

Effects of Nuclear Radiation and Cryogenic Temperatures on Nonmetallic Engineering Materials

E. C. MCKANNAN* AND R. L. GAUSE†
Marshall Space Flight Center, Huntsville, Ala.

Physical property data were obtained from the investigation of the combined effects of radiation and cryogenic temperatures on nonmetallic materials. The materials discussed are representative of those most likely to be used on spacecraft employing nuclear power systems, and they were selected from the categories of structural laminates, structural adhesives, thermal insulations, and electrical insulations. Although many of the materials were stable at the combined environmental conditions employed in the tests, a few unpredicted results were observed. Epoxy-nylon adhesive lost strength at cryogenic temperatures. Glass-to-resin bonds in plastic-glass laminates broke because of differential thermal expansion at cryogenic temperatures, thereby increasing the apparent elongation. Plastic foams that were made from stable polymers had poor radiation resistance, a condition that seemed to be a function of the foaming process.

Introduction

THE Marshall Space Flight Center (MSFC) conceived a program to obtain engineering data on the changes produced in the physical properties of nonmetallic materials by exposure to the various combinations of cryogenic temperatures, nuclear radiation, and vacuum. The program was implemented on November 9, 1961, with the awarding of Contract NAS8-2450, entitled "Investigation of the Combined Effects of Radiation and Vacuum on Engineering Materials," to the General Dynamics Corporation, Fort Worth, Texas (GD/FW). Initially, the contractor was directed to irradiate selected engineering materials in a vacuum (10^{-6} -mm Hg) environment only. Subsequently, the contract was modified to include the combined effects of 1) nuclear radiation and cryogenic temperatures and 2) nuclear radiation, vacuum and cryogenic temperatures. To date, approximately 80 materials from the following functional categories have been evaluated in one or more of these combinations of environments: 1) structural adhesives, 2) structural laminates, 3) thermal insulations, 4) electrical insulations, 5) potting compounds, 6) seals, 7) sealants, 8) lubricants, and 9) thermal control coatings.

Various chemical classes of materials (e.g., polyurethanes, epoxies, phenolics) were included in the program in the expectation that it would be possible to characterize specific chemical classes as being acceptable for use in a particular environment or combination of environments. An approach of this type was deemed necessary because of the economic limitations to the classification of the multitude of individual materials applicable to nuclear-powered spacecraft. The results on vacuum-radiation effects were presented recently by Lucas.¹ The second phase of the program, the effects of cryogenic temperature and radiation, are discussed in this paper.

Materials Selection and Testing Criteria

The materials tested (Table 1) are representative of those likely to be used in a nuclear-powered spacecraft; selection criteria included past history of use in nonnuclear vehicles; potential use in nuclear vehicles; and knowledge of their resistance to radiation and cryogenic temperatures, singly.

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* Deputy Chief, Engineering Physics Branch, Materials Division.

† Chief, Environmental Physics Section, Materials Division.

The test specimens for each material were fabricated from one batch and procured from the manufacturer or prepared according to the manufacturer's recommended procedures. Wherever possible, the specimen configuration corresponded to the American Society for Testing Materials (ASTM) specifications. The specific property measurements made depended upon the application of the particular material and included tensile shear strength, ultimate tensile strength, ultimate elongation, stress-strain relationship, compressive strength, and spectral reflectivity. Tests for each material were made generally at each of nine different environmental conditions consisting of the possible combinations of three different temperatures (50°C , -196°C , and -253°C) and three radiation levels (unirradiated, relatively low, and relatively high). Room-temperature radiations were made in air where the ambient temperature was from 21° to 25°C ; however, nuclear heating increased the specimen temperature to 50°C . The radiation source was the Ground Test Reactor (GTR) at the GD/FW Nuclear Aerospace Research Facility (NARF). It is a heterogeneous, highly enriched 3-Mw thermal reactor, which utilizes water for neutron moderation and reflection, radiation shielding, and cooling.^{2,3} The ratio of neutron flux to gamma dose was approximately 2×10^5 neutrons for each erg-g^{-1} (C). Throughout this paper the radiation is described in terms of the gamma dose only.

All of the specimens for the low-temperature phase of the program were irradiated while immersed in liquid nitrogen (LN_2) or in liquid hydrogen (LH_2) and were tested in the cryogen without intervening warm-up. This test procedure complicated the experiment by requiring the design and fabrication of special remotely operated, tensile test equipment,⁴ which could be mounted in the cryogenic dewars.

Table 1 Materials selected for evaluation

Material application	Trade name	Chemical class
Structural adhesive	Hexcel 1252	Polyurethane
	Metibond 406	Epoxy-nylon
Structural laminate	Conolon 506	Phenolic
	Paraplex P-43	Polyester
Thermal insulation	Stafoam AA402	Polyurethane
	Styrofoam 22	Polystyrene
Electrical insulation	Mylar C	Polyester
	H-Film	Polyimide
	Teflon (TFE)	Fluoroethylene
	Kel-F-81	Fluorocarbon

Such a procedure was deemed necessary to provide meaningful data, since it eliminated the possibility of erasing or annealing the radiation-induced effects, which could result from allowing the specimen to warm to room temperature prior to making property measurements.

Data Analysis Criteria

Reversible temperature effects on stable materials, as shown typically in Fig. 1, will produce no permanent property change caused by the thermal history except at the very high end of the temperature range; that is, there is no perceptible change in the molecular weight. As the temperature is decreased, the mobility of long, chain molecules is sharply decreased, thus causing an increase in the modulus and breaking strength. The material is said to be "embrittled" when the elongation is sufficiently small to erase the yield point, even though the breaking strength may be quite high, and the room temperature properties are unaffected.

Reversible effects caused by plasticizers (including water, residual monomer, or selected additives) are exemplified by the freezing effect. For practical purposes, all of the known plasticizers are immobile or frozen at LN_2 temperature ($-196^\circ C$) and below. When freezing occurs, the plasticized polymer decreases sharply in elongation and increases in modulus and strength. A similar effect on the change in physical properties may be caused by the loss of plasticizer by volatilization at higher temperatures. In the special case of water in condensation polymers, a specimen, although dried, may regain water from the atmosphere rapidly, and its physical properties will change reversibly with the water (plasticizer) content.

Irreversible effects that are generally temperature-dependent are 1) crosslinking and 2) degradation by chain scission. For any given polymer specimen, an increase in crosslinking usually is related to an increase in breaking strength and modulus, and a decrease in elongation. Crosslinking may be produced merely by heating a specimen if it contains residual-curing (crosslinking) additives. It may be effected in some polymers (e.g., polyethylenes, polystyrenes, and silicones) by irradiation at a low temperature, even in the absence of a chemical crosslinking agent. Chain scission irreversibly decreases molecular weight and, hence, breaking strength and modulus. When condensation polymers, such as polyamides and polyethyleneterephthalates, are subjected to high temperatures in the presence of water, scission may occur by a reverse polymerization reaction. Radiation inherently causes chain scission in polymers containing vinyl side groups, such as polyvinyl chlorides and polytetrafluoroethylenes.

One of the greatest limitations to an exact analysis of complex, organic, engineering materials is the degree of uncertainty as to the exact composition and molecular structure of the starting material. In some of the cases, the reactions that occur on irradiation of even, well-characterized, pure polymers are not well understood. Therefore, at this stage of the program, a thoroughly rigorous interpretation of the combined radiation and cryogenic effects is impossible.

Discussion of Results

Structural Adhesives

Structural adhesives are used extensively in space vehicles, particularly in the cryogenic insulation system and in honeycomb structures; therefore, their strength is of vital interest. The ultimate tensile lap-shear strength was the property chosen for measurement and was determined according to ASTM-D-1002-53T (slightly modified) by averaging the breaking strength of four specimens at each of eight environmental conditions. Almost all of the failures were adhesive rather than cohesive. All of the specimens tested at GD/FW

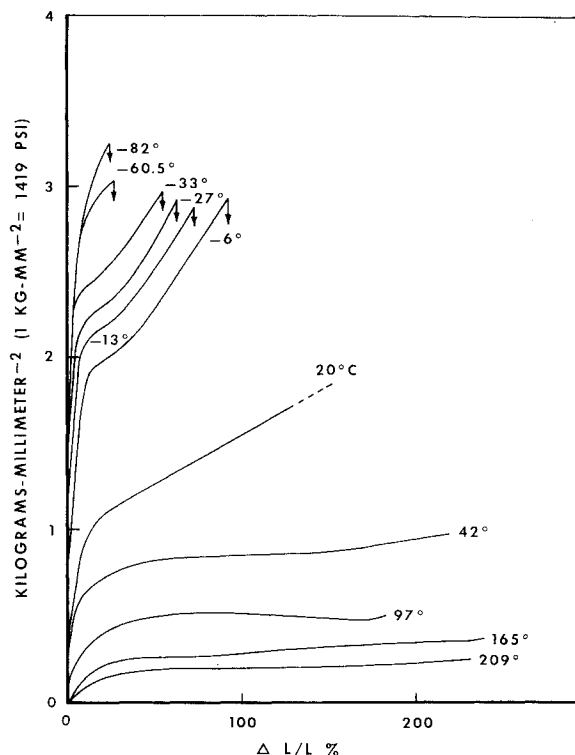


Fig. 1 Stress-strain relationships for polytetrafluoroethylene (Teflon).

and at MSFC were from the same batch and were prepared at MSFC. The adhesive area was 0.5 in.² on carefully cleaned adherends of 2024-T3 aluminum. Application and curing procedures used were those suggested by the manufacturer.

A polyurethane adhesive (Hexcel 1252) was chosen for evaluation, even though no previous radiation data were available for comparison. Interest in this material was based on the high radiation stability of other polyurethane resins. The data shown in Fig. 2 indicate that the shear strength increased with decreasing temperature in the absence of radiation as might be expected from a typical modulus-temperature relationship where the material does not become embrittled. Also, the shear strength increased with increasing radiation dose in air, even though the specimen temperature increased slightly because of radiation heating. The strength increase possibly can be attributed to radiation-induced crosslinking.

Quite the opposite trend was observed with increasing radiation exposure in LN_2 and in LH_2 . The crosslinking re-

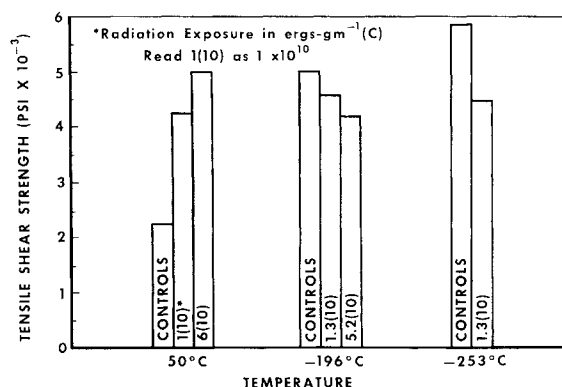


Fig. 2 Radiation-cryogenic temperature data for a polyurethane adhesive (Hexcel 1252).

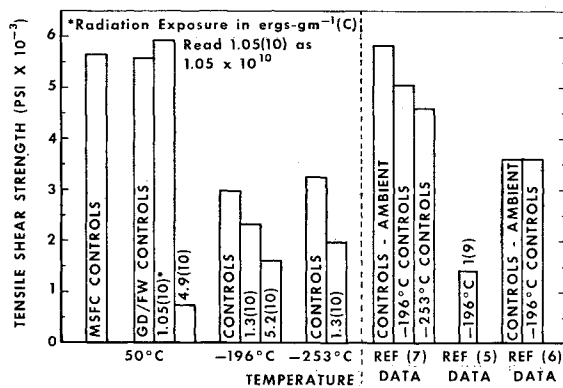


Fig. 3 Radiation-cryogenic temperature data for an epoxy-nylon adhesive (Metlbond 406).

action may have been inhibited by the low temperatures and the inert or reducing environments (the role of oxygen in the crosslinking reaction appears to be important but is not well understood). The effect of decreasing temperature at the radiation levels of approximately 1.3×10^{10} ergs-g⁻¹ (C) and 5×10^{10} ergs-g⁻¹ (C) indicates that there is a competing mechanism reducing the shear strength. Compare this with the fact that both irradiation at room temperature and decreasing temperature in the absence of radiation improve the shear strength. Fortunately, all of the observed reductions in shear strength were small at all of the temperatures and irradiation levels to 1.3×10^{10} ergs-g⁻¹ (C). Therefore, this material may be recommended for use in the evaluated environments at a design value equal to or less than its initial shear strength of 2240 psi.

An epoxy-nylon adhesive (Metlbond 406) was evaluated, although no previous radiation data were available for comparison, because of its high shear strength at normal temperatures and because it can be cured at relatively low temperatures. Subsequent radiation data obtained from the Lockheed Missiles and Space Company (LMSC)⁵ and the Aerojet-General Corporation (AGC)⁶ closely corroborate the data presented in this paper. Figure 3 shows that the shear strength decreased with decreasing temperature in the absence of radiation. At low temperatures, the total elongation decreased to a value below the yield point. According to one interpretation of stress-strain curves, this type of response is defined as embrittlement and is caused by the decreased molecular mobility. Entrapped moisture in the nylon may have contributed to the embrittlement. This polymer may be very sensitive to curing conditions because specimens tested by the manufacturer⁷ and cured only 15 min at 177°C degraded 29%. The identical material, when cured 60 min at 177°C, degraded 43% when tested at GD/FW under the same conditions as those used by the manufacturer.

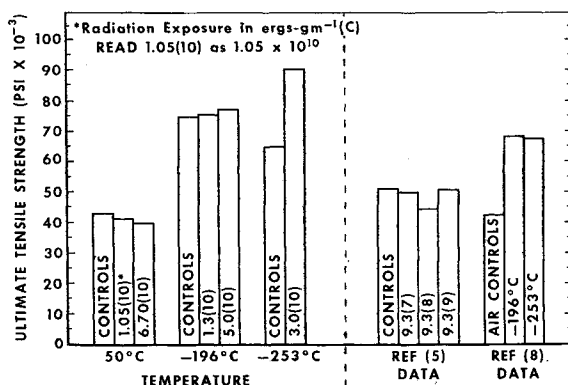


Fig. 4 Radiation-cryogenic temperature data for a glass-phenolic laminate (Conolon 506).

Irradiation in air at room temperature and pressure caused little change in shear strength up to an integrated dose of 1×10^{10} ergs-g⁻¹ (C). This information is corroborated by similar tests at AGC up to an exposure of 5.6×10^9 ergs-g⁻¹ (C). However, at 4.9×10^{10} ergs-g⁻¹ (C), the shear strength degraded 86%, indicating a sharp damage threshold caused by chain scission between 1×10^{10} and 4.9×10^{10} ergs-g⁻¹ (C).

Irradiation in LN₂ and LH₂ produced approximately the same amount of degradation: a 50% decrease from the unirradiated value in LN₂ and a 62% decrease from the initial room-temperature value. However, the degradation at high irradiation levels [5.2×10^{10} ergs-g⁻¹ (C)] in the cryogenics was not as severe as it was in air. This identical trend in the data probably indicates that chain scission occurred in all of the three environments.

This adhesive is not recommended for use above an exposure of 1×10^{10} ergs-g⁻¹ (C) at room temperature in air. The tensile shear strength at room temperature is approximately 5000 psi but is greatly dependent upon the amount and type of curing. At cryogenic temperatures, the tensile shear strength is 2900 psi unirradiated and 1400 psi for an exposure of 1×10^{10} ergs-g⁻¹ (C). No further investigation of the polymer is planned.

Structural Laminates

Structural laminates are used in space vehicles for support of components, for pressurized bottles, and for panels; therefore, their tensile strength is important. Tensile stress-strain curves were determined at eight different environmental conditions according to ASTM-D-638-61T. Specimen pulling rates varied with test conditions from 0.011 to 0.096 in./min because of environmental effects on the hydraulic system. However, there was no correlation between the measured properties and pulling rates over the narrow range of variations experienced. Reproducibility at constant conditions was excellent in all of the cases. All of the specimens were made from the same batch and prepared at MSFC into 0.125-in.-thick sheets according to the manufacturer's suggested procedures.

A 25% by weight phenolic resin-75% glass laminate (Conolon 506) was chosen for evaluation because of its widespread use and because previous information showed it to have excellent temperature and radiation stability. The data shown in Figs. 4 and 5 indicate that the tensile strength (unirradiated) increases at reduced temperatures and agree with those obtained by Hertz.⁸ However, the increased strength was accompanied by increased elongation at an almost constant modulus. The latter two facts are abnormal for cryogenic temperatures. These effects might be explained by the breaking of glass-resin bonding caused by the difference in thermal expansion between the glass and the resin. No

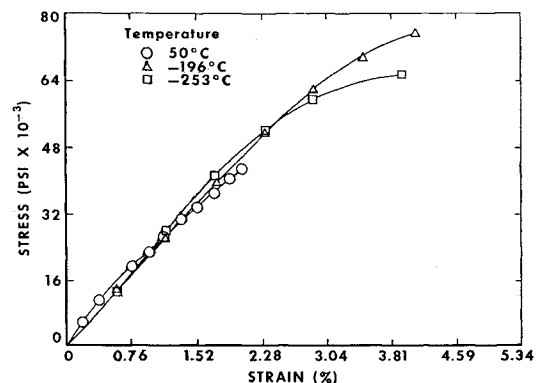


Fig. 5 A typical stress-strain temperature response curve for a structural laminate (Conolon 506).

other explanation is offered for this effect other than to note that the high glass content and the mechanical complexity of a glass-resin matrix tend to mask the specific effects, which occur at reduced temperatures. Irradiation up to 6.7×10^{10} ergs-g⁻¹ (C) in air at room temperature or in LN₂ caused no significant change; the stress-strain curves were identical at a given temperature, regardless of dose. Significant increases in strength and modulus resulted from irradiation up to 3.0×10^{10} ergs-g⁻¹ (C) in LH₂. Smith⁹ attributes this change to reactions between ionized hydrogen and constituents of the adhesive, which improve the bonding between the resin and the glass. This material may be recommended for use at cryogenic temperatures and in radiation up to 6.7×10^{10} ergs-g⁻¹ (C), because its tensile strength is equal to or greater than the room temperature strength of 40,000 psi.

A 30% by weight polyester resin-70% glass laminate (Paraplex P-43) was evaluated because previous information showed it to have good radiation stability. In general, it had much the same response to the test environments as the phenolic laminate, but it had only two-thirds the strength and two-thirds the stiffness. The tensile strength (unirradiated) increased about 60% in LN₂ and decreased slightly in LH₂ (Fig. 6). The same temperature effect was observed at a constant radiation dose of 1.0×10^{10} ergs-g⁻¹ (C). There was no significant change in this material when irradiated up to 6×10^{10} ergs-g⁻¹ (C) in air, LN₂, or LH₂. It can be recommended for use at cryogenic temperatures and in radiation up to 6.0×10^{10} ergs-g⁻¹ (C), because its tensile strength is equal to or greater than the room-temperature strength of 28,000 psi.

Thermal Insulations

Rigid, closed-cell foams are employed in space vehicles for thermal insulation of cryogenic fuel tanks and for protection of delicate components against mechanical shock. Compressive stress-strain curves were measured according to ASTM-D-1565-59T. Although the compressive strength is not the primary property for which thermal insulating foams are employed, this property was used to evaluate their response to radiation and low temperatures. The mechanical strength is so low that most foams cannot support loads and, therefore, they are reinforced with a matrix or honeycomb. A higher strength foam, which could be employed without reinforcement, would provide a lighter insulation system with lower thermal losses. The more pertinent property, thermal conductivity, will be the subject of a future environmental evaluation.

In the work described here, all of the specimens were cut from the same sheet, and two or three specimens were measured at each condition. Some difficulty was experienced in eliminating the hydraulic system tare loads, but comparisons with other data taken on standard testing machines indicate

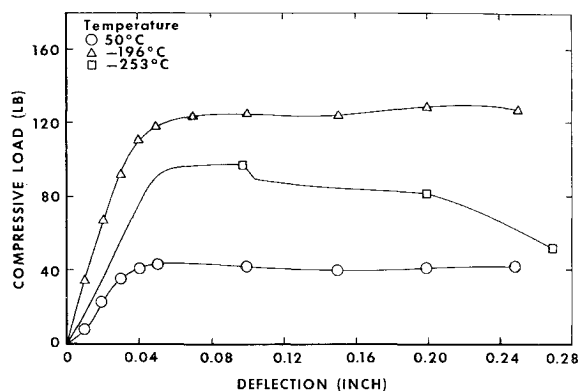


Fig. 7 A typical compression-deflection curve for a thermal insulation (Stafoam AA402).

that these data are valid. A typical compressive stress-strain curve for a foam, shown in Fig. 7, indicates that, after the material yields, it supports a constant load while continuing to deform. One interpretation of this curve is that all of the cells deform slightly up to the yield point, but beyond that point one "layer" of cells fails at a time. This interpretation implies that the constant load is that load required to break one "layer" of cells. The cell failures are discrete on a microscopic scale, but in the aggregate, they produce the appearance of bulk plastic flow. In this manner, both the constant load on deformation and the high value of deformation can be explained. The following results were obtained on the two types of foam material, polystyrene and polyurethane, which were evaluated.

A polystyrene foam (Styrofoam 22) was chosen for evaluation because of its common usage in many applications (it is an excellent thermal insulation) and because bulk polystyrene has excellent resistance to radiation damage. According to manufacturer's data, the specimens had a density of approximately 2 lb/ft³ and a thermal conductivity of 0.25 Btu-in./ft²-°F-hr at 10°C and 0.04 Btu-in./ft²-°F-hr at -240°C. Compressive strength and modulus increased with decreasing temperature, as expected, with or without radiation (Fig. 8). However, irradiation in air at room temperature caused severe degradation, even at a dose as low as 5×10^9 ergs-g⁻¹ (C). This surprising response may occur because most foams are produced by mixing into the polymer a thermally unstable compound, or blowing agent, which decomposes and gasifies upon heating. Even small quantities of residual blowing agent or its degradation products may degrade further and attack the polymer when irradiated at room temperature. However, the reaction would be inhibited at cryogenic temperatures. A typical blowing agent, methyl chloride, decomposes to form an acid or chloride ion, which attacks polystyrene. For this reason, polystyrene foam can-

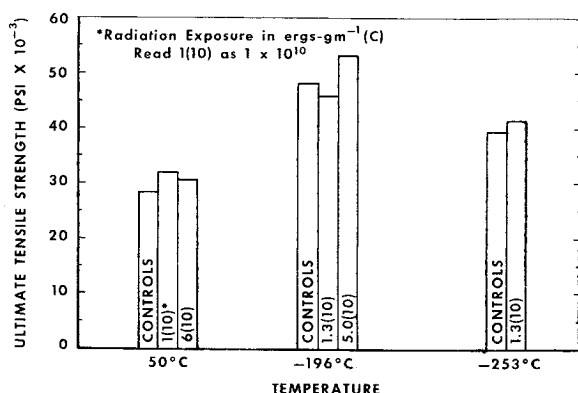


Fig. 6 Radiation-cryogenic temperature data for a glass-polyester laminate (Paraplex P-43).

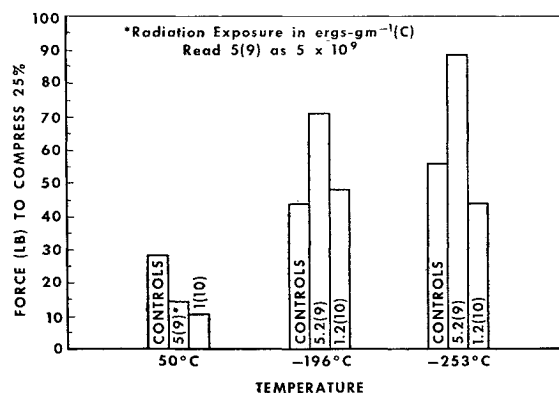


Fig. 8 Radiation-cryogenic temperature data for a polystyrene foam (Styrofoam 22).

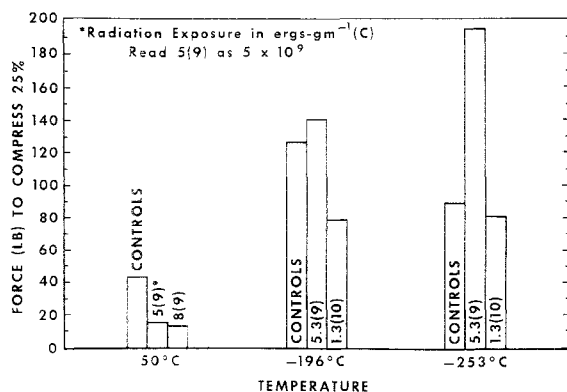


Fig. 9 Radiation-cryogenic temperature data for a polyurethane thermal insulation (Stafoam AA402).

not be recommended for applications involving irradiation up to 5×10^9 ergs-g⁻¹ (C) or greater.

A halocarbon-blown, *polyether-resin urethane foam* (Stafoam AA-402) was evaluated because it has higher strength and a wider service temperature than polystyrene foam and because a similar material, "Adiprene L," is stable at radiation doses up to 2×10^9 ergs-g⁻¹ (C). According to manufacturer's data for this rigid, closed-cell foam, $\rho = 2$ lb/ft³ and $k = 0.15$ at 21°C. Compressive strength and modulus increase with decreasing temperature, with or without radiation, in much the same manner as the polystyrene foam, except that the urethane foam was stronger (Figs. 7 and 9). Irradiation in air caused severe degradation but irradiation at cryogenic temperatures did not. This fact is explained on the basis of room-temperature reactions with the residual blowing agent, just as in the case of the polystyrene foam. Under increasing radiation in LN₂ or LH₂, maximum strength is reached at less than 1.3×10^{10} ergs-g⁻¹ (C); it appears that a competing degradation reaction becomes predominant over radiation-induced crosslinking at a dose less than 1.3×10^{10} ergs-g⁻¹ (C). As with the polystyrene foam, this material may be recommended for use at cryogenic temperatures but not for applications involving irradiation up to 5×10^9 ergs-g⁻¹ (C) or greater.

Electrical Insulation

Fluorocarbons are primary candidates for electrical insulation of wire and cable systems in space vehicles because of their stability over a wide range of service temperatures and their low dielectric losses. Plans were made for evaluating these materials according to ASTM-D-412-51T in the form of typical dumbbell-shaped tensile specimens. The pulling rate was modified to 0.5 in./min, which is an accepted practice

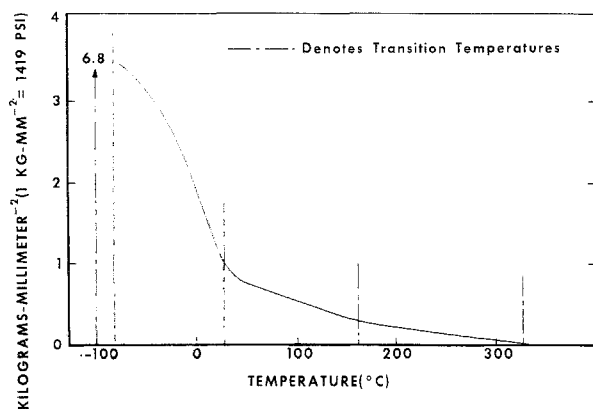


Fig. 10 Tensile strength vs temperature for polytetrafluoroethylene (Teflon).

for fluorocarbons. At the conditions at which data were obtained, reproducibility of duplicate specimens cut from the same sheet was very good. The following results were obtained for the two most widely used fluorocarbon polymers.

Polytetrafluoroethylene (Teflon 6, TFE) is widely used as a wire and cable coating because of its high temperature stability and low dielectric loss properties. Also, in another form, it is being considered for zero-g expulsion bladders in cryogenic fluid tanks because of its resistance to reactivity with liquid oxygen (LOX). In spite of its desirable properties in most environments, this material has notoriously poor radiation resistance. The degradation of its tensile properties when it is irradiated in air or in a vacuum has been documented by Jolley and Reed,¹⁰ Lucas,¹ and others. The dose causing complete degradation or zero strength in air at room temperature has been reported variously from 10^8 to 10^{10} ergs-g⁻¹ (C)^{10, 11}; but in the present work, it was observed in 0.010-in.-thick specimens at a dose of 4.6×10^9 ergs-g⁻¹ (C). Unfortunately, data at cryogenic temperatures were not obtained in this study because of equipment problems, particularly those involved in gripping the specimens. However, stress-strain curves have been measured carefully at temperatures as low as -100°C by Chatain;¹² even at this temperature, the large effect of increasing modulus and strength with decreasing temperature is obvious, as seen in Figs. 1, 10, and 11. The observed inflections, or "second-order transitions," in these curves are the central point of Chatain's thesis and his theoretical basis for classifying material properties. Much more work needs to be done on this material at cryogenic temperatures.

Polychlorotrifluoroethylene (Kel F-81, CTFE) is considered for use in many of the same applications as TFE. Although it does not provide the same wide service temperature range, it has somewhat better radiation resistance. However, part of the difference between the two materials observed in this study was caused by major differences in specimen thickness and in processing conditions. The CTFE was 0.125 in.

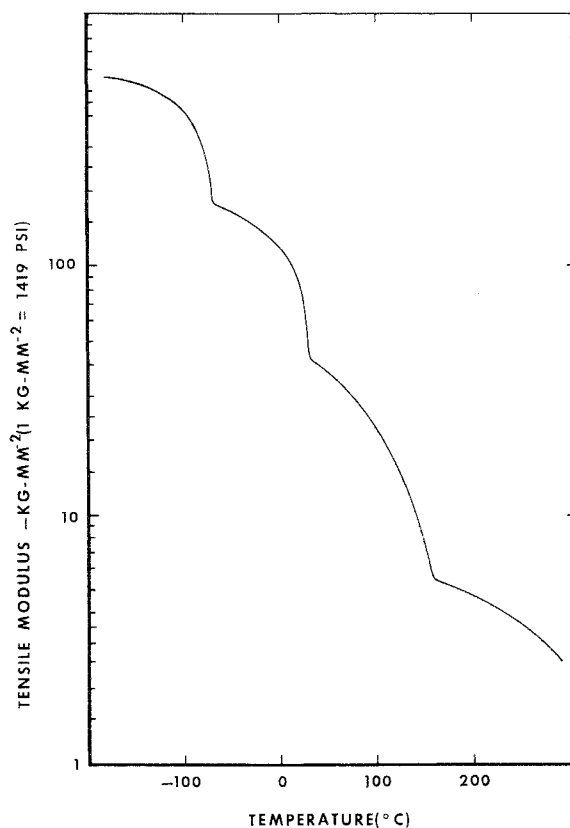


Fig. 11 Tensile modulus vs temperature for polytetrafluoroethylene (Teflon).

thick and was processed by melt extrusion; the TFE was 0.010 in. thick and was processed by cold pressing and subsequent sintering of a powder. Figure 12 shows that the tensile strength of unirradiated CTFE increases with decreasing temperature, but the material degraded rapidly by chain scission when irradiated at room temperature in air. The zero-strength dose was less than 1×10^{10} ergs-g⁻¹ (C). The degradation reaction appears to involve splitting off a halogen atom which leaves a secondary radical to combine with oxygen in a series of reactions that cause scission of the carbon chain. Irradiation in LN₂ degraded the tensile strength only 12% at 1.0×10^{10} ergs-g⁻¹ (C). Again, the degradation reaction involves splitting off a halogen atom which leaves a secondary radical. However, because of the lack of sufficient oxygen, the halogen probably recombines with the secondary radical to end the reaction series. Therefore, the degradation rate is mild when compared with the avalanche of reactions which occurs in the presence of oxygen. Irradiation in LH₂ again degraded the tensile strength rapidly, but the degradation started from very high initial value because of the cold temperature. This degradation reaction probably is caused by the combination of a split-off halogen with hydrogen to form an acid. Normally, the acid does not attack the material, but the complexity of secondary radicals and other reactive groups provides for the appearance of an avalanche effect similar to that observed during radiation in air.

At 5×10^9 ergs-g⁻¹ (C), the material degrades at room temperature but retains a high strength in LN₂ and LH₂. At a constant high dose of 1×10^{10} ergs-g⁻¹ (C), the material has zero strength at room temperature in air, high strength in LN₂, and moderate strength in LH₂. The explanation for these differences was suggested in the previous discussion. The elongation at all of the cryogenic conditions, with or without radiation, was about 1.0%.

This material can be recommended for use at cryogenic temperatures, and in an inert environment such as nitrogen, it can be irradiated up to at least 1×10^{10} ergs-g⁻¹ (C). The strength at these conditions is greater than its initial value of 5600 psi. It is not recommended for irradiation in air or in hydrogen, even at low doses.

Electrical Insulations: Films

Thin plastic films are used extensively in space vehicles as dielectrics in many electrical components and as vapor barriers in cryogenic insulation systems. A spectacular space application is represented by the Echo Balloon Satellites. Tensile strength and elongation were determined for two films, a polyester and a polyimide, at the nine environmental conditions according to a modification of ASTM-D-882-61T. Two or three duplicate specimens were tested at each condition, except in a few cases in which measurements were obtained for only one specimen because of equipment problems. The latter cases will be discussed separately. A 4-in. grip separation was used, and the specimen width was 1 in. without reduction in the gage length. All of the specimens were taken from the same roll of film and tested as received. The specimen grips were spools on which the film was wrapped. The initial values of ultimate tensile strength and elongation compared favorably with manufacturer's data.

A 0.001-in.-thick, oriented, *polyester film* (Mylar), based on polyethylene terephthalate, was tested. Irradiation data from the literature indicate that it is stable up to 10^9 or 10^{10} ergs-g⁻¹ (C). Although the tensile strength is considered in this report, the electrical properties are, at least, equally important. The electrical properties of Mylar and several other interesting polymers are now being evaluated and will be reported in the near future. Figure 13 shows that the tensile strength (unirradiated) at cryogenic temperatures is about five times the room-temperature value, with no difference between LN₂ and LH₂ exposure. The latter observation may occur because a small amount of moisture is en-

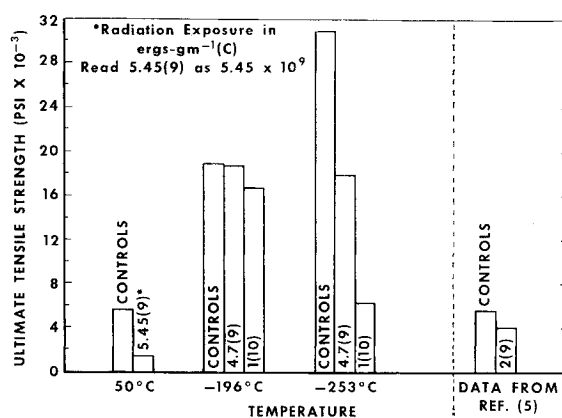


Fig. 12 Radiation-cryogenic temperature data for a fluorocarbon seal (Kel-F-81).

trapped in the material and acts as a plasticizer at room temperature but is frozen out at both LN₂ and LH₂ conditions. In the presence of radiation, the strength apparently increases at cryogenic temperatures, but equipment problems invalidated most of the data. Maximum strength is reached at a dose below 8×10^9 ergs-g⁻¹ (C); this peaking effect also may be caused by entrapped moisture, which would cause chain scission by hydrolysis where sufficient energy was applied. Mylar, therefore, may be recommended tentatively for use at cryogenic temperatures and at irradiation doses up to 5×10^9 ergs-g⁻¹ (C), because the strength did not decrease significantly below the initial value (18,000 psi) in any event where data were obtained.

Polyimide (H-Film), a new film material that is expected to be commercially available in 1964, was evaluated because of its high strength, wide service temperature, and radiation stability as reported by the manufacturer. The thinnest film available at the time the experiments were made, 0.0025 in., was supplied by the manufacturer. The data in Fig. 14 indicate that the tensile strength increased with decreasing temperature as expected according to the typical modulus vs temperature relationship. The same trend with temperature was observed at constant radiation doses up to 1.3×10^{10} ergs-g⁻¹ (C). In another study,¹³ this material proved to be the most flexible of all of the films tested for a mandrel bend evaluation in liquid helium and in LH₂. Irradiation in air at room temperature and in LN₂ caused no significant change in the tensile strength. This fact is corroborated by data from the manufacturer and from the Langley Research Center of NASA. Irradiation in LH₂ resulted in a high peak in the tensile strength at around 5×10^9 ergs-g⁻¹ (C), a fact that might suggest the possibility of a hydrolysis reaction where sufficient energy was applied to the material while in a reducing environment. This material is recommended for use at cryogenic temperatures and at doses up to, at least, $1.3 \times$

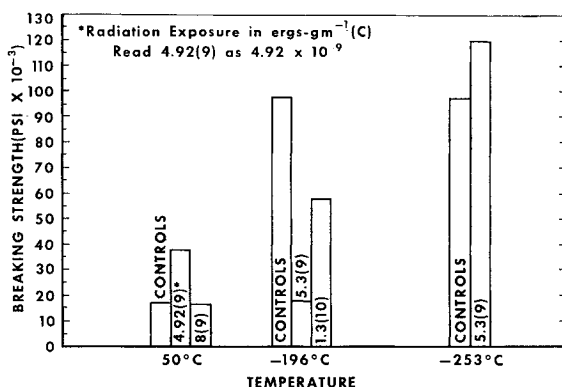


Fig. 13 Radiation-cryogenic temperature data for a polyester electrical insulation (Mylar C).

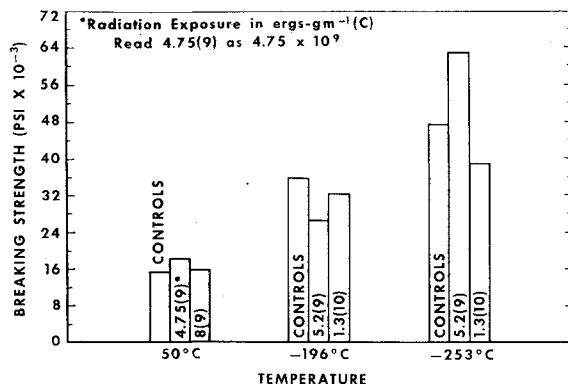


Fig. 14 Radiation-cryogenic temperature data for a polyimide electrical insulation (H-Film).

10^{10} ergs-g⁻¹ (C), because the tensile strength did not fall below its initial value of 15,000 psi in any event.

Recapitulation of Conclusions

Conclusions and recommendations concerning each of the materials evaluated were reported in the same sections in which the results were discussed. However, it is appropriate to summarize the new information gained in order to assess the general value of the program. Of course, a large amount of data was collected on materials of interest in environments where only educated guesses existed previously. This collection of data is required in order to provide actual strength values-in-use for designers. Another vital service of a program like the one described is to discover unusual effects, which would not have been expected. Some unpredicted effects are summarized as follows:

1) An epoxy-nylon adhesive loses strength at cryogenic temperatures, a trend that is contrary to most materials. One reason why this material is of interest is because of its relatively high strength at room temperature, but this advantage is lost at cryogenic temperatures.

2) A phenolic-glass laminate and a polyester-glass laminate changed little in strength and modulus at cryogenic conditions, but the elongation increased. The latter effect probably is dependent upon the breaking of glass-to-resin adhesion because of differential thermal expansion.

3) Polystyrene and polyurethane resins, which normally have high radiation resistance, are poor in this respect when expanded into a foam. It is suggested that this effect is dependent upon the foaming agent.

4) A fluorocarbon resin, which has poor radiation resistance in air and LH_2 , has exceptionally good resistance in LN_2 . An explanation of these differences is based on reactions with the medium rather than on temperature.

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