FZK-319
3 October 1966

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Quarterly Progress Report
30 June 1966 through 30 September 1966

E. E. KERLIN

Prepared for
George C. Marshall Space Flight Center
Huntsville, Alabama

Contract No. NAS8-18024
Request No. DCN 1-654-01167 (1F)

NUCLEAR AEROSPACE RESEARCH FACILITY

operated by

GENERAL DYNAMICS
Fort Worth Division

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This report was prepared by the Fort Worth Division of General Dynamics Corporation under Contract No. NAS8-18024, Evaluation of Cryogenic Insulation Materials and Composites for Use in Nuclear Radiation Environments, for the George C. Marshall Space Flight Center of the National Aeronautics and Space Administration. The work was administered under the technical direction of the Propulsion and Vehicle Engineering Division, Engineering Materials Branch of the George C. Marshall Space Flight Center, with R. L. Gause acting as project manager.

SUMMARY

A contract was entered into between the Fort Worth Division of General Dynamics Corporation and the National Aeronautics and Space Administration on 30 June 1966 for a 1-year study of thermal insulation system components of the type for use on a Ground Test Module. The program is to consist of three main phases: (1) selecting candidate test materials, (2) exposing materials to combined environments of nuclear reactor radiation and liquid hydrogen, and (3) testing and evaluating the results.

During the first quarter of operation, efforts were directed toward selecting candidate test materials, developing the test plan, and modifying the existing test equipment from a previous NASA contract, NAS8-2450.

The materials tested in the program were chosen from lists compiled independently by General Dynamics and NASA as the result of a survey and analysis of existing literature and related tests. These materials, discussed in Section II, fall into three categories: cryogenic insulations, adhesives, and vapor barriers.

Two types of tests are planned for evaluating the radiation dose of 1×10^{11} ergs/gm(C) on materials: (1) mechanical property evaluation of the individual components and combined systems, and (2) functional evaluation of a composite system applied to a test dewar. The mechanical property test measurements to be made are the following: lap-shear tensile strength, T-peel strength, thin-film tensile strength, thermal insulation compression-set test, flatwise tensile test, and thin-film permeability test.

After sufficient thermal cycles to establish the integrity of the bonded insulation system to the test dewar, the system will be irradiated to 1×10^{11} ergs/gm(C) and subsequently exposed to 6 hr of acoustic noise. The system will be visually inspected for cracks, splits, and sealed insulation.

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I. INTRODUCTION

One of the predicted material problem areas in the NERVA Engine ground test program is the thermal insulation for the LH_2 tank. The insulation will be located close to the nuclear rocket engine and will receive a high radiation dose during each engine operating cycle. The many operating cycles planned for the ground test program will expose the material to a total dose of approximately 10^{11} ergs/gm(C). In addition to the radiation dose, the material will be subjected to vibration loads, desert environments, and thermal contraction stresses.

Several research programs have been initiated by NASA to study and develop methods of applying insulation materials to cryogenic tanks. As a result of these programs, a number of materials with special fabrication procedures are available for use as thermal insulation on LH_2 tanks. However, the radiation stability of these materials has not been determined.

The Fort Worth Division of General Dynamics has entered into a contract agreement (NAS8-18024) with the George C. Marshall Space Flight Center of NASA to test materials suitable for thermal insulation systems. The 1-year contract is for evaluating the effects of radiation on the mechanical properties of several components of thermal insulation systems and on a composite thermal insulation system applied to a test dewar. Complete control tests will be performed to separate the various effects of cryogenic temperature and the combined effects of radiation and cryotemperature.

The test samples are to be fabricated by NASA from material selected and recommended jointly by General Dynamics and NASA.

The LH_2 control and irradiation testing is scheduled for a 2-week period beginning 15 February 1967. The materials will be irradiated to a total dose of 1×10^{11} ergs/gm(C), with some tests being conducted in the LH_2 irradiation environment with special test equipment. The remainder of the samples will be removed to the IML for testing in the Instron.

II. TEST MATERIALS

One of the predicted problem areas in the NERVA Engine ground test program is the suitability of materials for the insulation system of the liquid-hydrogen (LH_2) tank. The insulation is to be bonded to the tank with adhesives and protected from moisture and gases by a vapor barrier. A complete insulation system, as well as specimens of the individual components, is to be tested in the nuclear environment.

In this section, test materials are discussed under the following aspects: (1) materials selection, and (2) special specimen fabrication procedures.

2.1 Materials Selection

The materials to be tested in the nuclear environment fall into three categories: cryogenic insulations, adhesives, and vapor barriers. The original candidate materials submitted to NASA by General Dynamics included four cryogenic insulations, eight adhesives, and six vapor barriers. However, consultation with vendors and review of the listed materials by NASA personnel resulted in further selectivity, so that the final list comprises two cryogenic insulations, four adhesives, and three vapor barriers. These nine materials are discussed below in terms of (1) criteria for selection of individual products, (2) information sources for material review and evaluation, and (3) candidate material selection.

2.1.1 Criteria for Selection of Individual Products

Commercially available cryogenic insulations, adhesives, and vapor-barrier materials were reviewed to determine their suitability for use in a composite insulation system for cryogenic tankage. Individual products are selected in accordance with the following criteria: (1) nuclear-radiation and/or cryogenic-temperature stability, (2) suitability of properties for the desired application, (3) relative ease of handling and fabrication, and (4) cost of materials and production. Relative merits and limitations need to be appraised, since any one material seldom has all the desired or specified properties for the required environmental stability and production requirements in the application under consideration.

Important in space programs is the development factors to be considered in the production of test modules and advanced vehicles for flight programs. In the review, the potential applications of the materials selected for space flight and pre-launch stability is important, as well as the suitability for the ground test installation.

2.1.2 Information Sources for Material Review and Evaluation

Several research programs initiated by NASA and aerospace contractors have evaluated insulation materials and developed feasible methods of applying them on the inside and outside of cryogenic tanks. As a result of these programs, a number of materials and several fabrication procedures are available for consideration as composite cryogenic insulations for the LH_2 tanks. However, the radiation stability of these materials, individually or as part of the composite LH_2 cryogenic insulation system, have not been adequately determined.

The information surveyed included NASA Contract NAS8-2450 technical documents, data files at NARF, and documents of the Division Research Library at General Dynamics. These information sources were adequate for the initial selection of materials. Many important key references were on hand and others are being requested. Materials evaluation in support of the program is being vigorously pursued.

One document that has been found invaluable in the study and evaluation of plastics is Plastec Report 20, edited by A. H. Landrock and published in July 1965 by the Plastics Technical Evaluation Center (PLASTEC) at the Picatinny Arsenal

(Ref. 1). This document, compiled with the cooperation of the Cryogenics Data Center of the National Bureau of Standards at Boulder, Colorado, and the Defense Documentation Center, reviews the effects of cryogenic temperatures on plastics and related materials. It includes an annotated bibliography that contains 319 references representing reviews, symposium and conference papers, government agency and contractor reports, and journal articles. Specific sections of the report pertinent to this contract are Section 3; "Cryogenic Insulation"; Section 6; "Adhesives"; Section 7; "Plastic Films, Film Laminations and Vapor Barriers"; and Section 12; "Radiation Effects, Combined Effects". This report has been of material aid in evaluations and selections needed for this study on cryogenic insulations.

A voluminous source of information for the evaluation of cryogenic materials is the series of conferences on the Advances in Cryogenic Engineering (Ref. 2). In the appraisal of the radiation effects to be anticipated in the various material categories, the reports covering the work done under Contract NAS8-2450 are essential (Refs. 3, 4, 5, 6, and 7). Another source of essential information are several papers that were presented at the Seventh National SAMPE Symposium on Adhesives and Elastomers for Environmental Extremes, held in Los Angeles in 1964 (Ref. 8).

The large amount of information that was gathered on various materials for specific tests will not be presented here. Rather, a general discussion of the materials properties considerations is given, along with references of the reports containing the detailed information.

2.1.3 Candidate Materials Selection

2.1.3.1 Cryogenic Insulation

The insulation materials for cryogenic tankage were reviewed. Multiple-layer insulations and powder insulations are feasible from the thermal-conductivity viewpoint, but they are not applicable for the construction and service intended. Cryogenic insulation types selected for testing are cryogenic corkboard and polyurethane spray foam.

Cork is good for insulation at cryogenic temperatures and will be easy to fabricate and install on the flat surfaces of the tank. Boeing conducted thermal cycle tests on typical corkboard insulation panel for the intended application (Ref. 9). A major disadvantage of most corkboard is its high density - approximately 20 lb/ft^3. However, Armstrong

Cork Company has a new lightweight corkboard, Insulcork 7326 Cryogenic Insulation, of 7-8 lb/ft^3 density. The effects of radiation on the structural properties of wood have received little attention. Bolt and Carroll (Ref. 10) have summarized the limited information available on the effects of radiation on cork. In 1951, several types of wood, including cork, were exposed in the Canadian NRX (Ref. 11); samples exposed showed only slight darkening after a dosage of 6 x 10^{11} ergs/gm(C). West and Carroll exposed ordinary stopper corks to gamma radiation of about 5 x 10^{10} ergs/gm(C) (Ref. 12); the stoppers showed no discoloration or increased brittleness. In 1958, De Zeih exposed cork in air to 1 x 10^{10} ergs/gm(C) gamma radiation (Ref. 13); there was a slight (4%) increase in tensile strength, flexibility was not impaired, and compression and recovery from compression were reduced only about 5%. From these experiments, it is concluded that cork has adequate radiation resistance as a cryogenic insulation material for evaluation in the proposed radiation environmental application.

Another consideration regarding cork insulation is the possible deterioration of the cork binder materials. The

Insulcork 7326 is a clean grade of prime cork with a thermosetting resin binder. The phenolic, polyester, and other thermosetting resins are usually of high-radiation resistance; therefore, the binder is not expected to be a source of failure.

The corkboard selected for testing is the Insulcork 7326 Cryogenic Insulation distributed by the Industry Products Division of Armstrong Cork Company, Lancaster, Pa. Standard sheet size is 26 by 47 in. (rough, trimmed edges) and available in thicknesses of from 1/16 through 1-in. (Ref. 14).

A primary use of rigid foams is the cryogenic insulation of low-temperature containers, tanks, and lines. The relative merits of the various basic plastics that may be foamed is discussed by Ferrigno in Rigid Plastics Foams (Ref. 15). Epoxy, polystyrene, phenolic, vinyl, silicone, and polyurethane foams have been considered in the RIFT (Reactor-In-Flight-Test) program (Ref. 16). Desirable properties for the monolithic insulation systems when exposed to cryogenic temperature are (1) low tensile and compressive moduli, (2) low thermal expansion coefficient, (3) low Poisson's ratio, (4) low thermal conductivity, and (5) low density. However, the foaming characteristics, handling and fabrication problems, and radiation resistance are of major concern.

The foam selected is from a group of isocyanate-polyol resins commonly known as polyurethanes. They are formed by the reactions of compounds containing two or more active hydroxyl, amino, or carboxyl groups, with diisocyanates. The principal polyols used today are either polyesters or polyethers. The highly branched polyesters give rigid polyurethanes.

The rigid, small, uniform closed-cell structure of the spray foams gives low thermal conductivity. The foam has an extremely low moisture-vapor permeability and a high resistance to water absorption. The structural stability prevents costly heat leaks caused by shrinkage, buckling, or cracking. The spray system produces a uniform layer and tight adherence to pipes, supports, etc., on the tankage installation. These foams have low-density (nominal 2.0 lb/ft^3) and high-strength that means a saving in space, weight, and supports structure. A wide choice of adhesives is permissible because of the solvent resistance. However, a vapor-barrier coating or film composite is still considered as a necessary covering on the cryogenic insulation system.

The REIC Report No. 21 (Ref. 17) reported that Tomashot (Ref. 18) tested the ultimate flexural strength and flatwise compressive strength of polyurethane foam sandwich constructions after irradiation. The foam sandwich specimens showed no reduction in mechanical properties up to 1×10^{11} ergs/gm(C), the largest dose to which the specimens were subjected.

The polyurethanes as a chemical class are considered to be among the best in radiation resistance as elastomers as well as in foams. It may be stated that Harrington's studies (Ref. 19) indicate that these elastomers are capable of giving satisfactory service to at least 8.7×10^{10} ergs/gm(C). The work by Schollenberger at the B. F. Goodrich Co. (Ref. 20) concluded that limited dynamic applications might be practical after an exposure of 1.7×10^{11} ergs/gm(C) and static applications might be feasible after exposure to 4.4×10^{11} ergs/gm(C). Additional information is contained in the original articles and REIC review. The urethanes are high on the list of radiation-resistant elastomers and plastics. Since there are many chemical combinations, some variation in reported results in the elastomers and foams is to be expected. The Addendum to the REIC Report 21 (Ref. 21) reviews recent irradiations of urethane foams and elastomers. A safe radiation level is 1×10^9 ergs/gm(C), with a possible service level to a little over 1 x 10^{11} ergs/gm(C). This variation is to be expected in a group of compounds that vary in basic chemical compositions, temperature cures, additives, and fabrication procedures.

The thermal conductivity of polyurethane foams after irradiation was investigated at the Fort Worth Division. The results were not all that had been desired, since a factor in the experimental equipment gave results that were highly questionable. Both the CPR-20-2* and CPR-1021-2 polyurethane foams were irradiated. Details of the experiment are presented in Reference 6. The compressive strength of CPR-200 and CPR-1021 foams was tested. Both of these foams have given satisfactory service in the Saturn S-1 vehicle. The CPR-200 is a polyester, fluorocarbon-blown, rigid urethane foam. CPR-1021 is a polyfunctional, polyether-base, carbon-dioxide-blown, rigid urethane foam. When these foams were irradiated in air and vacuum in the GTR radiation field, there were no significant changes in the compressive strength at 25% compression after the maximum dose levels of 1.36 x 10^9 ergs/gm(C) in air and 9.1 x 10^8 ergs/gm(C) in vacuum. Complete results are presented in Reference 5.

Testing of these urethanes were continued in the subsequent NAS8-2450 program, with results of thermal conductivity

*CPR-20-2 and CPR-200-2 are the same CPR product.

and compression tests reported in Reference 7. The foams were not sufficiently affected after 3×10^{10} ergs/gm(C) gammas exposure to the reactor radiation field to be considered damaged.

Based on the general survey, it is predicted that the polyurethane foams will be satisfactory at 1×10^{11} ergs/gm(C) in both thermal conductivity and mechanical strength. The polyurethane adhesives will be discussed in the following section. The general behavior of these adhesives confirms results on elastomers and foams, i.e., that the urethanes, as a family of plastics and elastomers, are considered relatively high in radiation resistance as organic compounds.

Additional insulation information is contained in the references cited in Plastec Report No. 20 and papers in the Advances in Cryogenic Engineering series of conferences. Both the cork and urethane foams have advantages. It is anticipated that either could be used in the GTM LH_2 tank insulation - the cork on cylindrical flat surfaces and the urethane spray foam in areas where attachments or piping are located.

A recommended spray foam is the CPR 368 Isonate System. CPR 368 is a rigid, nonburning, PAPI-based spray system designed for either airless or air-atomized spray equipment for particular use at low ambient temperature conditions. The vendor is CPR Division of the Upjohn Company.

2.1.3.2 Adhesives

Since the need for structural adhesives for cryogenic applications in aerospace programs has been a major concern for several years, there has already been considerable developmental effort in this field. At the SAMPE symposium in 1964 (Ref. 8), papers of special interest on adhesives were presented by Kausen (Arthur D. Little, Inc.), Kuno (Whittaker Corp., Narmco Division), and Roseland (Douglas Aircraft Company), as reported in References 22, 23, and 24, respectively. The Plastec Report 20 and the series of conferences on Advances in Cryogenic Engineering (Refs. 1 and 2) also carry excellent information on the aerospace adhesives and their development.

The adhesives being considered in this program are for bonding the cryogenic insulation to the tank, bonding insulation blocks together, and bonding the vapor-barrier material to the outside of the insulation composite. It should be borne in mind that the fabrication and handling requirements established for the program eliminate the most desirable types of adhesives, i.e., those that require high-temperature and/or high-pressures for proper curing. The adhesives selected for testing will be applied as room curing putties or pastes having a relatively long pot life that need only contact pressure during bonding.

Many of the problems associated with bonded joints at cryogenic temperatures are the result of stress concentrations and gradients developed within the bond. Some of the principal causes are: (1) difference in thermal coefficient of expansion between adhesives and substrates, (2) shrinkage of adhesive on curing, (3) trapped gases or volatiles evolved during bonding, (4) difference in modulus of elasticity and shear strengths of adhesive and substrate, (5) differences in thermal conductivity of adhesive and substrate, (6) residual stresses in substrates as a result of the release of bonding pressure, (7) inelasticity in the adhesive or substrate, and (8) plasticity in the adhesive or substrate. Several classes of adhesives that have the desired properties enumerated above are polyurethanes, epoxy-polyamides, epoxy-nylons, modified epoxies, and epoxies.

According to the test plans (Section 3) the adhesives selected must not only be established cryogenic adhesives with the above requirements, but must also be radiation-resistant as well. The adhesives are to be exposed to the radiation field of the GTR while in air and while submerged in LH_2. Postirradiation testing will be in air and in LH_2 for the lap-shear specimens after a dose of 1×10^{11} ergs/gm(C) gammas.

The epoxies and phenolic-base adhesives have a higher radiation tolerance, but most of these are cured at elevated temperatures. The epoxy-nylons are good cryogenic adhesives but they also have a high-temperature cure. However, the polyurethanes are good radiation-resistant plastics (see previous discussion on cryogenic insulations), and the epoxy-polyamides, in general, are better in the radiation environment than many other adhesives.

The adhesives that have been selected for final testing are either polyurethanes or epoxy-polyamides. All of these adhesives may be applied by hand and bonded at room-temperature cure conditions. Furthermore, they have been used in previous aerospace programs. The polyurethanes are NARMCO 7343/7139 (Whittaker Corp., Narmco Materials Division) and APCO 1252 (Applied Plastics Co., Inc.). The epoxy-polyamides are Lefkoweld 109/LM-52 (Leffingwell Chemical Co.) and NARMCO 3135/7111. The flatwise-tensile strength composite insulation test will be assembled with the NARMCO 7343/7139 and Lefkoweld 109/LM-52 to represent the two adhesive classes selected. The Lefkoweld 109 Epoxy Adhesive is blended with 74 parts (weight) of Lefko LM-52 polyamide curing agent. It is suitable for end use applications from the low cryogenic (-423°F) temperature to 300°F service temperature. It is being used in diverse applications in the Apollo-Saturn program by major contractors. NARMCO 7343/7139 is a 2-part, room-temperature-curing polyurethane adhesive developed specifically for cryogenic applications. It provides high-shear, compressive, and tensile stresses when used as an adhesive for bonding nonmetallics and thermosetting plastics at temperatures as low as -423°F. NARMCO 7343 can easily be applied to a wide variety of substrates by troweling or through conventional sealant guns. Typical applications for this adhesive have been the bonding of doublers, clips, and brackets to skin portions of cryogenic fuel and oxidizer containers and the bonding of Mylar, Tedlar, and H-film (Kapton) barrier films to nonmetallic cores where a nonpermeable insulation was required. The NARMCO 3135 is compatible with plastic sandwich cores, polyurethane foams, and phenolic laminates. The curing agent to resin mixing portions is 50:50 by weight. The APCO 1252 has similar properties. It is also recommended for bond plastics, foams, metals, or elastomers to themselves or to other materials.

Chafey at General Dynamics/Astronautics reported on five classes of adhesives that were selected for evaluation at cryogenic temperatures on the basis of promising high lap-shear strengths at -65° and 75°F (Ref. 25). The results are summarized in the Addendum to REIC Report No. 21, (Ref. 21). NARMCO 3135, Resiweld No. 4 (epoxy-polyamide adhesives), and APCO 1219 (polyurethane adhesive) were included. Lap-shear specimens were tested at -423°, -320°, -100°, and 75°F. As has been previously stated, Chafey found that the room-temperature-cured adhesives are generally inferior to those that are heat cured. Of the three room-temperature-cured adhesives tested, the polyurethane gave higher lap-shear strengths than the epoxy-polyamides with an aluminum adherend and approximately the same strengths with stainless-steel adherends. All the adhesives tested had their highest lap-shear strengths at -100°F.

Of the four adhesives, two have been irradiated. General Dynamics irradiated APCO 1252 and Lockheed irradiated Lefkoweld 109/LM52.

The polyurethane adhesive (APCO 1252) irradiations covered the entire NAS8-2450 contract period from 1961 to 1966. Air irradiations were reported in References 4, 5, and 7; cryogenic irradiations (LN_2 and LH_2) in Reference 4; vacuum irradiations in Reference 5; and cryo-vac irradiations in Reference 7. In all controls, the decrease in temperature increased the tensile shear strength. One air control was 2240 psi and the LH_2 control was 5928 psi; but, on the other hand, another air control was as high as 3228 psi. During air irradiations to 3×10^{10} ergs/gm(C), the lap-shear specimens increased in tensile shear-strength. In vacuum and at cryogenic temperatures, only slight decreases in shear strength were noted at this same radiation level. The highest irradiation in air was to 1.7×10^{11} ergs/gm(C) (Ref. 7). For this high dose the shear strength decreased from 3228 psi to 2750 psi; however, this irradiated value was no lower than an air control value previously reported, i.e., 2743 psi (Refs. 4, 5, and 7). Here is an excellent illustration of the complexity of obtaining, reporting, and comparing nuclear irradiation data in combined environments by the use of commercially produced adhesives. Manufacturing parameters, specimen preparations, irradiation dosimetry, testing environments, and testing parameters influence reported data.

The effects of radiation on adhesives, as well as on thermal insulations and vapor-barrier materials, was investigated in the RIFT Radiation Effects Program (Refs. 26 and 27). The materials tested are similar to those being investigated in the General Dynamics program; however, the radiation level was too low to cause any serious radiation damage.

The Lefkoweld 109/LM52 epoxy-polyamide adhesive was tested in the RIFT program (Ref. 27). Three identical sets of test specimens were prepared. One set was conditioned and tested in ambient laboratory condition to provide reference data for comparison with ambient control data from other sources. The second set was conditioned and tested in liquid nitrogen to provide base-line control data for comparison with the data from the irradiated specimens. The third set of specimens was irradiated and tested in liquid nitrogen. The radiation level was 2.6×10^7 R. Lap-shear and tensile specimens were prepared and tested. The lap-shear ultimate strength was 3900, 2100, and 1900 psi for the respective test conditions, while the flatwise-tensile strength was 3900, 7000, and 7700 psi, respectively, for the three test conditions of air-ambient control, LN_2 control, and LN_2 irradiated. Since this is a low radiation level, irradiation in LN_2 had very little effect. The temperature effect is more pronounced and is of interest in that the change was a decrease for the lap-shear and an increase in flatwise-tensile strength as shown by the values presented above.

Based on radiation effects and cryogenic temperature test results reported to date, it is predicted that in the tests planned, the polyurethane adhesives will function best. The flatwise-tensile composite specimens should indicate the relative bonding capabilities of the polyurethane and epoxy-polyamide adhesives under a simulated service condition.

2.1.3.3 Vapor-Barrier Materials

The vapor-barrier materials are placed over the cryogenic insulation to keep moisture and air out of the porous insulation. For this test program, design parameters include radiation stability, gas permeability, low-temperature flexibility, film thickness, tensile strength, tensile moduli, linear expansion, bondability, cost of material, and method of installation. The successful operation of a foam depends upon the maintenance of a high-vacuum inside the closed cells of the foam. This vacuum is created in the cells by the condensation of the blow gas, such as Freon, when the tank is filled with LH_2. Since moisture or air permeating through the vapor barrier into the foam will considerably increase the thermal conductivity of the foam, it is important to have a vapor barrier of low permeability. The same principles apply for the corkboard.

Section 7 of the Plastec Report 20 (Ref. 1) reviews the development of the plastic films, film laminations, and vapor barriers for use in the insulation of cryogenic tanks. These studies are quite varied in purpose and scope and too lengthy to be reviewed here in further detail; however, brief comments will be made. The combined environmental conditions of cryogenic temperatures and high radiation levels of up to 1×10^{11} ergs/gm(C) gammas limit the available film materials.

Vapor-barrier materials may be bare films in various gages, films combined as composites, films laminated with such reinforcements as glass cloth or epoxy-fiberglass, and plastic films vapor-deposited, spray coated, or bonded with metal films.

A survey of available information on the effects of radiation and cryogenic temperatures on these films indicates that the following plastic films are the most promising candidate vapor-barrier materials: a polyamide (Kapton); a polyester (Mylar); a polyvinyl fluoride (Tedlar); and epoxy-glass cloth, either bare film or film coated with aluminum as composites or combined laminates.

Dahlin selected a complete vapor-barrier system (Ref. 9) for covering corkboard insulation. The vapor-barrier system includes a troweled or sprayed coating of polyurethane rubber-base mastic, aluminized Mylar film with seams sealed with a pressure-sensitive adhesive tape (3M 850) that has a 1-mil aluminized Mylar film backing. Miller et al. presented information on vapor-barrier films in support of the RIFT program (Refs. 28 and 29). The low-temperature tensile properties of films have been reported by Caren et al. (Ref. 30), Mowers and (Ref. 31), Toth and Barber (Ref. 32). The linear thermal contraction has been compared by Toth and Barber (Ref. 32). Bailey (Ref. 33) and Caren (Ref. 30) determined the hydrogen permeability of plastic films. Labovits in March 1966 surveyed the permeability of polymers to gases, vapors, and liquid (Ref. 34). Lynch (Ref. 35) compared the results of single monalayers of bare films and composite films.

The effects of radiation on the tensile strength and flexibility has been investigated at General Dynamics by Kerlin and Smith (Refs. 3, 4, 5, 6, and 7). The RIFT program mentioned previously in the adhesive section (Refs. 26 and 27) also irradiated films to 10^7 R. The results of these studies on tensile strength, thermal contraction, permeability, and radiation resistance indicate several bare films, metalized films, and laminates should be evaluated in this program, since no one material is outstanding in all desired vapor-barrier properties - chemical class or product.

Mylar has outstanding tensile properties but failed when irradiated before reaching the desired 1×10^{11} ergs/gm(C). Kapton has outstanding radiation resistance and could be used at 1×10^{11} ergs/gm(C); however, it is still expensive and does not equal other films as a vapor barrier to moisture, hydrogen, and air. Tedlar is between Mylar and Kapton in radiation resistance at 1×10^{11} ergs/gm(C), as shown on tests of films at General Dynamics. The aluminized Mylar films have lower vapor penetration values than the bare film, but there is not sufficient radiation effects test data to establish a feasible threshold of damage. Tedlar and the fiberglass laminates have low-gas permeability. The laminates and film composites are strong, radiation resistant but are expensive to fabricate. For thermal contraction, the laminates are lowest and are followed by Mylar, metalized Mylar, Kapton, and Tedlar. The coefficient of thermal contraction for Tedlar from room temperature to LN_2 temperature is twice that of Mylar A and a third higher than Kapton (Ref. 32).

It is evident from these brief remarks on the various film and vapor-barrier materials that none could be a "first" choice.

Mylar, Kapton, and laminate materials have been selected because of their strength, low permeability, and radiation resistance, respectively. Because of its high thermal contraction, Tedlar could have stresses and could crack over surface areas of the NERVA Ground Test Module LH_2 tank when it becomes cold. However, it will be kept in reserve as a back-up, in case Mylar does not qualify after irradiation to 1×10^{11} ergs/gm(C). In fact, Tedlar could be tested in place of Kapton as a tensile specimen film if present NASA tests with Tedlar on the LH_2 dewar are successful.

Additional engineering design and installation factors that need consideration in future tests include film thickness vs radiation stability, moisture penetration, ease of application, and bondability to the cork, foam, or mastic coating.

The selection of the vapor-barrier material, the application of the film to the insulation, and the successful sealing of film seams and edges are the "weakest link" in a composite-insulation system. It is recommended that new materials and material composites continue to be evaluated and that the actual design and method of attachment of these films - vapor barriers - to the cryogenic insulation be given further consideration not only for this insulation test dewar but also for the LH_2 tank cover for the NERVA Ground Test Module.

2.2 Special Specimen Fabrication Procedures

The GTM cryogenic insulation must be firmly attached to the skin of the LH_2 aluminum tank. To prevent oxygen penetration of the insulation and to exclude moisture from it, the insulation is covered with a suitable vapor barrier material. To test this composite insulation system that is bonded with adhesives, a special composite sandwich specimen has been designed which is a modification of the flatwise-sandwich tensile specimens of ASTM C 297-61. The proposed arrangement of the materials in the assembly for testing the polyurethane spray foam and corkboard insulations is illustrated in Figure 4.4. The method of attachment of the vapor barrier materials, the tensile strength of the selected adhesives, and the adherent properties of the spray foam and corkboard to the aluminum skin need investigation before a final composite insulation panel system is fabricated on the LH_2 test dewar in this program.

In this test, the specimens will have the vapor-barrier film or sheet material bonded to the insulation; however, on the GTM tank and LH_2 composite test panel, a sprayed or troweled mastic coating may be applied to the surface of the insulation. Furthermore, the vapor-barrier material film or sheet will have to be sealed with adhesive tapes of suitable material or be overlapped and wrapped as it is assembled. This test is mainly a qualification of adhesives.

The limited irradiation space in the LH_2 test dewar permits only two types of cryogenic insulations to be tested. One is a low-density cryogenic corkboard insulation - Insulcork 7326; the other, a rigid low density polyurethane spray foam - CPR 638. Both are recommended for these flatwise-tensile specimens.

The insulation composite is attached to the loading blocks that fit into the loading fixture of ASTM C 297-61. Detailed drawings of the loading fixture and loading blocks were forwarded to NASA. It is recommended that the upper and lower blocks of each specimen be treated differently to simulate possible conditions of the LH_2 tank surface. If one block is chemically cleaned with a solvent and left bare while the other block is etched, the relative adherent properties of these aluminum surfaces may be compared under similar fabrication, radiation, and test conditions.

Many excellent adhesives are eliminated by the requirements of this test. The adhesives selected for bonding the aluminum blocks, the cryogenic insulations, and the vapor-barrier materials require specifications, not only for fabrication of the tensile composite specimens, but also for endurance of severe environmental conditions. For example, fabrication of the assembly requires that the adhesives be applied by hand, cured at room temperature, and bond together different substrate materials.

The irradiation performed in LH_2 to 1×10^{11} ergs/gm(C) and followed by a room-temperature tensile test further restricts the qualified candidate adhesive systems. This is a high radiation level for most plastics. Two classes that have been tried in cryogenic space programs are the polyurethane-base adhesives and the combined epoxy-polyamide 2-component system. The insulation/vapor-barrier combinations will be bonded with these cryogenic adhesives. Table 2.2 lists the two cryogenic adhesives. The room-temperature-curing polyurethane is Narmco 7343/7139, and the epoxy-polyamide is Lefkoweld 109/LM52. Each adhesive bonds various materials; therefore, they should be good for this test.

The vapor-barrier materials for the composite tensile specimens present several different substrates that need to be bonded to the insulations. The Mylar-Al-Mylar gives the Mylar surface and the Al-Mylar-epoxy/glass gives both Al and epoxy surfaces for bonding to the insulation. If the Mylar-based materials prove unsatisfactory in the radiation field at the test temperatures, then Kapton (Type H) film will be considered. In this initial test program, these tensile specimens are not using Kapton because of the cost factor. Kapton is known to have excellent radiation resistance and cryogenic properties, but it is costly relative to the other available vapor-barrier films. Table 2.1 lists the selected vapor-barrier materials. The Schjel-Clad Laminate Type X-501 is a Mylar-Al-Mylar thin film. The Laminate II is aluminized Mylar bonded to an epoxy/glass laminate with an epoxy adhesive; but if it is not available, Schjel-Clad composites using epoxy/glass laminates are available.

The test plan for the T-peel specimens that this test replaces allowed four combinations of each insulation, with four specimens in each combination. By selection of two aluminum surfaces, two insulations, two adhesives, and two vapor barriers there will be a representative composite combination of each tested as air controls, air irradiations, LH_2 controls, and LH_2 irradiations. The LH_2 control and LH_2 irradiated specimen pulls will be performed at ambient-air conditions in the Instron after the specimens have been removed from the LH_2. This allows the effects of cryogenic temperature and/or radiation to be compared. There will be 16 specimens of each combination, 64 of each insulation, 64 of each adhesive, and 64 of each vapor-barrier material. This makes a total of 32 test specimens to be pulled in the Instron at ambient-air temperature after each exposure to the four environmental test conditions.

The modified test plan for the Flatwise Tensile Pie-Composites is summarized in Tables 2.1, 2.2, and 2.3. Table 2.1 lists the materials selected for the Flatwise-Tensile Pie-Composite specimens; Table 2.2 presents the details of the pie-composite arrangements; and, Table 2.3 separates the eight sets of composite specimens into the four environments of this test, the total for each set, and the total number of specimens for the Flatwise Tensile Composite group.

The method of preparation of the composite flatwise tensile specimens is an important factor to consider in this test. The

Table 2.1

COMPOSITE MATERIALS FOR FLATWISE-TENSILE SPECIMENS

	Cryogenic Insulations
TI-1	CPR 638, polyurethane spray foam, PAPI and polyether base
TI-2	Insulcork 7326, cryogenic cork board, phenolic binder
	Cryogenic Adhesives
CA-1	Narmco 7343/7139, polyurethane, room temperature cure
CA-2	Lefkoweld 109/LM52, epoxy-polyamide, low-temperature cure
	Vapor Barriers
VB-1	Schjel-Clad Laminate, Type X-501, Mylar-Al-Mylar
VB-2	Laminate II, or Schjel-Clad composite, Al-Mylar-epoxy/glass
	Loading Blocks
LB-1	Aluminum 2219, bare, chemically cleaned
LB-2	Aluminum 2219, etched overall or at specimen composite end

Table 2.2

COMPOSITE ARRANGEMENT OF FLATWISE-TENSILE SPECIMENS

Polyurethane Foam Insulation*

Spec.	Prep.	Block	Insulation	Adhesive	Barrier	Adhesive	Block
FWT-1	18	LB-1	TI-1	CA-1	VB-1	CA-1	LB-2
FWT-2	18	LB-1	TI-1	CA-1	VB-2	CA-1	LB-2
FWT-3	18	LB-1	TI-1	CA-2	VB-1	CA-2	LB-2
FWT-4	18	LB-1	TI-1	CA-2	VB-2	CA-2	LB-2
	72						

Cryogenic Corkboard Insulation**

Spec.	Prep.	Blk./Adh./Ins./Adh./Ins./Adh./Bar./Adh./Ins./Adh./Ins./Adh./Blk.
FWT-5	18	LB-1/CA-1/TI-2/CA-1/TI-2/CA-1/VB-1/CA-1/TI-2/CA-1/TI-2/CA-1/LB-2
FWT-6	18	LB-1/CA-1/TI-2/CA-1/TI-2/CA-1/VB-2/CA-1/TI-2/CA-1/TI-2/CA-1/LB-2
FWT-7	18	LB-1/CA-2/TI-2/CA-2/TI-2/CA-2/VB-1/CA-2/TI-2/CA-2/TI-2/CA-2/LB-2
FWT-8	18	LB-1/CA-2/TI-2/CA-2/TI-2/CA-2/VB-2/CA-2/TI-2/CA-2/TI-2/CA-2/LB-2
	72	

Total Requirements: 144 specimens of Flatwise-Tensile Composites

*Since the polyurethane foam is sprayed directly on the aluminum block, there is no need for the adhesive to join insulation to substrate. Also, the foam may be applied to required thickness by spraying over entire specimen set.

**Since the corkboard is procured in a specified thickness, it must be bonded to the aluminum substrate by an adhesive. The corkboard application to the GTM will require that more than one layer of insulation be used to overlap the seams; therefore, the cork insulation will be bonded to a second piece of cork insulation in these tests.

Table 2.3

CONDITIONS AND SPECIMENS FOR FLATWISE-TENSILE TESTS*

Specimen Type and ASTM Designation	Test Spec. Code	Test Conditions** Control Air	Control LH_2	Irradiated Air	Irradiated LH_2	Spares	Total for Each Composite and Group
Foam							
Composites	FWT-1	4	4	4	4	2	18
ASTM C 297-61	FWT-2	4	4	4	4	2	18
Flatwise-Tensile	FWT-3	4	4	4	4	2	18
	FWT-4	4	4	4	4	2	18
	(Foam)	16	16	16	16	8	72
Cork							
Composites	FWT-5	4	4	4	4	2	18
ASTM C 297-61	FWT-6	4	4	4	4	2	18
Flatwise-Tensile	FWT-7	4	4	4	4	2	18
	FWT-8	4	4	4	4	2	18
	(Cork)	16	16	16	16	8	72
	TOTALS	32	32	32	32	16	144

*All irradiations to 1×10^{11} ergs/gm(C) gamma in 10 Mw GTR at NARF.

**All tensile tests in Instron at ambient-air temperature.

specimens should be as uniform as possible in quality. If all specimens of a series could be cut from a single composite panel, there would be less variations in the sets tested - which improves the reliability of the environmental test data. Differences in the physical state of the cork insulation boards and the polyurethane foam requires separate procedures in preparation of the composite panels.

Cork insulation is generally applied to the cryogenic container in several layers by an adhesive/sealant in order to overlap the seams between the boards and to provide the necessary thickness for temperature control. Also, the vapor-barrier material is applied on the outside surface of the cork insulation. Two procedures seem feasible for preparing the cork composite panels. The cork composite could be fabricated layer by layer, starting with the bottom aluminum block and finishing with the top block. An alternate procedure is to bond the cork boards to form two cork sheets, i.e., to insert the vapor barrier between the cork sheets using the same adhesive as that for bonding the cork sheets. After the composite adhesives have cured, the set of blocks could be attached and the composite specimens separated by cutting or sawing and finishing on the edges. The blocks should be attached to the cork as an assembly of blocks. Attachment of one block at a time would reduce the uniformity of the adhesive bond between the cork and the blocks. The composite panel needs to extend beyond the block assembly on all sides to allow for the discarding of the edge of the panel.

The specimens for the polyurethane-foam insulation could be fabricated by assembly of the blocks into a unit, or set, with spacers between the blocks to allow for later cutting or sawing of the blocks apart after completion of the composite panel. Each set of blocks - the top set and the bottom set - is easily sprayed with the foam to the desired thickness. After the foam is cured and ready for application of the vapor-barrier material, the foam surfaces could be smoothed by any suitable method if the surface is considered too rough to apply the vapor barrier material. The adhesive can then be applied to the foam and the vapor barrier bonded between the foam insulation panels, so that individual specimens can be cut from the composite panel assembly and smoothed on the edges.

In assembly of the blocks to the panels, special care should be taken to have the top and bottom blocks in the proper relationship, so that the pin holes will line up with the pull-assembly pin holes which are at right angles in the top and bottom pull assemblies, as illustrated in ASTM C 297-61.

The above discussion on the preparation of the specimens is not intended as specific instructions. This is a feasible means of obtaining more reliable data by production of a uniform set of specimens from a single panel. Variations caused by fabrication procedures are reduced.

Regardless of the method of specimen preparation, the final specimen configurations should have the edges of the loading blocks in the direction of pull in the same plane. The surfaces of the two loading blocks attached to the composite specimen should be parallel to each other. These requirements are to prevent eccentric loading and shear forces on specimens during tensile pull. Several procedures for panel and specimen assemblies have been suggested by personnel at General Dynamics. These methods should be discussed with NASA personnel before fabrication of the panels.

III. TEST PLAN

Two types of tests are planned for the evaluation of the thermal insulation materials. The first is a evaluation of the mechanical properties of the components of the thermal insulation system. The second test is an application test of the composite thermal-insulation system to a dewar.

Samples from the same batch of materials will be subjected to the following test conditions: (1) qualification in air, (2) evaluation in LH_2, (3) irradiation in air, and (4) irradiation in LH_2. Tables 2.3 and 3.1 present the test plan for the program. Some of the specimens irradiated in LH_2 will be tested in LH_2 immediately after irradiation, without intervening warmup; the others will be brought to room temperature before testing. The specimens tested in LH_2 will be tested with Cryotensile Tester described in Section 5.1. The specimens to be tested at standard laboratory conditions will be tested with the Instron.

The selection of the two test environments was based on the required cross-head travel to pull the test specimens. Because of the limited test-pull travel of the dynamic test equipment, very few T-peel type materials samples could be tested. By irradiating them in LH_2 with subsequent testing in air, a statistical number of specimen can be evaluated.

Table 3.1

TEST TYPES AND TEST SPECIMENS FOR MATERIALS EVALUATIONS

Specimen Type and ASTM Designation	Test Material	Test Conditions: Control Air	Control LH_2	Irradiated Air	Irradiated LH_2	Total for Each Test and Group
Metal-to-Metal Adhesives Lap Shear D-1002	NARMCO 7343/7139	8[a]	8[b]	4	8[b]	28
	Lefkoweld 109/LM-52	8[a]	8[b]	4	8[b]	28
	APCO 1252	8[a]	8[b]	4	8[b]	28
	NARMCO 3135	8[a]	8[b]	4	8[b]	28
	Total No.	32	32	16	32	112
Metal-to-Metal Adhesives T-peel D-1876-61T	NARMCO 7343	4	4	4	4	16
	Lefkoweld 109/LM-52	4	4	4	4	16
	APCO 1252	4	4	4	4	16
	NARMCO 3135	4	4	4	4	16
	Total No.	16	16	16	16	64
Thin-Film Vapor Barrier Tensile D882-61T	Mylar	8	8	8	8	32
	Laminate II	8	8	8	8	32
	Tedlar	8	8	8	8	32
	Total No.	24	24	24	24	96
Thin-Film Vapor Barrier Permeability D1434-63	Mylar	2	2	2	2	8
	Laminate II	2	2	2	2	8
	Tedlar	2	2	2	2	8
	Total No.	6	6	6	6	24
Thermal Insulation Compression Set D1621-59T	CPR-368	4	4	4	4	16
	Insulcork 7326	4	4	4	4	16
	Total No.	8	8	8	8	32

[a]1/2 of the specimens are to be tested in Instron and 1/2 tested in Cryotensile Assembly at ambient conditions.

[b]1/2 of the specimens are to be tested in air in the Instron and 1/2 tested in LH_2 in the Cryotensile Assembly.

Table 3.1 (cont'd)

Specimen Type and ASTM Designation	Test Material	Test Conditions				Total for Each Test and Group
		Control		Irradiated		
		Air	LH_2	Air	LH_2	
Metal-Ad-TI-Ad-Metal Lap-Shear D-1002-64 Modified	A-1-TI-1[c]	4	4	4	4	16
	A-1-TI-2	4	4	4	4	16
	A-2-TI-1	4	4	4	4	16
	A-2-TI-2	4	4	4	4	16
	A-3-TI-1	4	4	4	4	16
	A-3-TI-2	4	4	4	4	16
	A-4-TI-1	4	4	4	4	16
	A-4-TI-2	4	4	4	4	16
	Total No.	32	32	32	32	128
Composite Flatwise Tensile Strength[d] C-297-61 Modified	Corkboard	16	16	16	16	64
	Spray Foam	16	16	16	16	64
	Total No.	32	32	32	32	128

[c]A-1-TI-1 = Adhesive No. 1 with Thermal Insulation No. 1

[d]Details in Table 2.3

Table 3.2 shows the comparisons to be made in evaluating the data from the mechanical-property test on the material specimens.

The thermal-insulation test dewar will be maintained full of LH_2 during irradiation, then allowed to warm up before movement from the irradiation area to the acoustic test area. From there it will be subjected to 6 hr of acoustic noise on each of two sides as shown in the test procedures section 4.4.3 and test equipment section 5.3.

The radiation dose for the test of 1×10^{11} ergs/gm(C) was selected as using higher than the expected dose to be received by the materials in the GTM application.

The ASTM and special test procedures to be used for the test are discussed in Section IV.

Table 3.2

SCHEDULED LAP-SHEAR TEST

Test No.	Test Equipment	Test Environment	Specimen Configuration
		Control Test*	
1	Instron	Ambient	Standard
2	Aluminum Tester	Ambient	Modified
3	Instron	Ambient after LH_2 Dip	Standard
4	Aluminum Tester	LH_2	Modified
		Irradiated Test*	
5	Instron	Ambient Condition	Standard
6	Aluminum Tester	LH_2	Modified
7	Instron	Ambient after LH_2 Dip	Standard

*Test results will be compared as follows:

Tests 1 & 2 to give specimen configuration and tester difference
" 1 & 3 " " thermal cycle to LH_2 effect
" 2 & 4 " " cryogenic embrittlement at LH_2
" 1 & 5 " " irradiation effect at ambient temperature
" 3 & 7 " " combined effect of irradiation and LH_2 with testing at ambient condition
" 4 & 6 " " combined effects of irradiation and LH_2 with testing at LH_2
" 5 & 7 " " synergestic effect in LH_2 and irradiation

IV. TEST PROCEDURES

Two general types of experimental evaluations are planned for this program. The first is to evaluate the mechanical properties of components of the thermal insulation systems; the second, to evaluate the functional characteristics of the composite in a service test.

4.1 Mechanical-Property Test Specimen and Procedures

The test-specimen configurations presented here apply to the mechanical-property test part of the thermal-insulation test program. The configurations derived from applicable ASTM test specifications are designed to be compatible with the test equipment. Two new specimen configurations were designed for the tests: (1) a double lap-shear specimen to test a composite foam-adhesive system in tensile shear and (2) a modified flat-wise tensile specimen to test the insulation-adhesive-film composite system for maximum overall tensile strength. These specimens were designed similar to standard ASTM test configurations.

The vendor's recommended fabrication procedures will be used in preparing the test samples. Since each material will be tested in several different configurations (with optimum compound procedures different for each) the same compounding procedures will be used for all of the test samples for that material. The number of samples for each material to be tested in the program is given in Table 3.1 of the Test Plan (Section III).

4.1.1 Standard Lap-Shear Test

The standard ASTM D-1002-64T test specimen and the procedures are to be used in this test. This test will be conducted at room temperature on the the Instron Tester using standard wedge-action jaws. The rate of loading will be by a free crosshead speed of 0.05 in./min.

4.1.2 Modified Lap-Shear Test

Special specimen configurations are required for the dynamic test equipment used in testing in the combined environments of radiation and LH_2. A fabrication panel for these specimens is shown in Figure 4.1. Figure 4.2 shows the requirements for the final specimen configuration. The exact slot length depends on the loading sequence in the dynamic-test equipment. The ½-in. by 1-in. bonded area is in accordance with the ASTM designation, and the spacing between top and bottom doublers is the same as the jaw-grip

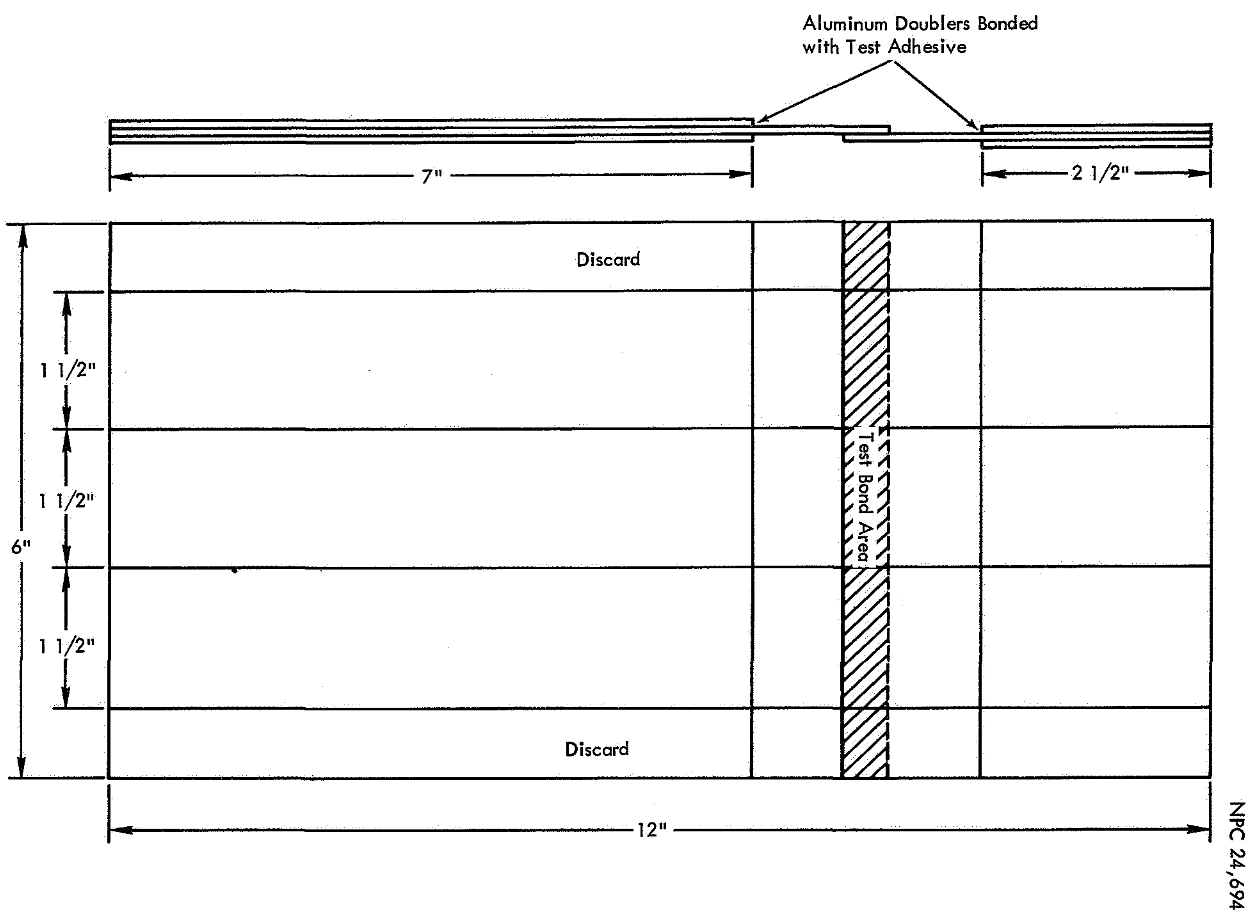

Figure 4.1 Modified Lap-Shear Bond Panel

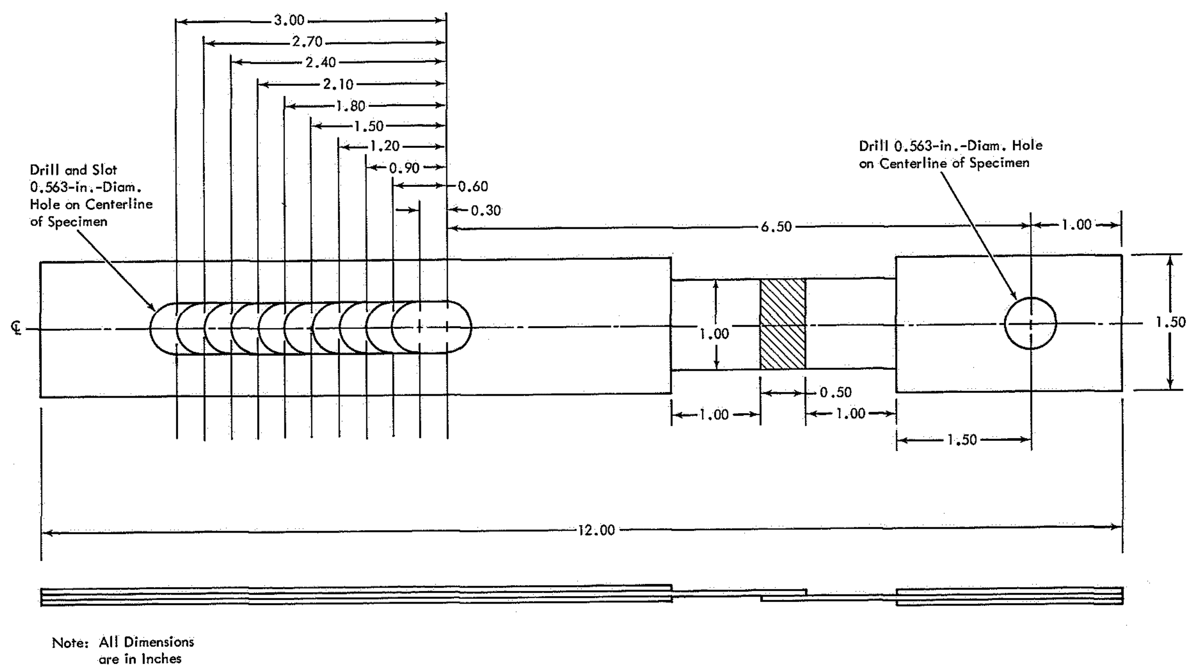

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Figure 4.2 Typical Lap-Shear Adhesive Specimen spacing specified by ASTM. Doublers are used for reinforcement of the holes and slots on the ends of the dynamic-test specimens. The testing speed will be 0.05 in./min.

4.1.3 Standard T-Peel Test

The ASTM D-1876 test specimen and the procedures are to be used in this test. The individual specimens are to be 1-in. wide and 12-in. long. The adherent material is to be 0.012 in.-thick 2024-T3 clad aluminum.

All of the T-peel specimens are to be tested in the Instron tester at standard conditions.

4.1.4 Thin-Film Tensile Test

Since all of the thin-film specimens are to be tested in the Instron, one of the standard specimen configurations prescribed in ASTM D-882-64T will be used. The selected specimen configuration is 0.010 in. thick, 1.00 in. wide, and 6 in. long.

4.1.5 Thin-Film Permeability Test

The standard test procedure described in ASTM E96-63T will be used for this test. The exact sample configuration is not given for these test procedures; the sample selected for the test is 0.010 in. thick and 4.0 in. square.

4.1.6 Thermal-Insulation Compression-Set Test

The test procedures and one of the standard test specimen prescribed in ASTM D-1621-59T will be used in this test. The selected specimen configuration is 3 in. wide, 3 in. long, and 1.0 in. thick.

4.1.7 Composite Double Lap-Shear Test

The specimens for the composite test of adhesive and thermal insulation material are to be a special configuration, since there is not a standard ASTM test specification for this test. The specimen design is shown in Figure 4.3. The specimens will be tested individually in the Instron at standard temperature and pressure, which will require the use of only one specimen pin-hole configuration. The specimens will be tested at 0.050 in/min. crosshead rate.

4.1.8 Flatwise Tensile Specimens

A composite test specimen will be fabricated to represent a cross section of the GTM tank insulation. The test sample will be fabricated as shown in Figure 4.4.

4.2 Control Tests

4.2.1 Mechanical-Property Tests

Control tests will be performed on specimens of all materials in the "as received" condition in an ambient-atmospheric environment in the IML. An additional control

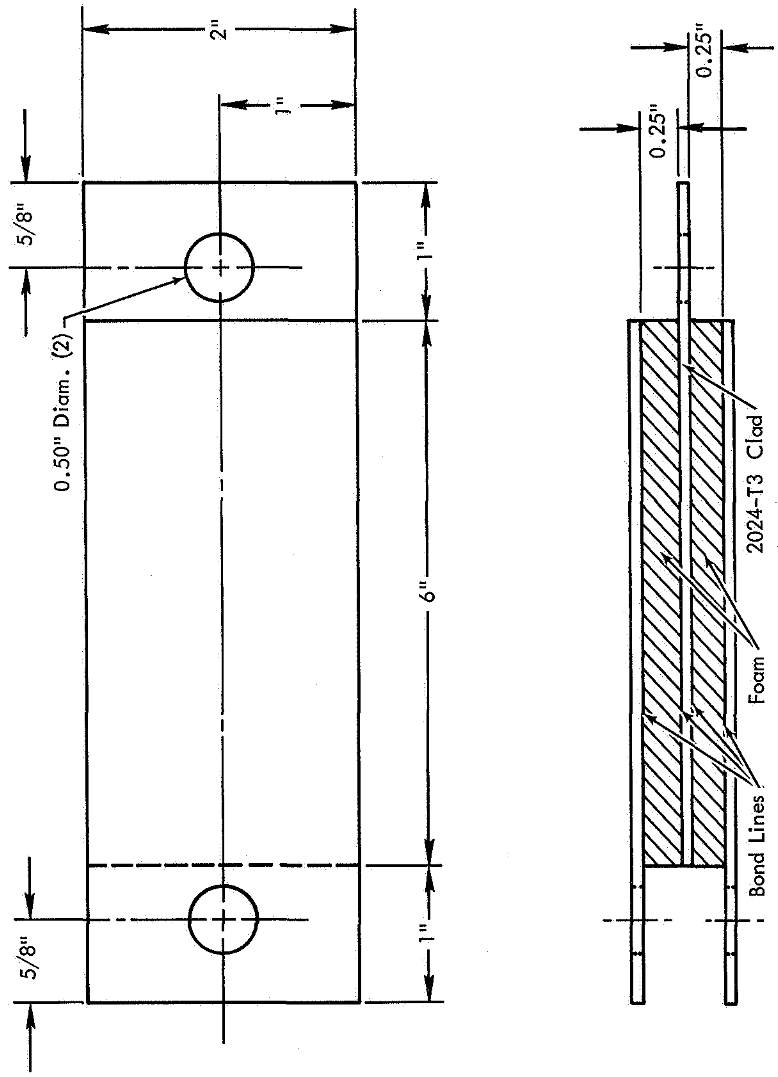

Figure 4.3 Composite Double Lap-Shear Test Specimen

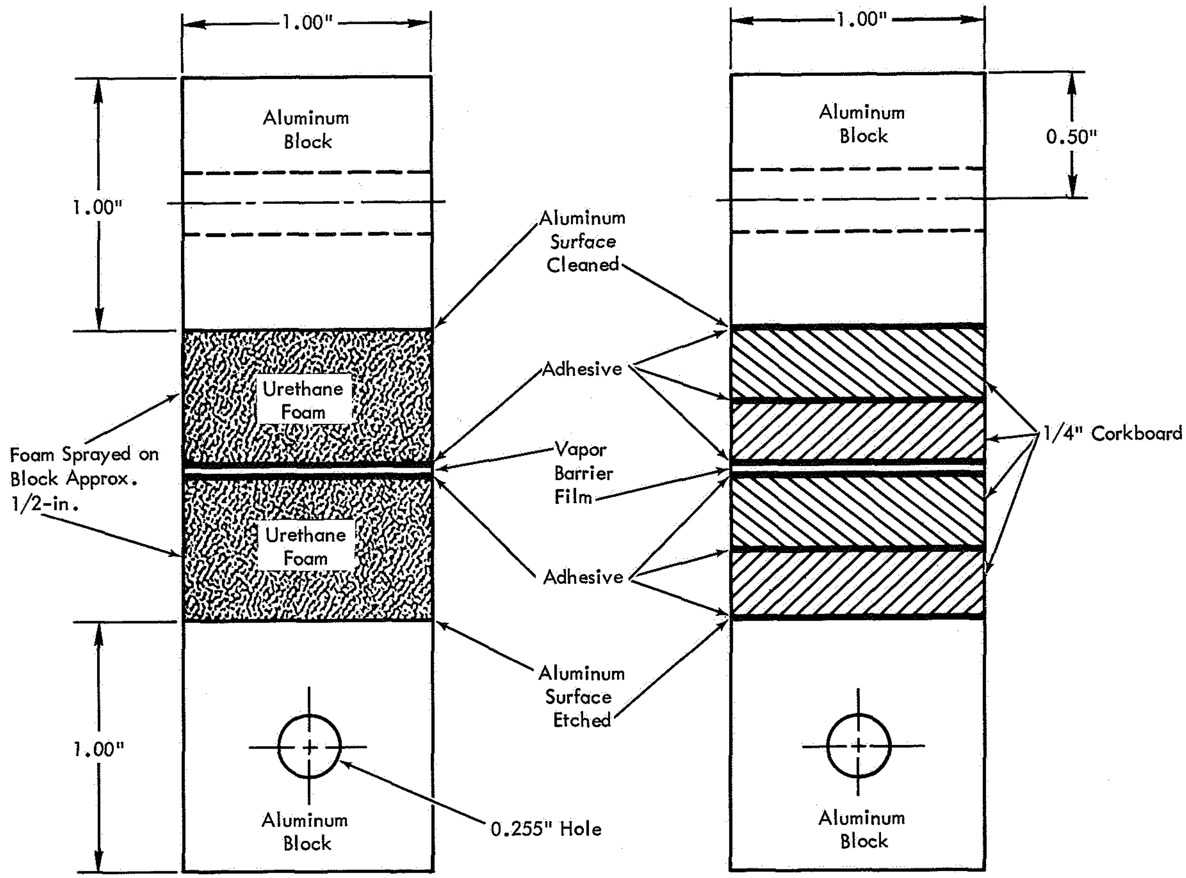

Figure 4.4 Typical Composite Flatwise Tensile Specimens

test using dynamic lap-shear specimens will be performed with the Instron to determine whether any difference exists between the test values from the standard ASTM test sample configuration and those from the modified version used in the dynamic test equipment. A cryogenic-temperature control test will be performed on lap-shear specimens of the four adhesives while submerged in LH_2. An ambient-temperature control test will be performed in the Instron with specimens that have been stored in LH_2.

4.2.2 Thermal Insulation Functional Test

After installation of the necessary control equipment and hardware on the NASA-fabricated test item, three thermal cycles will be run in Nuclear Area 1 to establish the integrity and safety of the test dewar at liquid-hydrogen temperatures.

4.3 Irradiation Exposure

The Ground Test Reactor will be operated at 3 Mw for approximately 50 hr to give a dose of 1×10^{11} ergs/gm(C) on the samples. The items discussed below will be on the three irradiation pallets as specified.

4.3.1 North Position

The Cryotensile tester with a circular dewar will be used for the LH_2 test condition. One pull rod will be used in the standard operating manner to pull 16 lap-shear specimens. The remainder of the test volume will be filled with 128 static specimens for subsequent testing in the IML. The LH_2 level and dewar pressure will be monitored during the radiation exposure.

4.3.2 East Position

A thermal insulation test dewar will be irradiated to a total dose of 1 x 10^{11} ergs/gm(C). The LH_2 level and dewar pressure will be monitored during the radiation exposure and temperatures within the thermal insulation will be continuously recorded. The system will be surveyed with television cameras and the picture recorded on tape throughout the irradiation.

4.3.3 West Position

An expanded metal rack set in a frame will be used to support static samples for the air exposure test. Approximately 128 samples will be exposed to a dose of 1 x 10^{11} ergs/gm(C).

4.4 Postirradiation Tests

4.4.1 Tensile Tests in LH_2

The sixteen dynamic lap-shear test specimens will be tested in liquid hydrogen immediately after irradiation. The crosshead speed will be 0.05 in./min for the test. The dewar will be warmed and the static test samples removed for testing in the IML.

4.4.2 Tensile Test in Air

All of the statically irradiated specimens from both the LH_2 dewar and the ambient rack will be removed for testing in the IML. The standard-tensile specimens will be tested in the Instron in accordance with established ASTM test procedures. The modified specimens will require only small modifications in the test procedure and will be tested within the framework of the ASTM test procedure.

4.4.3 Acoustic Noise Application Test

The postirradiation acoustic noise exposure of the thermal insulation dewar will be conducted in Nuclear Area 1. After radiation, the dewar will be drained of LH_2 and moved about 10 ft from the north end of the escalator tracks for the test. The condition of the thermal insulation will be visually inspected for cracks, unsealed vapor-barrier film, and loose insulation before the acoustic exposure is started. If any cracks or other irregularities are visible, they will be photographed before the acoustic exposure. If adhesive failure in large areas is such that the composite insulation material has large cracks, is loose, or has obviously deteriorated, the test will be terminated.

The LH_2 test dewar with the attached insulation panel will be enclosed by an acoustic reverberation chamber as shown in Figure 5.3. The chamber will be fabricated of plywood and shaped so that no two walls are parallel. The generated noise will be passed through a 10-ft catenoidal horn and into the reveberation chamber. Since the presence of the dewar will cause the sound field to be unevenly diffused, one insulated side of the dewar will be exposed directly to the incident noise at a time. The noise spectrum will be measured and controlled from a microphone located near the surface of the insulated side exposed to the incident noise. The spectrum will be recorded for the opposite insulated side during the run. The physical characteristics of the horn and chamber arrangement limit the low-frequency energy available; however, the noise energy above the peak point can be maintained equal to or above the required levels. During the acoustic exposure, the dewar will be filled with liquid nitrogen. After the exposure for 6 hr, the dewar will be drained of LN_2 and brought to room temperature for visual inspection of the thermal insulation.

V. TEST EQUIPMENT

5.1 Cryotensile Tester

The Cryotensile tester that will be used to test the lap-shear test specimens in LH_2 is shown in Figure 5.1. The tester was designed to apply vertical tensile loads to the slotted specimens located in four clevis assemblies. Each clevis assembly can contain as many as 20 tensile specimens, the quantity being limited by specimen thickness and slot lengths.

The overall height of the tester is approximately 8 ft, the width 3½ ft, and the depth 1½ ft. The structure is made of aluminum which, because of rapid radioactive decay rates, permits repeated use of the system in neutron irradiation tests over relatively short time periods. A horizontal dewar-mounting flange is located approximately one third of the way up from the bottom.

Tensile data in the form of applied tensile load in pounds and linear pull-rod travel in inches are obtained with the equipment. The applied loads to the specimens are obtained through actuation of a vertically mounted hydraulic cylinder. The cylinder has a net piston area of 6.8 $in.^2$

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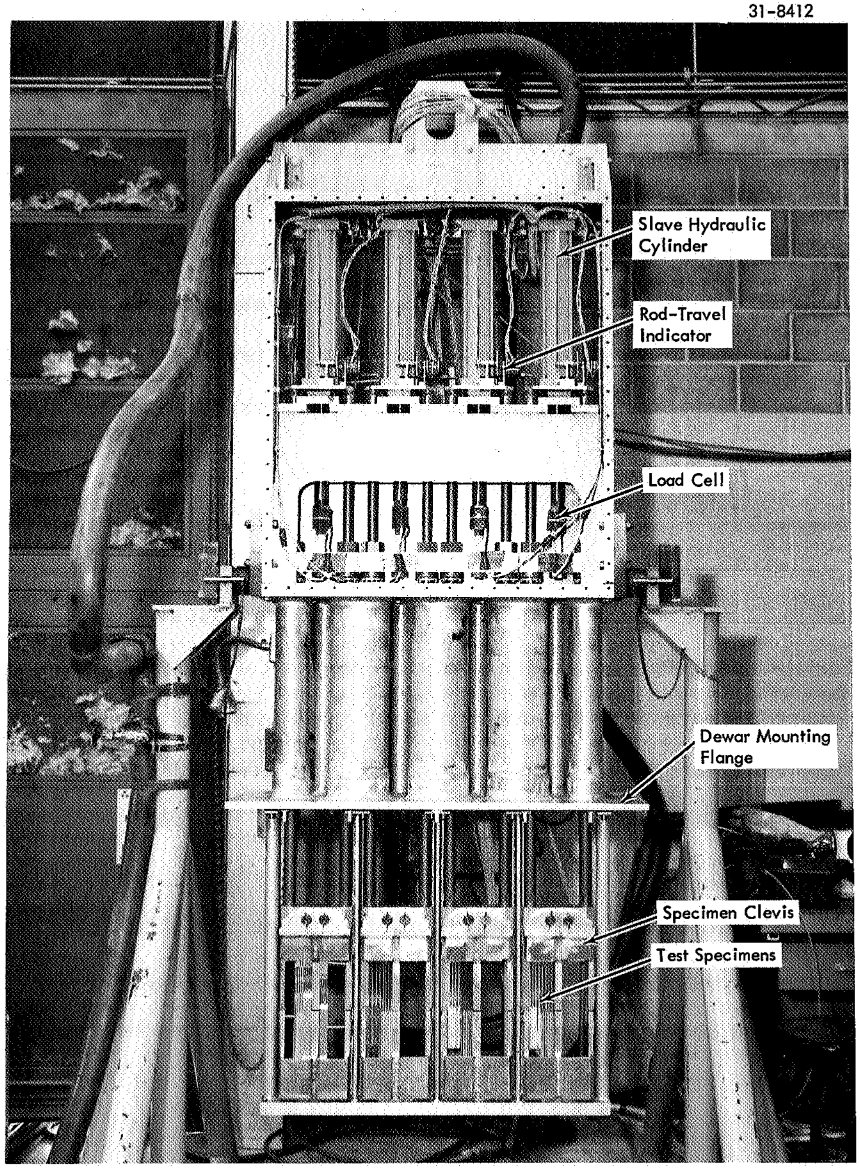

Figure 5.1 Cryotensile Tester

and a stroke of 9.0 in. The cylinder is mounted in the upper section of the tester. The piston rod of the hydraulic cylinder is coupled to a strain-gage-type load cell that provides electrical signals to instrumentation for determining loads to specimens. Double pull rods extend from the load cell, through the dewar mounting flange, to the clevis assemblies. These pull rods are sealed to the flange with stainless-steel bellows to ensure a vacuum-tight connection. A gear rack is mounted to the upper portion of the pull rod and engages a pinion gear mounted on the shaft of a potentiometer. Rod travel is thus transmitted to instrumentation in the form of varying potentiometer voltages. The entire upper portion of the tester is covered with an aluminum housing, or shroud. All connections to the tester are routed through this shroud.

The lower section of the double-pull-rod system connects to the upper end of the clevis assembly in which the tensile test specimens are loaded. The specimens themselves are the connecting link between the upper and lower sections of the clevis assembly. The lower section of the clevis assembly is secured to the lower extremity of the aluminum structure, forming a continuous tensile linkage between the lower clevis and the actuating hydraulic cylinder.

Each tensile test specimen contains, near the bottom of the specimen, a circular mounting hole through which the lower-clevis connecting pin passes, thus securing it to the clevis. The upper end of the tensile test specimen contains a slot for attachment with the upper-clevis connecting pin. The slots are varied in length to permit sequential loading of successive specimens by the upper-clevis connecting pin as the upper clevis is moved vertically upward by the hydraulic cylinder. Actuation of individual hydraulic cylinders in the tester is accomplished by slaving them to one master hydraulic cylinder mounted between the load-cell fitting and the crosshead member of an Instron machine. The crosshead downward motion in the Instron machine results in upward linear motion of the upper section of the clevis assembly, thus applying a tensile load to the test specimen. An accurate control of the Instron machine crosshead speed yields precise vertical movement rates for the upper section of the clevis assembly and enables compliance with established ASTM tensile-testing rates for the specimens.

5.2 Static Air Racks

Static specimens to be irradiated in ambient air will be mounted on expanded-metal racks that fit into an aluminum framework designed for use on the remote-controlled shuttle system of the GTR Radiation Effects Testing System. The amount of radiation received by each specimen depends not only on total exposure time but on its location on the rack and the location of the rack in the framework. By use of the shuttle system, the framework containing the racks can be removed from the radiation field at any time while the reactor is at power. Figure 5.2 is a photograph of static air racks located in the aluminum framework.

5.3 Thermal Insulation Dewar

The LH_2 dewar that is to be used in the tests described in Sections 4.2.2, 4.3.2, and 4.4.3 will be designed and fabricated at NASA-Huntsville. It will be a 30-in.-long cubic box constructed with 0.030-in.-thick sides of 2219-H86 aluminum. Two of the test panels will be Insulcork 7326 Cryogenic Insulation and two panels will be CPR 368 spray foam.

5.4 Support Equipment

A Sanborn Recorder (Model 50) will be used to indicate and record load-cell and rod-movement data during the dynamic testing.

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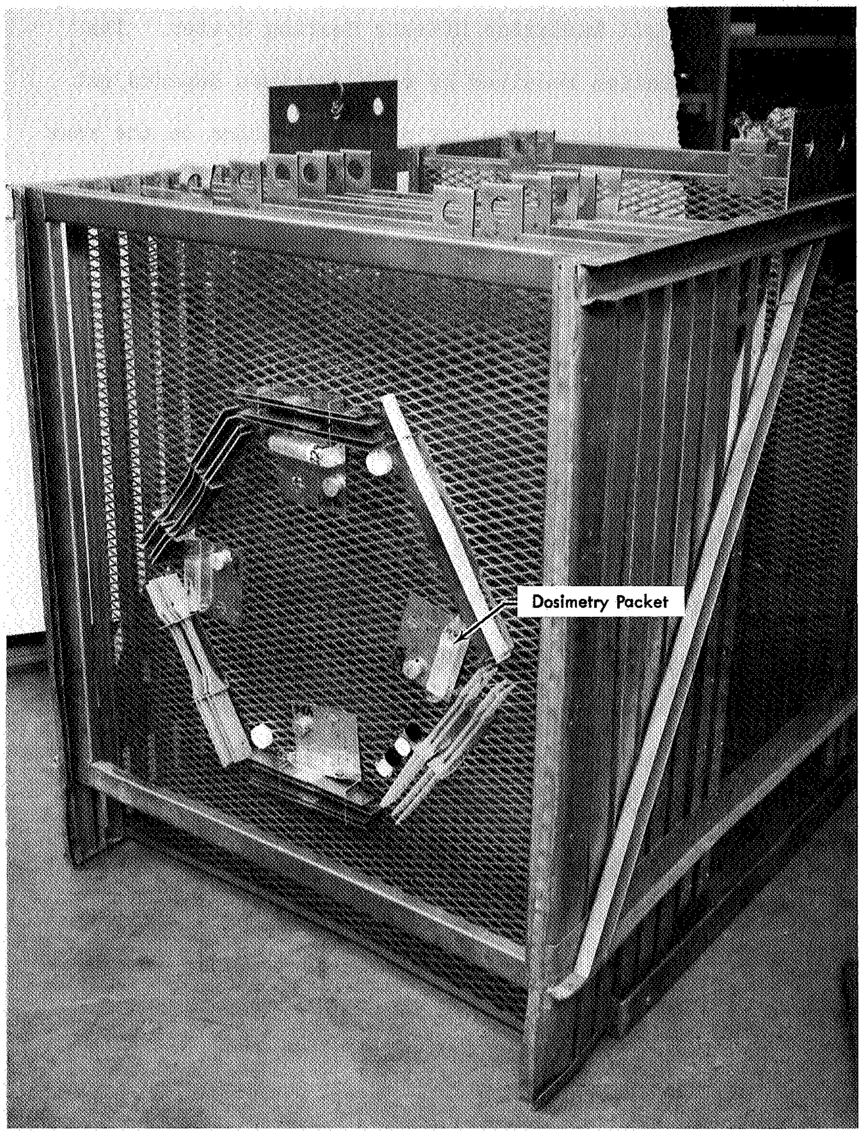

Figure 5.2 Static Samples on Rack for Ambient-Air Irradiation

An Instron tester (Type TT-C) will be used to drive the master hydraulic cylinder and to serve as a secondary load-indication system during the dynamic testing. It will also be used for postirradiation testing of the static test specimens.

Ambient temperatures of representative static air specimens will be monitored on a Brown temperature recorder. A separate recorder will be used to measure the output of the thermocouples in the thermal-insulation dewar.

The LH_2-control system in the NARF area will be used in these tests. The equipment required to supply, monitor, and control LH_2 for two test positions will be used.

The acoustical noise test equipment shown in Figure 5.3 will be used for postirradiation testing of the thermal-insulation dewar.

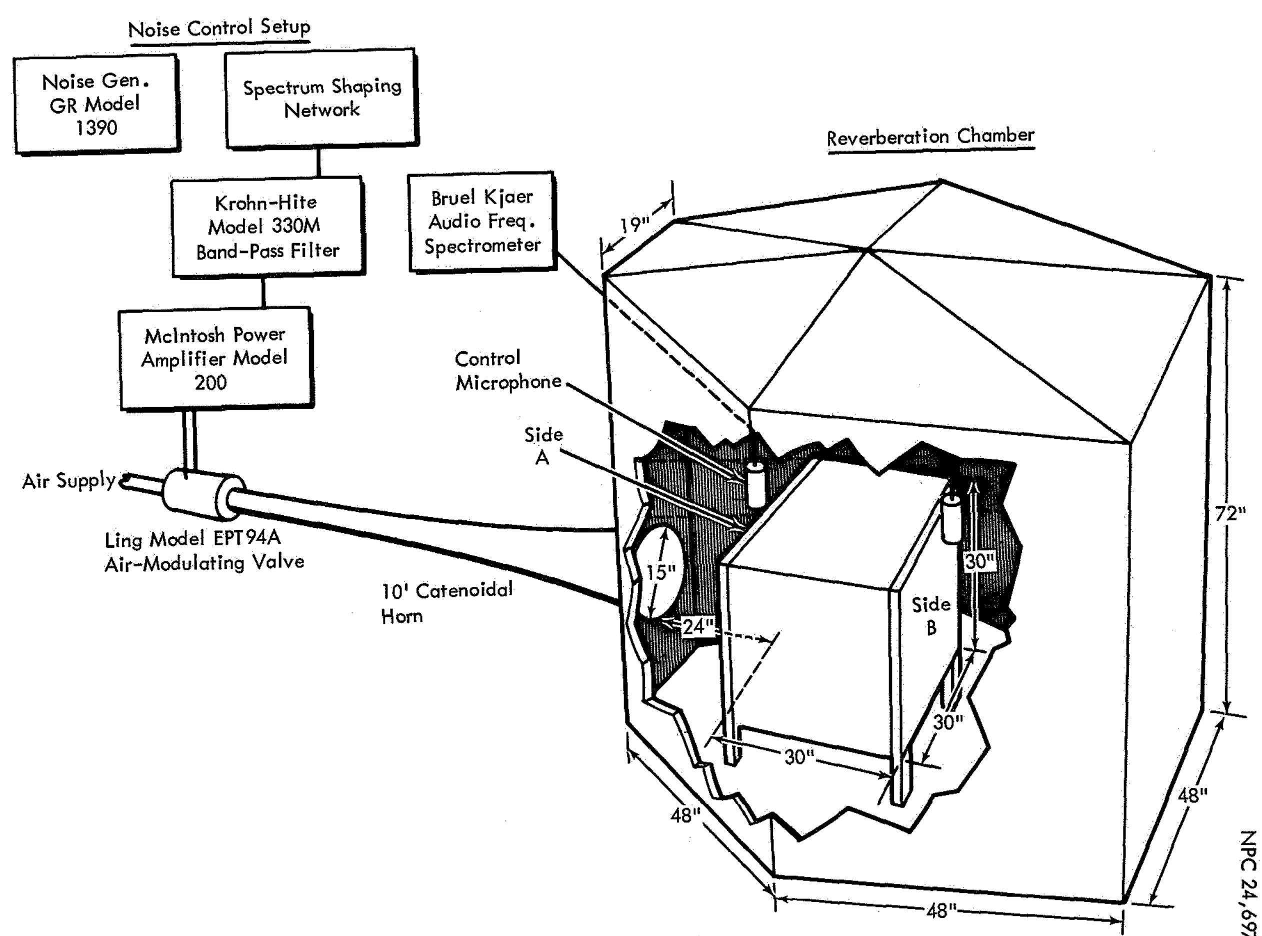

Figure 5.3 Acoustic Noise Setup

VI. RADIATION ENVIRONMENT

6.1 GTR Neutron Spectrum Determination

6.1.1 Analytical GTR Neutron Spectrum

The spectrum (Ref. 5) of the GTR in a water moderator has been measured to be Maxwellian at thermal energies ($E < 0.48$ ev), approximately E^{-1} from about 0.5 ev to 0.1 Mev, and essentially a fission spectrum for higher energies. In Figure 6.1, this spectral shape has been mathematically altered to account for the attenuation of the neutron flux by the boral surrounding the reactor in the dry-pool configuration. The resulting analytical spectrum has been shown to represent the actual spectrum fairly accurately.

Flux measurements have been made in the thermal, epithermal, and fast-energy ranges by use of a variety of thermal, resonance, and threshold detectors. Measurements made in the dry side with the boral in place and the energy range above 2.9 Mev agree well with those in the wet-pool side. The measured thermal flux is in general agreement with that obtained by integration of the analytical curve shown in Figure 6.1. Measurement of the epithermal flux by use of resonance detectors is very difficult, and values

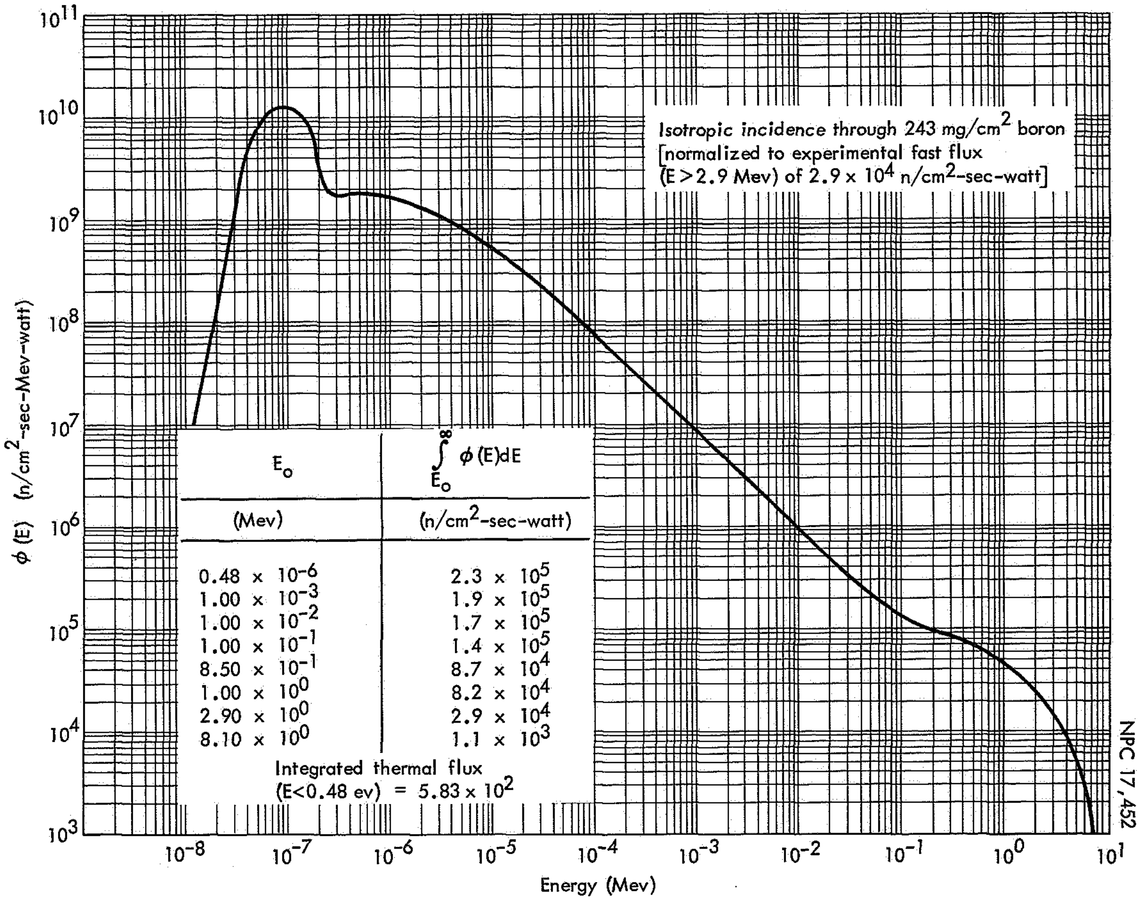

Figure 6.1 Analytical GTR Neutron Spectrum

differing by as much as a factor of 2 are obtained by the various detectors.

6.1.2 Experimental GTR Neutron Spectrum

Figure 6.2 shows a representative neutron-energy spectrum in the east cryogen chamber at a point midway (front to back) between the center sample positions. This spectrum is normalized to an experimental fast flux of $\phi(E < 2.9 \text{ Mev}) = 2.9 \times 10^4$ n/cm^2-sec/watt and shows that, in the energy interval given, the spectrum shape is affected only slightly, if at all, by attenuation through LN_2 or LH_2 (cf. Figure 6.1).

6.2 Nuclear-Measurement Procedures

Determination of the neutron flux and gamma dose inside the experimental assemblies will be according to NARF standard procedures used on all irradiation tests. Radioactivants used for neutron flux measurements were standard types and are outlined in the succeeding sections. Gamma-dose levels were monitored with nitrous-oxide dosimeters.

6.2.1 Ambient-Temperature Irradiation Dosimetry

For the air irradiation (west position), the nuclear measurement packets will be attached at strategic locations on the metal rack with bare and cadmium-covered foils for

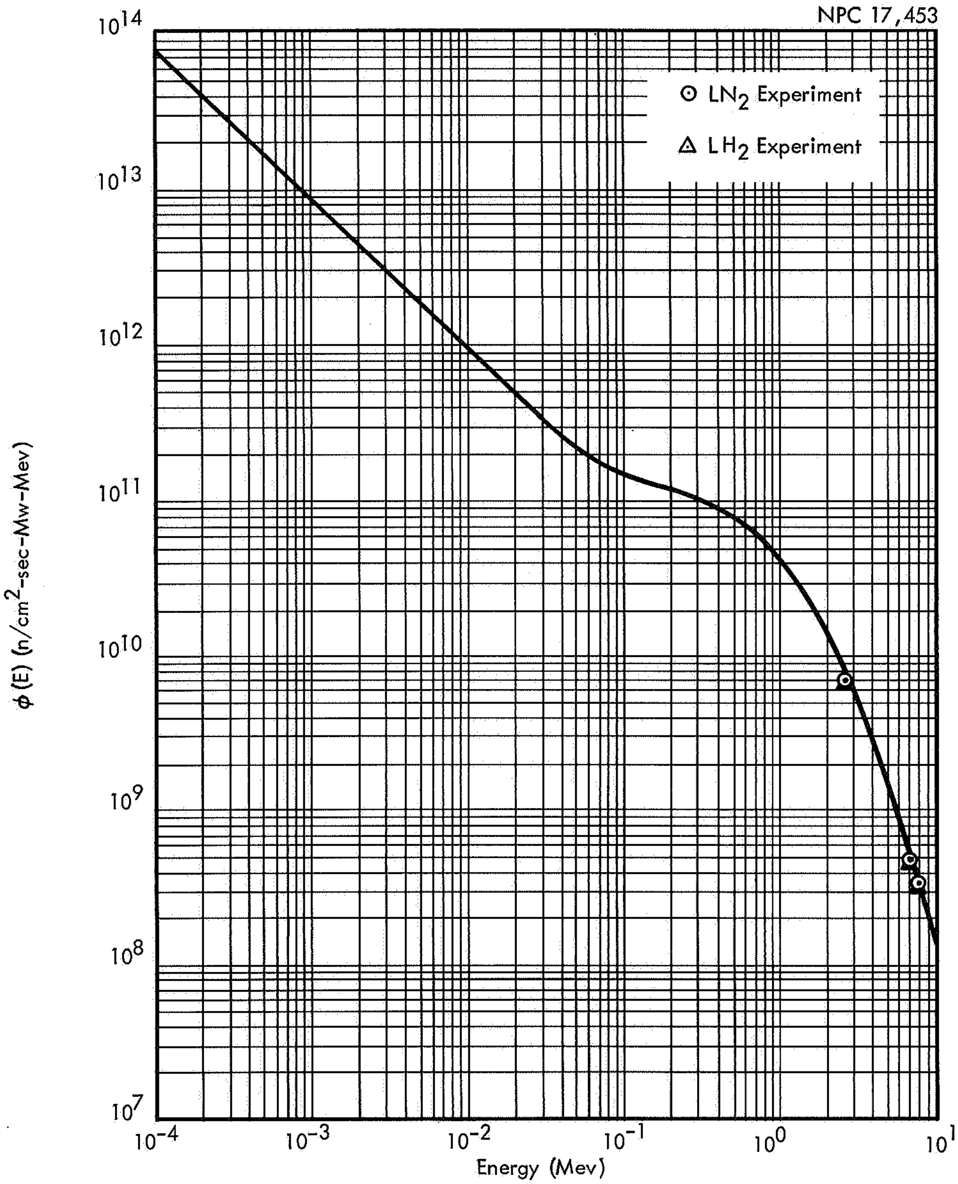

Figure 6.2 Representative Neutron Spectrum in the East Cryogen Chambers Midway Between Front and Back Sample Locations on Vertical Centerline

the thermal flux, with sulfur pellets for fast-neutron flux ($E < 2.9$ Mev), and with nitrous-oxide gamma dosimeters for the gamma dose.

6.2.2 Liquid-Hydrogen Irradiation Dosimetry

The neutron radioactivants (sulfur, and bare and cadmium-covered phosphorous) and the packets containing nitrous-oxide gamma dosimeters will be mounted on 0.040-inch-thick expanded-aluminum sheets. For the cryotensile tester (north position), the nuclear measurement packets will be located in front of and behind the test specimens and irradiated while submerged in LH_2. Packets for the composite cryogenic insulation test dewar will be attached to the panel at strategic locations and mounted inside the dewar behind the panel.

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