics - Knowledgeable Government Personnel." Here are compiled 700 names in 94 laboratories and offices not only in DOD but NASA and several other agencies. The material is indexed by subjects and by people and is arranged by laboratories of the agencies. The first edition came out in early 1961. The present one was published in 1963. There will be a new edition in 1966.

PLASTEC's fourth publication of wide general interest is its annual bibliography of conference papers. The coverage is papers on plastics presented at technical conferences throughout the year, ending with the national SPI Reinforced Plastics and SPE meetings. The fifth annual volume (PLASTEC Report 21) is titled "Subject Index, Bibliography and Code Description of Technical Conference Papers on Plastics: 2 March 1964 - 5 March 1965."

Volume six will be published in early summer of 1966. Earlier volumes cover 1960, 1961, 1962 and 1963. They may be purchased for $3.00 each from the Clearinghouse, Department of Commerce, Springfield, Virginia 22151. We do not try to keep in stock reports more than two or three years old. Such may be obtained from DDC or the Clearinghouse.

The present volume lists 550 papers presented at 23 conferences. The publication compiles listings by materials, type of data, military application, and author. In the analysis on trends it is observed that the number of families of plastics has almost doubled since the 1960 volume, indicating the growth of the technology.

Keeping track of the papers given at these conferences enables the reader to gain a considerable insight into work being accomplished in government laboratories or by contractors. These people frequently use open meetings to present and discuss their work. They are, in fact, encouraged to do so. One of the important benefits is the dissemination of information from government sponsored work to the general public.

Distribution of Reports

You may receive PLASTEC reports automatically by returning to us the Field-of-Interest-Register (FOIR) in the brochure. Please supply all the information requested. Distribution is automatic, according to the fields of interest indicated. The FOIR may be duplicated if there is more than one group desiring reports. The library in each company or agency should be included.

Input

PLASTEC has built a library of about 8000 documents, growing at a rate of 2000 a year. We have concentrated almost entirely on reports from government agencies, contractors and technical conference papers. We believe these are the first reporting of most new engineering data and also among the most difficult to acquire. For articles in the open literature we use commercial abstract services, one of which will be described later.

Each document accepted for the collection (half of those received are discarded) is indexed and abstracted. Several times each year a complete new index is printed, comprised of a source index and a subject term index. Care is taken to provide in each abstract the materials studied, their properties and their application, and to index these terms. Pre-coordination is used extensively here to combine materials and properties, materials and tests and to a lesser extent materials and applications.

The PLASTEC collection of documents is intended for the use of its specialists and the documents themselves are the basic tools. To find the documents that one wants in the collection, the Index is consulted. The document accession number or numbers is listed for each index term. The user may then go directly to the shelf for the document or he may go to a card file of abstracts. Visitors to the Center are welcome to study the documents and consult with the specialists, but we have no lending service for reports.

A computer has been used to alphabetize the index terms and provide a rapid print-out, a copy of which is given to each specialist. Each new printing has been cumulative (but bi-annual volumes may become necessary) and the computer is utilized in making additions and corrections in the most efficient manner. The index has been prepared by Franklin Institute.

Acquisition

Effective communication, recognized as one of the more difficult problems in the conduct of any business, is near the very heart of the PLASTEC operation. We not only have to tell you what we want and what you can get from us, but we have to keep telling you. People move away, people forget, policies change, new problems come along, inertia exerts its force: all obliterate the established smooth relationships. This we know from experience. Agencies that have been faithful in sending us reports suddenly are not heard from. Rarely is the omission or interruption deliberate - it is one of the reasons given above. The biggest thing each of you can do for us is to see that we get copies of documents originating in or contracted by your agency that deal with the subject of plastics. The selective distribution mentioned above is utilized by the Army Electronics Command and is recommended for study. Key terms or phrases describing areas of interest are established for those whom the Electronics Command wishes to distribute to. When the contract people see these key terms in a new contract, they automatically add the proper names to the distribution list.

Placing PLASTEC on distribution lists for contractors progress reports might seem like a request almost automatic in nature. But unless you have set up a selective distribution procedure, judgment is required in deciding which contracts are of interest and the automatic possibilities disappear. Further, someone must remember to exercise this judgment. Our best bet appears to be in the development of contacts in each agency and enlist their - and your - cooperation in seeing to it that we are placed on distribution lists. Trying to get on a distribution list after the contract has started is not usually possible. The Office of the Assistant Secretary of Defense, in the original establishment of PLASTEC, provided the authorization for this report distribution.

After six years of operation, we continue to uncover new sources of information. With the tremendous amount of activity in this country and abroad in research and development and the flood of documents resulting, a full-time operation for the storage and retrieval of information in any given field is required. Laboratory and engineering personnel simply do not have the time and resources to do the job. PLASTEC performs this information operation for the Defense Department in plastics.

PLASTEC was recently assigned the project of preparing a completely new edition of MIL-HDBK-17, Plastics for Flight Vehicles; Part 1 - Reinforced Plastics. We are hopeful that through this project we can put together the best collection of data on current and new resins and reinforcements and design information ever assembled. Either as part of the handbook or as separate volumes we plan coverage of repair and joining procedures, nondestructive testing, quality control and filament winding. Representatives from the Marshall Space Flight Center and Lewis Research Center sit with the advisory group for this project. Through the Industry Advisory Group we hope to receive useful data for the handbook. The project has added significance in that the handbook is specified as a contractual document by several branches of the government.

Other Information Sources

Other DOD information centers for materials are the Defense Metals Information Center at Battelle Memorial Institute, Columbus, Ohio and the Ceramics and Graphite Information Branch, Materials Laboratory, WPAFB, Ohio. Property-oriented centers are the Electronic Properties Center at Hughes Aircraft Company, Culver City, California; the Mechanical Properties Data Center at Belfour Engineering Company, Traverse City, Michigan and the Thermophysical Properties Research Center at Purdue University. Others are in the fields of chemical propulsion, infrared, radiation, shock and vibration, nondestructive testing, counterinsurgency, oceanography and four tied in with specific missions.

Information on Information Centers

The National Referral Center (NRC) for Science and Technology was organized 2 1/2 years ago as an "information center on information centers". A part of the Library of Congress, the center has listed 6000 sources of information in science and technology. NRC does not have data itself, but refers the inquirer to the most likely source of information.

Engineering Index Offers Plastics Abstracts

A greatly expanded abstract service in plastics is now available to the public from Engineering Index, under the technical guidance of the Society of Plastics Engineers (SPE). Engineering Index is a non-profit organization affiliated with the Engineers Joint Council, providing an abstract and index service for the world's engineering literature for many years. Their address is 345 E. 47th Street, New York, N. Y., 10017.

The new program, now in its second year, is a pilot-mechanized effort in two selected areas: plastics and the electrical-electronic area. The former is the result of a drive by the SPE to provide a comprehensive coverage of the world-wide plastics literature; the latter is motivated by a similar interest in the Institute of Electrical and Electronic Engineers.

The plastics services will include a monthly abstract bulletin with index and semi-annual cumulated indexes, and a central mechanized document retrieval service based on a deep index, which can also be the basis for selective dissemination of information. The subject index includes adhesion, adhesives and adhesive bonding. It is estimated that the plastics field will involve 10,000 articles per year and the electrical-electronics field 20,000 articles per year.

Each selection in the pilot study will comprise a list of categories, a list of abstracts arranged by categories, a subject index and an author index. Subscription rates for the Plastics Section range from $25 per year for SPE members to $100 for corporations and industrial libraries.

DEPARTMENT OF COMMERCE PROGRAMS

Clearinghouse for Federal Scientific and Technical Information

In February 1964, the White House announced that the Federal Council on Science and Technology had recommended that the Department of Commerce expand its "Clearinghouse" function, building upon the existing information services of the Office of Technical Services. Subsequently, the Clearinghouse for Federal Scientific and Technical Information was established.

The Clearinghouse is a national center for the dissemination of Government-generated information in the physical sciences and engineering, comparable to similar centers in agricultural and health research.

The Clearinghouse is a system for supplying the industrial and technical community with unclassified information about Government-generated science and technology in defense, space, atomic energy, and other national programs. It thus makes readily available, at low cost, research information which may aid in the development of a new product, solve a processing problem, or increase productivity through technical improvement.

The main services of the Clearinghouse are:

1. Research Reports. These consist of:

   reports (more than 50,000 a year) based on federally sponsored R&D and translations (about 25,000 a year) of foreign technical material.

   After the documents are cataloged and indexed, they are put on sale in two forms, printed pages or microfiche - a 4" x 6" unitized sheet form of microphotography covering up to 70 pages.

   New documents are announced semi-monthly in:

U. S. Government Research Reports - Department of Defense and other agencies except AEC and NASA (which announce their reports separately as indicated below). Sold by the Superintendent of Documents, U. S. Government Printing Office, Washington, D. C., 20402, $15 a year ($18.75 foreign).

Nuclear Science Abstracts - All new Atomic Energy Commission research reports and nuclear science literature world-wide. Sold by Superintendent of Documents, $30 a year ($37 foreign).

Scientific and Technical Aerospace Reports - All new National Aeronautics and Space Administration and other agency research reports in aerospace fields. Sold by Superintendent of Documents, $25 a year ($31 foreign).

Technical Translations - Translations of significant Slavic and Oriental research available from the Clearinghouse and other sources. Sold by Superintendent of Documents, $12 a year ($16 foreign).

The Clearinghouse also offers a free "Fast Announcement Service" to announce by direct mail reports of special industrial significance. To get this service, write to the Clearinghouse, (Attn: Customer Relations Branch), U. S. Department of Commerce, Springfield, Virginia 22151.

Selected documents are also announced through the technical, trade, and business press.

2. Literature-searching Service. This service has two parts:

   Bibliographies in selected content areas are prepared and distributed free.

   Special literature searches are made to order. The searched literature includes items in both the Clearinghouse collection and other Government agencies. The hourly fee for this service is $11.

3. Government-Wide Index. Beginning in 1966, the Clearinghouse will issue a monthly consolidated index to the Government-sponsored technical literature.

The Index will contain subject, author, source, and report number - the standard points of access to the total report literature. Such an Index will permit scientists, engineers, and research managers to scan one publication in reviewing report literature in their fields of interest.

4. Referral Services. The Clearinghouse is setting up a master file of sources of information in the physical sciences and engineering. These sources include both Government-sponsored centers and private industry. Inquirers are referred to the sources most likely to have the information needed on a given subject. The Clearinghouse cooperates with the National Referral Center (Library of Congress) in providing the service.

5. Research in Progress. The Clearinghouse will be the single point of contact for information on current Government-supported unclassified research and development work in progress. The aim is to help the industrial and technical community avoid duplication of technical work by announcing who is doing what in the unclassified area of Government research. The Science Information Exchange will cooperate in this effort. The SIE and other agencies will notify the Clearinghouse of new research projects. This information will then be cataloged and made available. The service will offer listings by project, title, performing organization, principal investigators, term of contract or grant, and descriptive terms for the new research reported. The file created through input of current research will also be used in general Clearinghouse reference services to answer inquiries in specific areas.

A Warning

You can be swallowed alive by the Information Monster if you don't look out. The number of papers and reports is steadily increasing, and perhaps more importantly, the availability and distribution of these documents is increasing. The government, the professional societies and the people in the information business all have plans to pile up on your desk reports in areas where you've shown an interest. The trade term for this is selective dissemination of information (SDI). You'll know more about your field than you ever did before - if only you can find ways to swallow the information before the monster swallows you.

Supervisors and their management must recognize the need for reviewing the literature in their budgets and planning. A study of the previous work and current work in a given project area is as essential as the fresh work that may be carried out. Specialized information centers, selective dissemination, and networks of information facilities are eliminating the need for the scientist and engineer to scan the entire output of publications, but only the individual scientist and engineer can read and absorb those reports selected as essential to his work. And management must provide the incentive and the climate for the individual to do this.

SESSION III. - PAPER 1.

APPLICATION OF AN ADHESIVELY BONDED CRYOGENIC INSULATION SYSTEM

8:30 - 9:10 A. M.

WEDNESDAY - MARCH 16, 1966

N66 34134

APPLICATION OF AN ADHESIVELY BONDED CRYOGENIC INSULATION SYSTEM

by

J. B. Rittenhouse
Staff Scientist, Materials Sciences Laboratory
Lockheed Palo Alto Research Laboratory

ABSTRACT

A materials and processing technique has been developed for the successful adhesive bonding of a cryogenic insulation system to the 2219T 87 aluminum alloy welded liquid hydrogen vessel for the Saturn V Apollo system. The materials and processing variables in the development of this cryogenic insulation system have been worked out for the adhesive bonding, the insulation materials system, the structural support system, through design analysis, laboratory testing of materials and fabrication and testing of a subscale cryogenic tank. The testing program will proceed to the fabrication and test of an insulated 82.6-inch diameter (half-scale) spherical liquid hydrogen vessel.

The design analysis trade-offs in the selection of the double aluminized Mylar-Dexiglass spacer system, with button retainers mounted over a helium purged fiberglass adhesively bonded substrate as opposed to the crinkled aluminized Mylar shingle system will be discussed. Installed system weight, ascent and space mission boil-off characteristics were developed analytically for these systems. Laboratory cryostat data were used to support and verify the analytical procedures. These heat transfer data as well as the fabrication variables were further developed in 26-inch diameter tank tests. As a result of the 26-inch diameter tank fabrication experiments the processing difficulties in controlling the density of the shingle system resulted in the selection of the button retainer system for installation on the half-scale tank. The button retainer multilayer insulation system thermal performance has been validated in the 26-inch diameter cryogenic tank tests.

For the test program it was necessary to adhesively bond platinum resistance thermometers and pressure transducer with their associated wiring to the test vessel interior. It was also necessary to bond thermocouples and transducer to the tank exterior and to several layers of the insulation materials system. The techniques for successfully accomplishing this task to assure accurate test results will also be discussed.

The fabrication and testing of the 82.6-inch diameter cryogenic vessel will proceed to simulated environmental conditions expected for an 8-day lunar mission. The vessel thermal performance with liquid hydrogen will be evaluated in the cryogenic flight and environmental simulation facility at the LMSC Santa Cruz test base for simulated ground hold, ascent and space environmental conditions.

Additional materials and processing techniques are required to validate the fabrication details as well as the thermal performance of the crinkled Mylar shingle insulation using a 60-inch diameter cryogenic vessel.

The quality assurance and optimized fabrication techniques used by LMSC to achieve a validated reliable adhesively bonded cryogenic vessel insulation system with its support structure will be presented.

APPLICATION OF AN ADHESIVELY BONDED CRYOGENIC INSULATION SYSTEM

by

John B. Rittenhouse*

For the Conference on

STRUCTURAL ADHESIVE BONDING

To be held at
Marshall Space Flight Center
Huntsville, Alabama
15 – 16 March 1966

*Staff Scientist
Materials Sciences Laboratory
Lockheed Palo Alto Research Laboratory
Lockheed Missiles & Space Company

ILLUSTRATIONS

APPLICATION OF AN ADHESIVELY BONDED CRYOGENIC INSULATION SYSTEM

by

John B. Rittenhouse

INTRODUCTION

The major objective of this program was to develop an optimum thermal protection system based upon the use of adhesively bonded multilayer insulation materials for the liquid hydrogen tank of the cryogenic spacecraft propulsion module.

The higher specific impulses available from chemical rocket systems employing cryogenic propellants make such propulsion systems extremely attractive for space missions. Since 1962, LMSC has been conducting feasibility, conceptual, and preliminary design studies of various cryogenic stages for use in Earth orbital, lunar, and interplanetary missions. Optimum performance for missions in excess of one day has required the use of multilayer insulations. However, the actual performance capability has been significantly dependent on the effectiveness of multilayer insulation in its applied condition (as opposed to laboratory determined values). In other words, the degradation of multilayer systems resulting from plumbing penetrations, deleterious effects of the ascent environments, methods of attachments, etc., could markedly reduce the payload capability of a given cryogenic vehicle. In all cases, the insulation system must be optimized to result in minimum payload penalty, assuming a vented system, and considering the combined effects of propellant loss from boiloff plus the inert weight resulting from insulation and additional tank weight required to accommodate the lost propellants. The need for multilayer insulation is obvious. Equally evident is the significance of insulation degradation on payload weight capability.

Propellant boiloff results from heat transfer to the cryogenic tanks through the structural supports, as well as through the insulation system. The influence of structural heat leaks is obviously more significant for the highly effective multilayer insulations.

Significant developments have been made toward demonstrating the practicability of a thermal protection system based upon the use of adhesively bonded multilayer insulation materials for a liquid hydrogen vessel. The helium purge-bag-encased aluminized Mylar-Dexiglas separator insulation system installed on a subscale liquid hydrogen tank was used to verify the results of the analytical and cryogenic laboratory studies. This insulation system remained undamaged after having been subjected to simulated vehicle ground hold purge, launch acceleration, vibration, and rapid ambient pressure drop. In a separate investigation the basic insulation was undamaged by an acoustic environment. The vacuum thermal conductivity performance for the system (less penetration heat leaks) was the same before and after having been subjected to these environments. The fiberglass reinforced plastic hat sections around the penetrations were adhesively bonded to the tank with Lefkoweld 109/LM52 epoxy.

OPTIMIZATION OF MULTILAYER INSULATION THICKNESS

The optimum number of radiation shields for three different techniques for meeting ground hold and ascent requirements were investigated on a parametric basis. The ground hold and ascent insulation systems were passive or sealed foam substrate, helium purged bag, and helium purged substrate.

The effective weight of multilayer insulation thickness for a range of effective thermal conductivity is defined as the sum of the insulation system weight and the prorated boiloff weight. The insulation weight includes not only the weight of the multilayer system but also that of the augmenting system. The factor used to prorate the boiloff is dependent upon the mission profile. The optimum thickness is that for which the effective weight is a minimum.

In the two systems using a substrate, the thickness of the sublayer is variable, but always such that the temperature of the coldest shield is 150° R during ground hold, to insure against cryopumping within the radiation shields. In the ground hold environment, the apparent conductivity of the multilayer is essentially that of the gas in between the layers. Therefore, the space flight evacuated condition performance has no effect on the ground hold performance. As a result, the better the insulation in space, the higher the boiloff rate during ground hold, since it requires a smaller thickness of insulation.

Approximately nine systems had been listed as possible candidates for use on the tank. Some of these systems, such as those employing taped parallel radiation shields and those requiring a vacuum bag for ground hold, were eliminated due to manufacturing and operational difficulties. Optimization analyses were made for the other systems. These systems were subsequently narrowed down to two candidates:

(1) Double aluminized Mylar-Dexiglas spacers, with button retainers mounted over a helium purged fiberglass substrate
(2) Crinkled aluminized Mylar shingles, applied over a helium purged fiberglass substrate

CRINKLED MYLAR SHINGLES

Other arrangements of radiation shields, spacers. attachment methods and ground hold and ascent insulation materials are possible. These systems, in addition to being analytically evaluated, were investigated experimentally as applied to subscale liquid hydrogen tanks. Preliminary manufacturing experience with the vessel indicates that the taped parallel layer crinkled Mylar system was the most difficult to manufacture. For a 25 radiation-shield system, ideally 25 separate patterns would be required or 8 separate patterns in a compromise design. It is anticipated that an advantage of the shingle system will be easier venting of trapped gases than can be expected for the similar multilayer button blankets.

Modified Button Layer System

Cryostat data were developed on a multilayer system comprised of insulation blankets held together with nylon monofilament lines which penetrate the insulation and are connected to small Teflon buttons at each boundary. The blankets were composed of 10 layers of double aluminized Mylar alternately spaced with a quartz spacer material (Dexiglas No. 239-9-1/2). Results were obtained for blankets using a 4-in. center-to-center button spacing. Structural screening tests conducted on centrifuge samples indicated an 8-in. center-to-center spacing would be adequate for structural support, and a cryostat test was made of such a blanket system. The test specimen consisted of four blankets, each blanket having ten layers of double aluminized Mylar and nine layers of quartz spacer. The blankets were individually applied to the cryostat with each blanket having a longitudinal butt joint held together with Teflon tabs between buttons at each edge of the blanket. The blankets were successively rotated 90 deg each, so as to stagger these butt joints circumferentially around the cryostat.

TESTING

Environmental testing of the insulation was performed as follows:

- Purge test – to determine whether the purge system would be adequate at low purge rates
- Vacuum/thermal tests – to evaluate the effectiveness of the insulation in a simulated space-thermal environment prior to the mechanical tests
- Acceleration – to simulate an ascent g loading
- Vibration – to simulate sinusoidal loading similar to launch conditions
- Ground hold thermal and ascent (rapid depressurization tests)
- Acoustic test

Purge Tests

Two tests were conducted in which air was purged from a prototype insulation system with helium. The concentration of air in the insulation as a function of time from purge start was obtained. The results show that approximately 1 hr was required to reach minimum air concentration (< 1% vol). In addition, a test was run in which the purge gas flow was stopped and the purge bag exit was sealed off after the minimum air concentration was achieved and the back diffusion rate determined from the air concentration after 1-1/2 hr.

Rapid Depressurization Test

The multilayer insulation is filled with either nitrogen or helium gas during the ground hold. This gas must be evacuated during ascent and achieve interstitial pressures below 10^{-5} Torr as quickly as possible, for maximum effectiveness of the insulation. Ideally, the interstitial pressure decay would be the same as the ambient pressure decay; however, the flow impedance imposed by the layers will retard the outgassing and penalize the system.

When the pressure is below approximately 5 Torr, the gas flow is by free molecular transport and the apparent conductivity varies linearly with pressure. Gas flow tests have been conducted to determine the effect of the insulation system design on the time required or the insulation to reach a simulated space vacuum during the vehicle ascent.

The materials used for the structural support and thermal protection system tested are listed below:

- A semi-monocoque conical support system of titanium is best suited to the vehicle and mission. This design was employed in the half-scale test vessel and integrated into the thermal protection system.
- The insulation system for the half-scale hydrogen tank and support system consisted of alternate layers of 1/4-mil Mylar, aluminized on both sides, with Dexiglas spacers and button retainers. This multilayer insulation was installed over a fiberglass mat sublayer which was purged with helium gas. The sublayer was separated from the multilayer insulation with an Aclar gas-tight purge bag. The sublayer was approximately 1/2-in. thick with 26 effective layers of Mylar-Dexiglas multilayer insulation and a Dacron netting overlay.

- The space between the cryogenic tank and the outer structural fairing was dry-nitrogen purged.
- The insulated fiberglass domes covering the upper access-cover with pressurization and venting penetrations and the lower fill and drain penetration were separately purged with helium to minimize the effects of possible minute leaks in these areas.

In the design of the adhesively bonded insulation system for the full-scale tank three blankets, each having ten reflective shields and nine spacers, were used. This design was based on subscale tank test data for a system having 4-in. button spacing, without correcting for the difference in boundary temperature expected in the space environment. In this design, one reflective shield is effectively lost because the bottom shield on one blanket is in direct contact with the top shield of the adjacent blanket. This was done so that the delicate fiberglass spacers were completely contained between the stronger shields, thus reducing the possibility of damage when the blankets were being installed on the tank. Thus, the 30 layers of aluminized Mylar are considered as 26 effective layers.

The nylon thread and button retainers which hold the insulation blankets together serve two important functions. First, they permit the individual layers to be assembled into blankets before being installed on the tank. Second, they provide the structural support needed under high load conditions and prevent relative movement of the individual shields and spacers. The use of a number of thin blankets instead of one thick one insures against a direct heat leak path to the tank at the points where the threads penetrate the layers. Relative movement between the blankets was prevented by the use of pieces of double-backed tape spaced over the surface of each blanket, thus forming a bond between adjacent blankets. The shear loads within the purged sublayer are taken by the Velcro fasteners that are placed at intervals over the tank surface and over the surface of the sublayer. In addition, these Velcro fasteners act as standoffs, forming inner and outer annuli for the helium purge gas, and should aid in the evacuation of the sublayer during ascent. The entire assembly was tied down at a number of points around the periphery with Dacron cord and fiberglass clamps that transmit the tensile loads to the continuous tank support system.

About three years ago, LMSC applied an expanded plastic insulation layer on the inside of a 9-ft liquid hydrogen tank. The 2219 Aluminum tank wall was etched with sodium dichromate and sulphuric acid. A polyethelene and wood "greenhouse" was constructed around the vessel to provide temperature and clean-room control. The inside wall of the tank was coated with a 0.00021-in. primer consisting of Lefkoweld 109/LM52 adhesive, diluted with acetone to a viscosity of approximately 100 centipoise. The insulation material was polyurethane moulded with a three-dimensional matrix of fiberglass. This material was formed into "tiles" about 1 ft^2, with a "ship-lap" bevel on all sides. These were fitted into the tank and individually shaped so that the final piece in each row had to be forced into place. When the keystone piece was in place, the row was self-supporting even before the adhesive was applied. The pieces were numbered in order and marked with their individual weight in grams and the square inches of contact surface. A callender roll adhesive applying device was used to assure a controllable and uniform application of adhesive. Based on the square inches of area, the exact weight of adhesive to assure the optimum bond thickness was calculated. The callender roll was adjusted until the required weight of adhesive was applied.

The adhesive was applied to the "tile" for each row, and the tiles were then fitted into position. The row was covered with a sheet of polyvinylchloride film that was in turn sealed to the tank wall with pliable chromate sealing tape to form a vacuum bag for curing (20 mm Hg vacuum), to apply a steady uniform pressure, and to provide a uniform bond thickness. A specimen tile was mounted upside down under a sheet of plexiglas and within the vacuum bag to permit visual inspection of the adhesive flow and coverage for quality control purposes. A second control specimen, using adhesive from the same batch, was affixed to the outside of the tank in the general area of the newly emplaced row.

The "greenhouse" temperature was raised to 110° F for the desired 4-hr curing. At 1-hr intervals, the exterior sample was removed from the tank and tested for adhesive hardness to provide information on the progress of the curing. After each row was bonded in place, the following row was coated with adhesive, interlocked into place, and covered with an extension of the vacuum bag. Internal and external control specimens were used with each row. Between curing cycles, the ambient room temperature was lowered to 45° F (±5°) to retard the setting of the adhesive during the application process. After cure, each row was checked for gap space between tiles. All gaps large enough to be visable were coated with Lefkoweld resin reinforced and filled with chopped glass fibers.

When the tiles were all in place, they were coated with a resin "tack" coat; then a 10-mil fiberglass epoxy resin vapor barrier was hand laminated in place. A 3-mil, 1-component, air-dried, polyurethane seal coat was sprayed on and air dried. This final surface was lightly sanded in place, and thermocouples, strain gages and other instrumentation devices were fastened in various positions, using the Lefkoweld 109/LM 52 adhesive and the vacuum bag technique to assure tight contact.

The tank has subsequently been moved to several locations and filled with LH_2 several times. The only apparent change that has occurred in the bonded polyurethane insulation was the development of stress lines in certain areas of the fiberglass inner seal but without evidence of leakage.

Quality Assurance and Repeatability of Manufacturing Techniques

Throughout the development of the multilayer thermal protection system for the one-half-scale cryogenic vessel, a complete case history log book with photographs was maintained. Results of the materials evaluation, manufacturing and processing techniques optimization, as well as the environmental and cryogenic testing, were recorded and analyzed so that this information could be incorporated into a set of reliable materials, processing and manufacturing specifications, and standard documents. Thus, because all of the required information was well documented, the repeatability and reliability of the insulation system was assured.

Molded Fiberglass Covers

These assemblies were prefabricated from resin-impregnated fiberglass cloth using molds specially designed to provide two mating-halves, one inboard half and one outboard half, for each cover assembly required. After curing in the molds, these assemblies were trimmed to net, mating holes drilled, and platenut fasteners installed on the inboard halves. The inboard halves were then bonded directly to the tank wall with Lefkoweld 109/LM52 epoxy adhesive.

Substrate Retainer Rings

Lightweight aluminum retaining rings with specially pressed grooves were prefabricated in circular sections and bolted to the inboard and outboard sides of the conical support structure at both the forward and aft extremities in preparation for installation of the helium-purged substrate.

Helium-Purged Substrate

The helium-purged substrate was assembled into two hemispherical shaped assemblies, prefabricated and subassembled from flat gore patterns cut out of the Dacron-fabric and fiberglass-matting materials. The Dacron-fabric 60-deg gore sections and the fiberglass-mat gores with silicone resin binder were stitched together using Dacron thread. The PF105-700 type fiberglass mat with silicone resin binder was 1/2-in. thick with a density of 0.6 lb/ft^3. The Dacron cloth which was used to contain the mat as a sandwich had a density of 0.8 oz/yd^2. These substrate units were installed onto the tank with the Velcro fasteners. The hook side of the Velcro fastener was bonded to the 2219T87 aluminum vessel wall with Lefkoweld 109/LM52 epoxy adhesive. The tank had previously been cleaned with a sulfuric acid-sodium dichromate solution. An area around each 1-$in.^2$ surface on the tank to which the Velcro fastener was to be attached was cleaned with methyl ethyl ketone prior to bonding. The Lefkoweld adhesive was applied with a spatula, the fastener was squeezed onto the adhesive, and the excess was scraped away; then, the fastener was held in place with masking tape during the 90°F overnight air-cure. On each Velcro fastener there is about 1/2-lb load which includes the pressure in the purge bag and the weight of the insulation.

Conical shaped sections of the purged substrate for the support structure insulation were fabricated in much the same manner and attached to the conical support with clamps on the retaining rings.

Sealed Aclar Purge Bag

The sealed purge bag (Fig. 1) was prefabricated from hemispherical and conical shaped gore sections of Aclar which were thermal-impulse fusion-bonded into two hemispheres and one conical-shaped bag assembly. The 5-mil fluorohalocarbon polymer film, Aclar, was fabricated into hemispheric sections using 30-deg gore sections with a 3/4-in. overlap sealed by thermal-impulse heating. The hook portion of the Velcro fastener was bonded to the Aclar bag using the Lefkoweld 109/LM52 adhesive. The surface of the Aclar was pretreated by the vendor for adhesive bonding and carried their 22C designation. The surfaces to which the Velcro fasteners were to be bonded were cleaned with isopropyl alcohol prior to applying the adhesive. After the hook portion of the Velcro fastener was attached, the Lefkoweld adhesive was applied with a spatula and the excess was squeezed out and scraped away. The fastener was held in place with masking tape during the 90° F overnight air-cure. Tearing

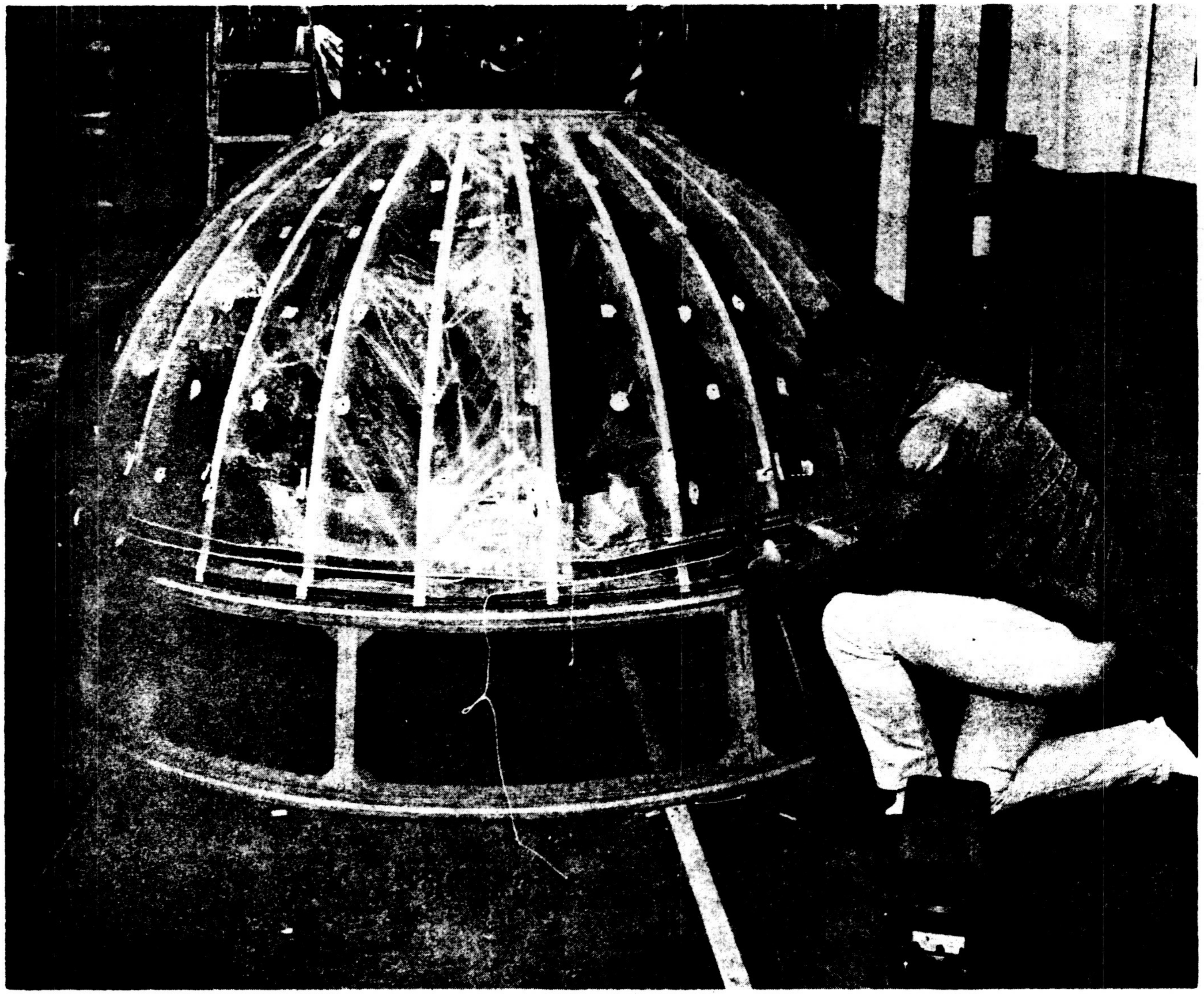


Fig. 1 Fabrication of Aclar Helium Purge Bag

away of the masking tape without simultaneously removing the fastener was one quality control technique for determining the effectiveness of the bond.

A screening program to determine the most efficient adhesive for bonding to the Aclar purge bag was conducted. Specimens of several adhesives were used to attach Velcro fasteners to an experimental Aclar bag. The adhesives were identified and properly cured. The experimental purge bag was pressurized with 0.5 psig of helium and then subjected to a blast of liquid nitrogen (Fig. 2). The only fasteners which remained attached under a 3-lb load after this treatment were those bonded with the Lefkoweld 109/LM52 which was the material selected. Table 1 gives a list of the adhesives screened for the tank and Aclar purge bag bonding application.

Radiation Shields, Multilayers With Button Retainers

The radiation shield multilayers were assembled by removing alternate layers of double aluminized 1/4-mil-thick Mylar together with the 2.8-mils Dexiglas 233-9-Y2 quartz paper spacer from the rolls to form a sandwich on a 60-deg gore template. Each blanket consisted of ten layers of Mylar and nine layers of Dexiglas. The gore section blanket was stitched together using the 40-lb test nylon monofilament and the 10-mil-thick Teflon button retainers on 8-in. centers (Fig. 3). At the edge of each gore, rectangular Teflon tabs were stitched on 4-in. centers. This latter method was used to make the butt joint between the six gores forming the hemisphere. The lower Mylar layer was attached to the Aclar purge bag with the Velcro fasteners (Fig. 4). The felt sides of the Velcro fasteners were stitched to the underside of the lower multilayer insulation blanket. Each Velcro-fastener Teflon-button nylon-thread assembly was tested with a 3-lb load as part of the quality assurance procedure. Three layers of these radiation shields were assembled over the purge bag and substrate. The gore sections in each layer were staggered circumferentially so that the butt joints (Fig. 5) of any underlying layer did not coincide (Fig. 6). The multilayer blankets in the penetration covers were secured with double faced pressure-sensitive adhesive Mylar tape P-94.

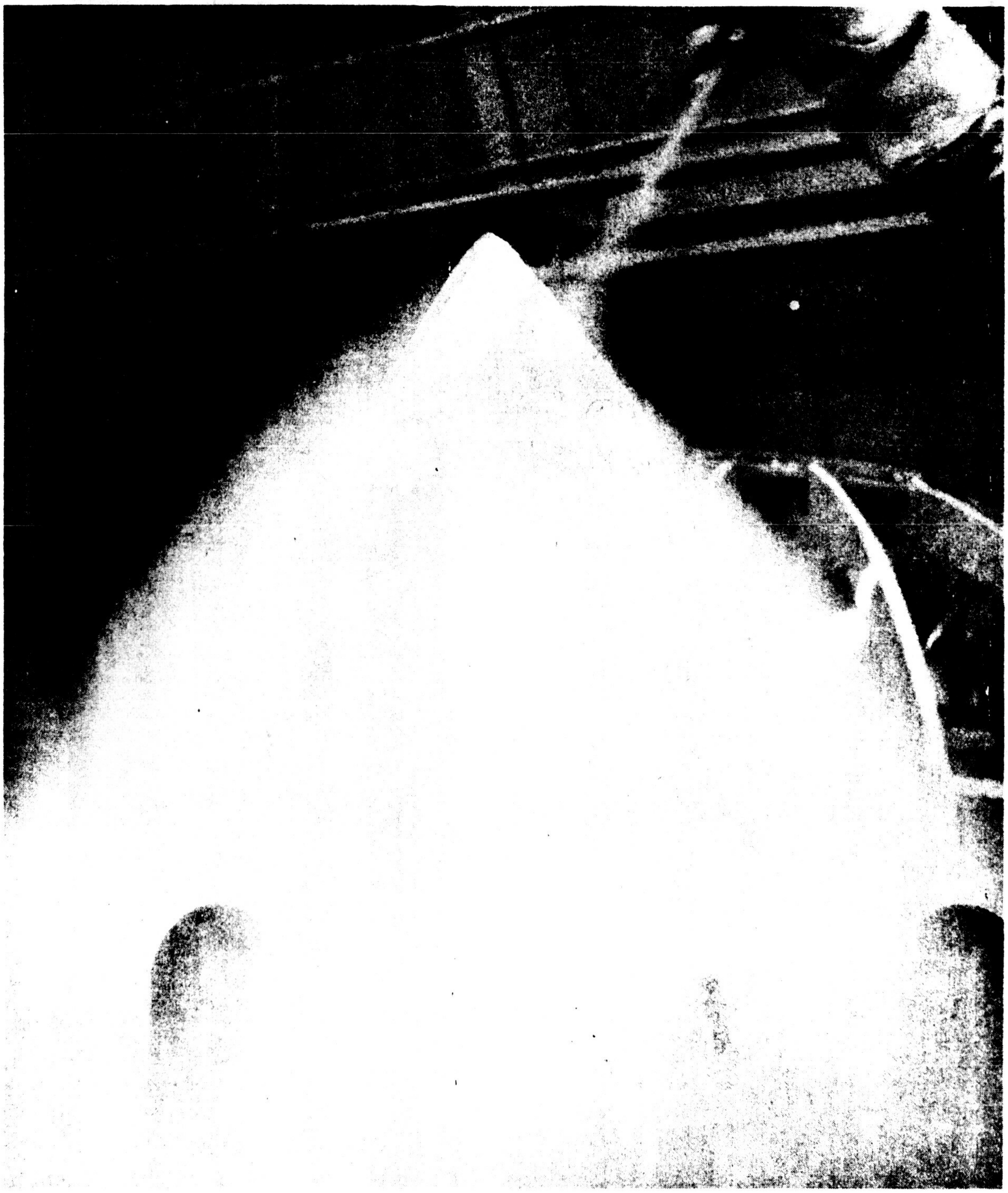


Fig. 2 Adhesive Bonded Screening Test

Table 1

ADHESIVES SCREENED FOR BONDING NYLON VELCRO FASTENER ALUMINUM TANK OR ACLAR PURGE BAG

Adhesive Designation	Component		Adhesive Type	Manufacturer	Operating Temperature Range (°F)	Cure Cycle	Application
	Base Parts by Weight	Catalyst Parts by Weight					
K-218-34	One Component		Rubber Base	Pittsburg Plate Glass Adhesive Division	-180 to +250	R.T. 1/2 hr	Mylar, Al foil fiberglass
7343/7193	7343 (100)	7139 (11)	Epoxy	Narmco Material Division	-423 to +320	R.T. 3 days 4 hr at 160° F	Metal/metal; most plastics
46971	One Component		Polyester	Dupont	-423 to 350	R.T. 3 days	Mylar to vinyl fiber
46971/RC805	46971 (40)	RC805 (1)	Polyester	Dupont	-423 to +350	300°F to 5 min	Mylar to vinyl fiber
Lefkoweld 109/LM52	109	LM52	Epoxy	Leffingwell Company	-423 to +300	R.T. 24 hr 1 hr at 150° F	Metal, wood; most plastics
P-94 Tape	Pressure Sensitive		Rubber Base on Mylar	Dupont	-64 to 300	R.T.	Most materials

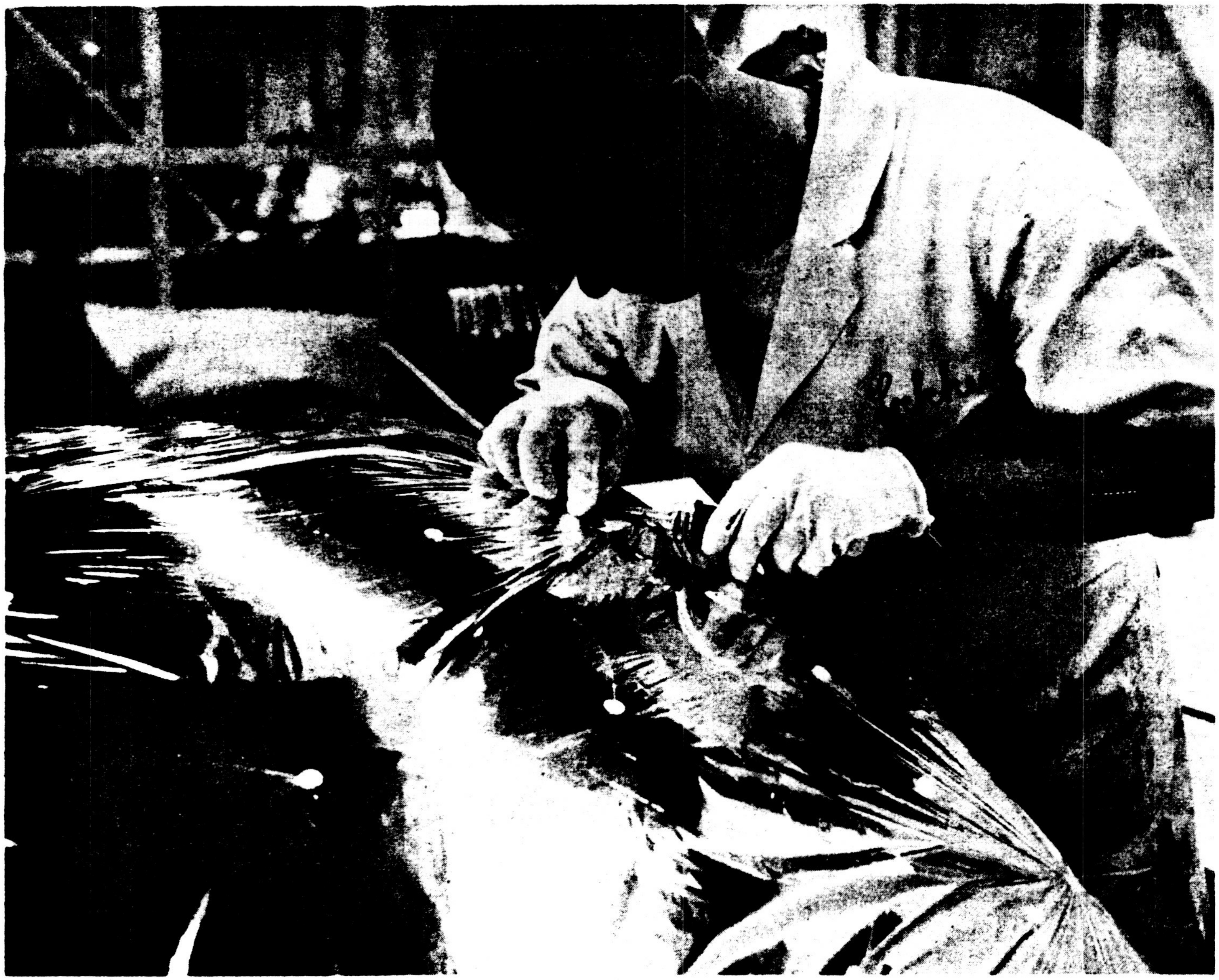


Fig. 3 Fabrication of Multilayer Radiation Shield Blanket

Fig. 4 Method of Attachment of Multilayer Blanket to Substrate Purge Bag

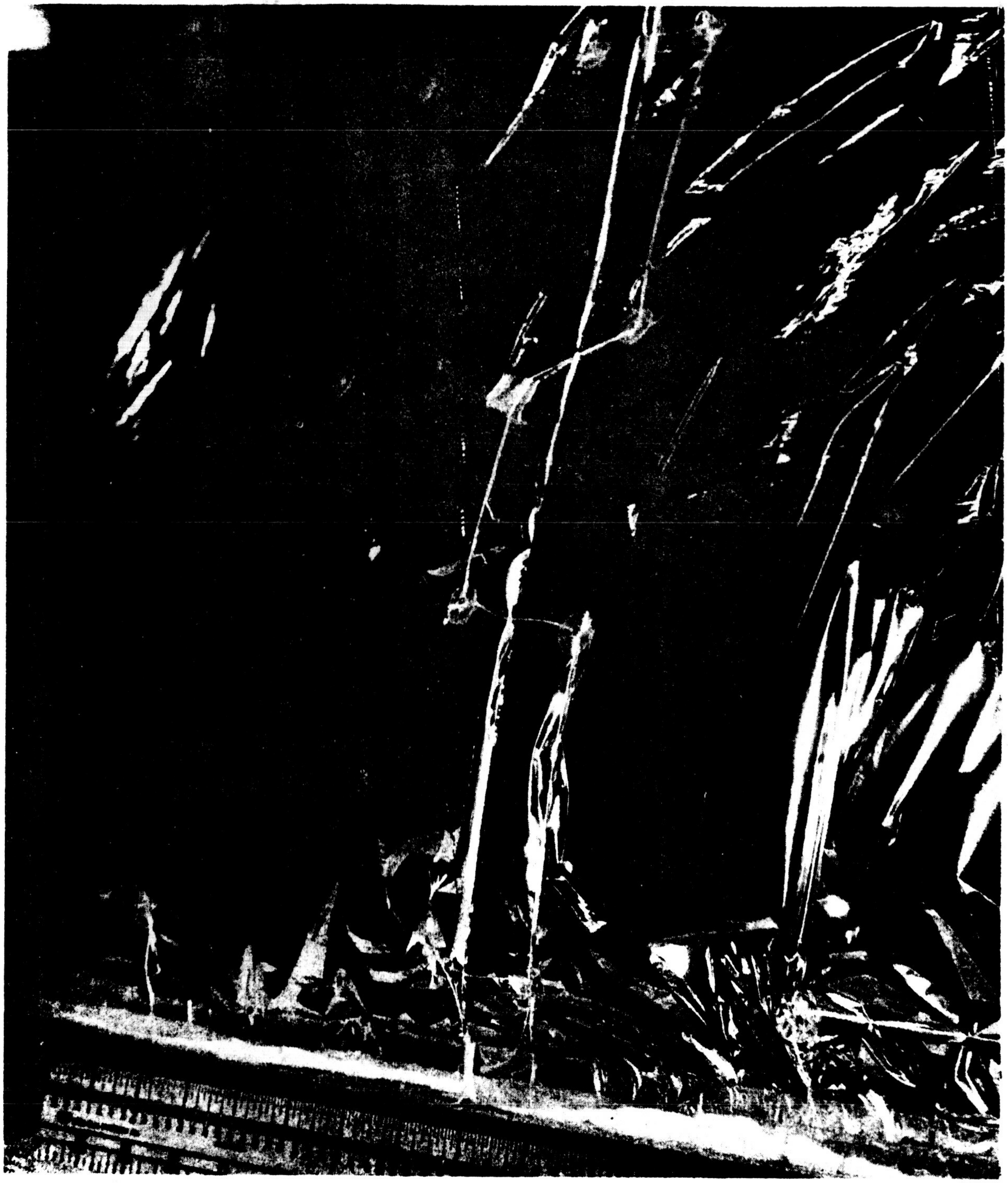


Fig. 5 Butt Joint of Multilayer Blanket After Environmental Tests

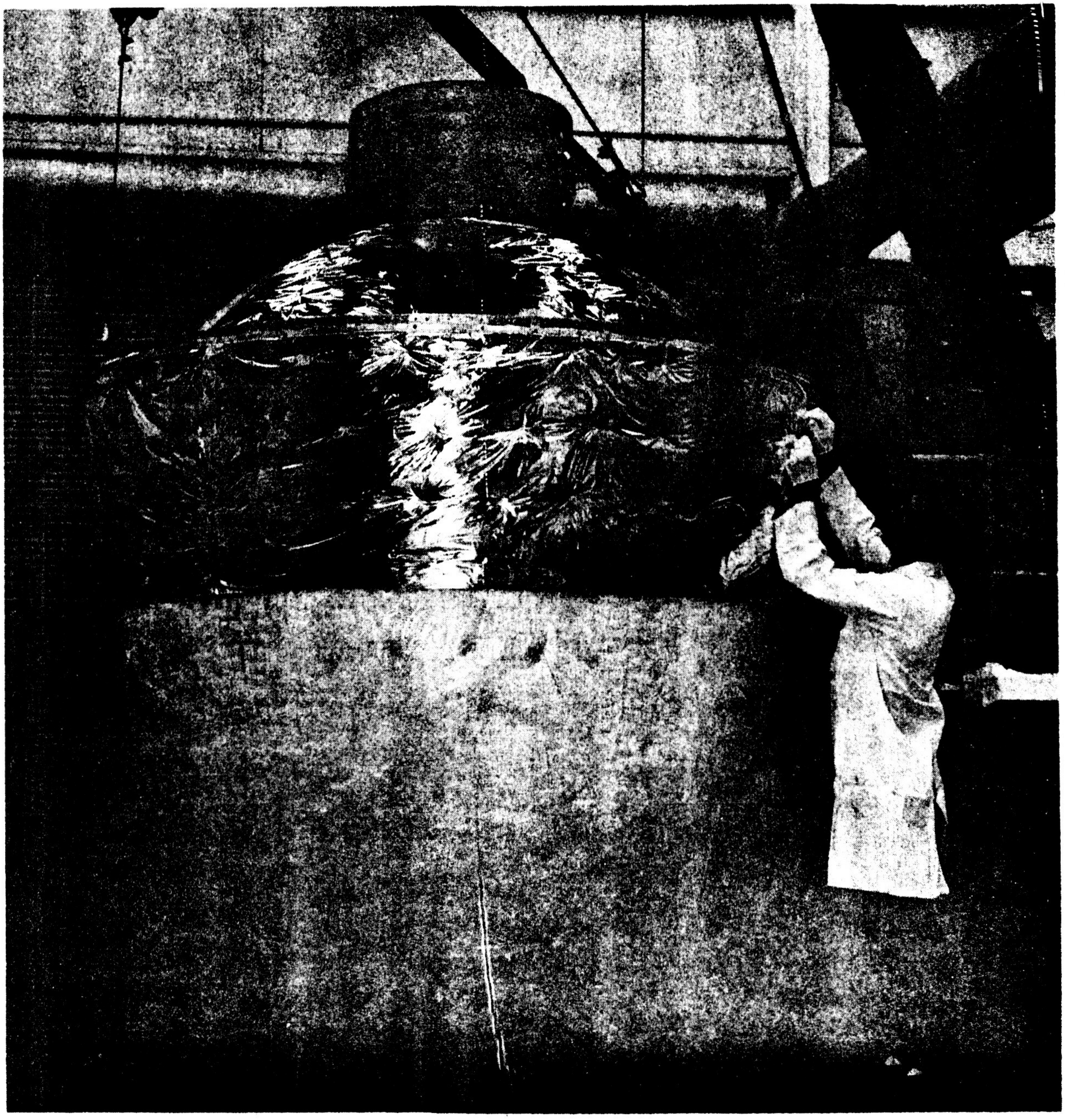


Fig. 6 Assembly of Insulation System on Half-Scale Liquid Hydrogen Vessel

Dacron Netting Envelope

A prefabricated Dacron-netting envelope consisting of two hemispheres and two conical-shaped sections was positioned over the insulated tank and secured by tying, clamping, and with a pressure-sensitive-adhesive Mylar tape aluminized on one side. Originally, a nylon netting envelope was used; however, the acoustic tests on the tank damaged this material. The Dacron net was not damaged in the environmental testing of the tank insulation system which used that material.

The region between the half-scale liquid hydrogen test vessel and the similarly scaled external shell structure was purged with dry nitrogen.

CONCLUSIONS

An adhesively bonded insulation system for a cryogenic propulsion tank has been developed. Analytical and experimental techniques were used in the evaluation, design, development, and final choice of two candidate insulation systems for qualification testing in subscale and half-scale vessels. The fabrication difficulties encountered in applying the crinkled Mylar shingle system to the subscale tank led to a choice of the multilayer button retainer system over the helium-purged fiberglass substrate as the insulation system to be evaluated with the half-scale tank. The insulation system on the half-scale tank and conical support section weighed about 0.43 lb/ft^2 over the 149 ft^2 tank and support area. The upper and lower tank penetration covers were not included in this weight.

Failure of reliability and quality assurance in any system usually occurs as a result of neglect of the obvious. One of the obvious details often neglected in an adhesively bonded materials system is the shelf life of the more volatile or reactive components in the adhesive. These reactive and volatile components, which improve viscosity, cure, bonding, and strength properties of the adhesive, have a limited lifetime. Thus, the adhesive's shelf life must be carefully controlled. One technique used in LMSC bonding operations for minimizing the neglect of the obvious in the shelf life of the adhesives is shown in Fig. 7 . This simple label, designed by LMSC materials processes and fabrication specialists, is being used by certain vendors on their products shipped to LMSC. It is believed that this simple but obvious device will go a long way toward minimizing current neglect of the obviously limited shelf life of adhesives used in bonding applications, thus helping to insure reliability and quality in the final product.

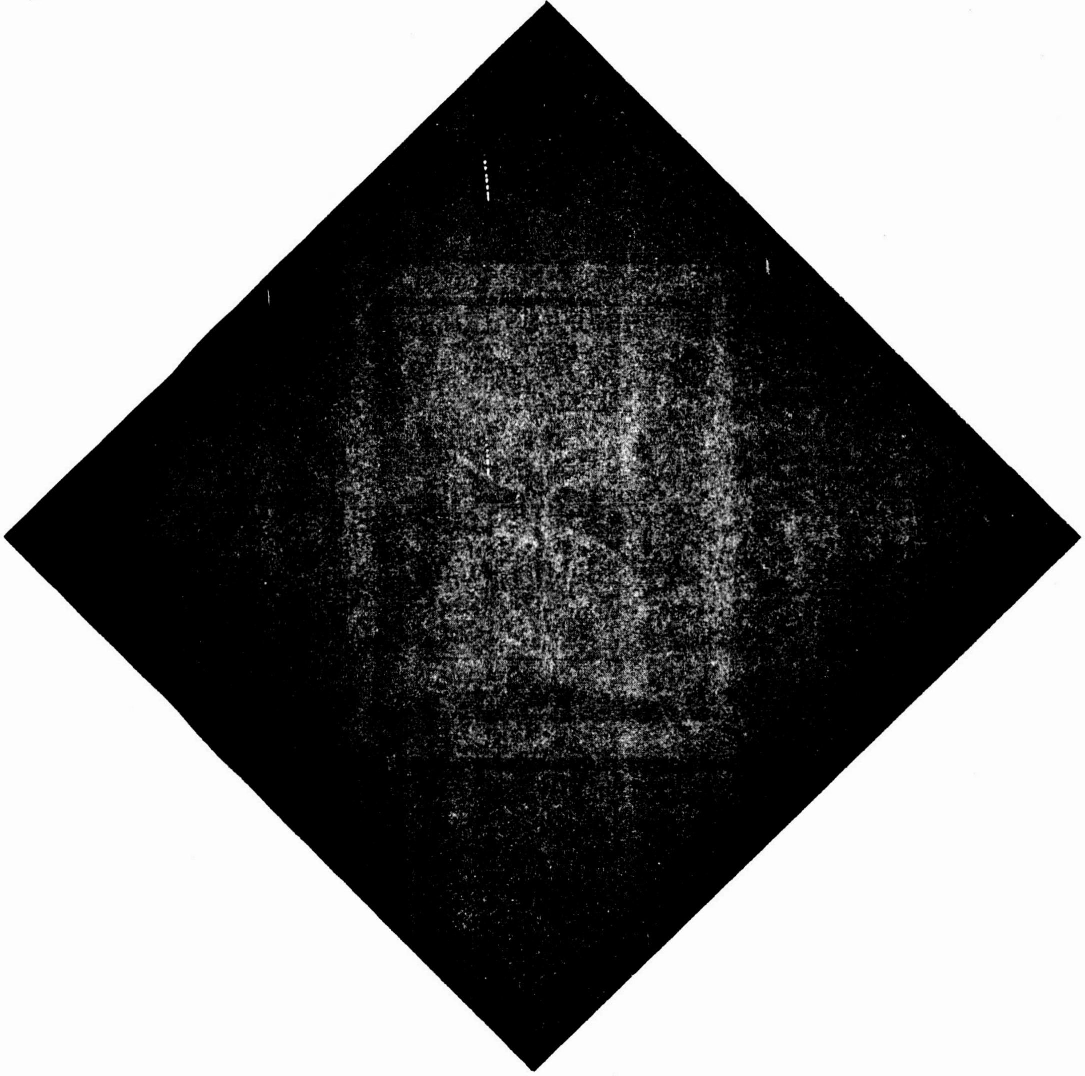


Fig. 7 Limited-Life Shipping Label

ACKNOWLEDGMENT

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SESSION III - PAPER 2.

SECONDARY BONDING

9:10 - 9:50 A. M.

WEDNESDAY - MARCH 16, 1966

N66 34135

SECONDARY BONDING

by

William S. Stiles
Technical Coordinator, Apollo Program
North American Aviation, Inc.
Downey, California

ABSTRACT

Secondary Bonding may be described as bonding operations which are done after the primary structure has been completed. It includes bonding general bracketry and miscellaneous hardware to any surface of the basic structure. Two types of bonding conditions are discussed:

1. In-station bonding where the assemblies are cured in conventional ovens and autoclaves.

2. Out-of-station bonding which utilizes cold-bonding techniques and localized heating devices.

This report highlights the Secondary Bonding procedures currently used on the Apollo capsule at the Space and Information Systems Division The adhesive system, prefit and indexing of parts, surface preparation and adhesive lay-up bagging and curing, and general manufacturing tricks-of-the-trade are described in detail.

SECONDARY BONDING

by

William S. Stiles

Technical Coordinator, Apollo Bonding and
Processing Department, Space and Information
Systems Division, North American Aviation, Inc.,
12214 S. Lakewood Blvd., Downey, California.

Delivered March 16, 1966, at the Structural Bonding Symposium at NASA Marshall Space and Flight Center, Huntsville, Alabama.

SECONDARY BONDING

by

William S. Stiles

Two of the most exciting and fascinating projects of our time are the Saturn and Apollo Programs, which are destined to lift and carry American astronauts to the Moon.

We at the Space and Information Systems Division of North American Aviation Company, Downey, California, are privileged to play an important role in these projects, under contract to the National Aeronautics and Space Administration. Prominent in this program is the Apollo spacecraft command module, a 12-foot-high cone-shaped structure designed around the bonded aluminum honeycomb sandwich concept. The inner skin of this crew compartment is a composite aluminum weldment, which forms an air-tight environmental chamber for the astronauts. During manufacture holes never penetrate this inner protective skin for any reason, because Man must take an earth-like environment with him on his adventures into space. Thus, this crew compartment we are building is of utmost importance. Therefore, to avoid leakage, we attach all details to the module by adhesive bonding.

Figure 1 shows the completed basic structure of the crew compartment with the secondary bracketry completely installed. The details next to the surface of the unit are attached by adhesive bonding. Other brackets are then bolted to the bonded brackets for assembly. Some of the lower structural brackets are bolted at the ends as well as being bonded. This particular section is being readied for the systems installation, which will fill most of the area visible in this figure.

Notice the number of secondary details on the structure. There is hardly a square foot that does not have a bracket of some kind bonded to the surface. The internal brackets are even more numerous.

More than 700 miscellaneous details are attached in this manner. They range from simple angles to extremely complex machined fittings.

I know of no other program which has such a high ratio of bonded brackets per square foot as does the Apollo crew compartment. You might be amazed at the number of technical problems involved in attaching these brackets—especially because of the shape of the module—not a straight line anywhere except on the forward deck. But we are meeting these challenges.

Attaching these details is known as secondary bonding, not because the bonding is secondary in importance, but because it is second in sequential buildup. It is also many times more difficult to accomplish than primary bonding, which is the buildup of the basic honeycomb sandwich structure. The Apollo command module is especially challenging because of its truncated and elliptical surfaces. The only straight surfaces are in the vertical lines of the side walls and on the flat forward deck. Figure 2 shows the general command module configuration with some of the external details already installed. Note the aluminum locating tool on the top deck. Approximately 10 sets of these egg-crate tools are used to locate the many details.

Rather than try to cover all phases of this complex subject, I will concentrate my remarks on:

1. The adhesive system
2. Prefitting and indexing parts
3. Surface preparation
4. Adhesive layup
5. Bagging and curing
6. Torque testing and final acceptance.

Now, I will explain some of the manufacturing tricks-of-the-trade developed in the Apollo Program.

The Adhesive System

Three adhesive systems, in combination with M-602 primer made by Pittsburgh Plate Glass Company, are being used for secondary bonding. They are tabulated on Table 1 for easy statistical comparison:

Table 1. Adhesive Systems

Item	Manufacturer's Name	Number	Type	Curing		Working Time
				Temperature	Time	
1	*Bloomingdale Rubber	HT-424	Film	290 F ±10°	3 hours	36 hours
2	*Bloomingdale Rubber	FM-123	Film	250 F ±15°	1 hour	120 hours
3	Shell Chemical	Epon 934	Paste	Room Temperature	1 week	15-60 minutes
				130 F ±10°	1 hour	

*American Cyanamid Company

These adhesive systems were selected especially for their curing temperatures and their ability to meet engineering design requirements. These systems are designed for temperatures ranging from -200 degrees Fahrenheit to +250 degrees Fahrenheit.

HT-424 is an old stand-by and work-horse of the industry. We use it exclusively for in-station bonding where ovens or autoclaves are used for curing. FM-123 is a relatively new adhesive having excellent mechanical properties. It cures in less time and at a lower temperature than HT-424. We use FM-123 in both in-station and out-of-station bonding operations where only localized heating devices can be used. Epon-934, which is only mentioned here, is used primarily for core filling, and where non-critical room temperature cures are permitted. The precure processing operations are the same for all three of these adhesives.

Prefitting and Indexing Parts

One of the most critical prebond operations is checking, adjusting, and measuring the gap between mating parts to be bonded. It is critical because of its relation to the adhesive film thickness. The amount of gap must be known always, and recorded before the layup operation is started. This is normally done by loading the parts into the holding fixture and physically measuring the actual gaps between the bonding surfaces. The gaps are usually irregular and may even taper, especially in a configuration like the Apollo crew compartment where most surfaces are curved and angular. As mentioned previously, this is why the operation is many times more difficult than the primary bonding. If the details can be adjusted for better fit, they should be adjusted at this time.

Figure 3 shows a typical interior operation for locating and measuring part mismatch. The mechanic is measuring the gap of a curved attaching angle with a feeler gage. Notice the egg-crate locating tool above the mechanic's head. It duplicates all attach points for the right-hand upper equipment bay assembly. We also use this same tool to recheck part location after the bonding is completed.

Mismatch is generally measured with feeler gages, dial indicators, and other standard measuring devices. The measurements must be verified by Inspection, and recorded. This recording is done on masking tape placed adjacent to both bonding surfaces. These dimensions are used later during adhesive layup operations to show when shims or additional layers of adhesive are required. Our testing has shown that multiple layers can be applied successfully without significantly reducing the strength of the bonded joint. Working with our design allowables, mismatch gaps up to .024 inch are filled with adhesive layers; gaps between .025 and .060 inch require a metal shim with adhesive on both sides. When the gaps exceed .060 inch, Engineering analysis is then required.

Shims are usually used where taper mismatch exists, because multiple layers of adhesive tend to out-flow during the cure cycle, causing the parts to tilt and mislocate. In other words, we must maintain a parallel glue line.

After prefitting the parts and recording the gaps, small aluminum locating tabs are placed against each detail to maintain its location during layup and cure. These tabs are held in place with double-back and lead tapes. Egg shell inspection decals are placed over the tabs to verify location and to reveal later disturbances. The term egg shell describes the sensitivity of these decals, which will crack, tear, etc., if the tabs are disturbed.

Figure 4 shows three typical areas with locating tabs, lead tape, and inspection decals in place. The details were removed for surface preparation.

Locating tabs replace the major locating tools during the bagging and curing cycles. Large tools cannot be used conveniently for these operations because of their size and the complications of bagging, which will be explained in more detail later. We know from experience that these little metal tabs have solved a serious problem in a cheap and simple manner. When they are carefully and accurately located and taped firmly in position, they maintain the location of the parts during the balance of the bonding operation. We leave the tabs on until after the parts are bonded to the assembly.

Surface Preparation and Adhesive Layup

When the details are prefit and the tabs firmly located, the next step is to carefully clean the bonding surfaces and apply the adhesive.

Surface preparation is the key to successful bonding. Maximum cleanliness is truly the watchword. The ideal condition is to process clean and layup adhesives in environmentally controlled rooms. However, when the assembly is too large, or facilities are too small, portable clear-plastic tents serve as locally controlled areas. To prevent hand contamination, suitable clean gloves are always a requirement for this operation. Hand oils, body oils, and acids transferred through the hands are a direct cause of many bond failures. The sight of shining metal produces the same compulsion in people as does a wet paint sign. They want to touch.

All aluminum surfaces of the crew compartment, including the secondary bracketry, are immersion cleaned and etched, then primed with M-602 primer while in the detail stage. This provides both a surface protection and a bonding surface for parts and assembly. Primed details subjected to only limited handling are cleaned with MEK just prior to adhesive layup.

On the primary structure, however, we remove primer from areas where brackets are to be bonded. Details are bonded directly to freshly etched bare metal. This procedure removes all outer surface contamination on the structure, which has been subjected to much more shop exposure during the preceding operations than the detail parts. We have experienced practically no bond failures in secondary bonds since establishing this procedure.

The etching operation is performed manually, using etchants mixed with Cab-O-Sil jelly, to retard acid evaporation-rate, which allows chemicals to stay wet. Frequent agitation is necessary to keep active chemicals in contact with the metal. We carefully monitor the exposure time of the acid to be sure that excess material is not removed, yet that the exposure time is long enough for maximum cleaning. We find from experience that 20 to 25 minutes of exposure give satisfactory results.

We remove the etchant by rinsing with polished water until all acid is removed. This we determine by testing with litmus paper. The cleaned surfaces are never wiped or touched after this operation. They are air dried.

This manual cleaning method is messy and hazardous to personnel and surrounding areas of the structure if special precautions are not followed. Careful masking of the surrounding areas with acid-resistant material is highly recommended. A simple trick we use on the Apollo is shown in Figure 5. Plastic catch bags are used to catch dangerous acid runoff.

As for the actual adhesive layup, the first and most important thing is to know the adhesive film you are using—to know it is qualified, properly identified, and has the necessary inspection stamps. Of equal importance is to know how old it is and how much working time is against the particular material; this tells you how much remaining working time you have in which to complete your layup.

We at North American have a rigid adhesive control program. Comprehensive records are kept on each roll and batch of adhesive entering our storage area. It is checked constantly by our Quality Assurance Laboratory well ahead of the time it will be used. Our inspectors maintain a constant vigilance over all adhesives being used in our layup operations—especially for work-time out of the freezer.

After obtaining the proper adhesive from the freezer, we take it to the zinc-top cutting table, where we do the cutting. A sheet of material large enough to cover the layup is removed from the roll, and the remainder is returned immediately to the freezer.

We place the detail part on the adhesive, and mark the trim line. We use scissors to cut and trim net to the trim line of the detail. This includes the additional layers noted on the part during the prefit operation. The protective film is removed from the side placed against the detail, as well as where extra layers of adhesive and shims are required. The outer layer of protective film is left on until time to install. There is nothing especially complicated about this operation, but we must be sure the protective film is removed between multiple layers of adhesive and shims. In addition, we must be sure the correct amount of adhesive is applied in the right locations. Trimming the adhesive net to the periphery of the part reduces excessive out-flash after curing.

Adhesive can be layed up to the detail parts early, and the parts returned to the freezer to await installation without jeopardizing the adhesive or part. This permits greater flexibility where a large number of parts is involved. All open work time against the adhesive is carefully documented to prevent over-aging. If you will remember, the table showed the working time for HT-424 as 36 hours, and FM-123 as 120 hours.

At the time of the actual layup, the detail parts are taken from the freezer, and, after removing the outer protective film, they are carefully nested between the tabs, and taped in position with lead tape. Lead tapes are especially helpful because the stiffness of the metal reduces liftoff during elevated temperature. We frequently hot tack the detail in position with a hot iron. It is also advisable to use dacron cloth over the part and glue line for interim protection, as well as to control adhesive flash.

Bagging and Curing, In-Station

In-station bonding is any bonding operation done in proper sequence utilizing fixed facilities and equipment; for instance, environmental-controlled layup room, autoclave, or oven. The term "proper sequence" means installing bracketry at a point of assembly when the structure and locating tools can be handled easily within the limitations of the bonding facilities.

Figure 6 shows a typical in-station curing operation with the crew compartment in our 15-foot-diameter Lacy autoclave. Notice the vacuum bag covering the unit, and the various vacuum lines protruding.

The most conventional method of applying bonding pressure is with a vacuum bag and vacuum. It is the only method that assures uniform pressure over every square inch of the part it covers. It can be used in combination with autoclaves and pressure multipliers when pressures greater than atmospheric are desired.

Most secondary bonding operations never require more than 10 psi, which is within the capabilities of vacuum only. Bonding pressure requirements are directly related to the bonding area and the amount of misfit between the parts being bonded. In other words, large areas create a condition whereby gasses become entrapped, and, if sufficient pressure is not maintained, void areas develop during the cure. In the case of poor fitup, pressure is sometimes used to align parts which otherwise would not bond. The practice of using pressure to take out misfit should be done with great discretion, because of the danger of preloading the assembly. As mentioned before under the prefit discussion, better bonds can be assured if the parts are adjusted to fit properly.

The word "bag" sounds simple, but don't be deceived. This bag, which is a pressure membrane, is the make-or-break of the entire bonding process. All processing labor can be lost if the bag fails to do its job during the critical curing cycle under vacuum.

For those who may not understand the application of the vacuum bag, let me explain and show you with Figure 7.

The bag forms a complete envelope over the entire part and is connected to a vacuum pump through suitable lines. The other essential ingredient is the generous layers of bleeder cloth placed between the parts and bag, which provide a passage for air evacuation. We use fiberglass boat cloth for this because it has a coarse weave and withstands the bonding temperatures.

The bag need not envelop the entire assembly. It can be placed against any surface of the structure where the skin becomes one side of the bag—providing there are no holes to cause leaking. Standard sealants—that is, zinc chromate putty or hi-temp buna-based materials— are used generally to seal bags against the assembly and elsewhere. We use a thin, transparent high-temp nylon-based film 2-mil thick for our vacuum bags. One is called Vac-Pac and is furnished by the Richmond Paper Company of Highland, California. The other is Capran 80, supplied by the Allied Chemical Company of Morristown, New Jersey. Both materials are acceptable for use with the adhesives systems mentioned previously.

One of the tricks of the trade is to use a generous amount of bagging material and plenty of dog ears. A typical dog ear will be shown later in Figure 8. Dog ears allow the bag to form freely around the parts without stretching, tearing, or puncturing. The cardinal rule in bagging is never allow the bag to bridge or lay over an unsupported area. If it does, the bag will surely fail and the assembly will have an inferior bond because of lack of pressure, depending upon the amount of pressure lost.

The technique of good bonding is an art, not a science, and it requires careful training to develop experienced mechanics in this field. Let me emphasize, the vacuum bag is the most practical tool at the disposal of the bonding technician.

In-station curing is accomplished in standard ovens or autoclaves having automatic heat-controlling and recording devices. The curing process itself is relatively simple and involves time-and-temperature factors of the specific adhesive being cured. If the cure is being done for the first time, it is recommended that a dry run be made without adhesive to establish and verify the process. This includes placing the monitoring thermocouples on the assembly.

Another critical phase of the curing cycle is to constantly survey the vacuum monitors to be sure adequate pressure is being maintained across the part at all times. Any loss in this pressure through a bag leak can result in a poor bond, or no bond at all.

Bagging and Curing, Out-of-Station

Out-of-station bonding is any bonding which uses portable curing equipment such as heat lamps, electric heater strips, portable ovens, or room temperature curing. Portable pressure devices must be used also; that is, mechanical vacuum tools, vacuum bags, and even spring-loaded mechanical tools.

Out-of-station bonding is usually necessary to accommodate special requirements after the unit has left the bonding area. Also out-of-station bonding is used for large assemblies which cannot be placed into ovens or autoclaves. That is the context in which we use the term at North American Other companies may use the term differently.

We advise other companies to avoid out-of-station bonding whenever possible because the bonding cycle must be performed under the same rigid controls as in-station bonding without the luxury of all the fine controls available in the fixed facility. Production costs are also considerably higher with out-of-station bonding—not only because parts are added to the structure in small quantities or even individually, but also because it is a slower, non-routine operation, and may adversely affect subsequent activity. Bagging techniques for out-of-station bonding are usually the same as described for in-station bonding, except that curing pressure is generally applied only through vacuum pressure.

When hot bonding is required, we use a technique newly developed at North American. This involves using a heater strip for the heat, and a vacuum bag for the pressure. We do not use hot bonding in areas where localized heat could damage surrounding components. Heat radiates rapidly through aluminum assemblies, and you must carefully evaluate each condition before proceeding.

Special strip heaters are placed directly on the bonding flanges and adjacent areas, then covered with the standard vacuum bag setup. The power supply lead runs out to a special controller which I will describe later.

The heater strips we use are manufactured by Briscoe Manufacturing Company, Columbus, Ohio. We like them because they are made in several watt-densities, and to almost any configuration. The proprietary feature of the heater is the stranded resistance wire which is woven into a uniform pattern, then encased in a Dow Corning silicone extrusion. It is fairly flexible and can be used repeatedly. They are made in widths of 1/2, 1, and 2 inches and almost any length. They are joined easily for making various widths. The one constraint on size area-wise is the total wattage needed to run the heater. This, in turn, is directly proportional to the power input. It is necessary to stay within the power limits of the controlling devices. Large amperages will require larger switch gear. We like to stay within 240 square inches maximum, which at 7 watts per square inch is equal to about 15 amps. This we can run very conveniently at 115 volts.

The heater can be obtained in several watt densities per square inch. We use 3, 5, and 7 watt heaters, and find they are satisfactory for most requirements.

A simple controller, handling up to 15 amps of power at 115 volts, also is available. It works on the principle of a matching heater coil and bi-metal switch which can be regulated by a percentage control knob. The controller is stable when used in a normal room environment.

I should mention that these controllers do not regulate power or voltage. They are strictly a bi-metal switch device that regulates by make-and-break of the circuit. If the heater exceeds the power rating of the controller, additional voltage dividers or switch gear must be connected into the heater circuit. I mention this because a pure resistance heater device must be designed to fit the requirements of part configuration, the mass weight, desired temperature, and available power source.

The advantages of using heater strips in combination with the vacuum bag are that the heater is held firmly against the part for maximum transfer of heat. This prevents developing hot spots which could damage both heater and parts.

One thing to remember when using heater strips and blankets is to never allow any part of the heater to operate without contacting a surface or heat-sink. This is one of the secrets of successful heater strip application.

As I mentioned for in-station bonding, it is advisable to make a dry run without adhesive to establish the controlling procedures on any first-time application. All parameters should be documented on a process control sheet so they can be repeated any time thereafter.

Figure 8 shows a typical bagging setup with the heater strip installed. After positioning thermocouples directly on the part to monitor the heat, we place layers of dacron over the parts and thermocouples, which act as a separator and help control flash. Strip heaters are then placed on the dacron over the bonding flanges, which are covered with several layers of boatcloth. We then complete the vacuum-bagging operation in the conventional manner.

Figure 8 shows the dog ears mentioned previously. You can see the dog ears along the outer periphery of the bag. Since it is not possible to seal every edge of the large bag to a small surface of the part or tool, it is necessary to use the dog-ear technique.

As you can see, we allow the vacuum bag to bunch so the material can unfold when vacuum pressure draws the bag in and around the details. Nylon bags also have a slight shrinking characteristic. Too few dog ears are an indication of insufficient bagging material. It is advisible to correct this condition immediately before beginning the cure cycle.

Torque Testing and Final Acceptance

The standard acceptance procedures include visually inspecting the glue line for obvious voids and discontinuities. Dimensional checks are made by reinstalling the egg crate locating tools to the assembly and checking the mismatch.

Actual bond integrity is checked with a torque wrench and special adapter tools designed to fit each configuration, which is shown in Figure 9. The part is tested over the entire length, usually every three inches. The torque wrench is moved in both directions at a given bond.

This method has proven very successful because each detail is actually torque tested and verified before attaching critical assemblies.

The torque values, however, must be calculated carefully for each cross-sectional configuration, because the torque force applied involves a peeling action which is not related to pure straight shear or flat-wise tensile loads.

Conclusions

I have discussed only a few of the highlights of this extremely complex subject. Each of the subheadings is truly a subject in itself. Secondary bonding, as I mentioned at the beginning, can be many times more challenging than bonding the basic structure because of the many variables in part configuration and bonding conditions. There is no one simple procedure that applies to all conditions. The fact that new and better adhesives are replacing the old standbys (the mechanical fasteners) make it imperative that we develop new and better methods for the bonding process.

I'm reminded of two men sitting on a roof during a flood. The water was about six or seven feet deep and swirling all around. As they watched, a hat floated down current about 100 feet. It stopped suddenly, and moved upstream. After about 100 feet in this direction, it stopped again suddenly, turning around it moved downstream as before. This was repeated several times before one of them exclaimed:

"Did you see that? Did you see that? I can't believe it."

"Don't get excited," advised the other man. "That's grandpa. Yesterday he said, 'Come hell or high water, I'm mowing that front lawn tomorrow!'"

That's how it is with us in the bonding business—come hell or high water, we'll bond what Engineering asks us to bond. And as you know, their demands necessarily are becoming more severe each day, and our job becomes correspondingly more difficult.

While I speak to you today, all over the country researchers are experimenting with new products—resins, adhesives, impregnated cloths, primers, sealers, cleaning agents, etc. And all over the country technicians are experimenting with unique tooling concepts, unconventional techniques, new parameters, and novel approaches to fitup, layup, bagging, and curing. The sole objective is to advance the state of the arts.

Also, symposiums such as this one today play a vital part in disseminating technical know-how. These factors—research, development, experiments, and communication—are the tools which will make it possible for us to stay abreast with ever-increasing Engineering demands for bonding, which will let us say confidently:

"Come hell or high water, we in bonding will do the job!"

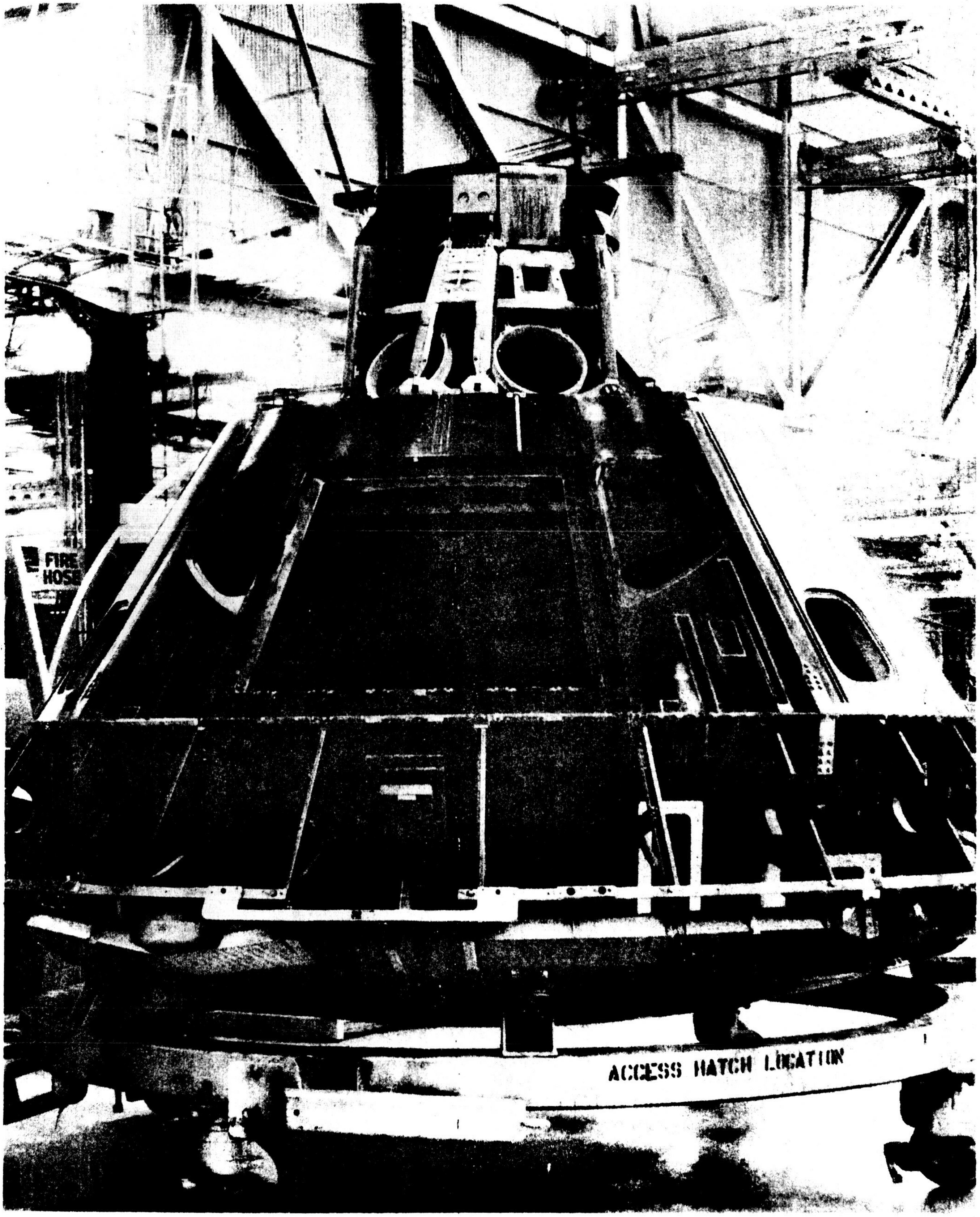

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Figure 1. Crew Compartment With all Bracketry in Place

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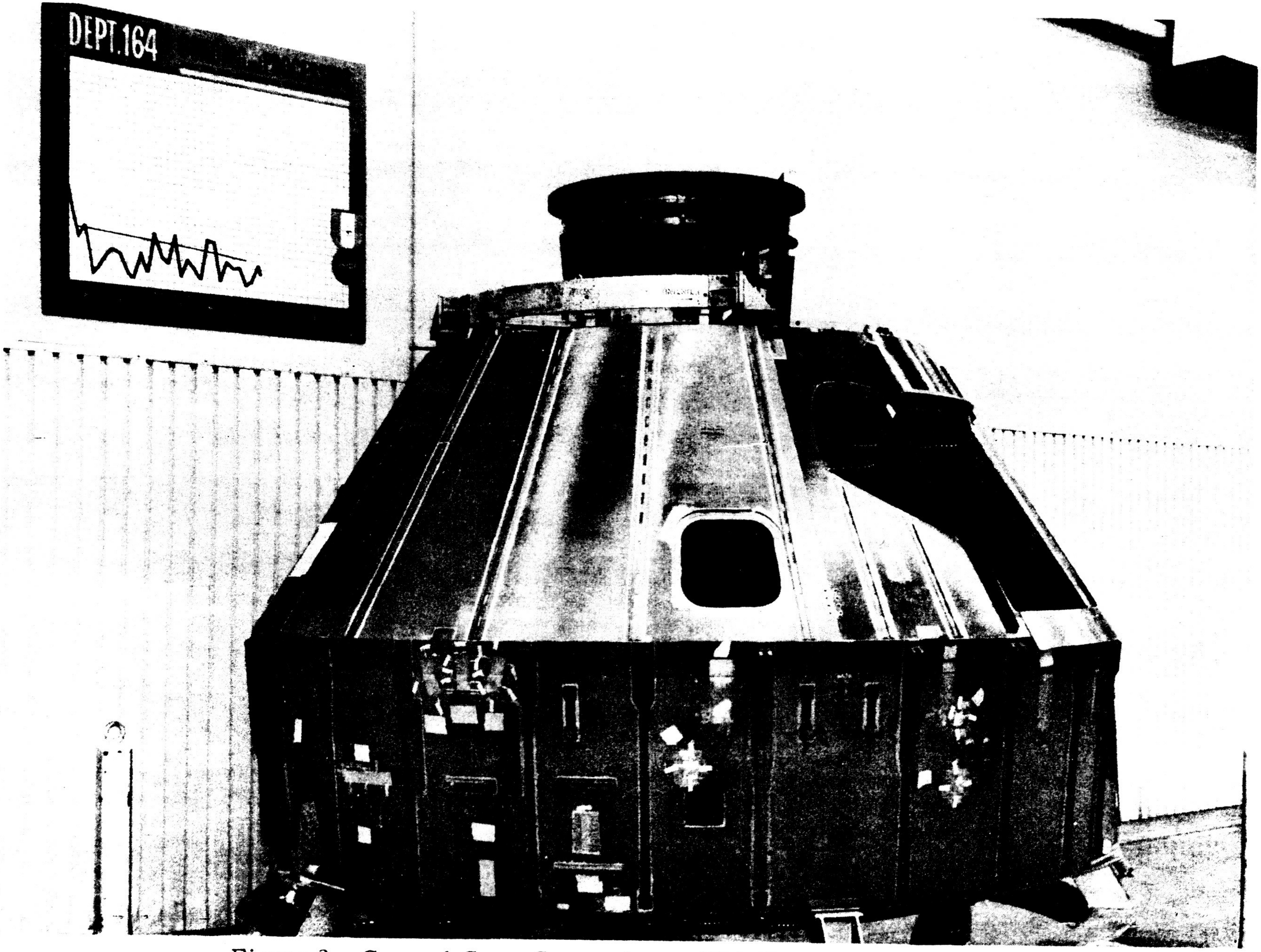

Figure 2. General Crew Compartment With External Brackets

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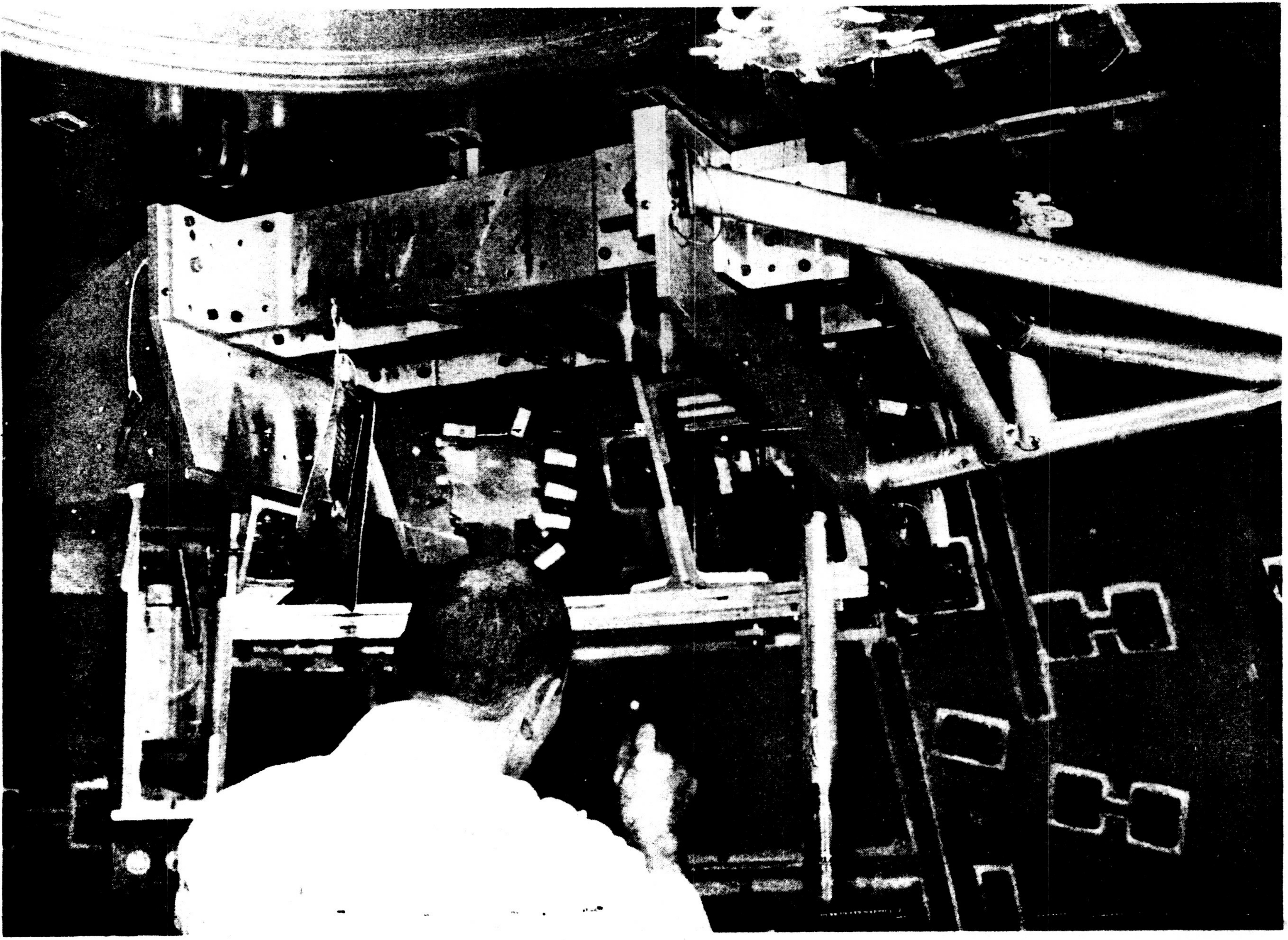

Figure 3. Typical Operation for Measuring Mismatch

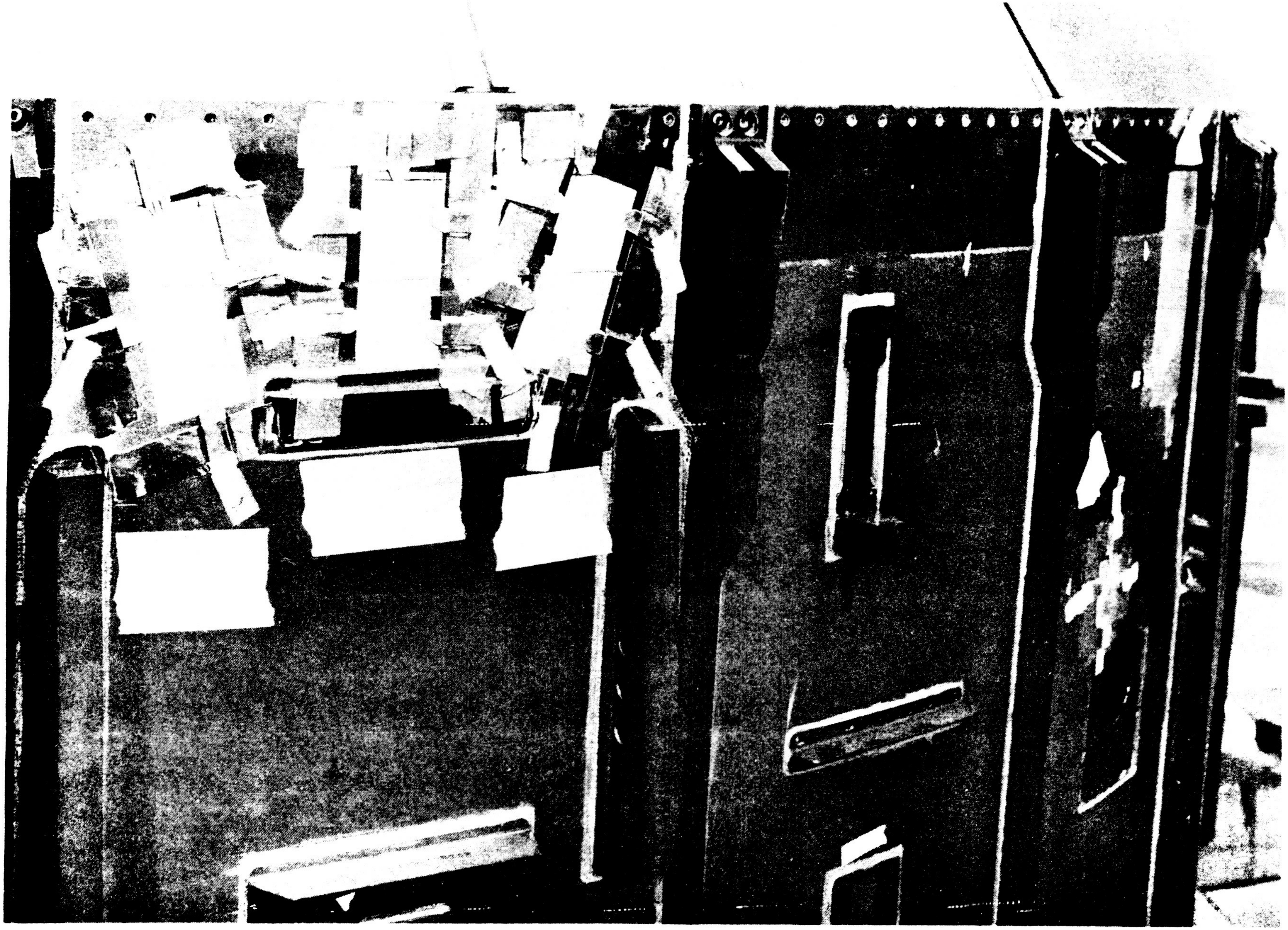

Figure 4. Locating Tabs, Lead Tape, and Inspection Decals in Place

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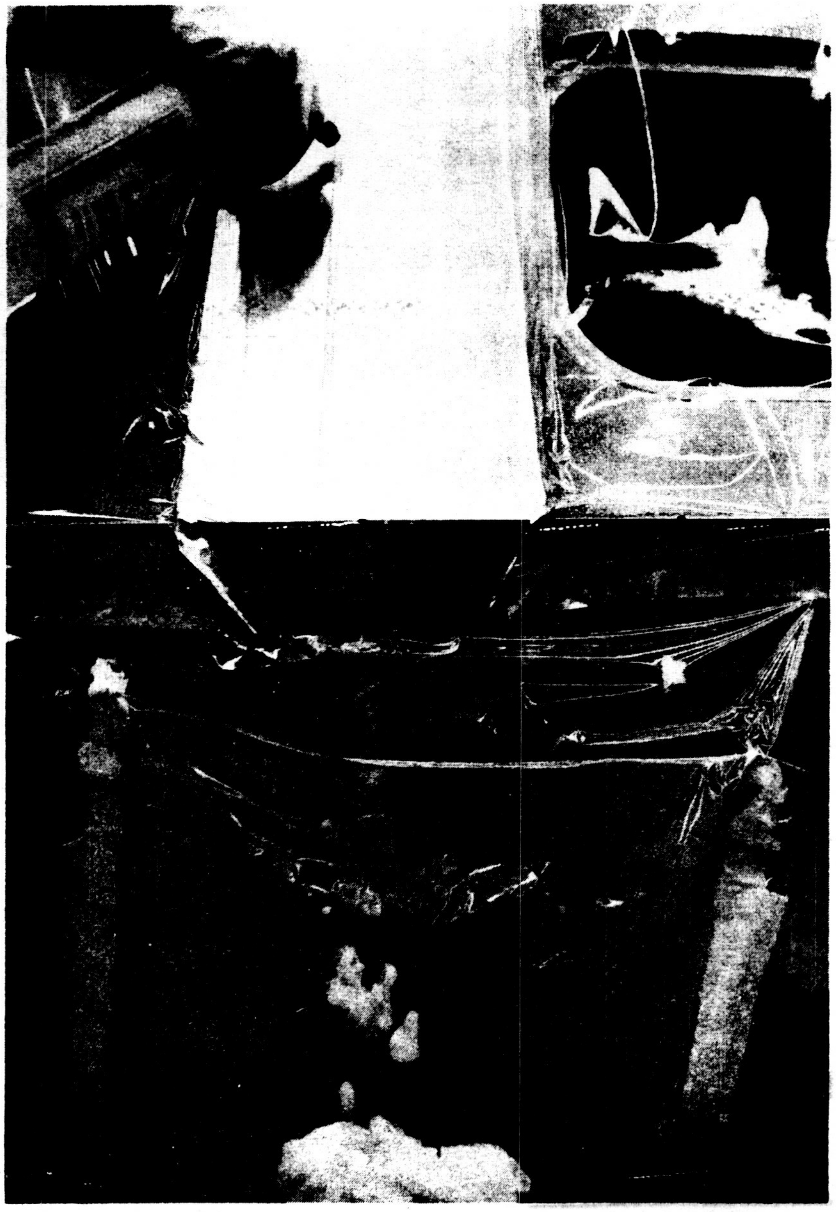

Figure 5. Paste Etch Operation

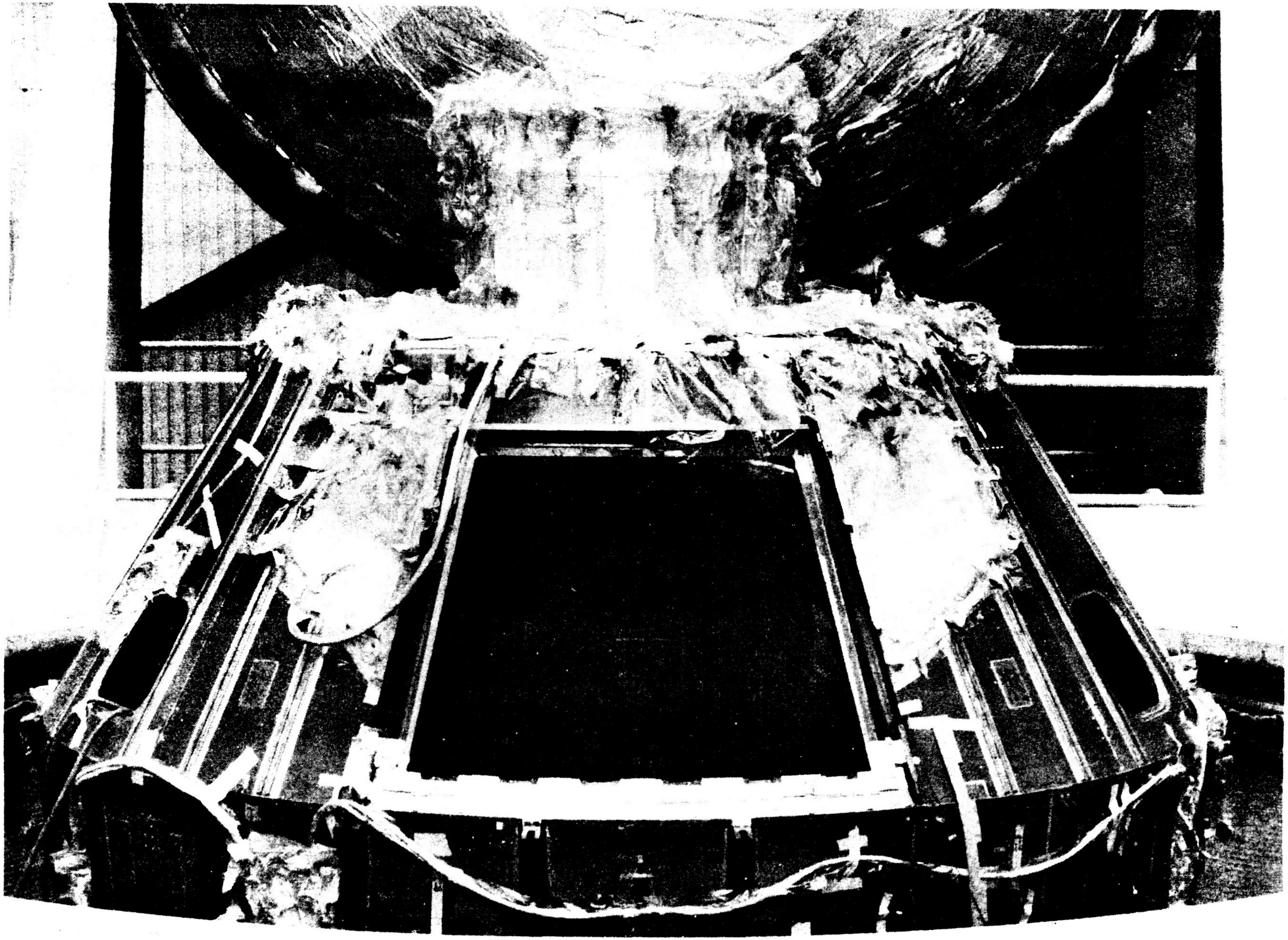

Figure 6. Crew Compartment in Autoclave for In-Station Cure

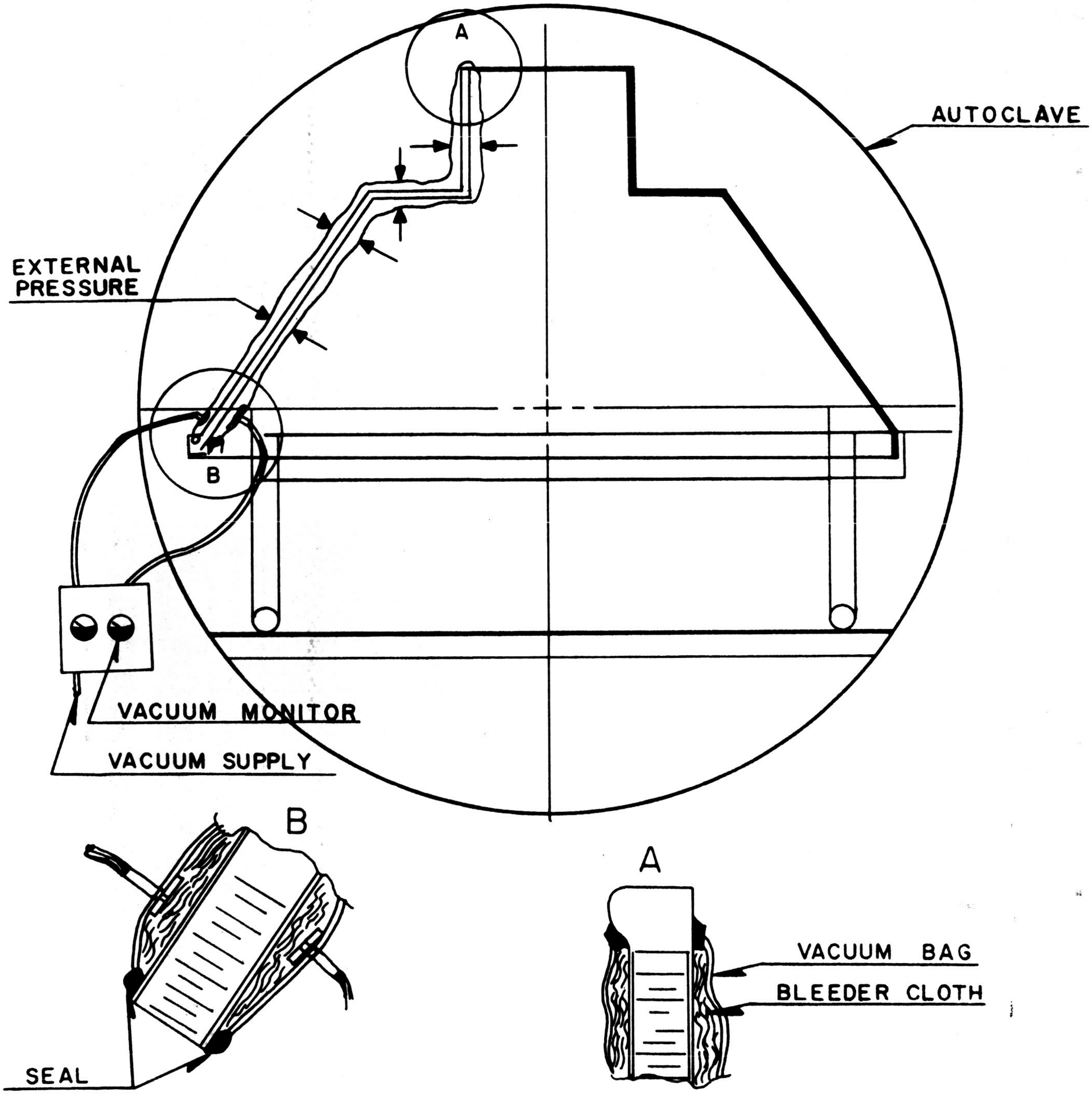

Figure 7. Illustration of Vacuum Bag Theory

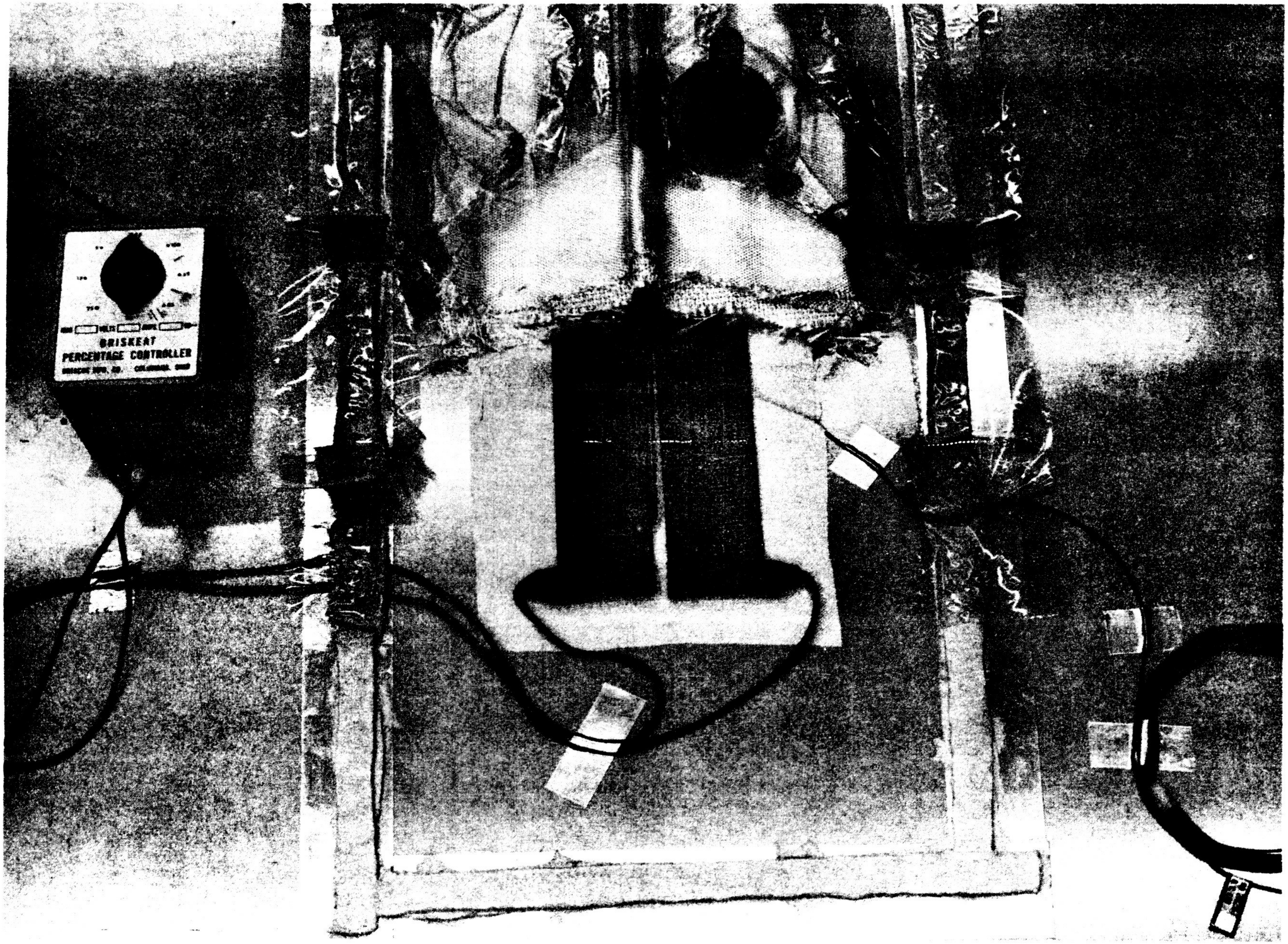

Figure 8. Bagging Setup With Heater Strip Installed

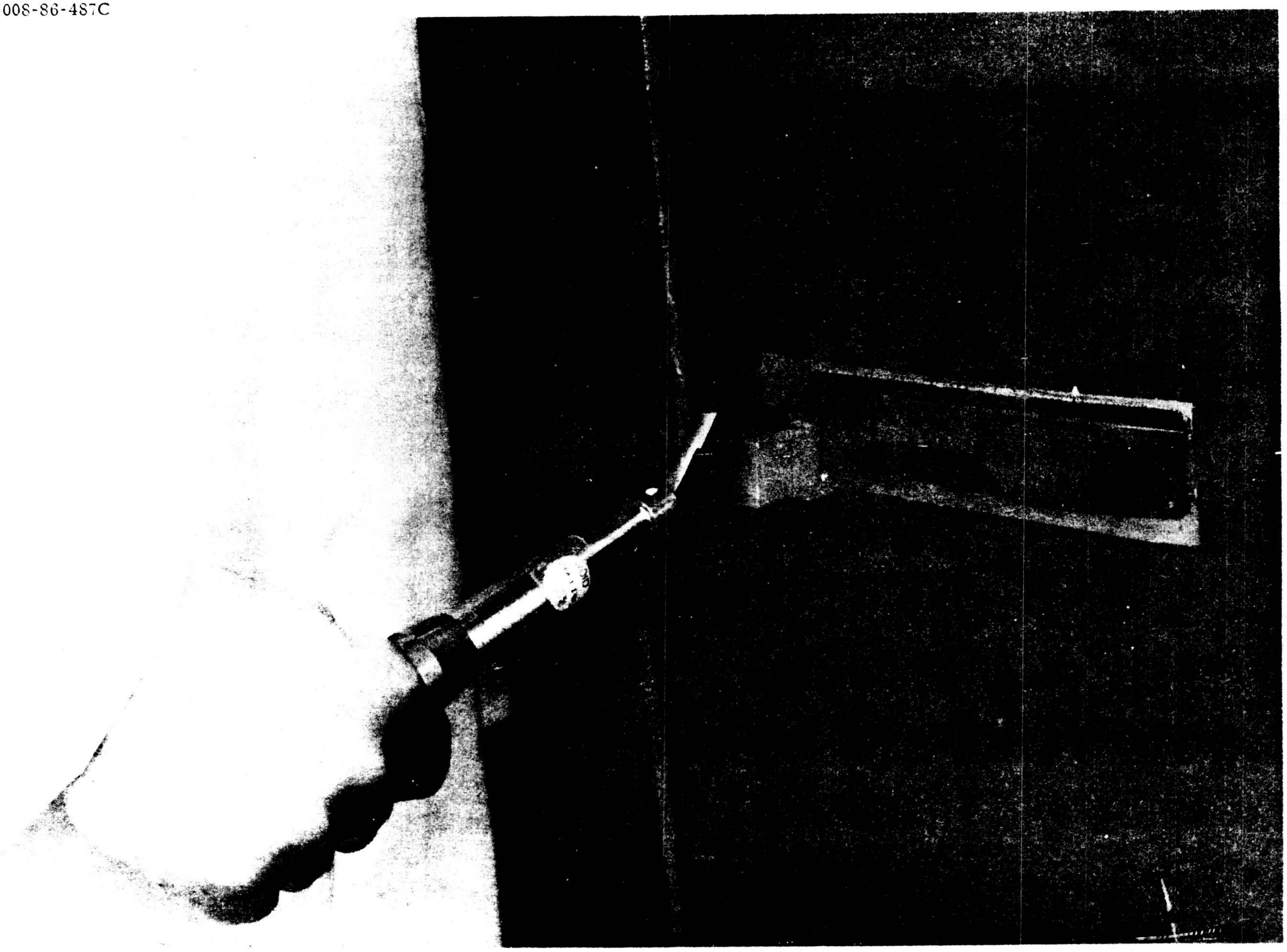

Figure 9. Typical Torque Test

SESSION III - PAPER 3

BONDED (ONE SIDE) COMMON BULKHEAD

10:00 - 10:40 A. M.

WEDNESDAY - MARCH 16, 1966

5

N66 34136

THE DEVELOPMENT OF A BONDED COMMON BULKHEAD FOR SATURN

by

A. C. Robertson
Chief Engineer, Special Programs (DAC)

and

E. L. Brown
Deputy Chief, Methods Development Branch (MSFC)

ABSTRACT

The fabrication of common bulkheads for Saturn vehicles (S-II, S-IV, and S-IVB) is complex and difficult to control. The bulkheads are a sandwich structure composed of aluminum alloy skins bonded to fiberglass honeycomb core. Therefore, the Manufacturing Engineering Laboratory at NASA/MSFC initiated a development program to simplify and improve the manufacture of these critical Saturn components. One approach selected for investigation was the use of bonding rather than welding to join the aluminum skins. Although a bonding material suitable for use at LH_2 temperatures has been available for several years, it's use has been restricted primarily to so-called non-structural applications - clips, brackets, etc. It's use to join primary structural components offers not only potential simplifications in fabrication but significant improvements in the repairability and replaceability of structure. This paper describes the development and fabrication of an experimental Saturn S-IV common bulkhead using this adhesive.

DOUGLAS PAPER NO. 3817

THE DEVELOPMENT OF A BONDED COMMON BULKHEAD FOR SATURN

PREPARED BY:
A.C. ROBERTSON
CHIEF ENGINEER, SPECIAL PROGRAMS
ADVANCE SYSTEMS AND TECHNOLOGY
DOUGLAS AIRCRAFT CO., INC.

AND
E.L. BROWN
DEPUTY CHIEF, METHODS DEVELOPMENT BRANCH
MANUFACTURING ENGINEERING LABORATORY, NASA
MARSHALL SPACE FLIGHT CENTER
HUNTSVILLE, ALABAMA

TO BE PRESENTED TO:
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HUNTSVILLE, ALABAMA
MARCH 15 – 16, 1966

DOUGLAS

DOUGLAS MISSILE & SPACE SYSTEMS DIVISION

INTRODUCTION

As a part of the development of the Saturn S-IV/S-IVB stage the Douglas Aircraft Company has pioneered in the development of the cryogenic common bulkhead. The term common bulkhead is derived from the design function of the bulkhead, which is to separate the two cryogenics, liquid hydrogen and liquid oxygen, in a single tank, thereby shortening the stage and eliminating the necessity for two separate bulkheads and the associated interstage structure. The common bulkhead is structurally adequate to withstand both the thermal and the pressure loads from both the hydrogen and the oxygen tanks, and it has sufficient insulation properties to prevent the liquid hydrogen from freezing the liquid oxygen. Another benefit from the common bulkhead is that it permits a reduction in the total length of the vehicle, thereby reducing the bending moments.

DISCUSSION

THE PRESENT BULKHEAD

Figure 1 shows the general arrangement of the Saturn S-IVB stage. The common bulkhead is shown installed on the aft dome. The bulkhead is designed to withstand the full pressure of either the liquid hydrogen tank or the liquid oxygen tank while the other tank is at ambient pressure. This is a rigorous requirement, considering the size of the bulkhead and the necessity for controlling weight. The basic components making up the common bulkhead are aluminum alloy forward and aft domes bonded to a phenolic fiber glass honeycomb core. The S-IV and S-IVB common bulkheads are spherical in shape and differ only in the basic diameter dimension and thickness, and in the details of the attachment to the aft dome. Figure 2 shows these S-IV common bulkhead components. Figure 3 shows the details of the meridian and the dollar joints and the ring attached joint. All these joints are single-pass methane inert gas (MIG) welds.

The present method of manufacturing the S-IV and S-IVB common bulkhead consists of the following major operations. First, the preformed aluminum gore segments are welded into complete forward and aft domes, including the dollar and the attach rings. Then, the aft dome is installed on a bonding fixture. The next assembly operation is to bond the honeycomb to the aft dome. However, because of slight distortions in the contour of both the forward and the aft domes, distortions arising both from forming operations and from welding of the segments together, it is necessary to sculpture the honeycomb to fit these distortions, or waves, in the fore and aft domes. To determine how to sculpture the core, the contour mismatch between the fore and aft domes is measured before the core is bonded to the aft dome. The method used at Douglas for measuring this contour mismatch is the Paulino block system. This method uses 350 small honeycomb blocks temporarily installed over the surface of the aft dome. Each small honeycomb block has a putty pad on top. The forward dome is then placed over the aft dome and lowered into its final position relative to the aft dome. In the process, the forward dome contacts all the putty pads. The dome is then removed and each small pad is measured to determine the variations in contour between the forward dome and the aft dome. Figure 4 depicts this measuring technique. Following this, the honeycomb core sections are fitted to the aft dome. After fitting, the core is removed, the dome is cleaned, and an adhesive film is placed on it. The core is then re-installed and bonded in an autoclave to the aft dome. Figure 5 shows the core bonded to the aft skin.

Following this bonding operation the core is sculptured. To accomplish this, the core is spot-faced according to the dimensions measured from the putty pads, as shown in Figure 6. This operation provides guide points for the manual sanding operation which is used to bring the core surface to contour. This hand-sanding operation is time consuming and subject to some human error. After this sculpturing operation is complete, the adhesive film is installed over the core, and the forward dome is placed over the assembly for the final bonding operation. The entire assembly is then put in the autoclave for the final adhesive cure cycle. A few problems have been encountered in this fabrication process; the major problems are welding and fitup. Some of the common bulkhead difficulties relating to these two problems are (1) weld porosity, (2) weld cracking, (3) skin debonding, and (4) skin wrinkling.

To some degree, weld cracks are now attributed to the aging of the weld metal during the adhesive bonding cycle. Figure 7 shows the effect of the bond cycle on weld ductility and, as this figure indicates, the ductility decreases considerably with the increasing numbers of bonding cycles to which the welds are subjected. Frequently, these cracks appear following the thermal shock induced by the initial propellant loading. Wrinkles have also been observed on the liquid hydrogen side of the S-IV-6 bulkhead and the S-IV dynamic vehicle. These wrinkles have been attributed to forcing the excess metal in the forward dome to conform to the honeycomb core surface. The records of difficulties experienced show that weld cracking or debonding has occurred during the cryogenic loading in most of the S-IV common bulkheads. Although all the S-IV stages performed successfully, this record indicates the desirability of improving the processing techniques.

As has been previously stated, the problems of fitup and weld-zoned cracks are inherent in the existing S-IV and S-IVB processes. The methods for fitting the domes to the core are time consuming and are marginal for providing the required bond-line fit. The repeated subjection of the weld-zone metal to the honeycomb adhesive bond cycles is an undesirable part of the existing process.

THE BONDED BULKHEAD CONCEPT

In an effort to overcome these difficulties, a program for investigating the bonded bulkhead concept was initiated. The most desirable approach is an all-bonded concept in which all the welding is replaced with bonding. However, no LO_2 compatible adhesive is available at the present time. Therefore, the program was limited to a bulkhead bonded on the hydrogen side only, hence, the one side bonded bulkhead. The basic concept is shown in Figure 8. The aft skin (the LO_2 skin) is identical to a standard S-IV bulkhead. The forward skin is made up in segments which are bonded, rather than welded, together. The joint details for this concept are shown in Figure 9. This design is based on the following four basic considerations:

1. The loads and design criteria will be the same as the S-IV.
2. The design should permit the maximum possible use of existing S-IV tooling. In fact, an S-IV size bulkhead was chosen because the tooling was available and was no longer needed for production use.
3. The bulkhead will utilize the same materials as the S-IV.
4. The finished bulkhead must have the capability of being installed in a S-IV aft dome for testing.

DESIGN

The primary design effort then went into the selection of the joint concept to join the hydrogen side segments, dollar, and attach ring. Three basic types of joints were considered. These types of joints, shown in Figure 10, included all bonded plus mechanical fastener joints and bonded plus mechanical fastener joints with an overseal. Including variations, this study covered approximately 25 different joint concepts. On the basis of a design evaluation, these 25 were narrowed to 3 joints, two bonded and one bonded plus mechanical fastener for specimen testing. The joints selected were joints numbers 3C, 4, and 8A, shown in Figures 11, 12 and 13, respectively. Test specimens were prepared for both tension and compression tests. The following are the five basic conditions from which the loads were derived (all loadings are limit):

1. Ground hold, LO_2 tank at ambient pressure--thermal shock from the start of the LH_2 tank fill, 0.5 psig (reverse).

2. Ground hold, LH_2 at ambient pressure--thermal shock from start of LO_2 tank fill, 0.5 psig internal in LO_2 tank.
3. Ground hold, the LO_2 tank loaded and pressurized--the LH_2 empty and at ambient pressure, 37 psig internal in LO_2 tank.
4. S-I burnout, LH_2 tank pressure minimum--LO_2 tank pressure maximum, 28.5 psig internal in LO_2 tank.
5. Ground hold, LH_2 tank full and pressurized--the LO_2 tank loaded and an ambient pressure. The resulting differential pressure = 23.8 psig reverse.

Condition I results in a tension stress of 72,000 psi with a nominal 0.025 in. thick LH_2 skin; 82,000 psi on a minimum 0.020 in. skin; and 58,600 psi on maximum 0.035 in. skin, or a critical tension load across the joint of 2,051 lb/in. This result takes place while the LH_2 skin and splice are at -423°F. Condition II results in a compression stress of 55,000 psi with nominal 0.025 in. thick LH_2 skin; 56,800 psi on minimum 0.020 in. skin gage; and 49,600 psi on maximum 0.035 in. skin gage, or a critical compression load of -1,736 lb/in. while the LH_2 skin and bond are at +120°F. A typical compression specimen is shown in Figure 14. The results of these tests are shown in Figures 15 and 16 for the tension and compression results, respectively. Included in the figures are the design requirements for the joints for ease of comparison. As these figures indicate, all the joints were satisfactory in tension and the scalloped doubler configuration, 4, and the mechanical fastener doubler configuration, 8A, were satisfactory in compression. The laminated doubler configuration, 3C, was a little under strength at the ambient temperature condition. On the basis of these results then, Configuration 3C was eliminated from further consideration.

To further evaluate the remaining two joint candidates, 3-ft test bulkheads were built to check the joint leak properties. Figure 17 shows one of these specimens. These bulkheads were tested in a test fixture in such a way that the top surface could be immersed in liquid hydrogen and pressurized to the pressures the full-scale bulkhead would experience. The all-bonded joint (4C) had leak rates less than 7×10^{-5} cc's per second of hydrogen after a hold time of 1 hour, which was considered an acceptable leak rate. Because the all-bonded joint concept was also preferable for production, this joint was selected for the full-scale experimental bulkhead.

FABRICATION

The fabrication of the bulkhead proceeded on the basis of this joint concept. Because the aft dome was identical to the production S-IV aft domes and a production type S-IV aft dome was available, the production type S-IV aft dome was used in the fabrication. This aft dome was fitted to the joining tool and cleaned in the standard manner with an etch solution consisting of sulphuric acid and sodium dichromate. An HT 424 prime coat was applied; then the HT 424 adhesive was applied to the prime surface and the HRP honeycomb core was assembled on the aft dome by means of a band of 6 lb/cu ft density honeycomb core 12 in. wide around the greatest circumference, and 4 lb/cu ft density honeycomb core for the remaining surface. The honeycomb was covered with a vacuum bag and bonded to the aft dome in the autoclave under 45 lb/sq in. pressure at 325°F. The bonding procedure up to this point was identical with previous production methods for the S-IV bulkhead except that the 6-lb density core was cut 1 in. wider to allow it to extend 1 in. further down under the forward ring for support of that ring. The other major difference was that the core was a net thickness of 1 in., rather than over-size, to allow for core sculpturing. This core was machined and hand sanded in the forward ring area to produce a proper fit with the forward ring, as shown in Figure 18.

After proper cleaning and adhesive application, the forward ring was bonded to the honeycomb core. Other than the fit of the forward ring to the honeycomb core, there were no sanding or machining operations performed on the core; thus, there was a considerable saving of manhours. Following this, the forward dome segments were individually fitted into place. Because of slight variations in the contour of the aft dome and the honeycomb, some trimming on assembly was performed on the segments to comply with the gap tolerances.

The forward gore segments and the dollar were then cleaned and primed in accordance with the same procedure used for the aft dome. The adhesive was applied and the bonding operation was completed with the same cure cycle used to bond the honeycomb to the aft dome.

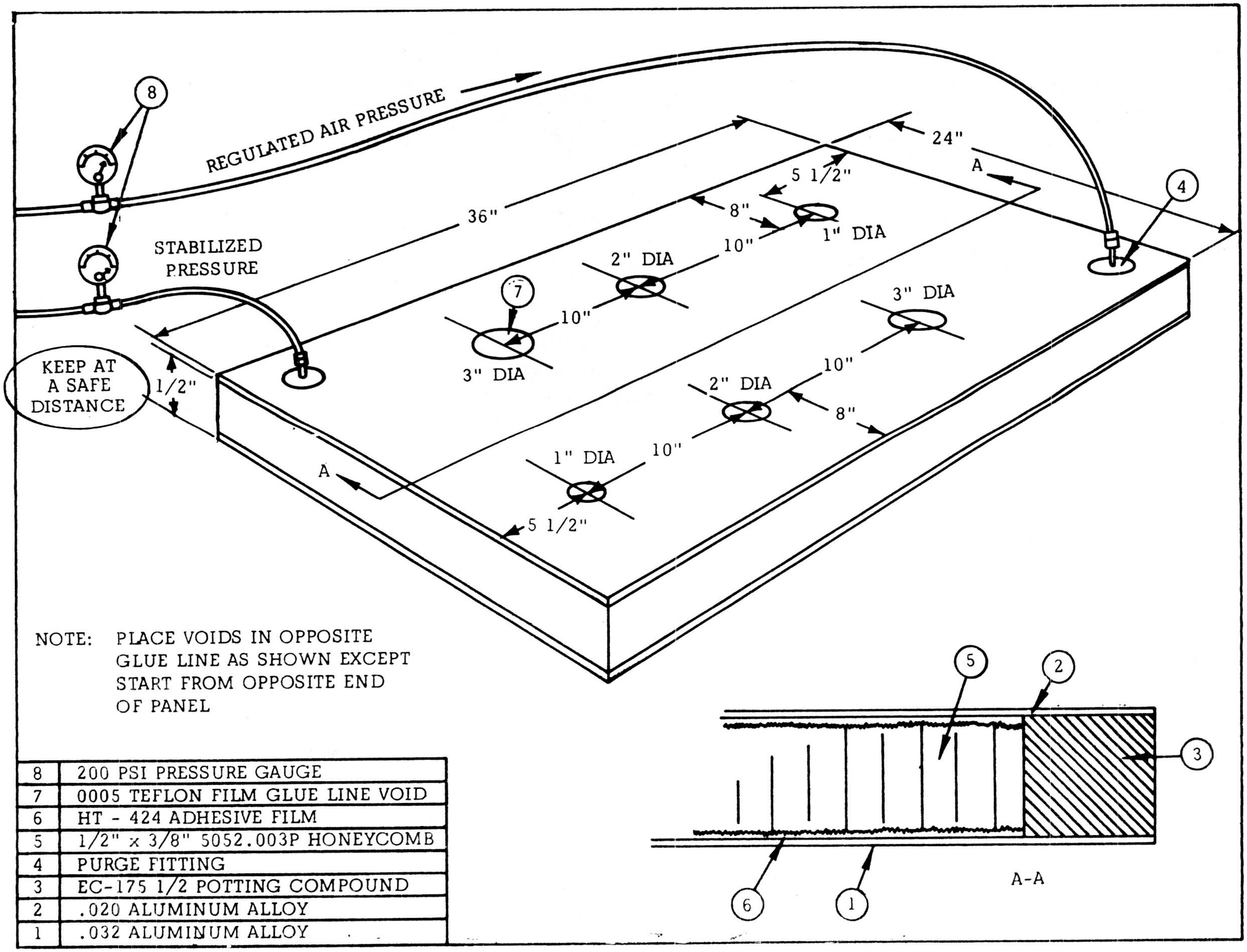
When heating a large composite structure, consideration must be given to the stresses produced in the structure resulting from this uneven heating of the various areas of the structure and the different coefficient of expansion of the materials. In the standard S-IV bonding process, the first bond of the honeycomb core to the aft dome is accomplished in 30 hours, including the heat up, bonding time at temperature, and cool down. The temperature differential between thermocouples during heatup and cooldown is held to a maximum of 10°F.

However, when bonding an integral forward dome core assembly, particularly during cooldown, the two aluminum domes must contract together to avoid creating stresses that might damage the core. Therefore, the temperature differential between thermocouples is held to a maximum of 5°F during this last cycle. This closer tolerance on the temperature spread necessitates a slower cooldown rate and; consequently, this second bonding cycle requires 60 hours. When the forward dome is composed of individual segments such as the experimental bulkhead, these segments are free to expand or to contract as separate parts, reducing the buildup of stresses and allowing, therefore, larger temperature differentials between thermocouples. Under these conditions, the bonding of the forward gore segments and the dollar to the aft dome core assembly of the research bulkhead is accomplished with a bonding cycle of 30 hours duration, thereby effecting a savings of 30 hours of autoclave time.

Figures 19 and 20 show the bulkhead with the forward skin bonded in place. At this point, the only remaining operation was the bonding of the doublers over the joints in the forward dome segments dollar and ring. The doublers were cleaned in accordance with the same procedures used in cleaning the aft and forward domes. The gaps between the segments, however, required sealing to prevent cleaning solution from entering the core area. These gaps were sealed with a room-temperature curing, modified epoxy paste adhesive (Epon 934). This material is resistant to high and low temperatures and to acid. After cleaning, the doublers were bonded over the joints by means of a polyurethane adhesive (Narmco 7343). A vacuum pressure of 10 to 15 in. of mercury was applied after bonding to hold the doublers in place during cure. The cure cycle consisted of 24 hours at room temperature, under vacuum, followed by 24 hours at room temperature, without vacuum, and then 24 hours at 160°F. Subjecting the polyurethane to a temperature of 160°F for 24 hours is required for completing the cure when it is necessary to secure full strength of the adhesive immediately after cure. The adhesive will reach the same strength after approximately 2 months without the elevated temperature cure.

Figures 21 and 22 show the completed bulkhead with the doublers bonded in place. Upon inspection and careful examination of the joint detail shown on Figure 22, it was decided that the joint should be reworked to improve both the fit of the parts and their strength. To accomplish this, the meridian and equatorial doublers were cut approximately 1 in. from the problem area and heat was used to loosen the adhesive; and then the doublers were peeled off. All of the doublers in these affected areas were removed. New ones were made and fitted, then cleaned and bonded in place with the same polyurethane adhesive. In this application, however, the higher temperature cure cycle was not used because it was undesirable to re-heat the entire structure and the full strength capability of the adhesive was not required immediately. The rework of these areas is shown in Figure 23.

The design of the bulkhead calls for a seal weld between the forward ring and the aft ring which not only prevents liquid oxygen or hydrogen from entering the core cavity but also provides a stronger and more rigid structure. In an attempt to follow this procedure on the DA-103 bulkhead, it was discovered that during the core-bonding operation, some of the adhesive had migrated into the weld area in several places, thereby making a complete seal weld impractical because of the impossibility of securing adequate welding cleanliness. Therefore, the two rings



NOTE: PLACE VOIDS IN OPPOSITE GLUE LINE AS SHOWN EXCEPT START FROM OPPOSITE END OF PANEL

8	200 PSI PRESSURE GAUGE
7	0005 TEFLON FILM GLUE LINE VOID
6	HT - 424 ADHESIVE FILM
5	1/2" x 3/8" 5052.003P HONEYCOMB
4	PURGE FITTING
3	EC-175 1/2 POTTING COMPOUND
2	.020 ALUMINUM ALLOY
1	.032 ALUMINUM ALLOY

FIGURE 1. PANEL ASSEMBLY DETAILS

FIGURE 2. STRESSCOAT PATTERN OVER 3" DIA. SKIN VOID (12 PSIG INTERNAL PRESSURE) .020" FACING SHEET.

FIGURE 3. STRESSCOAT PATTERN OVER 3" DIA. CORE VOID. (12 PSIG INTERNAL PRESSURE) .020" FACING SHEET.

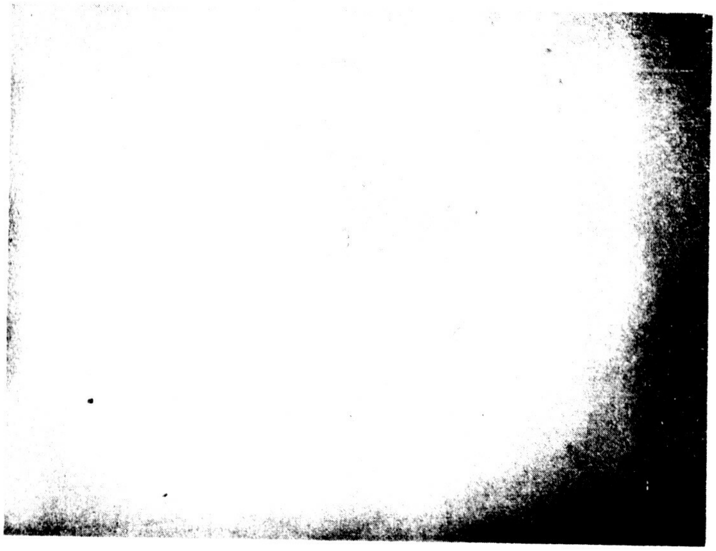


FIGURE 4. STRESSCOAT PATTERN OVER 2" DIA. SKIN VOID (12 PSIG INTERNAL PRESSURE) .020" FACING SHEET.

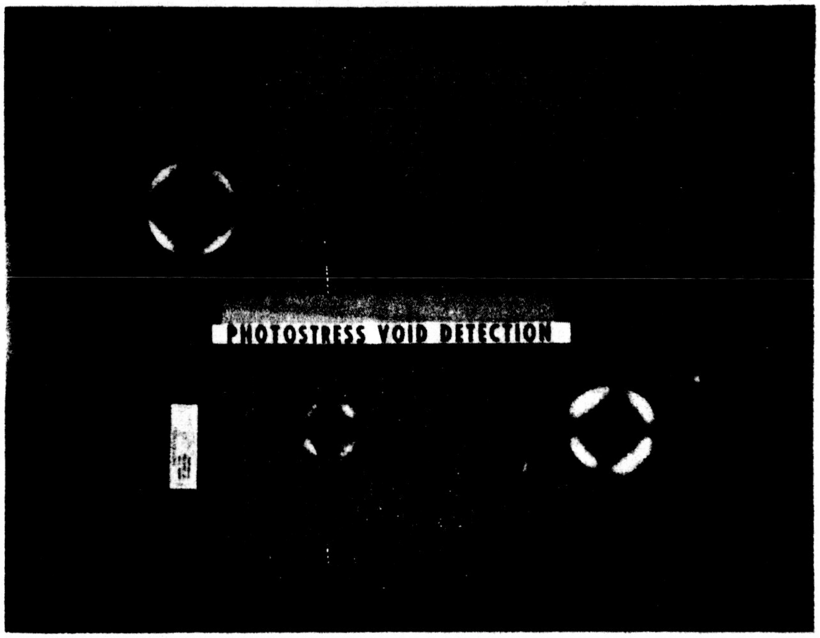


FIGURE 5. VOID INDICATIONS IN PHOTOELASTIC COATINGS; 10 PSIG INTERNAL PRESSURE

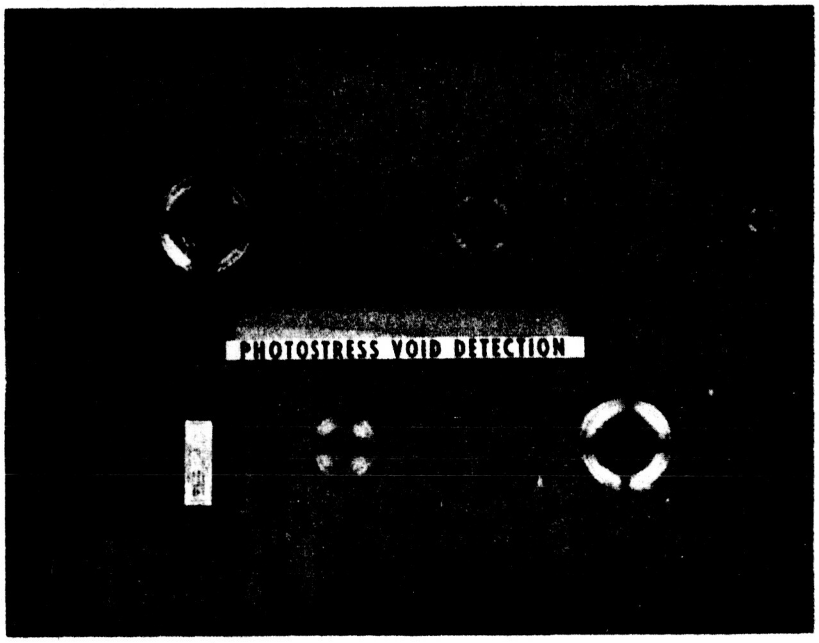


FIGURE 6. VOID INDICATIONS IN PHOTOELASTIC COATINGS; 20 PSIG INTERNAL PRESSURE

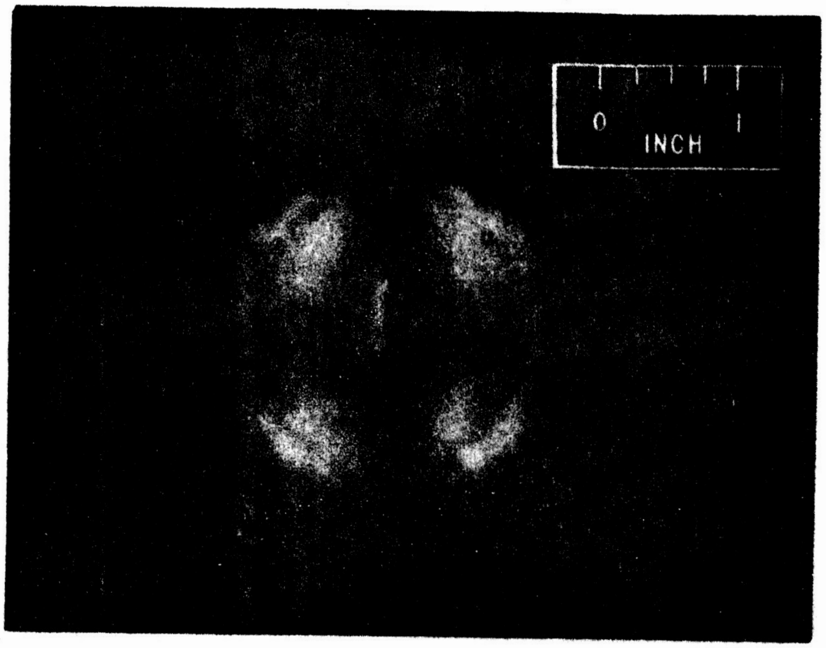


FIGURE 7. 2" DIAMETER VOID; 20 PSIG.
BONDED FLEXIBLE SHEET

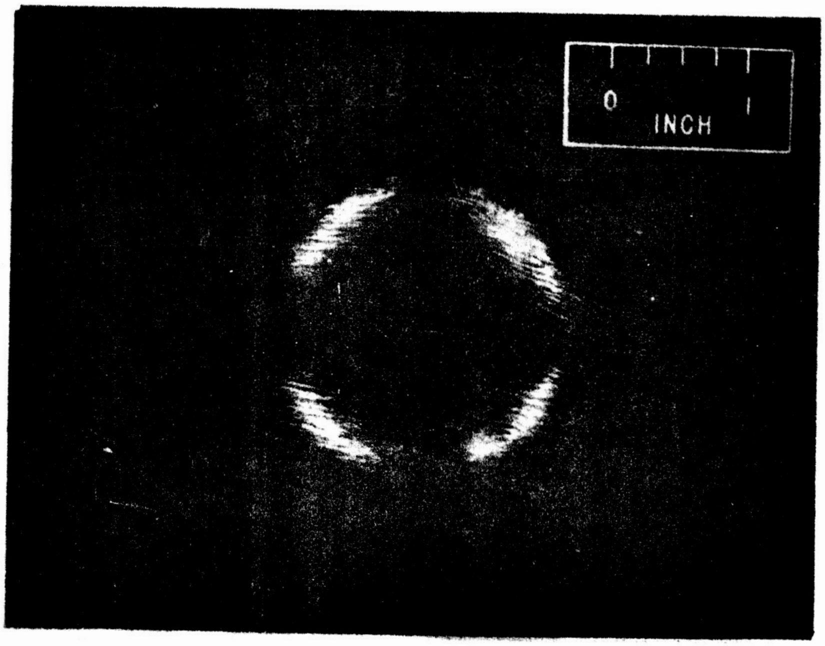


FIGURE 8. 2" DIAMETER VOID; 20 PSIG.
CAST RIGID FILM

were tack-welded at locations approximately 2 ft apart around the periphery to achieve the strength for handling and then were sealed with PR 1938, a silicone sealant.

As noted earlier, one of the requirements of the program was that the experimental bulkhead must be designed so that it could be installed in an S-IV aft dome for structural and functional tests. Because the bulkhead geometry and the attach rings (that attach the bulkhead to the aft dome) were essentially identical to production S-IV domes, this introduced no particular problem. The method of attachment, with huckbolts and welding, was also the same. However, prior to welding the bulkhead into the aft dome, a question arose concerning the possibility of deteriorating the polyurethane adhesive during the welding of the forward ring of the bulkhead to the aft dome as a result of the proximity of the adhesive and the heat from the welding operation. Strength tests were performed on lap shear coupons bonded with polyurethane adhesive and subjected to temperatures of 200°F, 250°F, 300°F, and 400°F for a period of 5 min. before testing at room temperature. The strength of these coupons remained constant through the 300°F exposure but fell sharply when exposed to 350°F and 400°F; consequently, 300°F was set as the critical limit. However, during the welding operation, as determined by temperature indicating materials, the temperature did not exceed 250°F.

INSPECTION

Nondestructive testing techniques were utilized throughout fabrication to ensure the quality of the finished product. Sonic (Growler) tests were performed on the gore segments to detect any unbonds between the aluminum domes and the honeycomb core; none were detected. The bond quality was as good or better than any production bulkheads. Both ultrasonic, pulse-echo tests and quality control test coupons were used to inspect the doubler bonding operations. Test coupons were processed with each doubler bond and pull tested to ensure the quality of the polyurethane adhesive. Upon completion of the doubler bonding operation, ultrasonic pulse-echo tests were performed on all the doublers. The calibration of the equipment for both the sonic and the ultrasonic inspections was accomplished by referencing to a standard containing an unbond of known size. The ultrasonic inspection of the doublers indicated approximately 80 areas of small unbonds along the meridian and circumferential doublers. These areas of unbond were scattered and did not individually exceed 1/2 in. in diameter. As a part of the rework of the doubler joints at the meridian-circumferential intersection described earlier, these ultrasonic inspection results were compared with the unbonds in the removed parts. The actual unbond size was found to be, in all cases, smaller than the indicated size. This exaggeration of size frequently occurs when the unbond area is smaller than the size of the ultrasonic transducer used in the test. The removal of the doublers did, however, reveal another problem. On some of the removed parts, there were small channels in the adhesive, most of them isolated, but some connected and occasionally extending to the edge of the doubler. The small size of these channels prevented them from being detected by the ultrasonic inspection procedure and although there would be no significant loss of strength as a result of these channels, they could conceivably serve as leak paths for liquid hydrogen into the interior of the bulkhead.

It would appear that the more costly and sophisticated ultrasonic C scan recording or infrared detector techniques could be employed to detect these small channel unbond areas. Both of these techniques have the capability of providing a tight scan recording of the entire part. The use of such equipment would be recommended for use on production bulkheads. For the experimental bulkhead, a seal coat of polyurethane adhesive was applied over all the doubler seams to seal these channels. This coat was left uncovered and was cured at room temperature.

In retrospect, throughout the entire bonding operation there were only three problem areas of any consequence: (1) the channeling of the polyurethane of adhesive, (2) the misfit of the doublers, and (3) the migration of the HT 424 foam into the seal weld area. These problems appear to be easily resolved in the future. The channeling of the polyurethane adhesive appears to be caused by entrapping air under the doubler when it is laid down on the adhesive, and it may be aggravated by the vacuum used to hold the parts together during cure. This condition could be resolved by the use of modified adhesive application techniques and perhaps by the use of weights or clamping pressure to hold the doublers in place instead of the vacuum pressure. The misfit of the doublers is merely a matter of proper production tooling. The migration of the HT 424 foam into the seal weld area could be resolved either by the use of a more thixotropic material in place of the foam or by the insertion of a blocking part to prevent the foam migration into the weld area.

RESULTS AND CONCLUSIONS

Since the structural tests have not been performed, it is not possible to draw final conclusions on this bonded bulkhead concept. However, on the basis of the work accomplished to date, certain preliminary results and conclusions regarding the fabrication, rework, weight, and cost of this concept indicate that the bulkhead has proved to be much easier to fabricate than the standard production design; potential problems and weight are reduced, and a considerable cost savings might be realized.

Significant advantages of this simplified and improved fabrication are as follows:

- Probably the most significant factor is the elimination of the core sculpturing. As noted earlier, the core sculpturing was eliminated and still the bulkhead sonic inspection showed excellent bond line fit and bond.

- In addition, the tolerances on the gore and dollar trim dimensions were relaxed. The gap tolerance (between parts) on the S-IV welded segments is 0 to 0.015 in. and the gap tolerance on the bonded segments is 0 to 0.090 in., which simplifies considerably the trim and fitup of the parts.

- Furthermore, this concept has the potential of eliminating the chem milling of the gores and dollars. This was not accomplished in the experimental bulkhead because aluminum sheets in the required thicknesses (0.025-0.032) are not available in the widths required (120 in.). Thicker sheets available in the required thickness were used and chem-milled to the design thickness. However, by increasing the number of segments and thereby reducing the sheet widths required, the chem milling could be eliminated.

- On the basis of the experimental fabrication, it would appear that the skin wrinkling problem has been solved although, admittedly, if the wrinkling is to occur, it probably will appear during cryogenic tests.

- The bulkhead is simple to rework, as demonstrated by the rework of the meridian equatorial doublers. The doublers can be removed by simply heating them, with a portable heater, and peeling them off.

- The weld cracking problems are obviously reduced because there are fewer welds in the bulkhead and in the ultimate, all-bonded concept there are no welds at all.
- Because the bonded joint is lighter than the equivalent welded joint, the bulkhead weight has been reduced. For the research bulkhead, it is approximately 25 lb lighter than a standard bulkhead, and for an all-bonded bulkhead, the weight saving would be of the order of 50 lb.
- The experimental bulkhead fabrication process also suggests that considerable cost savings might be realized. For example, the manhours used in the bonding operation of the research bulkhead were approximately 50% of the hours used in the bonding operation of a production bulkhead. This is particularly significant when it is realized that the bonding operations for the experimental bulkhead actually included the bonding of the doublers, over and above the normal honeycomb to skin bonding done on production bulkheads. To this saving could be added the saving in welding manhours. These have not been estimated.

The manufacturing of the bulkhead is complete. At the present time, the bulkhead is being prepared for structural testing. If the bulkhead does, in fact, perform structurally as it is designed to do, the program will have demonstrated a fabrication technique that would appear to offer significant advances in the fabrication of composite structures of this type.

SATURN IB SECOND STAGE

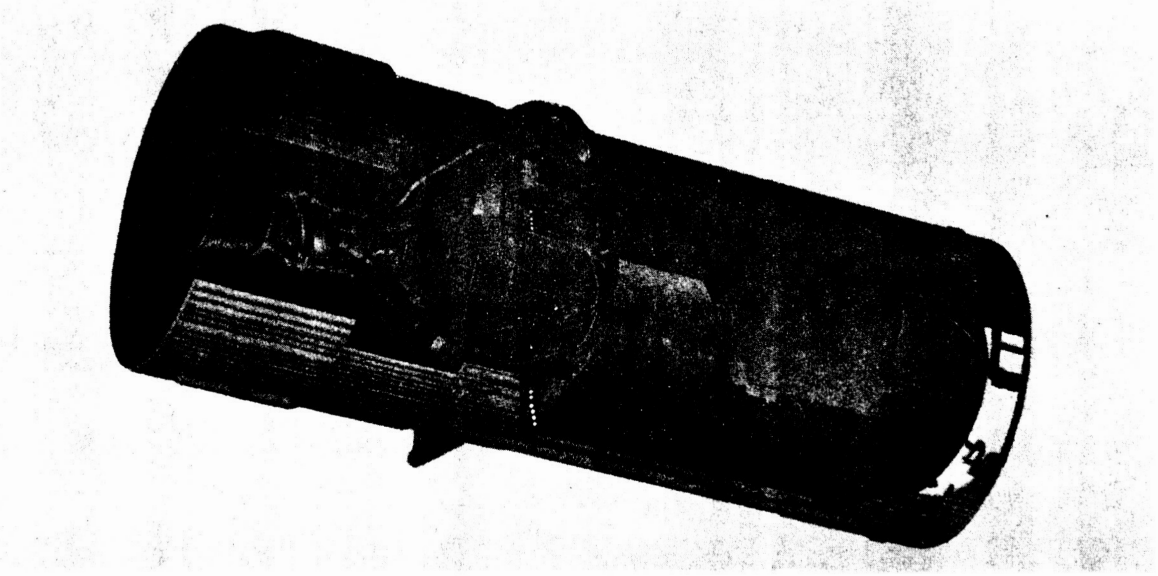


FIGURE 1

M-19561

S-IV BULKHEAD COMPONENTS

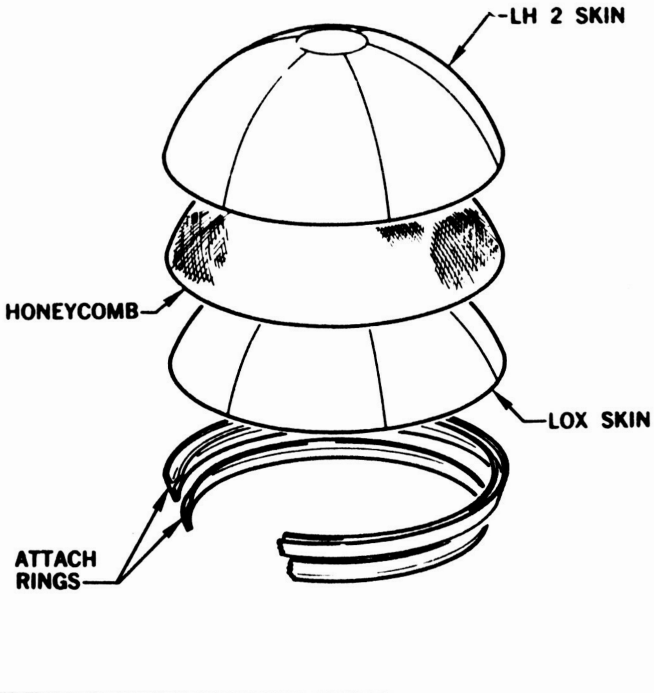


FIGURE 2

M-19558

S-IV JOINT DETAILS

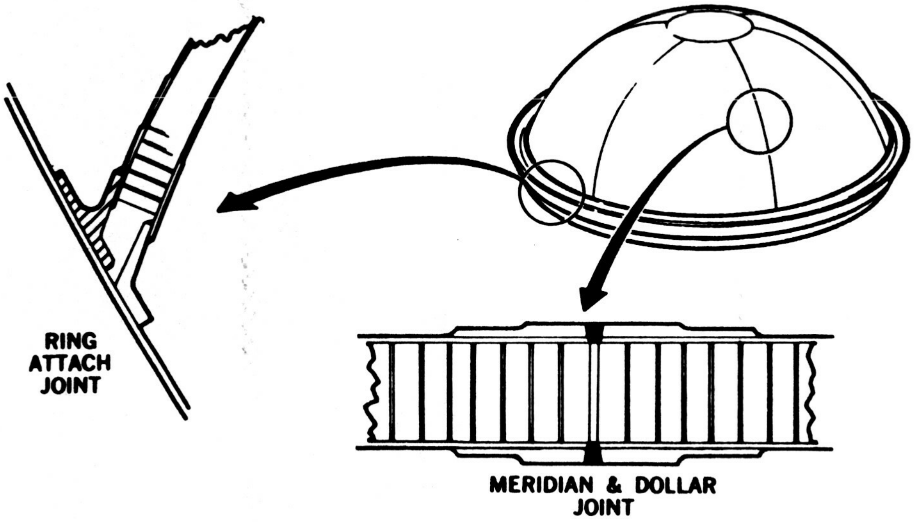


FIGURE 3

FITTING THE AFT DOME

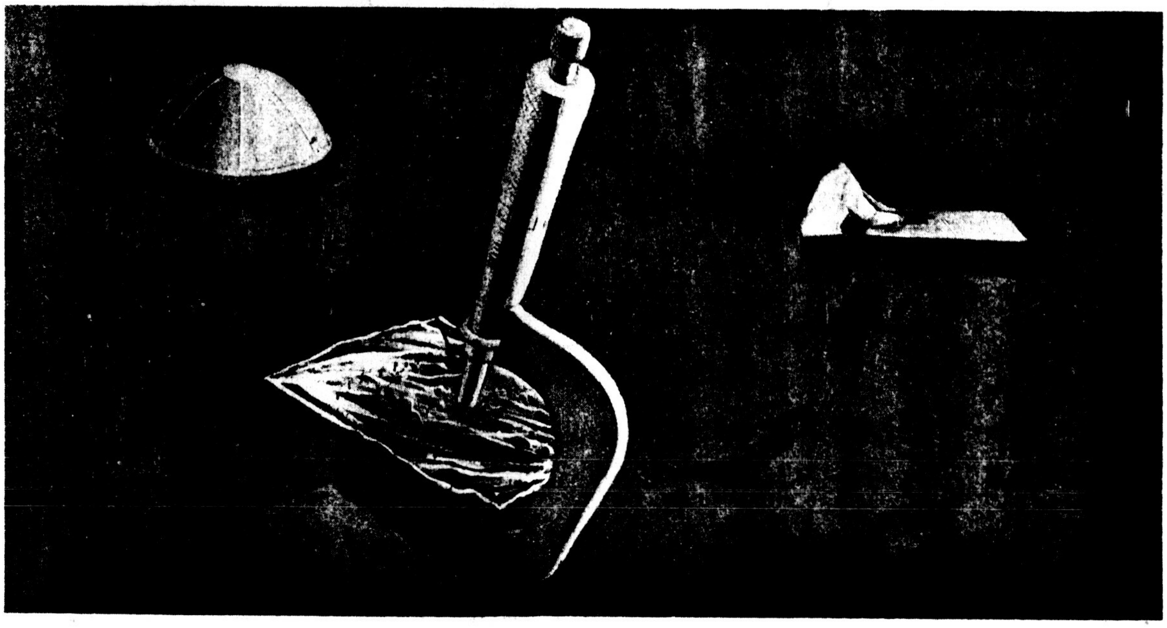


FIGURE 4

CORE BONDED TO AFT DOME

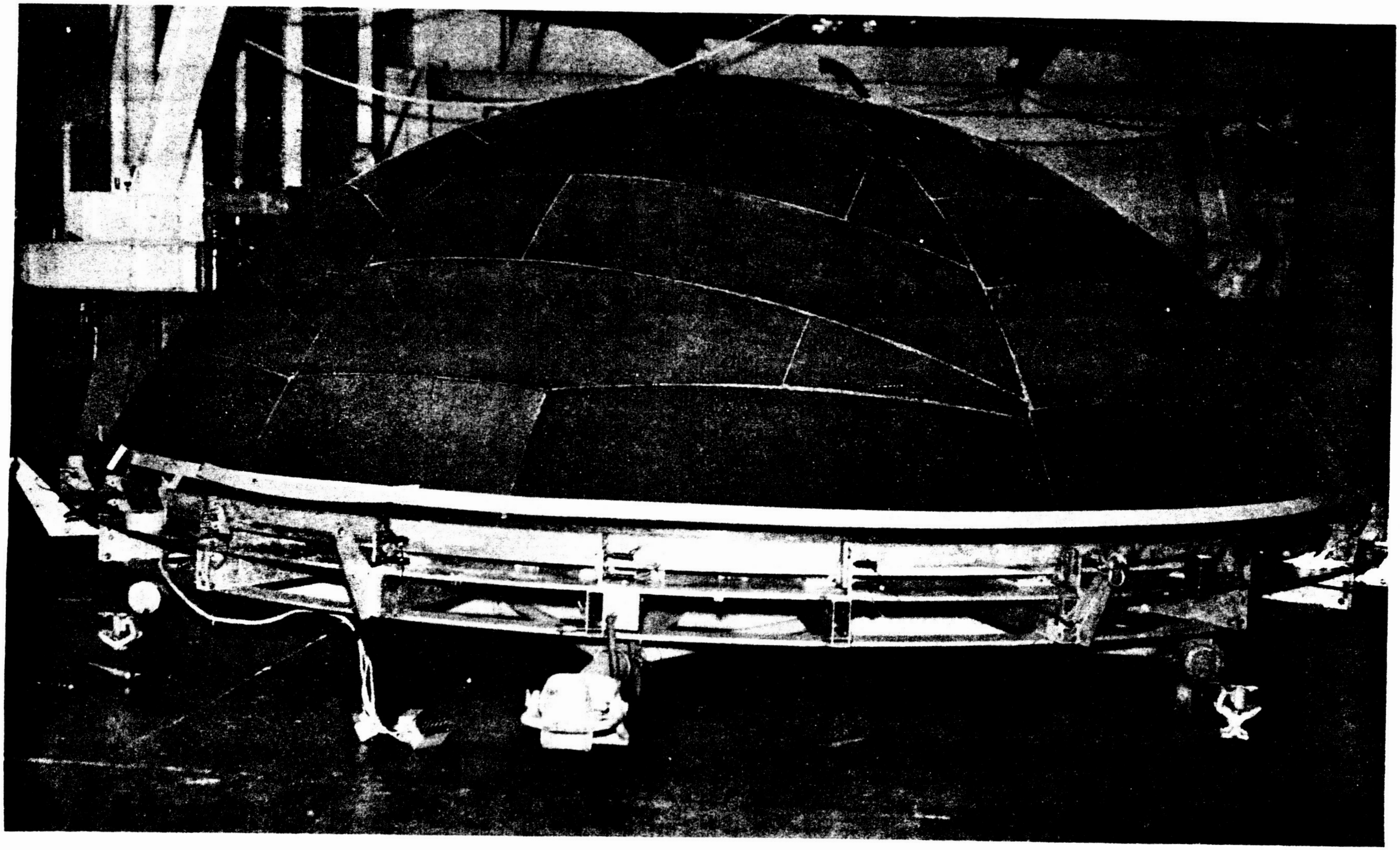


FIGURE 5

SPOT FACE SAND AND FIT CHECK FORWARD BULKHEAD

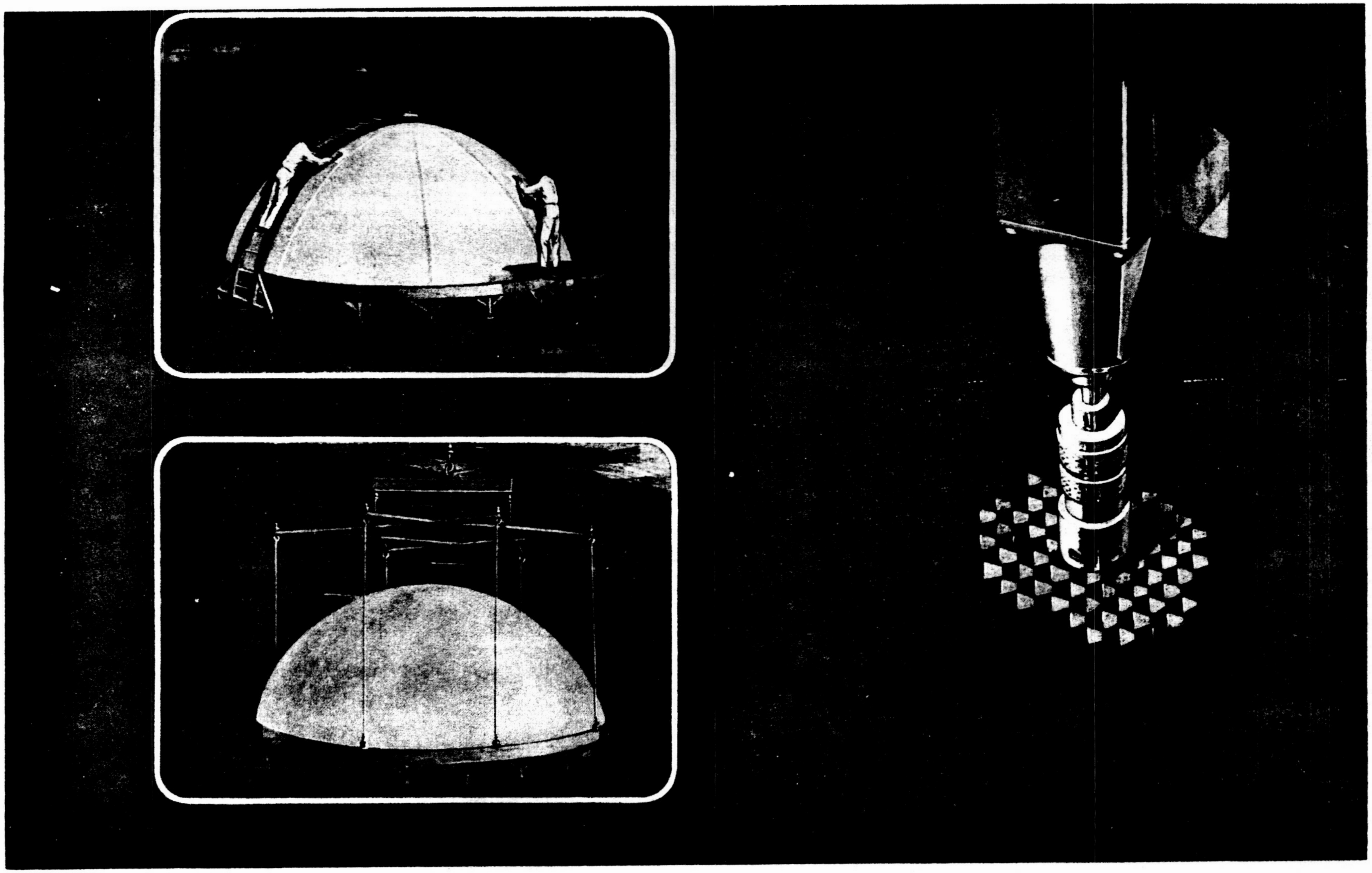


FIGURE 6

M-32087

EFFECT OF BOND CYCLE ON WELD DUCTILITY

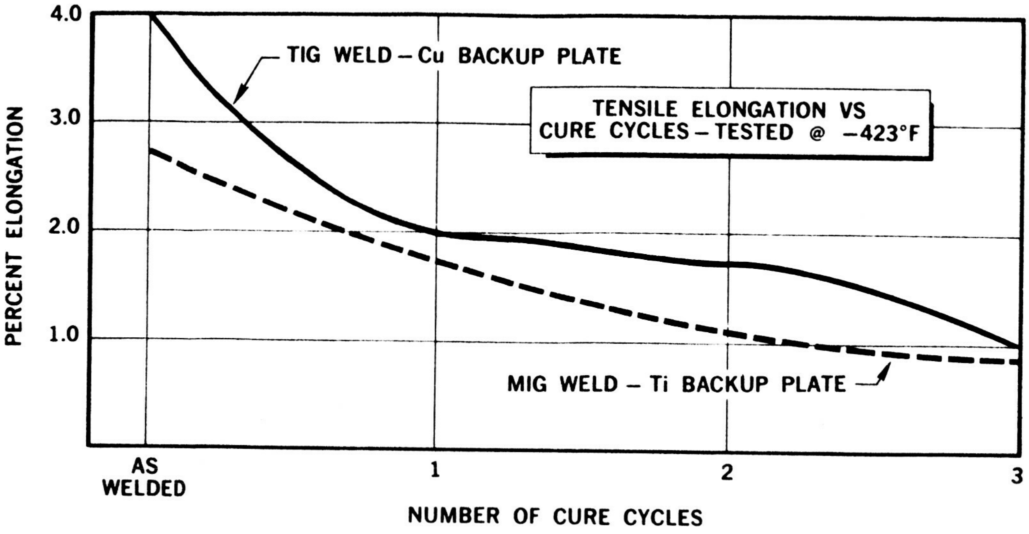


FIGURE 7

M-19559A

NEW BULKHEAD COMPONENTS

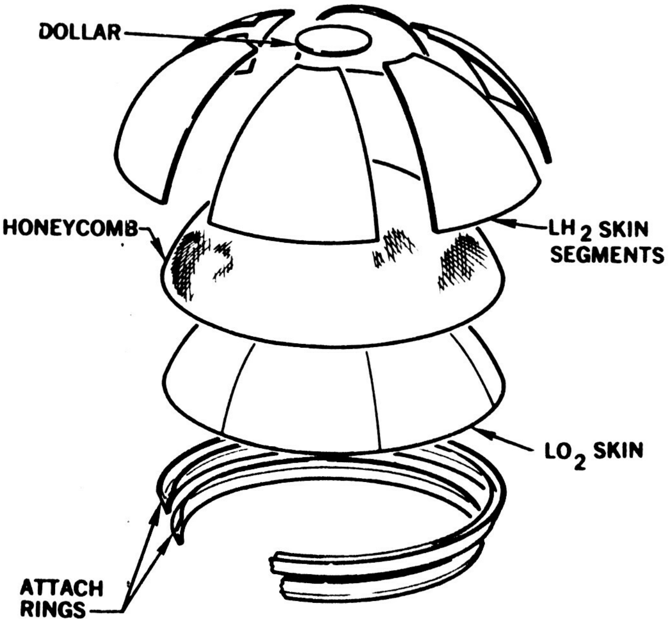


FIGURE 8

M-19560

NEW BULKHEAD JOINT DETAILS

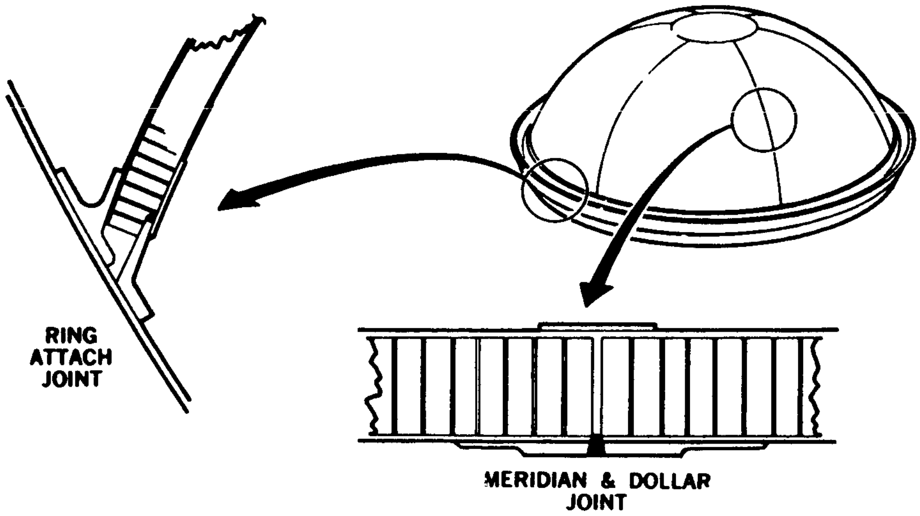


FIGURE 9

M-19556A

TYPES OF JOINTS

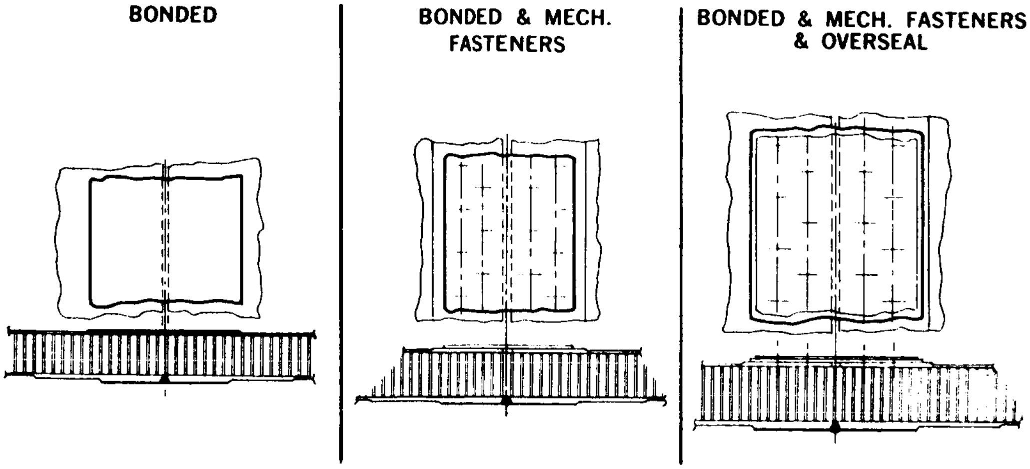
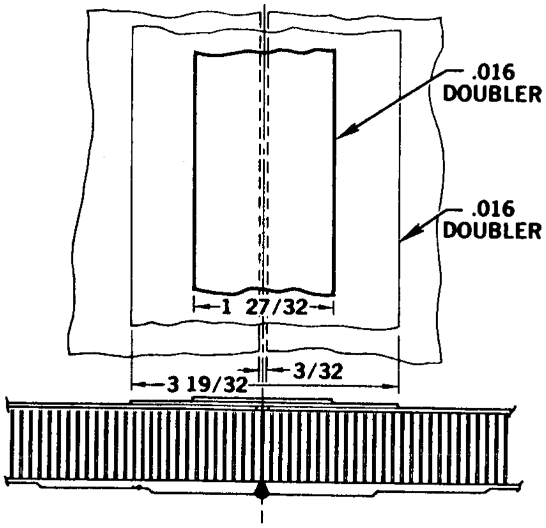


FIGURE 10

M-19557

CONFIG. 3C

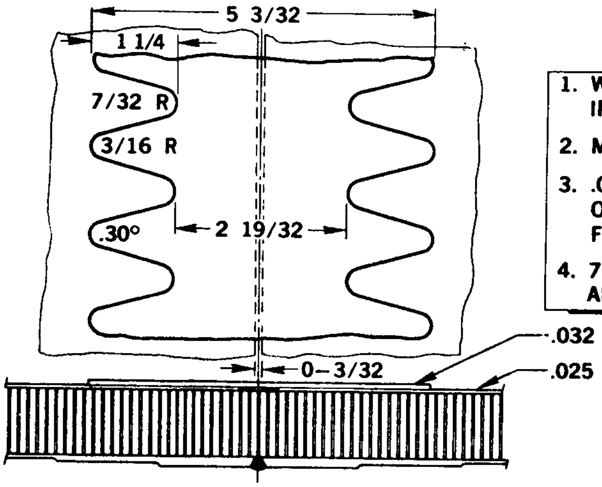


1. WEIGHT FACTOR = .230
2. MARGIN OF SAFETY = 24%
3. .016 DOUBLER + .016 DOUBLER OVER .025 SKIN. DOUBLERS TO BE INSTALLED FLAT (NO FORMING).
4. 7343/7139 ADHESIVE FOR STRENGTH AND SEAL.

FIGURE 11

M-19544

CONFIG. 4

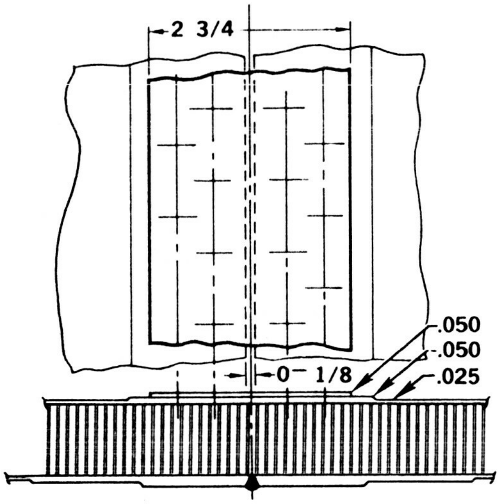


1. WEIGHT FACTOR = .315 IF .032; .395 IF DBR IS .040
2. MARGIN OF SAFETY = 28%
3. .030 OR .040 FINGERED DOUBLER OVER .025 SKIN, DOUBLER TO BE FORMED TO SPHERICAL RADIUS.
4. 7343/7139 ADHESIVE FOR STRENGTH AND SEAL

FIGURE 12

M-19546

CONFIG. 8A



1. WEIGHT FACTOR = .8
2. MARGIN OF SAFETY = 19%.
3. 5/32 A-286 STAINLESS STEEL FLUSH BLIND BOLTS WITH JACKETS.
4. .050 DOUBLER OVER .050 STEPPED LANDING ON .025 SKINS.

   DOUBLER TO BE FORMED TO SPHERICAL RADIUS.
5. 7343/7139 ADHESIVE ON FAYING SURFACES FOR SEAL.

FIGURE 13

COMPRESSION TEST PANELS

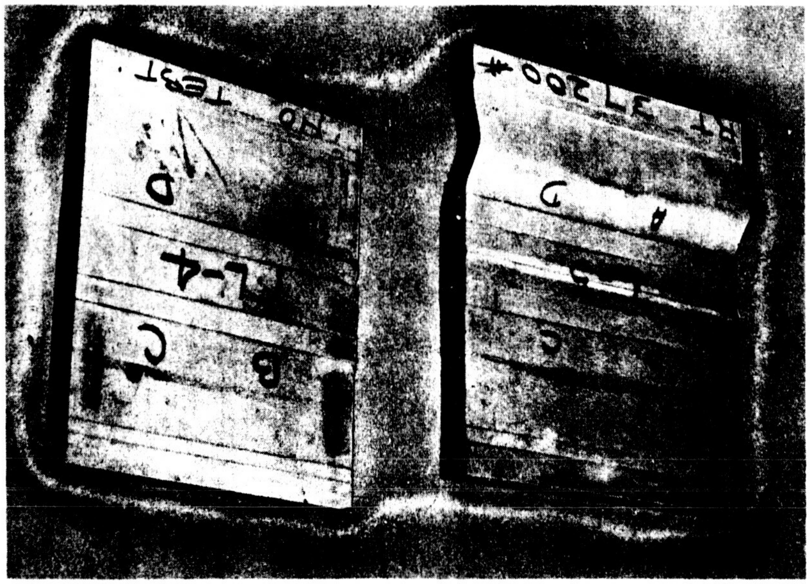


FIGURE 14

M-22156

TENSION PANEL TEST CURVES

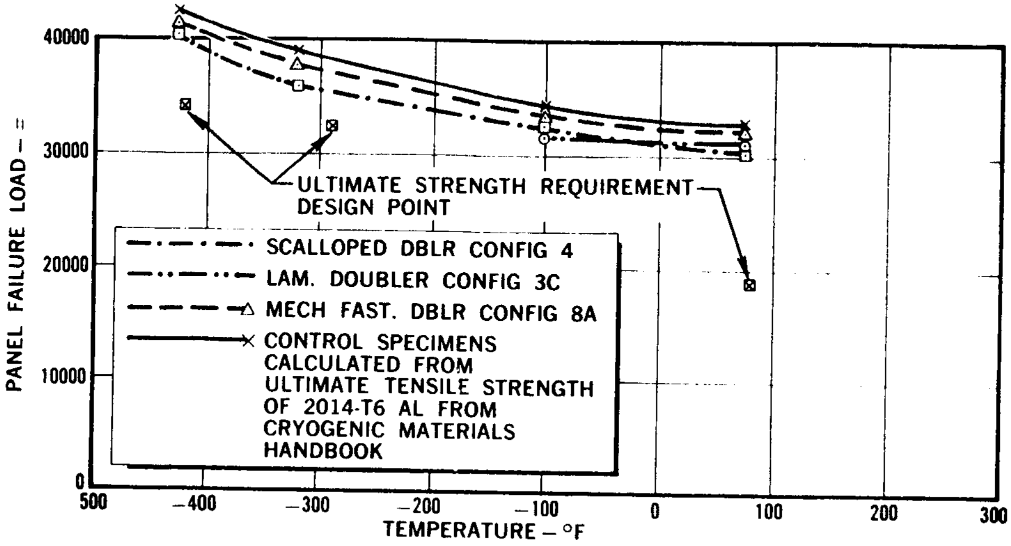


FIGURE 15

M-22157

COMPRESSION TEST PANEL CURVES

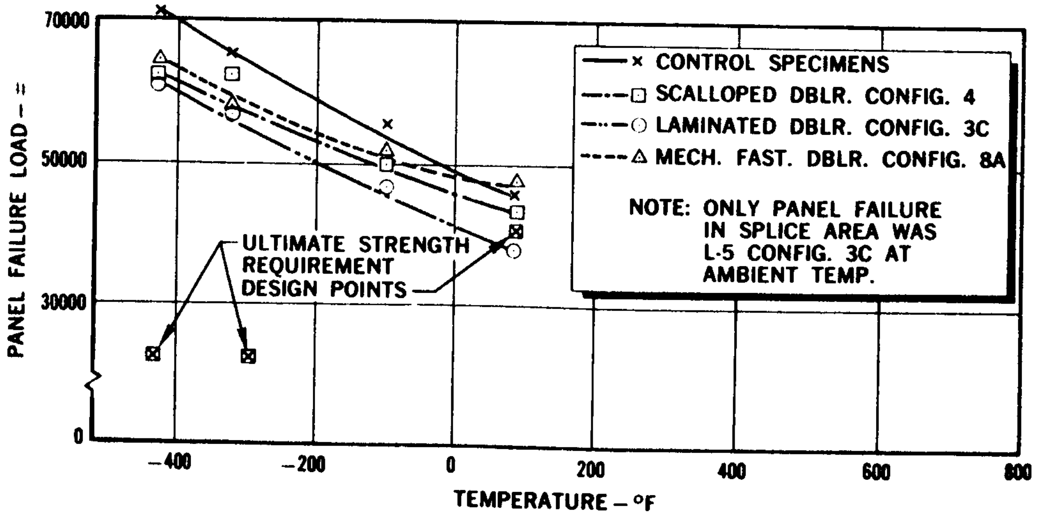


FIGURE 16

SUBSCALE BULKHEAD

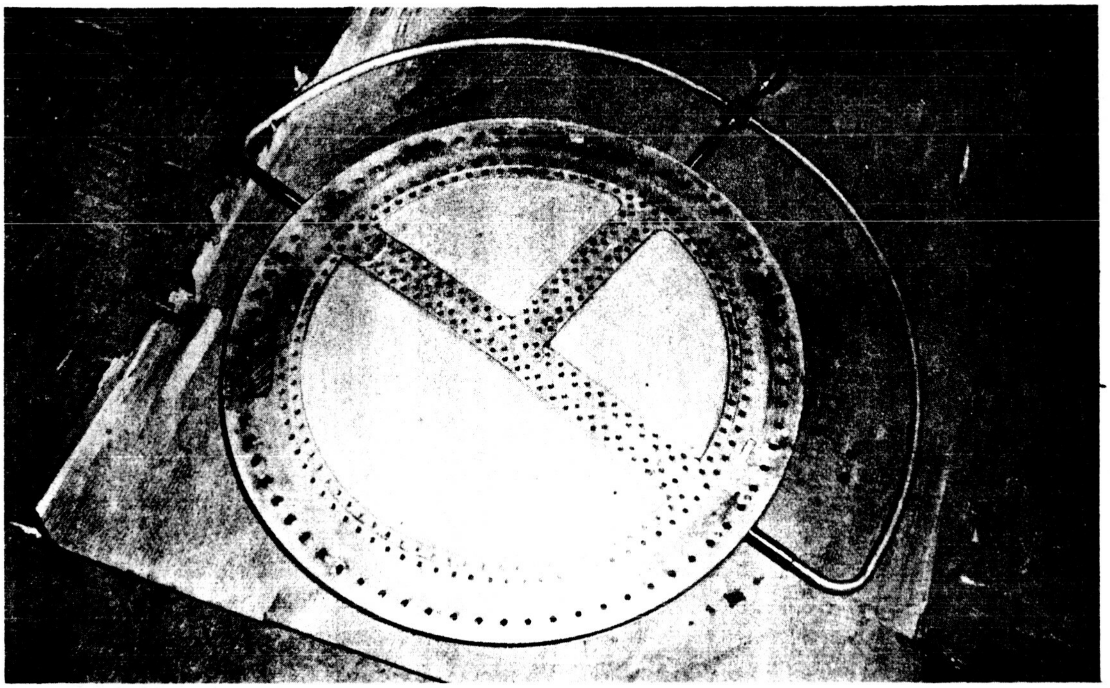
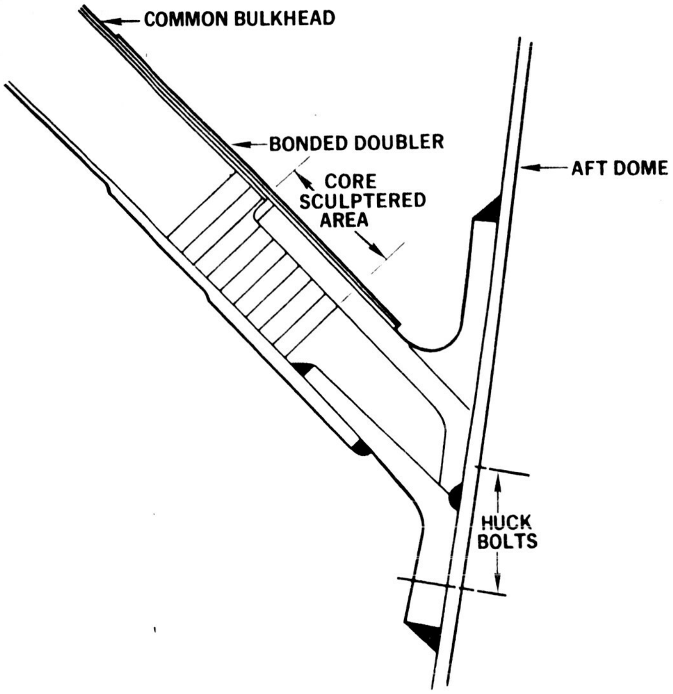


FIGURE 17

M-32104



RING ATTACH JOINT DETAIL

FIGURE 18

FORWARD SKIN BONDED IN PLACE

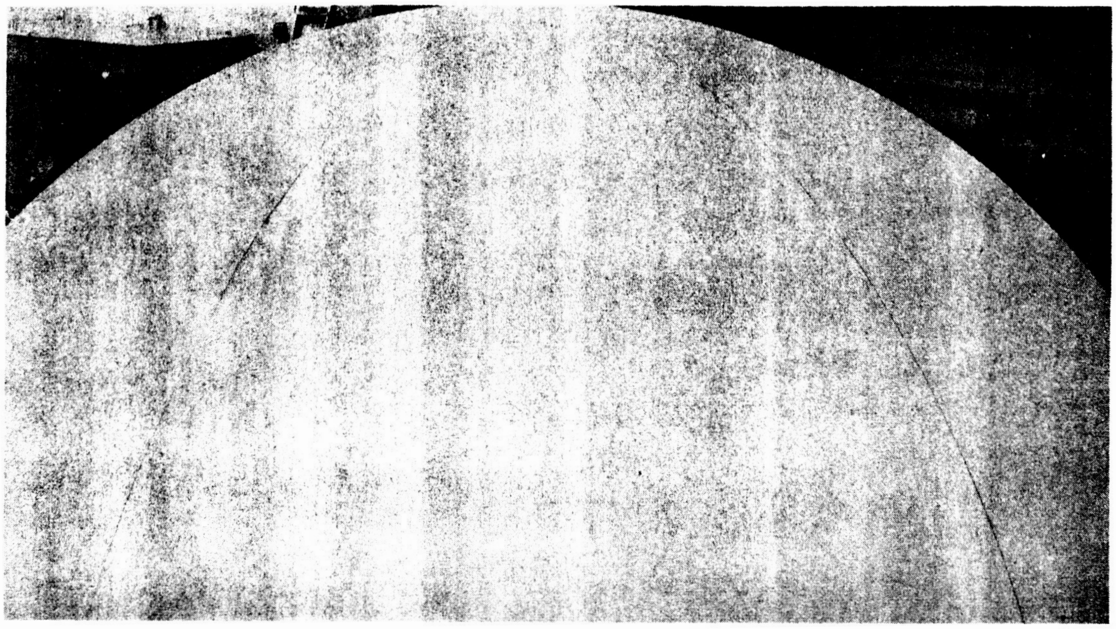


FIGURE 19

FORWARD SKIN
BONDED IN PLACE

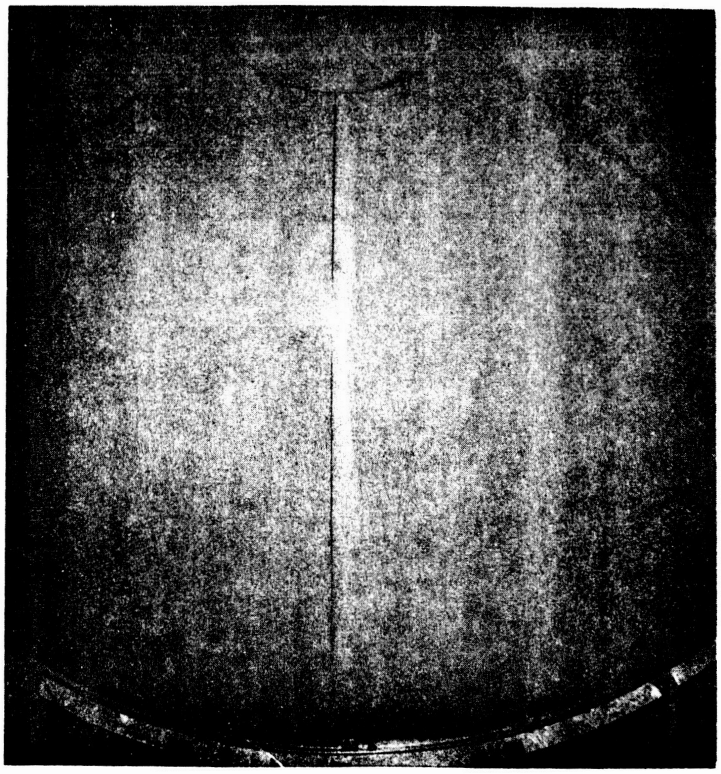


FIGURE 20

COMPLETED BULKHEAD

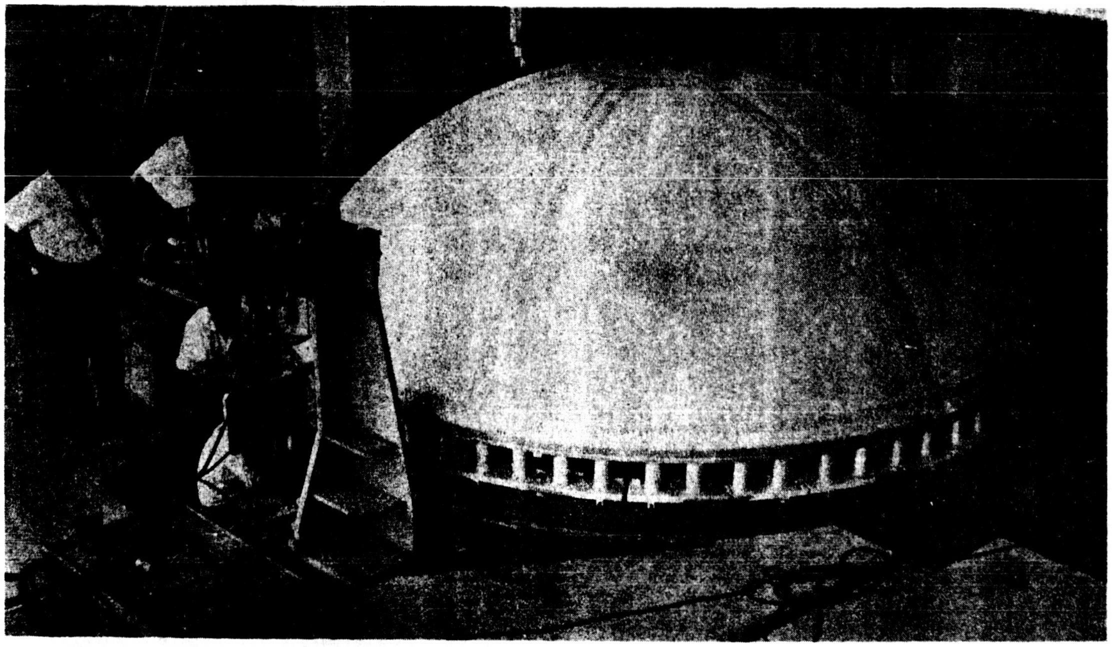


FIGURE 21

COMPLETED BULKHEAD JOINT DETAIL

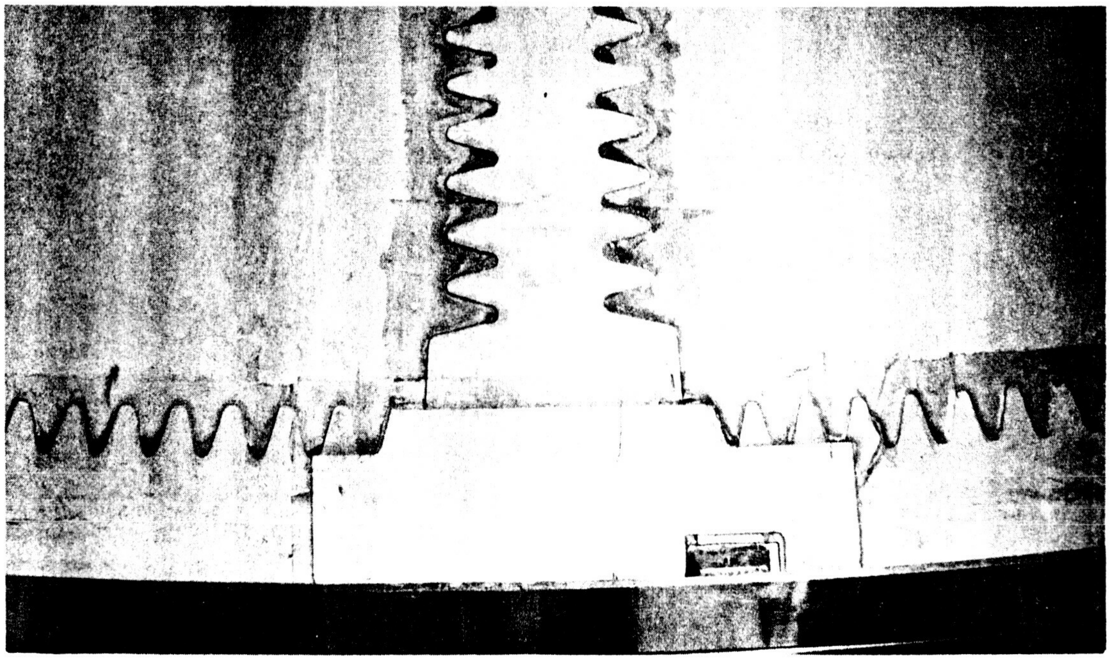


FIGURE 22

REWORK JOINT DETAIL

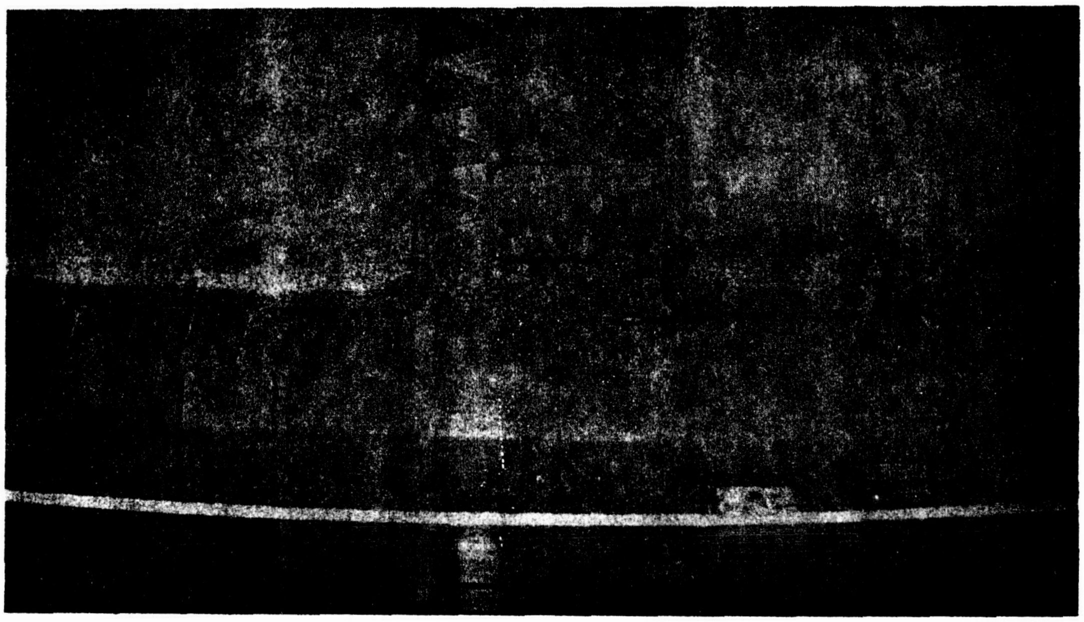


FIGURE 23

M-32088

RESULTS & CONCLUSIONS

- SIMPLIFIED & IMPROVED FABRICATION
  - ELIMINATE CHEM MILLING
  - TOLERANCES ON TRIM DIMENSIONS RELAXED
  - ELIMINATE CORE SCULPTURING
  - IMPROVED HONEYCOMB-SKIN BONDING
- NO SKIN WRINKLING
- REDUCED WELD CRACKING
- REDUCED WEIGHT 25 LBS
- SIMPLE REWORK
- REDUCED COST

FIGURE 24

SESSION IV - PAPER 1.

NON-DESTRUCTIVE TESTING OF BONDED STRUCTURES WITH LIQUID CRYSTALS

10:40 - 11:20 A. M.

WEDNESDAY - MARCH 16, 1966

N66 34137

NONDESTRUCTIVE TESTING OF BONDED STRUCTURES WITH LIQUID CRYSTALS

Presented by
LaMarr Sabourin
Research Engineer
Quality Control Research and Development Group
Quality Control Department
Commercial Airplane Division

THE BOEING COMPANY

Acknowledgment is expressed to Dr. Wayne Woodmansee of The Boeing Company for his assistance in preparing this paper and his work on liquid crystals.

Liquid crystals are a group of compounds that exhibit, in the liquid state, optical properties normally associated with solids.

Although their existence has been known for more than seventy years, substances that exhibit the liquid-crystal phase have until recently been regarded more as laboratory curiosities than as potentially useful or theoretically important objects of study.

In almost every way a crystal would seem to be the very opposite of a liquid. The molecules in a liquid are not arranged in any order that extends over a distance greater than a few molecules. The molecules in a crystal, on the other hand, are fixed in a regular three-dimensional array. Yet liquid crystals - substances that share some of the properties of both liquids and crystals - do exist. What is more, they cannot be considered rare, since they account for at least one out of every two hundred organic compounds synthesized in the laboratory.

Liquid crystals may be divided into nematic, smetic, and cholesteric mesophases. However, the cholesteric materials have the most obvious application in thermal nondestructive testing as a result of their unique optical properties. They are called cholesteric because their molecular structure is characteristic of a large number of compounds that contain cholesterol. (Cholesterol by itself does not have a liquid-crystal phase). The most striking optical characteristic of cholesteric substances is a response to subtle changes in thermal environment that produces variations in color. Although cholesteric substances are colorless as isotropic liquids, they pass through a series of bright colors when they are cooled through their liquid-crystal phase. In this phase they may first appear to be violet, then blue, then green, then yellow, then red, and finally are colorless again as the reflection maximum enters the infrared region.

Our work at Boeing on liquid crystals has been done with cholesterol esters. By varying the amounts in a mixture of the esters, it is possible to vary the temperature at which the color change will occur. The color-temperature behavior of some of the mixtures we have investigated is shown in Fig. 1. Mixtures A, B, and C are typical of materials employed for bond testing. Fig. 2 shows light scattering as a function of single cholesterol ester structure.

We have developed mixtures that change from red to blue over the range 29 to 30°C for testing bonded test panels, and mixtures that change from red to blue over a 4° range (24 to 28°C) at the low end of the series to some that change over a 2° range (37 to 39°C) at the high end. These materials have been developed to enable visualization of thermal gradients on electronic components, human skin, in display systems, and in other applications where the earlier mixtures were too sensitive.

The advantages of these materials as temperature indicators are:

1. Speed of response. Color changes may be observed in less than one second.
2. Reversibility. The color transition may be noted as the material is heated or cooled.
3. Reproducibility. A given mixture will exhibit the same color at the same temperature.

In order to visualize the colors associated with these materials it is necessary to paint liquid crystals on a dark background since the colors arise because of reflection rather than absorption of light. A water soluble black paint is used for the background. The crystal mixture is brushed or sprayed over the dark background of the part to be tested. Approximately one gram of these mixtures will cover one square foot to a thickness of about ten microns, which is sufficient to produce an accurate color indication. If less than ten microns is applied, colors will appear in an irregular pattern quite different in appearance from the colors obtained with a real temperature gradient. If liquid crystals have been left on the surface for some time, the colors will appear to be dull. The intensity of the colors may be restored by warming the surface and brushing the crystals.

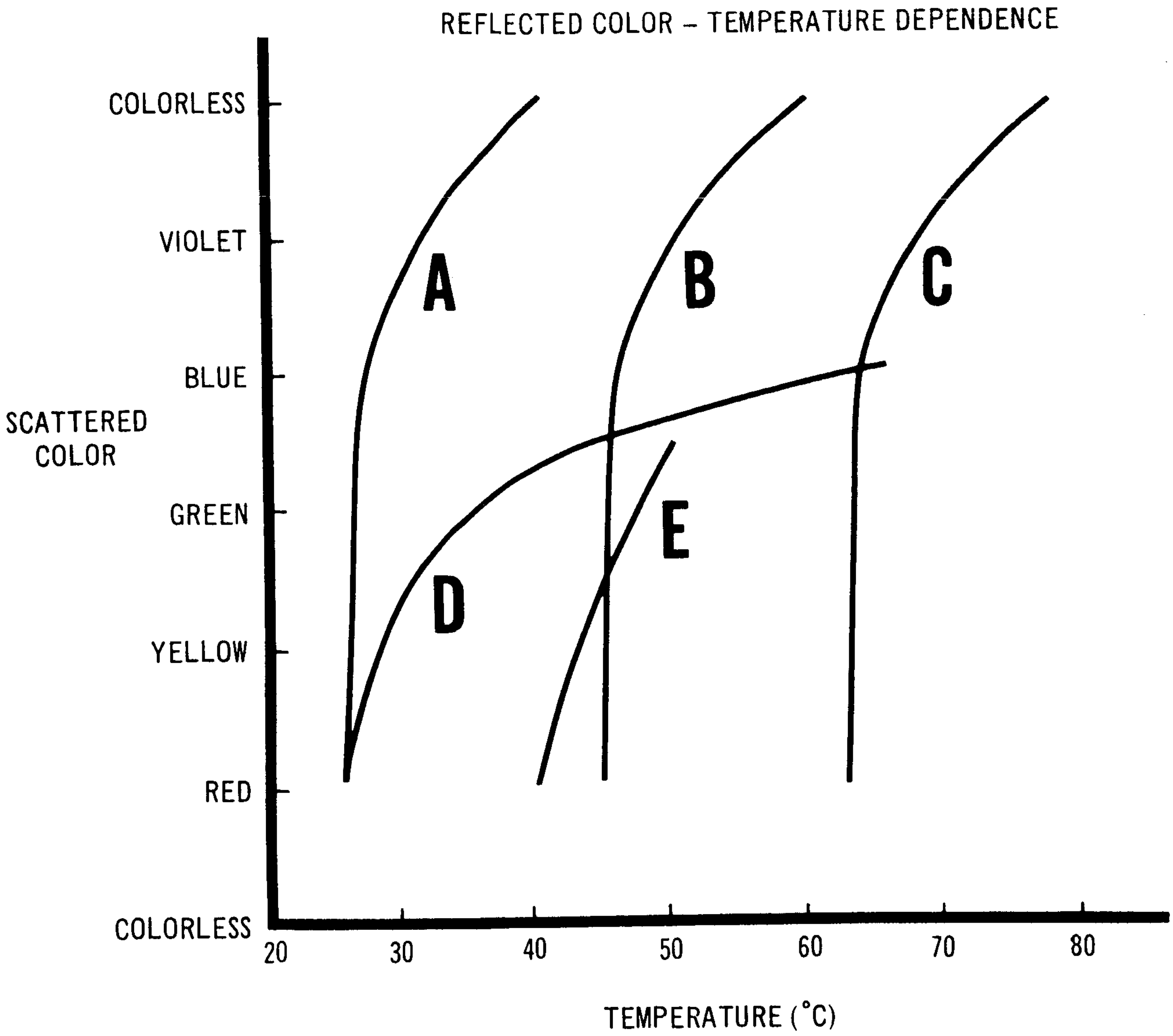


FIGURE 1 TEMPERATURE DEPENDENCE OF LIGHT SCATTERING FOR MIXTURES OF CHOLESTEROL ESTERS.

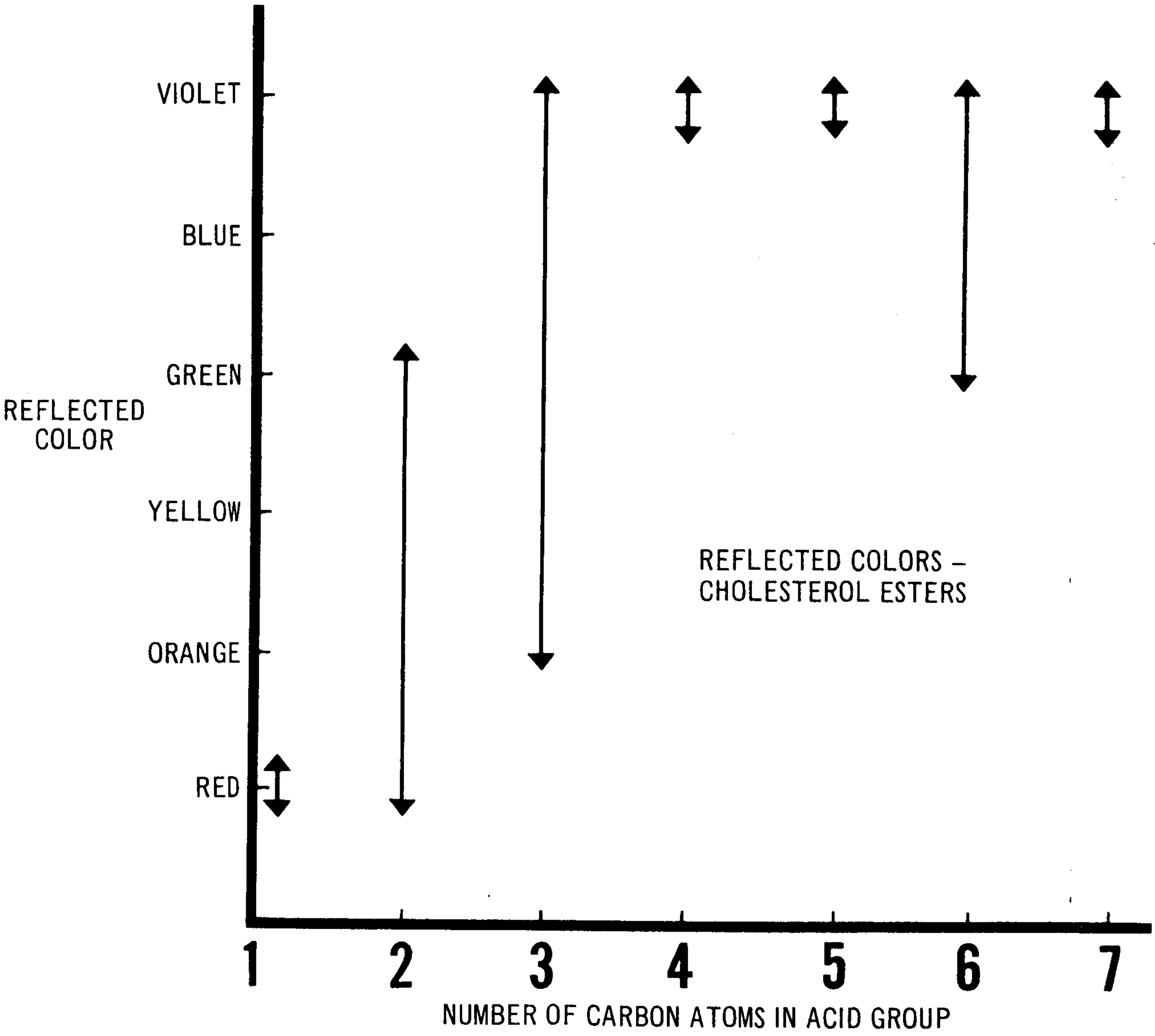


FIGURE 2 LIGHT SCATTERING AS A FUNCTION OF CHOLESTEROL ESTER STRUCTURE.

Although the colors are not changed by this process, that is, the reds are still red, their brilliance is greatly enhanced.

Another consideration related to the use of these materials is their stability upon standing. Fifty-four different mixtures of cholesteric liquid crystals were prepared to investigate compositional effects upon the stability of the color-producing plane texture. Eleven mixtures have remained in the plane texture for approximately one month without change.

To check the surface for bonding defects, the crystals are heated by heat lamps slightly above their transition range and are then cooled by a jet of cool air. Rapid heating and cooling of the surface results in the greatest sensitivity to bonding anomalies. If the part is well-bonded and free of defects, the color is uniform and cools consistently, changing from violet to red. The skin over defects cools more rapidly than the rest of the surface as the temperature is lowered quickly. Because this method is based on thermal flow, defect areas will also show up during the heating cycle. Defect areas will show up as warmer colors because of the slower transmission of heat through an unbonded area.

During the early stages of our work on liquid crystals we investigated metal-to-metal and aluminum honeycomb structures. As a result of the rapid dissipation of heat in aluminum face sheets, our results with aluminum honeycomb have not been too encouraging. Some success, however, was noted on metal-to-metal aluminum bonds. We feel at this time that we can achieve better results on aluminum honeycomb structures by using sonic and ultrasonic devices rather than liquid crystals. The majority of our work on liquid crystals has thus been confined to titanium panels and composite structures of titanium skins, HRP core, and polyimide prepreg. The thermal conductivity of titanium and ferrous alloys makes these metals ideal for thermal tests of this type.

Various ways of utilizing liquid crystals in the inspection of honeycomb panels are being considered. Following completion of the inspection, the liquid crystals and paint may be readily washed off with hot water. Since water and the cholesterol esters are immiscible, these phases can be separated by first filtering the carbon black out of the wash water, then by dissolving the liquid crystals in hot alcohol and recrystallization in an ice bath, they can be purified and reused.

The maximum skin thickness through which liquid crystals can detect defects in bonded panels is about 0.060 in. The time required to inspect a 4-foot by 4-foot panel is about one-half hour. This includes the time required for the black background paint to dry. The cost of inspecting bonded structures with liquid crystals is quite high at the present time because we are buying the cholesterol esters in small quantities. If larger quantities are bought, the price will be reduced considerably. The components used in our mixture cost about 50 cents per gram at the present time. If the raw materials are purchased in quantities of 10 kilograms, the price will be between 12 and 15 cents a gram.

The technique involved in inspecting with liquid crystals is not complex. Although the color transition is fast, it is a simple matter to go back over the area and check it again. If a permanent record is desired, a polaroid or movie camera can be used to take pictures of the color transition. It should not take more than a few days to train an operator to inspect structures with liquid crystals.

Liquid crystals have many other potential uses outside of checking for bond defects. They may be used in checking electronic circuits for hot spots. They can be used to check rivets in structures where they have to be insulated from dissimilar metals. A rivet that is improperly insulated will take at least ten times longer to heat up to the blue color than a rivet that is properly insulated. In the field of medicine they can be used for checking abnormal skin temperatures.

A great advantage of using liquid crystals for thermal nondestructive testing will be its low cost compared with that of more sophisticated and expensive equipment, particularly where the entire bonded surface area must be inspected. When this requirement is imposed, contact techniques become rather costly as a result of the total time required. At present, except on short production runs, liquid-crystal technique may not be the cheapest route. When liquid crystals are fully implemented the price per unit from the raw material suppliers should be reduced considerably because of a guaranteed market. The accuracy of liquid crystals is comparable with that of other methods of inspection. So far, we have only scratched the surface in regard to the use of liquid crystals in nondestructive testing.

The Boeing Company is continuing research work in the field of liquid crystals. I'm sure that in the future we will find many other uses for liquid crystals, as well as better and more refined application methods.

Figure 3 shows a schematic drawing of the method used to heat and cool the liquid crystals on the panel. Attached as an appendix to this paper are two color photographs of defects detected with liquid crystals. The first photograph shows three adhesive cutouts and the second shows a poor adhesive butt splice.

I want to thank the National Aeronautics and Space Administration for the opportunity of presenting this paper on liquid crystals at this symposium.

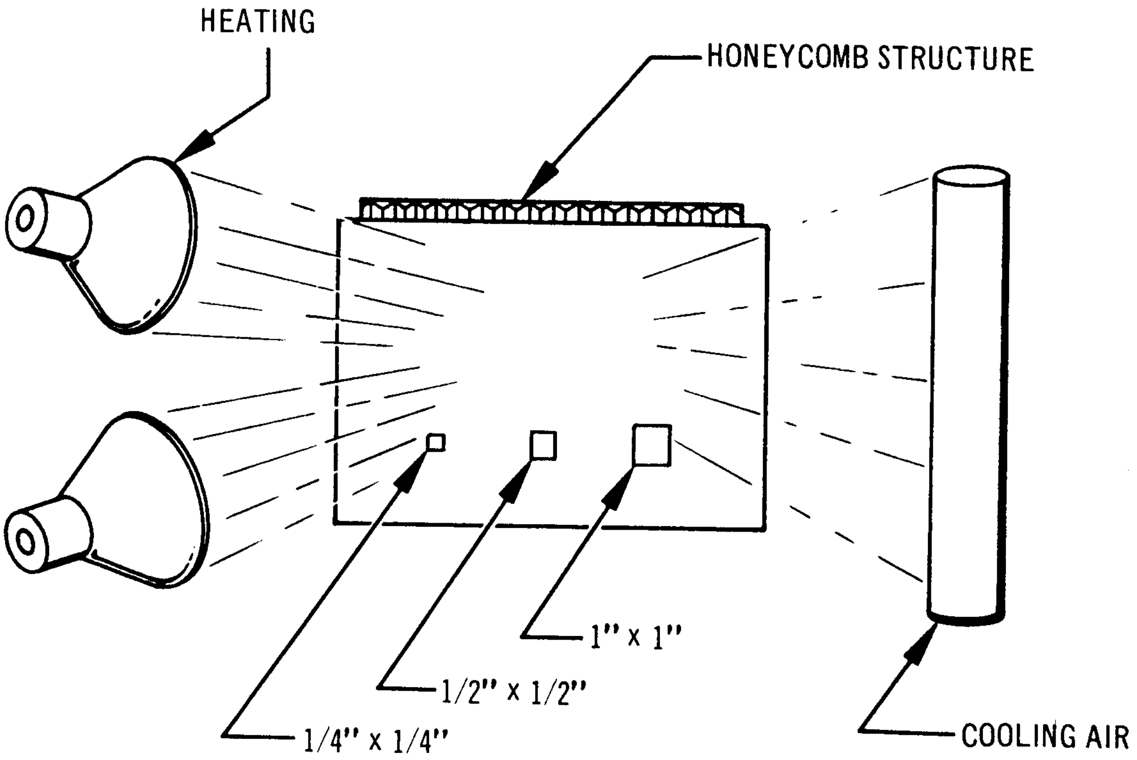
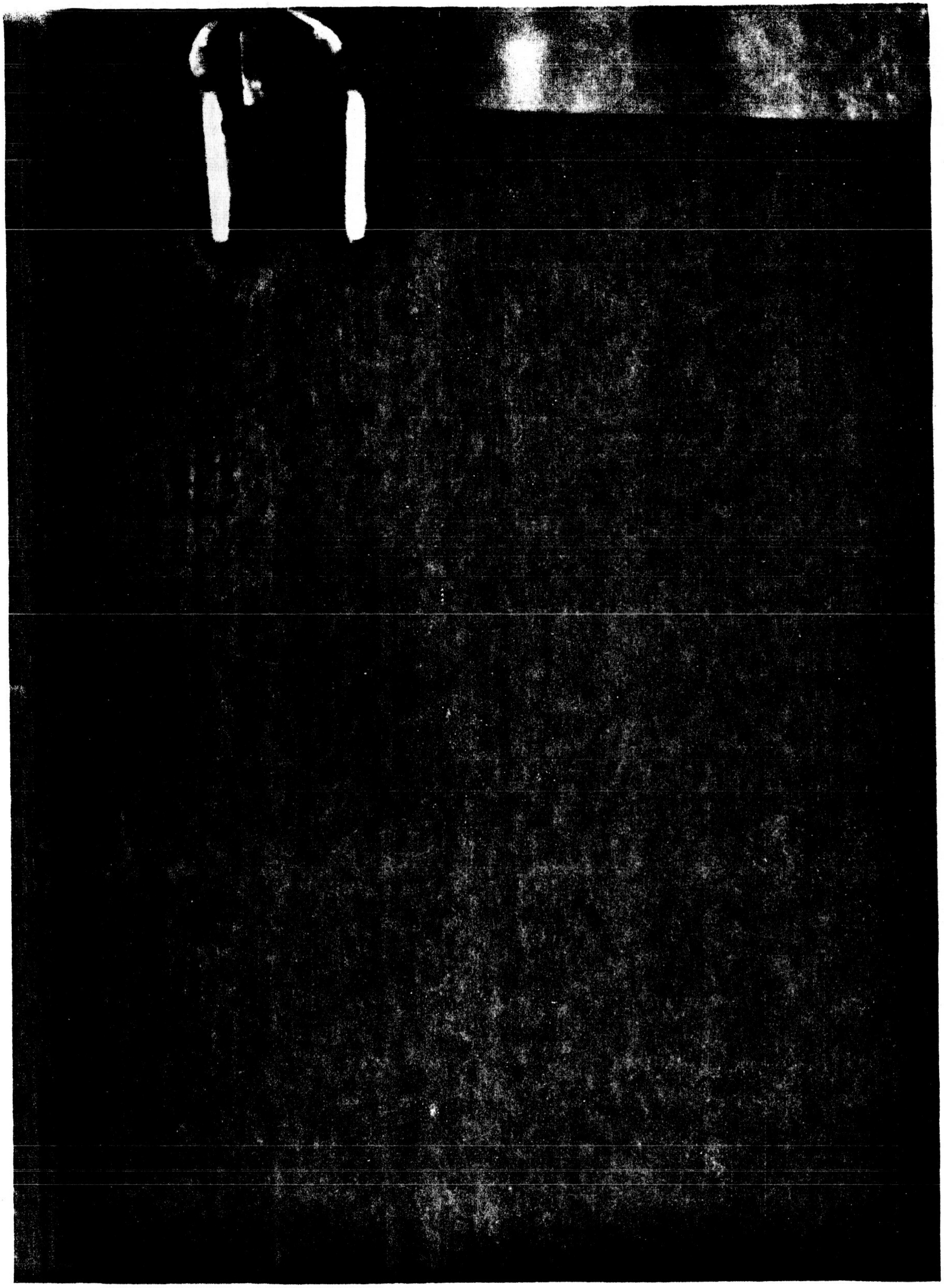


FIGURE 3 FLAW DETECTION IN TITANIUM – PHENOLIC HONEYCOMB SANDWICH STRUCTURE



SESSION IV - PAPER 2.

INSPECTION OF HONEYCOMB PANELS USING BI-REFRINGENT PLASTIC COATINGS

11:20 - 12:00 A. M.

WEDNESDAY - MARCH 16, 1966

N66 34138

INSPECTION OF HONEYCOMB STRUCTURES USING BI-REFRINGENT PLASTIC COATINGS

by

Hill M. Walker
Methods Development Branch
Manufacturing Research and Technology Division
Manufacturing Engineering Laboratory

ABSTRACT

In response to the urgent need for more and better methods for determining the integrity of bonded honeycomb sandwich structures, a technique has been devised whereby bond defects in the core-to-skin interface can be detected by simple, visual observation, without relying on expensive equipment or sophisticated operator technique. This inspection device involves the application of a transparent plastic film, employed in conventional photo elastic stress analysis investigations, to the surface of the honeycomb panel skin. Areas over defective bonds are caused to deflect outward by application of air pressure to the sealed, perforated core volume. These deflections carry through to the plastic surface film, causing peripheral strains which become visible when illuminated and viewed (or photographed) through polarized filters. Effectiveness of the techniques is dependent upon the proper interrelationship among the affecting variables of void size, skin thickness, coating thickness, and internal pressure applied. Contract and in-house efforts have carried this inspection method to what can be considered an effective, workable stage.

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HONEYCOMB STRUCTURES USING BI-REFRINGENT PLASTIC COATINGS

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Delivered March 16, 1966, at the Structural Bonding Symposium at NASA, Marshall Space Flight Center, Huntsville, Alabama.

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SUMMARY

The continuing demand for more and better methods for effecting manufacturing process control and inspection of bonded honeycomb sandwich structures has resulted in the development of a technique, which, without dependence upon complicated equipment or sophisticated operator techniques, can provide for economical, efficient inspection of honeycomb sandwich structures. Applicability is virtually unlimited, with operator participation being limited to simple visual observation of the inspected surfaces.

INTRODUCTION

In conceiving an ideal honeycomb sandwich inspection device, one could envision a system which would allow for detection of bond defects in unbonded areas, on flat or curved panel surfaces, by some method which would transfer an indication of the underlying defect to the panel surface, where it would be amplified and readily identified by visual observation. When the panel being inspected has perforated honeycomb core, and edge members provide a hermetic seal, pneumatic pressurization will, at some level, cause the facing sheet to bulge outward over unbonded areas. Further, if these disturbed areas are covered with a strain indicator coating, the void outline should be easily located on the panel surface.

Two such indicator coatings have been employed in demonstrating this inspection technique - a brittle lacquer and a photoelastic or bi-refringent plastic. The brittle lacquer, sprayed on the skin surface, will fracture in response to strain induced in the substrate. Strains occurring in and above defective bonds under the surface are located by crack patterns in the lacquer coating which occur when the panel is pressurized. The photoelastic plastic coating, applied and stressed in the same manner, provides indication of void areas by localized color changes which become apparent when the area under investigation is illuminated and viewed through polarized filters.

These two distinct indicator film types are familiar tools for stress analysis work, where skin strains are to be determined with a high degree of accuracy. However, in the application under discussion here, these qualities are of no significant value; relative sensitivity in visually indicating localized strains is the basic criterion by which the particular coating material is judged.

Initial Efforts

The first experimental work performed with this inspection technique involved the brittle lacquer coating system. Honeycomb panels were fabricated with potted or seal edge members and with intentional voids generated in the core-to-skin bond line by inclusion of half-mil circular teflon film inserts, varying from 1 - 3 inches in diameter (See Figure 1). A commercially available coating was applied by spraying to 0.005-inch thickness and cured in a closely-controlled atmosphere for over 18 hours (It should be noted here that these brittle lacquers are highly sensitive to changes in temperature and humidity; they are susceptible to changes in sensitivity and crazing if environmental conditions are allowed to vary even the slightest degree). Subsequent pressurization of the perforated, sealed cellular volume resulted in a clear definition of the underlying voids by crack patterns in the lacquer film (Figures 2, 3, and 4). The general technique was adequately demonstrated by this process; however, the above mentioned over-sensitivity of the lacquer film to environmental changes forecast undesirable limitations in practical application of the inspection method; utilization of bi-refringent plastic coatings seemed the obvious alternative.

One of the panels used in evaluating the brittle lacquer coating technique was stripped and cleaned, and three commercially available bi-refringent materials applied over the void areas to evaluate relative sensitivity. Included among these were a rigid sheet, 0.075-inch thick, bonded over a 2-inch diameter void, a flexible, rubber-like sheet 0.080-inch thick, bonded over 1-inch and 3-inch voids, and a catalysed liquid material, cast and allowed to cure over an area containing 1, 2, and 3-inch voids.

The "instrumented" panel was fitted with a pressure gage and, using an ordinary bicycle hand pump, was pressurized gradually until the void areas could be located. No indication could be found with the naked eye under ordinary illuminating conditions; however, when illuminated through a polarizing filter, and viewed through polarized film, the outlines of the 2-inch and 3-inch diameter voids became clearly visible at less than 3 psig. The outlines appeared as multi-colored rings at the periphery of each underlying void.

Increasing the pressure to 10 psig brought out the outlines of the 1-inch diameter voids, and further accentuated the outlines of the 2-inch and 3-inch voids. Similarly, the strain patterns on all void areas became proportionally brighter when visually observed at the 15, 20, and 25 psig pressure levels. Figures 5 and 6 indicate the clarity with which the various voids can be seen under 10 psig and 20 psig pressures; Figures 7 and 8 provide a more detailed illustration of the 2-inch void indications.

These initial development tests were carried out (with both the brittle lacquer and the bi-refringent plastic coatings) on a panel with 0.020-inch thick facing sheets. Interest was generated in application of the photo-elastic material to heavier gages, such as were incorporated in several designs applicable to Saturn V vehicles. A prototype S-IC intertank structure, 4-inches thick, having 0.070-inch facing sheets, was being proposed at that time to Marshall Space Flight Center by the stage contractor. A specimen panel with sealed edge members and void implants was obtained, coated, and tested in the manner of the above-described panel test. Results were encouraging; a 1/2 X 1-inch void was identified at less than 30 psig cavity pressure, and several larger voids of varying shapes were located at considerably lower pressure levels. In a more spectacular demonstration, a production instrument panel, having 0.063-inch facing sheets and <u>no</u> intentional void implants, was coated and tested; at less than 100 psi the entire core pattern was outlined, and single cell node de-bonds were detected.

Based on the achievements of these studies, it was quite apparent that this flaw detection technique utilizing bi-refringent coatings could find immediate application in the inspection of the extremely large honeycomb sandwich structures incorporated in or being considered for space booster vehicles. One could appreciate the advantages of being able to inspect the 33-foot diameter S-II common bulkhead, with a high degree of confidence, in a matter of hours instead of days. This could, of course, apply as well to the S-IVB bulkhead, the proposed S-IC inter-tank and thrust structures, skirts, etc. No special facility would be required; any darkened area would serve the purpose. The coated surfaces would be flood-lit through polarizing film. However, a certain amount of development work was still to be done before this inspection technique could be employed with the necessary degree of reliability:

1. Relationship of Variables. In order that maximum resolution be realized from any of the applied photoelastic indicator films, it is necessary to determine the inter-relationship among the four affecting variables - (a) anticipated minimum void dimension, (b) facing sheet thickness, (c) indicator film thickness, and (d) internal pressure to be applied. Higher pressure causes greater deflection; thinner facing sheets will deflect more sharply for a given pressure level; larger voids react under less pressure; and, to a point, the thicker the overlying indicator film, the more pronounced is the void indication.

2. Application. Preliminary evaluation had been carried out with indicator films either bonded in place or cast onto a horizontal surface.

Since it is quite likely that the coating will be applied to vertical or domed surfaces, development of a sprayable coating was considered mandatory.

3. Removal. A method was needed to insure easy, complete removal of all traces of the organic coating material from the inspected part, in the likely event that the surface in question should come in contact with liquid oxygen, as would be the case with the S-II and S-IVB common bulkheads.

4. Low Cost. It was assumed (correctly) that a highly sensitive indicator film could be developed by formulating commercially available polymers, an attractive feature when considering a potentially high-volume utilization rate.

A contract was entered into with the Lockheed-Georgia Company to perfect and extend this inspection technique, along these guidelines.

Theory of Photo-elasticity

At this juncture, it might be appropriate to enlarge upon the physical phenomena of photo-elastic coatings, a simple application of which forms the core of this project. Bi-refringence, resulting from deformation of optically isotropic materials, was reported in the early 19th century; the bi-refringent coating technique, however, was developed as a skin strain detector in recent years.

The theory utilized in general photo-elastic or bi-refringent coating work is based on physical principles involving light transmission through optically isotropic and bi-refringent materials. Optically isotropic materials transmit light at the same velocity in all directions, consequently, each material exhibits a single value for index of refraction. Most of these materials, however, become anisotropic or bi-refringent upon forced deformation due to the fact that two optical axes are formed at any point observed. These axes are orthogonal at every point, and the index of refraction of one axis is not necessarily equal to that of the other. It has been established that the difference between indices of refraction is proportional to the difference between principal stresses at the point considered, and that the optical axes coincide with the principal stress directions. If the indices differ at a point, part of the incident light amplitude will emerge behind its complimentary component. The term relative retardation is used to define this phenomenon, and a polariscope is used to measure this quantity in terms of a selected wave length of light. A proportionality constant between relative retardation and principal stress difference can be determed by a simple calibration.

In the bi-refringent coating technique the optically isotropic material is applied to the structure to be analyzed. The structure is subsequently loaded in some manner and the resulting surface strains are transmitted to the coating which then becomes anisotropic, or bi-refringent. The instrument used to detect this bi-refringence is called areflective polariscope since the observed light is reflected from the substrate surface. (It should be emphasized here that the surface to be analyzed must be reflective, or must receive a reflective coating prior to application of the indicator coating.) The simplest such polariscope is a sheet of circular polarized film, placed directly on the coating surface. The normal polychromatic lighting of the work area is usually sufficient to allow detection of stressed areas. When using polychromatic lighting, these areas are displayed by different colors, such as those occurring over the void areas shown in Figures 5 through 8.

For bi-refringent coatings, the proportionality constant which compares relative retardation (effectively, the observed color intensity) to principal strain difference (in this application, severity of deflection over underlying voids) is termed the strain-optic coefficient (K); this factor, together with certain mechanical and chemical properties of a material, is relied upon in determining utility of a given coating system for a specific application. For example, in applying this void detection system to a low-modulus or relatively thin substrate, one would look for a high "K" factor for lowest detectability threshold, low modulus or flexibility to avoid reinforcement, adequate but low adhesive strength to facilitate coating removal, and perhaps a consistency which would allow for spray coating.

Development Program

The emphasis upon application of indicator coatings to very large structures, as opposed to the considerable technical experience gained with laboratory specimens of limited area, indicated the need for development of a sprayable material with extended pot life, which would hang to an appreciable thickness on vertical surfaces, and which would cure on application at ambient temperatures. No available bi-refringent coating could offer this combination; dual component spray guns, with fluctuating resin-to-catalyst ratios, or premixed coatings with short pot life and post-cure requirements, were considered inadequate. In any method used it is essential that the hardener be uniformly and intimately mixed with the resin in order to provide a coating with uniform properties. Most resins are critical in this respect because hardener-to-resin ratios are quite low. Small fluctuations in hardener proportioning and mixing ratio can produce large variations in strain-optic coefficient, and the variation existing in a coated structure cannot be easily determined. Proportioning and mixing is further complicated by the enormous viscosity differential between hardener and resin.

Coating Development Objectives

Requirements were established which would yield the desired characteristics of an acceptable spray coating system; the most important were considered to be:

1. Pre-mix coating constituents and spray as a single component.
2. Provide high hardener-to-resin ratio.
3. Maximize solid content.
4. Provide long pot life in large quantity mixes.
5. Utilize conventional paint spray equipment.
6. Spray thickest possible coating on vertical surfaces.
7. Short, ambient-temperature cure.
8. Provide high strain-optic coefficient for cured coating.
9. Attain low modulus of elasticity in cured coating.
10. Stable properties after cure.
11. Good optical properties for clarity in analysis.

12. Allow for easy coating removal after analysis.

These twelve points were adopted as objectives in coating development, since their accomplishment would provide an ideal system for large area coverage.

Material Screening and Selection

The bulk of present bi-refringent coating technology hinges about the relatively new epoxy resin systems; in order that this technology might be best utilized, epoxy resins were considered as bases for this development effort. Epoxies can be cured in a variety of ways, and their cured properties can be varied widely by additives in formulation. However, the effects of various curing agents, accelerators, or other agents, are not well defined; consequently, a screening process was carried out to determine these effects, and to allow for selection of coating constituents and concentrations required to satisfy the previously established objectives.

Calibration beams were prepared and coated with resin compounds representing a seemingly endless variety of constituents and ratios. Two basic resin systems, Epon 828 and Ciba 6020, were used throughout, by virtue of their known compatibility with a variety of curing agents and their resultant high strain-optic coeffecients (K). Additives, in varying combinations and ratios, included accelerators, flexibilizers, viscosity modifiers, and volitilization inhibiters, as well as curing agents or hardeners.

Ketimines were employed throughout as curing agents; a relatively new compound, the Ketimines are formed by reaction of aliphatic polyamines and Ketones. They have a very low level of reactivity with the base resin in the absence of moisture; however, moisture converts the Ketimine to polyamine and Ketone. The Ketone is released and it evolves, leaving the polyamine to function as a reactive cross-linking agent for the resin in the same manner as conventional amine curing agents. The merits of the system are obvious - immediate initiation of cross-linking or curing process on spraying, due to atmospheric moisture present, yet with potentially extended pot life in the liquid mixture, particularly if the container is hermetically sealed.

As was to be expected in a controlled evaluation of this sort, many combinations were ruled out due to intolerable side effects - surface crazing, blushing, bubbles, low "K" factor, or time-dependent variation in "K" factor. The system selected for modification into a sprayable coating - one which with least compromise met the arbitrary screening requirements - was composed of pure Ketimine - EPON H-3, tri-dimethylamine methyl phenol - DMP, and Epoxy Resin - CIBA 6020.

Spray Evaluations

Evaluations were initiated to develop spray techniques necessary for maintaining the superior optical properties and high strain-optic coefficient attained in the selected formulation during screening. The selected compound was sprayed on vertical test panels, at an ambient temperature of 70° F. Poor atomization resulted, and spraying was tried at various higher temperature levels. Good spraying characteristics were demonstrated at 150° F; however, small bubbles were thickly dispersed in the film, trapped during cure, and seriously altered optical properties. Flow control agents were tried, as was an airless spray gun, but to no advantage. It became apparent that the problem of bubble evolution and entrapment was inherent with the Ketimine curing agent; the viscosity of the coating during Ketimine conversion was too high to allow the evolved Ketones to escape, and they remained in the film as bubbles during and after cure. Further evaluations included utilization of solvents to reduce viscosity of the sprayed coating.

Addition of Methyl Ethyl Ketone (MEK) in various concentrations was undertaken, and coatings were sprayed at temperatures ranging from 70° F to 125° F. Little advantage was realized in spraying at the higher temperatures because of complications due to rapid solvent flashing; it was found that good optical properties were gained in 70° F, spraying with a 29 percent (by weight) addition of MEK.

The MEK dilution, however, created another problem; the resultant reduction in viscosity lowered the wet film thickness obtainable on a vertical surface. Thixotroping agents were tried in varying concentrations. It was determined that a 3 percent addition of cab-o-sil (an inert powdered silica) did not impair optical properties, yet provided the consistency necessary to allow for spraying on a vertical surface to thicknesses in the 6-8 mil range.

The finalized formulation, utilized as the basic bi-refringent coating system in subsequent development tests, now became:

component	p. b. w.
97 percent Ciba 6020 Resin - 3 percent Cab-o-sil	100
EPON H-3 Ketimine	40
Tri-Dimethylamine Methyl Phenol	6
Methyl Ethyl Ketone	29

This formulation was sprayable to a wet film thickness, on a vertical surface, of 0.008-inch, built up by deposition of 0.0005-inch spray film on each pass. It cures to a tack-free condition in 3 hours, making it an excellent candidate for a coating film for analyzing large structures, since the desired thickness can be built up in successive layers, as was done in the subsequent test work.

That the derived formulation exhibited a relatively long pot life under realistic circumstances was demonstrated by mixing a 1-gallon batch of the compound, then placing it in a container having insulated walls and bottom to prevent heat loss from the exothermic reaction. The top of the container remained open to allow for entrance of atmospheric moisture necessary for Ketimine conversion. The material, held at 70° F and 30 percent RH for 8 hours, was considered sprayable. Based on this evaluation, it was estimated that a 5-gallon quantity in sealed metal cans could be afforded a pot life of seven days if refrigerated.

The foregoing outline of the coating development program is oversimplified, in the interest of brevity. Each step forward was taken at the expense of laborious and sometimes frustrating "side-steps". The effort was, however, rewarding; a cheap, effective, easily-applied material has been developed, to be used in carrying out a promising new honeycomb inspection technique.

Honeycomb Test Panels

Fifty honeycomb sandwich panels containing void implants were fabricated to evaluate the bi-refringent coating system for conditions simulating the intended application. Common denominators among the panels were core type and size (2 X 2-feet X 0.50-inch thick, 1/4-5052-.003P) and panel area - 2 X 2-feet. Also, all panels were closed out at the edges with phenolic strips, bonded or potted to effect a hermetic seal; in each panel, two opposing strips were fitted with tubes to allow for pressure application and detection.

Thirty of the panels had circular bond line voids produced by cutting holes in the HT-424 adhesive. Among the 30 panels involved there were five each of six different nominal face sheet thicknesses; 0.016, 0.032, 0.050, 0.063, 0.080, 0.100 inch. Two rows of voids were cut in the adhesive for one face sheet of each panel. For the 0.016, 0.032, and 0.050-inch face sheets each row has void diameters of 0.50-inch, 1.0, and 2.0 inches; each row of the thicker face sheets had void diameters of 1.0, 2.0, and 3.0 inches.

The remaining 20 panels were fabricated with a variety of defects, including voids generated by differing methods: marginal bonds; doubler voids; and areas of potential defect due to "bad practice" assembly. These last 20 specimens were fabricated to allow for demonstration of utility and flexibility of the void detection concept. The first 30 units were intended as the test articles through which to establish the interrelationship of affecting variables, one of the primary program objectives, as outlined earlier.

Each panel was divided into two equal areas, A and B, with each area containing a row of voids. This provided 10 identical test areas for each face sheet thickness, and the bi-refringent coating thickness was varied by a nominal 0.010 inch from one area to another. Thus, 10 different coating thicknesses covering an approximate range from 0.010 inch to 0.100 inch were provided for each of the six different face sheet thicknesses. Evaluation of these 30 panels (actually 60 different investigated areas) provided information necessary to determine the coating thickness required to maximize void detectability for any particular face sheet thickness within the range investigated. All panels were spray-coated in a vertical position, as specified earlier. Calibration beams were taped to an aluminum alloy sheet, and were sprayed along with the honeycomb panels. Beam specimens were removed periodically in order to determine the strain-optic coefficient for different thicknesses of the same coating used on the panels. The last spray coat applied to the beams and panels was allowed to cure a minimum of 24 hours at 80° F before calibration or void detection evaluations were initiated.

Void Detection Evaluations

The void detection evaluations were performed by observing the bi-refringent coating through a commercial reflective polariscope while the honeycomb panel was being pressurized internally with air. One of the tubes potted in the test panel edge members was connected to an air pressure gage.

The other tube, connected to the air supply line through a variable pressure regulator, was also fitted with a pressure gage. The polariscope was located approximately five feet from the panel surface, and the gages and regulator positioned so that the operator could observe both gages and panel while varying the pressure. Except for the polariscope light source, the room was in total darkness while void detection measurements were being made.

Prior to performing the evaluations, several methods of light conditioning and observation were investigated to evaluate their relative merits with respect to sensitivity in void detection. Sheets of circular polarized filter were placed on the coating surface and observed under illumination by a polychromatic light source. This method was found to be approximately equivalent to observations made through the reflective polariscope with a circular polarized field at normal angle of incidence. Observations were also made by illuminating the panel surface with polarized light at various angles of incidence other than normal, while observing the bi-refringence through a polarizer at the angle of reflection. Such observations at 45 degree angles made the voids detectable at pressures slightly lower than when using the circular field at normal incidence. However, the greater angles produced scattering of the reflected light, and poor definition of the bi-refringent patterns. As a result, it was concluded that more uniform and reproducible results would be obtained by utilizing the reflective polariscope at normal (90°) incidence.

Three different detection or observation pressure levels were determined and recorded (See Tables I thru VI) for each void group:

A. Minimum pressure for void outline detection using a 90 degree rotation of the polariscope analyser, which presented a mixed fringe pattern in apparent motion about the void area.

B. Minimum detection pressure using the stationary circular polariscope.

C. Pressure at which voids were clearly and immediately distinguishable, using the stationary circular polariscope.

The general trend of detection pressure levels in relation to panel skin thicknesses and coating thicknesses is enlightening; from this data collection we are given a critical coating thickness for maximum detectability, over a specified minimum void size, and a pressure level at which void areas can be easily seen.

Coating Removal

The task of removing this relatively rigid coating became, in the end, unexpectedly simple. After unsuccessfully trying various stripping agents, and an equally unsuccessful venture with thermal shock (flooding the surface with liquid CO_2 did craze and spall the coating, but left much unaffected and firmly adherred residue), it was found that the Ciba 6020 system was thermoplastic at approximately 160° F, and could be easily peeled off after heating the part to this temperature, or by heat gun application.

Flexible Coating Development

In addition to pursuing the initial objectives of this program, a concurrent effort was made to develop a highly sensitive, flexible coating, applicable to the relatively flexible plastic skin employed on the cellular honeycomb insulation for the S-II Liquid Hydrogen tank walls. After screening a number of candidate materials (mostly urethanes, which best filled the flexibility requirement), a Spencer-Kellogg formulation was found which offered the desired characteristics of high sensitivity (K=0.05), sprayability at ambient temperature on a vertical surface, and very easy removal. This material, designated M-86-50CX, is a one-part system, catalyzed by atmospheric moisture, and is tack free in one hour. Tests are presently under way to determine the effectivenss of the system in detecting single-cell defects in the 0.75-inch honeycomb cell-to-face sheet bond, a critical area in view of the considerable helium purge pressure requirement on the system.

Conclusion.

With reliance only on the techniques and materials developed to date, we feel that a workable and immediately applicable bonding evaluation system has been successfully demonstrated. The data shown in Tables I and II indicate that pressure differentials necessary to show even very small defects can be easily attained in a vacuum chamber, which would allow for inspection of non-perforated core structures, such as are found in high-performance aircraft components. The ultimate in material sensitivity has certainly not been reached.

Continuing development should result in definition of an inspection technique for any conceivable combination of materials and design configurations.

TABLE I
RESULTS OF BIREFRINGENT COATING THICKNESS EVALUATION ON HONEYCOMB PANELS HAVING 0.014 INCH THICK CLAD 7075-T6 ALUMINUM ALLOY FACE SHEETS

Panel Number	Coating Thickness, Inch	Detection Pressure, PSI, for 0.5-inch Dia. Void(1)			Detection Pressure, PSI, for 2.0-inch Dia. Void(1)			Detection Pressure, PSI, for 2.0-inch Dia. Void(1)		
		A	B	C	A	B	C	A	B	C
16-1A	0.010	12.0	(2)	(2)	0.5	1.5	2.5	0.1	0.5	0.5
16-1B	0.023	2.5	8.0	12.0	0.5	1.5	2.0	0.1	0.5	0.5
16-2A	0.029	2.0	9.0	12.0	0.1	1.0	2.0	0.1	0.1	0.5
16-2B	0.039	2.0	9.0	12.0	0.1	1.0	2.0	0.1	0.1	0.5
16-3A	0.048	2.0	12.0	15.0	0.1	1.5	2.0	0.1	0.1	0.5
16-3B	0.057	2.0	12.0	15.0	0.1	1.5	2.0	0.1	0.1	0.5
16-4A	0.070	2.0	13.0	20.0	0.1	2.0	3.0	0.1	0.1	0.5
16-4B	0.078	3.0	13.0	20.0	(3)	(3)	(3)	0.1	0.1	0.5
16.5A	0.086	5.0	20.0	(2)	0.5	4.0	7.0	0.1	0.5	1.0
16-5B	0.095	9.0	(2)	(2)	0.5	4.0	7.0	0.1	0.5	1.0

(1) A-Detection pressure when using plane polariscope and rotating analyzer.
B-Detection pressure when using circular polariscope.
C-Pressure required to clearly show void shape when using circular polariscope.
(2) Not detectable for pressure up to 20 PSI.
(3) Not measured.

TABLE II

RESULTS OF BIREFRINGENT COATING THICKNESS EVALUATION ON HONEYCOMB PANELS HAVING 0.030 INCH THICK CLAD 7075-T6 ALUMINUM ALLOY FACE SHEETS

Panel Number	Coating Thickness Inch	Detection Pressure, PSI, for 0.5 inch Dia. Void(1)			Detection Pressure, PSI, for 1.0 inch Dia. Void(1)			Detection Pressure, PSI, for 2.0 inch Dia. Void(1)		
		A	B	C	A	B	C	A	B	C
32-1A	0.008	16.0	(2)	(2)	3.5	12.0	15.0	2.0	4.0	6.0
32-1B	0.016	6.5	30.0	40.0	2.0	6.0	9.0	0.2	0.5	1.5
32-2A	0.027	5.0	20.0	22.0	1.2	3.5	5.0	0.1	0.5	1.0
32-2B	0.036	4.0	18.0	20.0	1.0	3.0	3.5	0.1	0.2	0.5
32-3A	0.051	4.0	18.0	22.0	0.5	2.0	3.5	0.1	0.1	0.5
32-3B	0.061	3.5	18.0	22.0	0.1	1.0	3.0	0.1	0.1	0.5
32-4A	0.068	4.0	19.0	24.0	0.5	3.0	5.0	0.1	0.5	1.0
32-4B	0.078	4.0	19.0	24.0	0.5	3.5	5.0	0.1	0.5	1.0
32-5A	0.085	4.0	22.0	30.0	0.5	4.0	6.0	0.1	1.0	2.0
32-5B	0.097	4.0	22.0	30.0	0.5	4.5	6.0	0.1	1.0	2.0

(1) A-Detection pressure when using plane polariscope and rotating analyzer.
B-Detection pressure when using circular polariscope.
C-Pressure required to clearly show void shape when using circular polariscope.

(2) Not detectable for pressure up to 30 PSI.

TABLE III

RESULTS OF BIREFRINGENT COATING THICKNESS EVALUATION ON HONEYCOMB PANELS HAVING 0.048 INCH THICK CLAD 7075-T6 ALUMINUM ALLOY FACE SHEETS

Panel Number	Coating Thickness Inch	Detection Pressure, PSI, for 0.5 inch Dia. Void(1)			Detection Pressure, PSI, for 1.0 inch Dia. Void(1)			Detection Pressure, PSI, for 2.0 inch Dia. Void(1)		
		A	B	C	A	B	C	A	B	C
50-1A	0.010	(2)	(2)	(2)	14.0	(2)	(2)	3.5	11.0	16.5
50-1B	0.020	23.0	(2)	(2)	6.5	17.0	22.0	1.0	3.0	5.5
50-2A	0.030	18.0	(2)	(2)	2.0	14.5	16.0	0.5	3.0	5.0
50-2B	0.036	14.5	(2)	(2)	1.5	9.0	11.0	0.2	2.0	3.0
50-3A	0.053	10.0	(2)	(2)	1.0	6.0	9.0	0.1	1.5	2.0
50-3B	0.059	7.0	35.0	(2)	1.0	6.0	9.0	0.1	1.0	1.5
50-4A	0.068	7.0	30.0	(2)	1.0	5.0	9.0	0.1	1.0	2.0
50-4B	0.076	7.0	30.0	(2)	1.0	5.0	8.0	0.1	1.0	2.0
50-5A	0.085	9.0	40.0	(2)	1.0	5.0	10.0	0.1	1.5	2.0
50-5B	0.100	11.0	40.0	(2)	1.5	8.0	11.0	0.2	2.0	2.5

(1) A-Detection pressure when using plane polariscope and rotating analyzer.
B-Detection pressure when using circular polariscope.
C-Pressure required to clearly show void shape when using circular polariscope.

(2) Not detectable for pressure up to 40 PSI.

TABLE IV
RESULTS OF BIREFRINGENT COATING THICKNESS EVALUATION ON HONEYCOMB PANELS HAVING 0.061 INCH THICK CLAD 7075-T6 ALUMINUM ALLOY FACE SHEETS

Panel Number	Coating Thickness Inch	Detection Pressure, PSI, for 1.0 inch Dia. Void(1)			Detection Pressure, PSI, for 2.0 inch Dia. Void(1)			Detection Pressure, PSI, for 3.0 inch Dia. Void(1)		
		A	B	C	A	B	C	A	B	C
63-1A	0.008	18.0	(2)	(2)	4.0	18.5	22.0	1.5	8.5	11.0
63-1B	0.018	10.0	20.0	35.0	1.5	3.5	8.0	0.5	2.0	3.0
63-2A	0.028	6.0	19.0	28.0	2.0	3.5	7.5	0.5	2.0	3.0
63-2B	0.037	5.0	15.0	22.0	1.5	3.0	5.0	0.5	1.5	3.0
63-3A	0.050	3.0	10.0	17.0	0.5	2.0	3.0	0.1	1.0	1.5
63-3B	0.058	2.0	9.0	14.0	0.3	1.5	2.0	0.1	0.5	1.0
63-4A	0.069	2.0	8.0	12.0	0.2	1.0	2.0	0.1	0.5	1.0
63-4B	0.077	2.0	8.0	12.0	0.2	1.0	2.0	0.1	0.5	1.0
63-5A	0.084	2.0	9.0	14.0	0.1	1.0	2.5	0.1	0.3	1.0
63-5B	0.096	2.0	8.0	14.0	0.1	1.0	2.5	0.1	0.3	1.0

(1) A-Detection pressure when using plane polariscope and rotating analyzer.
B-Detection pressure when using circular polariscope.
C-Pressure required to clearly show void shape when using circular polariscope
(2) Not detectable for pressure up to 40 PSI.

TABLE V

RESULTS OF BIREFRINGENT COATING THICKNESS EVALUATION ON HONEYCOMB PANELS HAVING 0.078 INCH THICK CLAD 7075-T6 ALUMINUM ALLOY FACE SHEETS

Panel Number	Coating Thickness Inch	Detection Pressure, PSI, for 1.0 inch Dia. Void(1)			Detection Pressure, PSI, for 2.0 inch Dia. Void(1)			Detection Pressure, PSI, for 3.0 inch Dia. Void(1)		
		A	B	C	A	B	C	A	B	C
80-1A	0.008	30.0	(3)	(3)	7.5	28.0	40.0	3.0	13.0	19.0
80-1B	0.016	15.0	(3)	(3)	3.5	11.0	15.0	1.5	6.0	9.0
80-2A	0.026	4.0	26.0	35.0	2.0	5.0	8.0	1.0	2.5	3.0
80-2B	0.041	4.0	15.0	23.0	(3)	(3)	(3)	0.5	1.5	2.5
80-3A	0.047	4.0	16.0	24.0	0.7	3.5	4.5	0.1	1.0	2.5
80-3B	0.059	4.0	13.0	20.0	0.5	3.0	4.0	0.1	1.0	2.0
80-4A	0.072	3.0	12.0	18.0	0.1	3.0	3.5	0.1	1.0	2.0
80-4B	0.080	3.0	12.0	18.0	0.1	3.0	3.5	0.1	1.0	2.0
80-5A	0.088	2.5	12.0	16.0	0.1	3.0	3.5	0.1	1.0	2.0
80-5B	0.096	2.5	12.0	16.0	0.1	3.0	3.5	0.1	1.0	2.0

(1) A-Detection pressure when using plane polariscope and rotating analyzer.
B-Detection pressure when using circular polariscope.
C-Pressure required to clearly show void shape when using circular polariscope.

(2) Not detectable for pressure up to 40 PSI.

(3) Not measured.

TABLE VI
RESULTS OF BIREFRINGENT COATING THICKNESS EVALUATION ON HONEYCOMB PANELS HAVING 0.097 INCH THICK CLAD 7075-T6 ALUMINUM ALLOY FACE SHEETS

Panel Number	Coating Thickness Inch	Detection Pressure, PSI, for 1.0 inch Dia. Void(1)			Detection Pressure, PSI, for 2.0 inch Dia. Void(1)			Detection Pressure, PSI, for 3.0 inch Dia. Void(1)		
		A	B	C	A	B	C	A	B	C
100-1A	0.008	40.0	(2)	(2)	17.0	40.0	(2)	9.0	23.0	31.0
100-1B	0.016	28.0	(2)	(2)	8.0	19.0	30.0	6.0	10.0	17.0
100-2A	0.025	10.0	30.0	(2)	2.5	10.0	13.0	1.0	5.0	7.0
100-2B	0.041	6.0	28.0	40.0	2.0	7.0	9.0	0.5	2.5	5.0
100-3A	0.047	6.0	28.0	40.0	1.5	6.0	8.0	0.5	2.5	4.0
100-3B	0.059	6.0	27.0	35.0	1.5	6.0	8.0	0.5	2.5	4.0
100-4A	0.067	6.0	22.0	32.0	1.5	5.0	8.0	0.5	2.5	3.0
100-4B	0.077	6.0	22.0	32.0	1.5	5.0	8.0	0.5	2.5	3.0
100-5A	0.086	6.5	24.0	35.0	1.5	5.0	7.0	0.5	2.5	3.0
100-5B	0.096	6.5	24.0	35.0	1.5	5.0	7.0	0.5	2.5	3.0

(1) A-Detection pressure when using plane polariscope and rotating analyzer.
B-Detection pressure when using circular polariscope.
C-Pressure required to clearly show void shape when using circular polariscope.

(2) Not detectable for pressure up to 40 PSI.