PROPERTIES OF NONMETALLIC MATERIALS AT CRYOGENIC TEMPERATURES

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SUMMARY

This paper presents property data and information describing the various cryogenic applications for nonmetallic materials. Until very recently, this classification of materials was usually ignored for structural applications at cryogenic temperatures.

Basically, there are three reasons for the reluctance of designers to use this family of materials. First, there were essentially no cryogenic mechanical and physical property data available. Few standards for samples or test methods existed to determine the properties, which made it extremely difficult to compare data obtained with the meager data available. Second, stress analysis of the most attractive structural materials, the filament-wound, fiber-reinforced composites, was extremely complex and specialized, and the concepts utilized to arrive at design analyses were still under development. Third, variations in the constituents and their effect on end-item properties made it less than attractive to utilize this type of material for cryogenic structural applications, although preliminary data indicated these materials had a great potential because of their high strength-to-weight ratio.

The impetus necessary to overcome this lack of information was provided by the aerospace industries' urgent requirements for structural materials with high strength-to-weight ratios capable of operating over wide temperature ranges. Minimum weight has always been important for aircraft applications, but is of the utmost necessity for hardware used in upper stage vehicles, satellites, and spacecraft.

Contract funds were provided to various organizations to obtain cryogenic property data, to develop sophisticated design analysis techniques, and to improve the materials available for use in manufacturing structural nonmetallic hardware for cryogenic service.

Today the results of these programs are being reduced to common practice. Other scientific disciplines, such as high-energy physics, which work in the cryogenic temperature region, are benefiting from the results obtained in the programs initiated for cryogenic aerospace applications.

INTRODUCTION

Many nonmetallic materials are either being used or evaluated for structural or semistructural applications at cryogenic temperatures. Some advantages that are considered when evaluating these nonmetallic materials for cryogenic temperatures include the following characteristics:

1. High strength-to-weight ratios.

2. Low thermal conductivities.

3. Low specific heats.

4. Ease of orthotropic fabrication.

5. Corrosion resistance.

6. Durability.

7. Self-lubricating properties of some materials.

8. Fatigue resistance.

However, these potential advantages are not achieved without simultaneously accepting certain design problem areas which result from the following characteristics of nonmetallic materials:

1. High coefficients of thermal expansion.

2. A tendency towards brittleness at low temperatures.

3. Complexities of the stress analysis of heterogeneous composite resin-fiber structures.

4. Lack of generally standardized test techniques for developing design data.

5. Variations in resins and fibers used to fabricate composite structural elements.

Some typical cryogenic applications for nonmetallic materials of construction can be separated into five classifications:

1. Reinforced plastic structures.

2. Adhesives, sealants, and coatings.

3. Bearings and dynamic seals.

4. Gaskets and static seals.

5. Film materials used as diaphragms, vapor barriers, etc.

A sixth major use classification for nonmetallic materials, cryogenic thermal insulation, has not been considered for this paper.

A short description of typical examples from each classification is presented below. This overview will be followed by a presentation of cryogenic property data available at the present time for materials from each of these classifications. More complete descriptions of current and potential applications will be presented in each section. Limitations of the data available and a discussion of the data not yet obtained will also be presented.

CLASSIFICATION OF NONMETALLIC MATERIAL USES

The first category of nonmetallic materials includes glass-fiber reinforced-plastic structures such as filament-wound pressure vessels used for storage of cryogens or as the basic structural element of liquid hydrogen bubble chambers (although fiber-glass is the principal current reinforcement, high modulus fiber reinforcements such as boron and graphite are being evaluated with various resin combinations in an attempt to develop a composite laminate material with a modulus at least as great as aluminum). Another cryogenic application for fiber-reinforced plastic materials is structural insulators and supports used between the insulation and outer walls of cryogen containing vessels. Sandwich constructions of fiber-reinforced plastic face materials in combination with honeycomb, foam core systems, and foam-filled honeycombs have also been used in various cryogenic systems. These combinations of materials are used as composite structural members of insulation systems where the heat leak through a honeycomb core must be tolerated to prevent structural failure of the foam insulation caused by thermal shock and strain.

The second category of nonmetallic materials for structural applications at cryogenic temperatures consists of adhesives, sealants, and coatings. The effects of cryogenic temperatures on the strengths of adhesive joints have recently been examined by many investigators. Sections of large cryogenic tanks have been successfully joined using adhesive bonding techniques. Dissimilar metallic materials are bonded together to prevent galvanic corrosion, and integral linings for filament-wound cryogenic storage vessels are adhesively bonded into place. The most critical problem to be overcome before filament-wound tanks can be used for multiple cryogenic pressure cycles is associated with the development of an adhesive system to attach the liner to the filament-wound wall. Adhesive sealants are also used at cryogenic temperatures to provide pressure-tight structural seals. Some organic coatings have been evaluated for reducing to zero the tolerances between rotating members in cryogenic turbomachinery applications. Thin coatings of some of these same insulating materials have been shown to alter the boiling characteristics of hydrogen so as to enhance the heat transfer from cryogenic fluid to the structural members, and in this manner to reduce the time required to achieve thermal equilibrium.

The third category for structural applications of nonmetallic materials at cryogenic temperatures includes dynamic seals and bearings. Typical cryogenic shaft-riding face seals utilize various proprietary compositions of carbon-graphite or ceramic oxide materials as the primary seal. A compromise is made between the amount of friction, face wear, and the leakage allowed past the seal face.

The fluorocarbon plastics, in particular, have inherently low friction coefficients at all temperatures. Many cryogenic bearings utilize various types of fluorocarbon resins, alone or in combination with other materials. These specialized items form a very small but important portion of many cryogenic devices.

Static gaskets and seals comprise the fourth category of nonmetallic materials used in cryogenic applications. Gaskets used at cryogenic temperatures, however, become harder as the temperature decreases. To maintain the sealing capacity accomplished at ambient temperatures, the gasket should maintain flexibility or compressibility. A further problem with most organic gasket materials evolves from their inherently larger thermal contraction coefficients compared to the joining metal surfaces. This results in a decrease in sealing force as the device cools to cryogenic temperatures and increases the potential for leakage past the gasket. Various special nonmetallic gaskets, composite metal-plastic gaskets, and special design techniques have been developed to meet cryogenic sealing requirements.

Many types of nonmetallic materials are used as static structural seals at cryogenic temperatures. These seals are used to control the flow of cryogenic fluids and gases under high and low pressures and from flowrates small enough to be measured with a mass spectrometer to those measured in tons/second. Many of these cryogenic seals have utilized fluorocarbon plastic materials. This family of materials maintain some ductility at extreme cryogenic temperatures even at extremely high strain rates.

A fifth category of nonmetallics used in cryogenic applications includes the film materials that are used as diaphragms and vapor barriers. Regulators with nonmetallic pressure sensing diaphragms have been used in cryogenic environments (fluid and pneumatic) at high and low pressures. High pressure differentials over small areas are common, and multi-ply polyester film diaphragms have been used effectively at high- and low-frequency rates at temperatures as low as $20^{\circ}K$. Over two-million cycles have been completed successfully at $20^{\circ}K$ using this multi-ply type of diaphragm as a small R&D positive displacement pump for liquid hydrogen.

Representative cryogenic test programs pertaining to all five use categories of nonmetallic materials are presented in the following sections. Each section includes the title of the program, its source, its objectives, the materials tested, the properties measured, the data obtained, and the conclusions drawn.

Reinforced Plastic Laminates

Reinforced plastic laminate materials have received the most experimental attention because their potential structural properties are very attractive to a designer. Comprehensive test programs have been completed by various organizations pertaining to the effects of cryogenic temperatures on mechanical and physical properties of reinforced plastic laminates.

Data from five programs have been selected for this report. One program was established to determine if reproducible data could be obtained for fiber-reinforced plastic materials at cryogenic temperatures; a second presents data obtained for glass fabric laminates utilizing a variety of resin systems; a third shows data for unidirectional filament-wound laminates; a fourth indicates the combined effects of nuclear radiation and cryogenic environments; and a fifth presents property data for filament-wound laminates being considered for liquid hydrogen bubble chambers.

Title. "An Assessment of Test Specimens and Test Techniques Useful to the Evaluation of Structural Reinforced Plastic Materials at Cryogenic Temperatures" (NAS 8-11070).

Source. Goodyear Aerospace Corporation, Akron, Ohio.

Objectives. The primary objective of this program was to establish industry standards for reinforced plastic test specimens and test techniques applicable to cryogenic temperatures. The ultimate objective was to obtain a design handbook containing data for a variety of reinforced plastic materials at cryogenic temperatures.

Materials. To minimize variables, two S/HTS glass fabric styles, 1543 and 1581, were used in this program. A single epoxy resin was preimpregnated onto both fabrics.

Properties. Tensile strength, elongation, and modulus; compressive strength, flexural strength, and modulus; shear strength and bearing strength; were measured parallel, normal, and at $45°$ to the warp direction of the fabric.

Test temperatures. Test temperatures were 298, 197, 77, and $20°K$.

Data. Some of the data obtained in this program are presented in Table I. These data show that the mechanical properties of fiberglass-reinforced plastics increase substantially at lower temperatures over the room temperature values. The specific strengths (stress-density) of the materials were found to be exceptionally high.

Conclusions. The spread of the data, expressed as the coefficient of variation, obtained during the program for samples cut parallel to the principal reinforcement direction were:

Tension	4.8%
Compression	9.6%
Flexure	5.6%
Shear	9.6%
Bearing strength	9.4%

These coefficients of variation were based on a small number of samples and include the rather large variation obtained in the strength of the S/HTS glass fibers themselves. It was concluded, therefore, that the test values appear in general to be within acceptable limits.

Title. "Determination of the Performance of Plastic Laminates Under Cryogenic Temperatures" [AF 33(616)-8289].

Source. Narmco Research and Development, San Diego, California.

Objectives. The objective of this program was to determine the mechanical properties for a wide variety of plastic laminate materials at cryogenic temperatures.

Materials. Glass fabric (181 style) laminates were fabricated using epoxies, phenolics, polyesters, high-temperature polyesters, silicones, flexible polyurethanes, polybenzimidazol, fluorinated ethylene propylene, phenyl-silanes, and nylon epoxies.

Properties. Tensile, compressive, and flexural strengths and moduli; tensile fatigue, and bearing strength tests were performed.

Test temperature. Tests were performed at 298, 195, 77, and 20°K.

Data. The data indicated that reinforced plastics generally increase in strength as temperatures decrease. Based on static test data, it was found that epoxy resins were best for cryogenic use; polyesters and phenolics were second. When subjected to tensile fatigue, however, the epoxies and phenolics continued to show good life while the polyesters fell behind. Average tensile strength of the glass-reinforced laminates is shown in Fig. 1. Average tensile, compressive, and flexural property data are shown in Figs. 2, 3, and 4. Bearing strength and tensile fatigue data are shown in Figs. 5 and 6.

Conclusions. It was concluded that decreasing the test temperature from 77°K to 20°K had little effect on properties, although decreasing from ambient to 77°K resulted in marked increases in strength. Further, it was concluded that modulus values did not increase as rapidly as strength data as test temperatures were reduced. Finally, the tensile fatigue strengths of fiberglass-reinforced plastics were higher at cryogenic temperatures than at room temperatures.

Title. "Cryogenic Properties of High Strength Glass-Reinforced Plastics" [AF 33(657)-9161].

Source. Martin Company, Denver, Colorado.

Objectives. The objectives were to determine the behavior of a variety of uni-directional filament-wound resin/glass combinations at cryogenic temperatures.

Materials. Ten resin systems and two glass types were used. Resins included four epoxies, an epoxy novolac, a phenolic, two polyesters, a silicone, and a phenyl-silane. E and S glasses were used.

Properties. Tensile strength, modulus, and static tensile fatigue data were obtained.

Test temperatures. Tests were performed at ambient, 77, and 20°K.

Data. The strength data for the various resin systems are shown in Figs. 7 and 8. Modulus data is shown in Fig. 9. These data show a general increase in strength as temperature is decreased from ambient to 77°K. Below 77°K, strength shows a tendency to level off or decrease. Modulus data show a continued increase down to 20°K as opposed to the strength data. Static fatigue did not occur at cryogenic temperatures, which adds evidence that ambient temperature static fatigue is in reality a stress corrosion process resulting from absorbed water vapor onto the glass.

Conclusions. The conclusions drawn from this study were: first, the resin made a large contribution to the tensile strength of NOL ring samples at cryogenic temperatures, and second, the effect of reinforcement type on the tensile modulus was significant. The E-glass material shows a much lower modulus than the S-glass.

Title. "Effect of Nuclear Radiation and Liquid Hydrogen on Mechanical Properties of Three Phenolic Materials."

Source. Aerojet-General Corporation, Sacramento, California.

Objectives. The objectives were to investigate the mechanical properties of three phenolic based materials in a combined environment of liquid hydrogen and nuclear radiation.

Materials. Phenolic asbestos-cloth laminates (Grade AA - MIL-P-8059A), phenolic linen-cloth laminates (Grade L - MIL-P-15035B, Type FBI), and phenolic glass-cloth laminates (MIL-F-9084, Type VIII impregnated with 91-LD phenolic resin) were used in this study.

Properties. Ultimate tensile strength and elongation data were obtained.

Test temperatures. Tests were performed at $20^{\circ}K$ plus one series at ambient temperatures.

Data. The data obtained in this study are presented in Table II. The data show that tensile strength for all three materials is not appreciably affected by the combined environment of cryogenic temperature and nuclear radiation. However, loss of ductility was significant after nuclear radiation.

Conclusions. Care should be exercised in selecting any of the three types of materials tested for a combined cryogenic-radiation environment if any appreciable strain must be sustained by the materials.

Title. "Physical Properties of Filament-Wound Glass-Epoxy Structures as Applied to Possible Use in Liquid Hydrogen Bubble Chambers."

Source. Stanford Linear Accelerator Center, Stanford, California.

Objectives. The objectives were to determine the feasibility of using filament-wound laminates for a structural, liquid hydrogen pulse system bubble chamber where eddy current heating makes a metal chamber impractical.

Materials. Materials tested were S/HTS roving impregnated with ERL 2256 epoxy and MPDA hardener. H-film sheets were incorporated into the structure to reduce permeability.

Properties. Fatigue (flexural), flexural strength and modulus, shear strength, low-temperature permeability, and thermal contraction were measured.

Test temperatures. Tests were performed at ambient to $4.2^{\circ}K$.

Data. Thermal contraction data obtained in this program are shown in Fig. 10. It is shown that the laminate contraction is basically that of the glass filaments. Flexural fatigue data are shown in Table III. The data indicate that inclusion of H-film in the structure had no adverse effects on the integrity of the laminate under flexural fatigue conditions. Flex and shear strength data of samples with Mylar,

Tedlar, and beryllium film showed a weakening of the matrix. Shear strength losses on the order of 50% were measured for all samples with barriers other than H-film. H-film resulted in ≃ 5% loss in shear strength.

Conclusions. Glass-filament epoxy structures exposed to cryogenic temperatures, particularly liquid hydrogen, are equal to or superior to stainless-steel bubble chambers with dc field or transient magnetic fields. Problems such as feed-through tubing into the chamber and sealing of the chamber must be investigated in more detail. The filament-wound chamber was lighter than a stainless-steel unit and had considerably less static heat loss than stainless steel.

Adhesives, Sealants, and Coatings

Structural adhesives

Structural adhesive evaluations for cryogenic temperature environments have been reported in the literature since the middle 1950's. The early reports were limited to descriptions of paste and liquid adhesive systems and usually to temperatures down to 77°K. Recent programs have considered the basic problems associated with bonded joints at cryogenic temperatures. Most of these problems have been attributed to stress concentrations and gradients developed within the bond. Some of the principal causes for these concentrations include:

1. Differences in the thermal coefficient of expansion between adhesive and adherends.

2. Shrinkage of the adhesive on curing.

3. Trapped gases or volatiles evolved during bonding.

4. Differences in elastic moduli and strength between adhesives and adherends.

5. Residual stresses in joints as a result of releasing bonding pressures.

For example, a low modulus adhesive may readily relieve stress concentrations at room temperatures by deformation, but at cryogenic temperatures it may become so brittle that these concentrated stresses are only relieved by fracture of the adhesive.

The results of two review papers from the same organization are presented to describe some of the recent information developed for cryogenic structural adhesives. The later report presents information obtained through 1964; the earlier report includes data obtained as early as 1961. Differences in the conclusions indicate continued increase in knowledge regarding adhesives for cryogenic temperatures.

Title. "Development of Adhesives for Very Low Temperatures" (NAS 8-1565).

Source. Narmco Research and Development, San Diego, California, 1961-1963.

Objectives. The objectives were to search the literature and evaluate various commercially available adhesives for cryogenic applications.

Materials. Research efforts were concentrated on nylon-epoxy, epoxy-polyamides, polyurethane, and fluorocarbon film systems. Aluminum and stainless-steel adherends were used.

Properties. Tensile shear, tee peel, mechanical shock, and butt tensile strengths were determined.

Test temperatures. Tests were performed at room temperature, 77, and 20°K.

Data. Tensile lap shear strength data for the commercial adhesives evaluated at the beginning of this program are shown in Fig. 11. These data indicate the general superiority of nylon-epoxy adhesive systems at cryogenic temperatures.

Conclusions. The nylon powder-filled epoxy-polyamide paste was the best system evaluated during this program. A polyurethane elastomer adhesive showed superior strength and toughness at extremely low temperatures. However, it tended to absorb moisture from the air, which caused it to foam during bonding and resulted in unsatisfactory joints.

Title. "Adhesive Bonding of Insulation for Temperature Extremes — Cryogenic to Re-entry."

Source. Narmco Material Division, Costa Mesa, California, 1964.

Objectives. The objectives were to determine mechanical property data for a variety of structural adhesives at temperatures from 20 to ∼ 800°K. The advantages of adhesives, design considerations, fabrication techniques and variables, and recommended steps for selecting an adhesive were also summarized.

Materials. Nylon epoxies, modified epoxies, silicone phenolics, epoxy-phenolics, polyaromatics, and polyurethane adhesives were investigated.

Properties. Tensile shear, tee peel, mechanical shock, and butt tensile strengths were determined.

Test temperatures. Tests were performed at 20 to 850°K.

Data. The effect of temperatures on the lap shear strength of different types of adhesives is presented in Fig. 12. These data show that at extreme cryogenic temperatures, the polyurethane adhesives are stronger than all others. However, at ambient temperatures the strength of the polyurethane adhesive is less than most of the other adhesive types. Figure 13 shows the lap shear strengths of eight different types of adhesive systems at a temperature of 20°K.

Conclusions. Polyurethane adhesives are very good for extremely low temperatures, but their strength decreases very quickly as they approach ambient temperatures. The nylon-epoxy materials, on the other hand, have a rather uniform strength at temperatures from 20 to 373°K.

Adhesive sealants

NASA has sponsored programs to develop adhesive sealants for use at cryogenic temperatures. These programs were directed toward finding materials that would be flexible down to a temperature of 20°K. However, preliminary screening tests indicated that even the most flexible materials were incapable of meeting the bending requirements described in MIL-S-8516 at liquid nitrogen (77°K) temperatures. Further testing indicated that the low-temperature sealing characteristics of polyurethanes and methyl-phenyl silicone elastomers could be improved by using them in conjunction with nylon or glass-fabric reinforcements. The higher modulus fabric reinforcements minimized the thermal strains in the sealants at low temperatures by accepting a larger portion of those thermal strains and transmitting them to the metal substrate rather than concentrating them in the sealant.

Various informal programs have been performed to evaluate specific cryogenic sealant requirements. One of these programs was directed toward the development of a sealant system to prevent moisture from penetrating into rigid polyurethane-foam-insulated liquid hydrogen lines. A polyurethane elastomer impregnated into circumferentially wrapped open weave nylon tape was satisfactory for this purpose, even when attached directly to the outer metal wall of the hydrogen line.

Very little quantitative data are available pertaining to sealants for use at cryogenic temperatures. Evaluation tests have to be tailored to the solution of particular problem areas. One of the few reports available is summarized below.

Title. "Development of Cryogenic Sealants for Applications at Cryogenic Temperatures" (NAS 8-2428).

Source. Hughes Aircraft Company, Culver City, California.

Objectives. The objectives were to develop organic sealants that may be used to seal or repair containers for cryogenic fluids.

Materials. Eight different polymer systems were investigated:

1. Polysulfide.

2. Silicone.

3. Epoxy.

4. Polyurethane.

5. Fluorosilicone.

6. Epoxy-silicone.

7. Butyl formal, butyl ether, and polysulfide blend.

8. Carboxyl terminated polybutadiene and epoxy blend.

Properties. Low-temperature bend characteristics, thermal contraction, and resistance to vibration were determined.

Test temperatures. Tests were performed at ambient to $77^{\circ}K$.

Data. Only reinforced silicones and polyurethanes met vibration and bend test requirements at $77^{\circ}K$.

Conclusions. The woven fabric provided reinforcement continuity throughout the structure, and was capable of transmitting loads to the metal substrate during the bend tests. Further, the reinforcement lowers the over-all thermal contraction to a level where thermal stress at the sealant-substrate interface is reduced to a minimum.

Structural coatings

Structural coatings have been considered for cryogenic turbomachinery applications. These coatings were evaluated for dual use as an internal wearing surface and as an internal thermal insulation. Investigations performed by several manufacturers of liquid propellant rocket engines indicated that some filled fluorocarbon plastic materials might be effectively utilized for this dual (wearing and insulating) purpose. Unknown factors included the possibility that thermal strains could be large enough to cause the coating to separate from the metallic substrate at cryogenic temperatures, and determination of the effects of fluid erosion on the adhesion of the coatings. Some

of the systems evaluated were quite promising, even though only preliminary tests were completed. One of these preliminary programs is described below.

Title. "Evaluation of KX635 for Turbomachinery Applications."

Source. Rocketdyne, a division of North American Rockwell Corporation, Canoga Park, California.

Objectives. The objectives were to determine if a filled fluorocarbon plastic, (KX635) might be developed for an insulating internal wearing surface for cryogenic turbomachinery.

Materials. Metallic substrates were K-monel and Tens-50 aluminum casting alloy. Coating material was KX635, a glass microballoon-filled Kel-F dispersion material.

Properties. Bond strength between coating and substrates at ambient and cryogenic temperatures was determined by static tensile tests and flexural fatigue tests. Thermal contraction and thermal conductivity were measured from ambient to cryogenic temperatures.

Test temperatures. Tests were performed at ambient, 77 and 20°K.

Data. Tensile tests indicated that the coating adhered to metallic substrates at all temperatures until metal failure occurred. Flexural fatigue tests at cryogenic temperatures showed that the coating adhered to substrates at total deflections up to 0.300 in. without coating-to-metal separation. Erosion rates of the coating material caused by fluid flow were not determined.

Conclusions. The various data generated were promising enough that further efforts could be justified to evaluate this type of internal insulation-wearing surface for cryogenic applications.

Dynamic Seals and Bearings

Nonmetallic materials are used as semistructural elements in many dynamic seal and bearing applications at cryogenic temperatures. Bearings and seals are required for any application with movement or where flow of cryogenic fluids or gases must be controlled. Examination of the literature reveals that materials from one polymer family, the fluorocarbons, are widely used for these applications. The fluorocarbon plastics have characteristics that make them very attractive for cryogenic sealing and wear applications. Some of these are:

1. Some ductility at cryogenic temperatures.

2. Adequate mechanical properties at cryogenic temperatures.

3. Chemical inertness.

4. Low coefficients of friction over total useful temperature range.

Many cryogenic seal applications utilize unmodified fluorocarbon plastics as sealing elements. Generally, cryogenic bearing applications have used fluorocarbons (particularly TFE Teflon) to achieve the appropriate friction, wear, and durability characteristics necessary for prolonged, trouble-free operation at cryogenic temperatures under widely varying stress conditions.

Data from four programs pertaining to fluorocarbon plastics have been selected

for this report. The first two describe efforts performed to develop self-lubricated bearings for cryogenic temperatures. The third describes a comprehensive program initiated to obtain design properties of various thermoplastic materials at temperatures down to 20°K. The fourth program presents information pertaining to strain rate, temperature, crystallinity, and surface smoothness on the mechanical properties of PCTFE plastics.

Title. "Bearing Lubrication at Low Temperatures."

Source. British Oxygen Research and Development, Ltd., London, England.

Objectives. The objectives were to determine the effectiveness of various Teflon-containing materials as solid lubricants in liquid oxygen and liquid nitrogen against stainless steel and lead-bronze shafts.

Materials. Shaft and bushing materials investigated were:

1. Shafts — Two types of shaft material were used.

   a. S.80 stainless steel.

   b. 20% lead-bronze.

2. Bushings — Three types of bushing were used.

   a. Porous bronze (0.010 in. thick) filled with TFE to a thickness of 0.0005 in. (Glacier DP).

   b. TFE, with graphite and bronze powder added (Glacier DQ).

   c. Porous bronze containing 80% bronze and 20% TFE (Bound Brook Polyslip).

Properties. Wear of bearings (measured as increase in diameter of bushing) was determined after 1.5 hours running time at stresses from 100 to 900 psi.

Test temperatures. Tests were performed at 90 and 77°K.

Data. Wear data for the three Teflon-containing bushing materials against the two shaft materials in liquid oxygen are shown in Fig. 14. The data indicate all three materials were essentially equivalent when running against a 20% lead-bronze shaft. The bearing material with the least quantity of exposed Teflon had the least wear against S.80 stainless steel.

Conclusions. It was concluded that a shaft with good thermal conductivity used in conjunction with TFE Teflon-containing bearing material was suitable for service in liquid oxygen and liquid nitrogen. The design of the bearing is such that adequate "dry lubrication" is maintained under operating conditions, and a free flow of liquid passes through the bearing for cooling.

Title. "Evaluation of Ball Bearing Separator Materials Operating Submerged in Liquid Nitrogen."

Source. CEL, National Bureau of Standards, Boulder, Colorado.

Objectives. The objectives were to develop a reliable, high-load-capacity (axial and thrust) bearing operating at moderate speeds.

Materials.

    1. Ball and Race — ball and race materials were 440C.

    2. Separators — separators were three types:

        a. Metallic coated with TFE.

        b. Filled TFE.

        c. Phenolic.

Properties. Bearing life (hours vs torque in in.-oz) was measured while operating under constant environmental conditions of thrust load, speed, and temperatures.

Test temperature. Tests were performed at 77°K.

Data. Sample identification information is shown in Table IV. Bearing life data in liquid nitrogen for all samples tested are shown in Fig. 15. These data indicate that the filled TFE separator (sample No. 8) had a much longer bearing life than all other materials tested. Torque decreased with time for this material. Torque for all others increased with time.

Conclusions. It was concluded that filled TFE separators were superior to the other separator materials when operating in liquid nitrogen.

    Title. "Final Report, Program of Testing Nonmetallic Materials at Cryogenic Temperatures" [AF 04(611)-6354].

    Source. Rocketdyne, a division of North American Rockwell Corporation, Canoga Park, California.

    Objectives. The objectives were to determine the mechanical properties of a variety of thermoplastics at cryogenic temperatures. The study included the determination of the effect of thermal treatments on the crystallinity levels of certain materials and the resultant effect on mechanical properties.

    Materials. Materials tested were TFE and FEP Teflon, Kel-F, Mylar, and nylon, as well as fabric, fiber, and powder-reinforced TFE and FEP Teflons.

    Properties. Tensile yield, ultimate, modulus and elongation; flexural strength and modulus; compressive strength and modulus; impact strength, modulus of rigidity, and coefficient of thermal expansion were determined.

    Test temperatures. Tests were performed at 300, 194, 144, 77, and 20°K.

    Data. A summary of the data from this program is presented in Figs. 16-20 and Table V.

    Conclusions. The conclusions are separated into the five types of materials tested:

        1. Kel-F (PCTFE) — Ambient yield strength variations between the three crystallinity levels was ≃ 20% (5500 psi/amorphous, 6500 psi/crystalline). At a temperature of 20°K, the spread was ≃ 60% (28 600 psi/amorphous, 18 000 psi/crystalline). At ambient temperatures the amorphous material was slightly weaker than the crystalline, but at cryogenic temperatures the more amorphous was much stronger than the crystalline.

The notched Izod impact strength of Kel-F remained relatively constant over the test temperature range. However, the amorphous samples had slightly higher impact strength than the crystalline.

2. Teflon (TFE) — The effect of crystallinity on the low-temperature properties of TFE was very similar to Kel-F. The crystalline materials had higher properties than the amorphous at room temperatures, but at cryogenic temperatures, the reverse was true. The effect, however, was less pronounced than for Kel-F.

3. Teflon (FEP) — Normal processing variables had little effect on crystallinity of material. FEP Teflon has about 50% higher impact strength than Kel-F at cryogenic temperatures. Cryogenic elongation of FEP Teflon was also higher than Kel-F; however, it had lower flexural and compressive properties than Kel-F. In addition, the contraction of FEP Teflon is greater than Kel-F at cryogenic temperatures.

4. Powder and Fiber-Filled Teflons — Advantages of powder and fiber-filled TFE and FEP Teflons were found to be less evident at cryogenic temperatures than those usually described for ambient and moderately elevated temperatures. Results obtained in this program indicate that these materials were usually inferior to the unfilled materials. Coefficient of expansion of filled materials was less than the unfilled.

5. Glass-Fabric Reinforced Teflons — Glass-fabric reinforced Teflons are used where the mechanical properties of the glass are combined with the lubricity of the Teflon. The materials are very strong and can be used in applications involving tensile and flexural strength, as well as compression and shear. The room temperature mechanical properties of these reinforced Teflons, both FEP and TFE, are controlled in great part by the strength of the glass fabric, rather than the resin. However, the increase in mechanical properties for these materials at cryogenic temperatures is much greater than normal for fiberglass reinforced plastic laminates. The great increase in the strength of the Teflon resins at cryogenic temperatures is seen in the strength of the laminates at these low temperatures.

Title. "Effects of Strain Rate, Temperature, Crystallinity, and Surface Smoothness on the Tensile Properties of PCTFE Plastics."

Source. Rocketdyne, a division of North American Rockwell Corporation, Canoga Park, California.

Objectives. The objectives were to determine by statistical analysis the independent and interacting effects of strain rate, temperature, crystallinity, and surface finish on the tensile properties of PCTFE (Kel-F) plastic.

Material. Test material was PCTFE (Kel-F).

Properties. Tensile yield, maximum and ultimate, elongation at maximum strength, and ultimate elongation properties were measured.

Test temperatures. Tests were performed at ambient and $77^{\circ}K$.

Data. Tensile data obtained over six decades of loading rates 0.02 to 10 000 in./min) at two temperatures (ambient and 77°K) using molded and machined surfaces on the samples are presented in Tables VI and VII. These data indicate that the maximum tensile strength at ambient temperatures increases in direct proportion to the strain rate, but at cryogenic (77°K) temperatures, the maximum strength was much less at 10 000 in./min loading rate than at the 1000 in./min rate. In fact, the maximum tensile strength of the material at 10 000 in./min loading rate was less at cryogenic temperatures than at ambient temperatures.

Conclusions. At the very high (1000 and 10 000 in./min) loading rates the effects of surface finish and crystallinity on the tensile properties of PCTFE plastic materials were much less apparent than at more conventional testing rates. At low and medium strain rates, the effects of crystallinity and surface were also found to be dependent on the loading rate at which the tests were performed. Further, strain rate effects were more significant at cryogenic temperatures than at ambient temperatures.

Gaskets and Static Seals

Until recently, flat gaskets were almost universally used for detachable flange seals. For cryogenic applications where flanges can be heavy, gaskets may still be the logical solution to a sealing problem. Gaskets used in cryogenic applications should not be brittle at use temperatures. Flanges and bolts must be selected so that cooling will not cause an excessive decay in sealing force as the gasket (because of its higher contraction) tries to shrink away from them. A further requirement, chemical inertness with liquid oxygen, must be met by gasket materials used in some aerospace cryogenic applications.

Very little thought has been given to the definition and standardization of cryogenic static seals. Generally, seals have been used that did an adequate job in a specific application, without regard to past or future seal designs or needs. Because of this history, there are many varieties of static cryogenic seals available today, and each claims superiority over rival designs.

A review of two specialized static seals has been selected for inclusion in this report. One is a gasket designed specifically for use in liquid oxygen, and the other, a plastic-coated-metal pressure-actuated seal, which is typical of the many new commercially available static seals on the market.

Title. "The Development of a New Cryogenic Gasket for Liquid Oxygen Service" (NAS 8-5053).

Source. Narmco Research and Development, San Diego, California.

Objective. The objective was to develop a superior flat gasket material for service in liquid oxygen.

Materials. The material tested was a laminated gasket composed of alternate layers of TFE Teflon film and glass cloth, encapsulated with FEP Teflon.

Properties. Compressibility and leakage measurements were performed.

Test temperatures. Tests were performed at ambient and 77°K.

Data. Laminate constructions that indicated higher energy absorption values at 77°K, with the least fall-off in the repetitive compressive energy absorption tests, were judged to be superior in compressibility. Figures 21 and 22 show the laminate

construction and the 77°K cyclic energy absorption data for the laminated Teflon-glass gasket construction compared to an asbestos-rubber gasket previously used in liquid oxygen applications. Leakage tests were performed by compressively loading the gaskets in a special flanged fixture to 3000 psi, and pressurizing the internal volume to 200 psi. Flange loads were reduced until leakage occurred to determine the ASME gasket m-factor. (Factors less than 1 only attained with pressure-actuated seals.) The same procedures were followed at ambient and cryogenic temperatures. The laminated material had m-factors as low as 1.31 at ambient and 1.56 at 77°K. The currently used asbestos-rubber gasket had values of 1.30 at ambient temperature, but at 77°K no seal was obtained at 3000 psi flange pressure.

Conclusions. The laminated TFE Teflon-glass fabric gasket material was superior to the currently used asbestos-rubber gasket in compressibility and sealing characteristics at 77°K.

Title. "Static and Dynamic Seals Used on Rocketdyne Engines."

Source. Rocketdyne, a division of North American Rockwell Corporation, Canoga Park, California.

Objectives. The objectives were to describe the various sealing concepts utilized in Rocketdyne liquid propellant rocket engines.

Materials. Test materials were various plastic, elastomeric, metal-plastic, and metal-elastomeric materials.

Properties. Detail seal performance was not given; sealing concepts were emphasized.

Test temperatures. Tests were performed at temperatures from ambient to 20°K.

Data. Data given are pertinent to the Naflex pressure-actuated seal. Rocketdyne designed and developed the Naflex seal in 1958 for the Atlas engine system. Many improvements have been made to the original design with the assistance of digital computers. The basic metal cup is made from steel alloys. The primary and secondary sealing surfaces are dispersion coated with Teflon resins fused to the metal substrate, Fig. 23. Leakage can be monitored between the primary and secondary seals. The Teflon coating provides the soft sealing surface and the metal portion, the spring force necessary to achieve a seal at pressures of 15 to 1500 psi at temperatures from 20 to 373°K.

Film Materials

High-strength plastic film materials have been used in a variety of structural and semistructural applications at cryogenic temperatures including diaphragms, bladders, and filament-wound tank liners. Shaft-riding lip-seals used at temperatures down to 20°K are often fabricated from multiple plies of various plastic films. Film materials are also used to form vapor barriers between cryogenic fluids and internal tank insulation materials such as polyurethane foams.

Liners for filament-wound pressure vessels designed for cryogenic temperature service must have enough biaxial ductility at cryogenic temperatures to be able to expand with the pressurized reinforced plastic shell. Not only must the liner materials have enough elongation at cryogenic temperatures to move with the shell, but the material must have enough additional low-temperature elongation to compensate for the higher thermal contraction of the liner over that of the filament-wound tank. These two additive requirements are very difficult to meet at cryogenic temperatures. Cryogenic

fluid vapor barriers for internally insulated cryogenic tanks have some of the same problems associated with liners for filament-wound tanks (although working pressure strains for large cryogenic metal storage tanks are considerably less than those of high-pressure filament-wound tanks). Further, a small leak of hydrogen through the vapor barrier into the internal polyurethane foam insulation greatly increases the thermal conductivity of the foam, reducing its efficiency.

Information presented from two papers pertaining to film materials as liners for filament-wound tanks for cryogens and as vapor barriers is outlined in the paragraphs below.

Title. "Structural Properties of Glass-Fiber Filament-Wound Cryogenic Pressure Vessels" (NAS 3-2562).

Source. Douglas Aircraft Company, Santa Monica, California, and NASA-Lewis Research Center, Cleveland, Ohio.

Objectives. The objective was to obtain a suitable liner for a filament-wound high-pressure tank for cryogenic service.

Materials. Materials tested were Mylar, Tedlar, H-film, and Seilon UR29E polyurethane.

Properties. Thermal contraction, biaxial cyclic tests, and uniaxial tensile tests were performed.

Test temperatures. Tests were performed at 297, 77, and 20°K.

Data. Uniaxial tensile data for the four film materials is shown in Figs. 24-27. These data show Mylar to have the highest strength at 20°K and Tedlar to have the greatest elongation. Thermal contraction data are shown in Fig. 28. The data show Mylar, of the four plastic film materials, to have the lowest contraction and UR29E polyurethane to have a contraction rate approximately four times as great. Table VIII shows the combined effects of the contraction differentials between the laminate, the liner, and the residual elongation of the liner at 20°K.

Conclusions. If the total theoretical elongations were realized by tank liners at cryogenic temperatures, the shell would only be carrying between 25 and 49% of its predicted ultimate load, not a very attractive limit. Only the Mylar-lined tank failed at its predicted stress. Other liner materials failed at stresses considerably lower than predicted. Because of this, the emphasis was switched to metallic foil liners.

Title. "Low Temperature Tensile, Thermal Contraction, and Gaseous Hydrogen Permeability Data on Hydrogen-Vapor Barrier Materials" (NAS 8-5600).

Source. Lockheed Missiles and Space Company, Sunnyvale, California.

Objectives. The objectives were to determine promising gaseous hydrogen vapor barrier materials.

Materials. Three film materials, two impregnated glasses, and three impregnated quartz types were evaluated.

Properties. Permeation, contraction, and tensile properties were measured.

<u>Test temperatures.</u>   Tests were performed at 297, 77 and 20°K.

<u>Data.</u>   Average permeation rates for films and impregnated fabrics are shown in Table IX. These data show the laminate films to be less permeable than the unsupported films. Contraction data are shown in Fig. 29 for film and laminate materials. Film materials had more contraction than the impregnated fabrics. Tensile properties of the film materials, Tables X, XI, and XII, show the mechanical properties of the film and impregnated fabrics.

<u>Conclusions.</u>   Although the ultimate strengths of the impregnated fabrics were higher than those of the film materials at cryogenic temperatures, the strain carrying abilities of the films were much better than the laminates. A further disadvantage of the impregnated fabrics was their poorly defined transition stresses in the 45° direction.

TABLE I

MECHANICAL PROPERTIES OF TWO TYPES OF EPOXY-GLASS CLOTH LAMINATES
WOUND ON FLAT MANDRELS AT AMBIENT AND CRYOGENIC TEMPERATURES

Laminate	Specimen Direction	Temperature, K	Tension, psi	Compression, psi	Flexure, psi	Shear, psi	Bearing Yield/Ultimate, psi	Tensile Elongation, percent	Tensile Modulus, percent	Flexural Modulus, psi
1581 Cloth (37.61% resin)	Parallel	298	92.506	62 587	104 186	8 237	45 925 / 79 017	3.37	3 286 670	3 328 300
		197	115 540	79 663	134 650	10.003	48 208 / 107 183	4.11	3 496 330	3 334 000
		77	144 543	103 202	170 638	13 495	67 330 / 121 530	5.01	3 980 000	3 911 500
		20	137 770	109 097	180 325	11 542	65 283 / 123 367	4.81	4 266 000	3 664 000
	Normal	298	84 474	59 161	96 297	7 638	44 433 / 76 333	3.30	3 166 000	3 127 200
		197	98.752	74 089	126 103	10 028	48 383 / 104 600	3.96	3 340 670	2 976 000
		77	128 010	94 826	157 132	12 240	65 933 / 121 200	4.64	3 974 000	3 589 333
		20	118 110	100 847	170 221	10.869	74 700 / 131 533	4.50	4 178 000	3 785 000
	45 degrees	298	32.716	31 370	48.529	6 186	41 750 / 86 200	10.0	1 498 670	1 631 900
		197	46 034	45 939	72 274	7 241	41 387 / 110 167	10.57	2 108 670	1 809 667
		77	50 684	69 935	105.156	9 442	67 733 / 121 300	6.71	2 730 670	2 600 667
		20	46 613	77 689	103 330	8 686	68 700 / 129 600	4.84	2 834 000	2 911 000
1543 Cloth (32.59% resin)	Parallel	298	169 316	89.503	133.264	9 861	54 358 / 82 625	3.27	5 495 000	5.194 900
		197	186 190	106 620	167 701	12 440	56 608 / 103 183	3.51	5 565 830	5 108 333
		77	232 385	122 448	207 525	15 966	81 158 / 138 780	4.48	5 858 330	5 405 000
		20	220 683	128.171	217 897	13 848	73 500 / 132 667	4.09	5 997 857	5.561 000
	Normal	298	28 613	35 753	43 665	*	36 567 / 83 917	3.71	2 144 670	2 060 900
		197	35 008	49 318	55 480	*	37 333 / 98 200	4.98	2 463 330	2 212 000
		77	42 190	69 359	76 035	*	61 750 / 104 200	5.58	3 237 330	2 845 000
		20	37.554	65.480	74.688	*	61 016 / 104 200	5.68	3 287 330	2 992 000
	45 degrees	298	19.937	31 245	44 824	*	42 200 / 90 017	5.48	1 894 670	1 876.700
		197	23.607	45 471	61 222	*	47 416 / 109 367	4.47	2 154 000	2 122 330
		77	27 861	66 456	75 756	*	67 900 / 114 900	1.11	3 011 330	2.815 330
		20	27 490	68 476	81 668	*	64 683 / 118 000	1.21	3 531 330	3 250 000

*Failure occurred in flexure instead of shear

TABLE II

NUCLEAR RADIATION EFFECT ON MECHANICAL PROPERTIES* AT -423°F

| Material | Control Test at -423°F | | | | Cryogenic Test | | | | | | | | | | | |
| | | | | | First Irradiation Test at -423°F Average Dose: 4×10^6 ergs/g(C) | | | | Second Irradiation Test at -423°F Average Dose: 5×10^8 ergs/g(C) | | | | Third Irradiation Test; Irradiated at -423°F, Tested at Room Temperature Average Dose: 5×10^8 ergs/g(C) | | | |
	Ultimate Strength, psi	Standard Deviation	Elongation, percent	Standard Deviation	Ultimate Strength, psi	Standard Deviation	Elongation, percent	Standard Deviation	Ultimate Strength, psi	Standard Deviation	Elongation, percent	Standard Deviation	Ultimate Strength, psi	Standard Deviation	Elongation, percent	Standard Deviation
Phenolic Asbestos	13 470	999	6.5	0.6	14 182	2381	2.0	0.14	12 444	903	1.0	0.14	8 921	1770	1.5	0.3
Phenolic Linen	16 598	1162	6.3	0.3	16 342	538	1.8	0.07	16 102	364	1.5	0.07	11 550	569	4.1	1.24
Phenolic Glass	42 099	1653	3.7	1.3	42 752	3663	4.2	0.78	40 579	2735.	0.4	0.001	24 816	570	0.8	0.04

*Average data for five specimens.

TABLE III

UNIDIRECTIONAL FATIGUE TESTS ON FLAT SPECIMEN

Sample Structure	Maximum Flexural Stress, psi	Maximum Deflection, inch	Fatigue Test Temperature, $^{\circ}$K	Number of Cycles	Deviation From Original Value
HTS--Filament structure (no film barrier)	± 3000	± 0.024	77.8	5×10^6	None
HTS--Filament epoxy structure with H-film barriers	± 3000	± 0.024	77.8	10×10^6	None
HTS--Filament epoxy structure with H-film barriers	± 6000	± 0.058	77.8	16.7×10^6	None
HTS--Filament epoxy structure with H-film barriers	± 9000	± 0.089	77.8	4×10^6	None
HTS--Glass cloth-epoxy structure (no film barrier)	± 3000	± 0.024	77.8	6.5×10^6	None

TABLE IV

TYPES OF BEARINGS TESTED

Bearing No.	Bearing Type	Ball and Race Material	Separator Type	Separator Material	Internal Clearance $x\ 10^{-4}$ in.
1	Radial*	52100	Riding inner ring	Phenolic	5.0 to 9.8
2	Radial*	440C	Riding inner ring	Phenolic	5.5 to 7.5
3	Radial*	440C	Riding balls	Phenolic	4.3 to 5.3
4	Radial**	440C	Riding outer ring	Phenolic	5.3 to 12.5
5	Radial**	440C	Two-piece, aluminum armored, riding outer ring	Phenolic	5.6 to 7.0
6	Radial**	440C	Two-piece pressed	Steel, PTFE Coated	8 to 18
7	Radial*	440C	None	Alternate undersized 440C balls	4.8 to 5.5
8	Radial**	440C	Riding outer ring	Filled PTFE	4.4 to 5.8
9	Radial**	440C	Riding outer ring	Phenolic	2.5 to 4.5
10	Radial**	440C	Riding outer ring	PTFE coated, Grade A, phosphor bronze	4.6 to 5.5
11	Radial**	440C	Riding outer ring	Grade A phosphor bronze	4.1 to 5.5
12	Angular Contact**	52100	Riding inner ring	Phenolic	2.5 to 4.5

*Full shoulder on both sides of inner ring and a counterbore on one side of outer ring.
**Full shoulder on both sides of outer ring and a low shoulder on one side of inner ring.

TABLE V

MECHANICAL AND THERMAL PROPERTIES OF FLUOROCARBON RESINS OF VARYING CRYSTALLINITIES

Mechanical Property	Temperature, °F	Kel-F Crystallinity, percent				FEP Teflon Crystallinity, percent			TFE Teflon Crystallinity, percent				
		40	55	60	70	44	49	51	47	49	52.5	66.2	71
Notched Izod Impact Strength, ft-lb/in.	-423	—	—	1.4	1.2	—	1.95	1.7	—	—	1.25	—	1.55
	-320	—	—	1.3	1.1	—	1.9	1.5	—	—	1.17	—	1.35
	(-297)	—	—	(1.25)	(1.07)	—	(1.9)	(1.5)	—	—	(1.15)	—	(1.39)
Modulus of Rigidity, psi x 10^3	-423	262	222	—	230	935	—	695	267	—	275	377	—
	-320	232	197	—	203	305	—	305	224	—	190	200	—
	(-297)	(229)	(192)	—	(195)	(275)	—	(275)	(216)	—	(175)	(175)	—
Tensile Yield Strength, psi x 10^3	-423	28.5	20.5	—	18.0	23.8	23.5	—	—	20.0	19.0	15.6	—
	-320	24.9	17.5	—	15.3	19.1	18.8	—	—	16.0	15.4	10.6	—
	(-297)	(24.4)	(17.0)	—	(15.0)	(17.9)	(17.6)	—	—	(14.2)	(14.1)	(9.0)	—
Ultimate Tensile Strength, psi x 10^3	-423	29.0	22.5	—	17.95	24.0	23.6	—	—	20.05	19.0	15.7	—
	-320	24.9	17.6	—	15.8	18.1	17.9	—	—	17.9	15.4	11.8	—
	(-297)	(24.1)	(17.5)	—	(15.3)	(17.0)	(16.8)	—	—	16.0	14.2	11.0	—
Tensile Modulus, psi x 10^6	-423	1.26	0.975	—	0.830	0.825	0.620	—	—	0.600	0.645	0.625	—
	-320	1.11	0.802	—	0.760	0.700	0.450	—	—	0.500	0.520	0.435	—
	(-297)	(1.08)	(0.800)	—	(0.750)	(0.652)	(0.430)	—	—	(0.465)	(0.480)	(0.400)	—
Elongation, percent	-423	ca 2	ca 1	—	1	ca 5	ca 5	—	—	ca 4	ca 3	ca 2	—
	-320	ca 4	2.5	—	ca 1	ca 7	ca 7	—	—	ca 9	ca 8	ca 4	—
	(-297)	(ca 4.5)	(3)	—	(ca 1)	(ca 8)	(ca 7)	—	—	(9)	(8)	(ca 5)	—
Flexural Strength, psi x 10^3	-423	74.0	55.5	—	50.5	35.9	39.6	—	—	33.8	25.7	22.8	—
	-320	58.0	42.7	—	37.0	25.7	27.7	—	—	30.0	24.0	21.5	—
	(-297)	(53.5)	(41.0)	—	(35.0)	(24.0)	(25.5)	—	—	(28.0)	(23.0)	(20.5)	—
Flexural Modulus, psi x 10^6	-423	2.09	1.94	—	1.84	0.78	0.75	—	—	0.778	0.72	0.695	—
	-320	1.83	1.70	—	1.64	0.68	0.68	—	—	0.720	0.70	0.64	—
	(-297)	(1.70)	(1.64)	—	(1.59)	(0.67)	(0.66)	—	—	(0.690)	(0.64)	(0.61)	—
		50*	55	60	70	44	49	51	47	50*	56*	68*	71
Compressive Strength, psi x 10^3	-423	42.5	—	44.5	28.8	36.3	35.2	—	—	30.4	32.5	33.2	—
	-320	35.0	—	37.5	38.8	30.0	30.0	—	—	20.0	21.2	22.0	—
	(-297)	(34.0)	—	(36.0)	(38.8)	(28.5)	(28.5)	—	—	(18.5)	(20.0)	(21.5)	—
Compressive Modulus, psi x 10^6	-423	1.67	—	1.76	1.48	1.01	1.04	—	—	0.81	0.90	0.97	—
	-320	1.48	—	1.52	1.65	0.91	0.91	—	—	0.75	0.79	0.84	—
	(-297)	(1.27)	—	(1.40)	(1.65)	(0.90)	(0.89)	—	—	(0.72)	(0.76)	(0.81)	—
Coefficient of Thermal Expansion (Total contraction from 70°F) in./in.	-423	0.011	—	—	—	0.017	—	—	—	0.0214	—	—	—
	-320	0.0093	—	—	—	0.0157	—	—	—	0.0205	—	—	—
	(-297)	(0.0090)	—	—	—	(0.0154)	—	—	—	0.0201	—	—	—

The above values were obtained by PLASTEC by reading off data points from the original data curves. The values in parentheses() were obtained by interpolation. No actual data was obtained with liquid-oxygen temperatures in the original study.

Note change in crystallinity, indicated by asterisk (*)

TABLE VI

MEAN VALUES OF TENSILE STRENGTH FOR PCTFE TEST SAMPLES

Sample and Strain Rate, in./min	Mean Value of Maximum Tensile Strength, psi				Mean Value of Tensile Stress at Failure, psi			
	Temperature				Temperature			
	-320°F		75°F		-320°F		75°F	
	Molded Surface	Machined Surface	Molded Surface	Machined Surface	Molded Surface	Machined Surface	Molded Surface	Machined Surface
40-Percent Crystallinity Sample								
0.02					20 939	21 290	4 710	5 165
0.2					24 343	24 155	4 818	5 884
2.0					26 533	24 241	4 208	4 661
20.0					22 924	22 211	4 808	5 572
1 000.0	22 507	22 799	8 548	9 294	22 500	22 800	6 210	6 890
10 000.0	9 313	8 496	13 698	13 296	4 020	4 120	7 830	6 800
55-Percent Crystallinity Sample								
0.02					19 833	19 835	5 264	4 933
0.2					23 119	22 732	5 313	5 684
2.0					24 581	24 174	5 068	5 306
20.0					22 247	21 192	5 400	6 007
1 000.0	22 293	21 417	9 016	9 431	22 290	21 400	6 380	6 820
10 000.0	9 816	8 179	13 618	13 226	4 700	3 440	12 770	13 230

TABLE VII

MEAN VALUES OF PERCENT ELONGATION FOR PCTFE TEST SAMPLES

Sample and Strain Rate, in./min	Mean Value of Percent Elongation at Maximum Tensile Strength				Mean Value of Percent Elongation at Failure			
	Temperature				Temperature			
	-320°F		75°F		-320°F		75°F	
	Molded Surface	Machined Surface	Molded Surface	Machined Surface	Molded Surface	Machined Surface	Molded Surface	Machined Surface
40-Percent Crystallinity Sample								
0.02					6.2	6.9	125.0	141.0
0.2					5.8	5.2	120.0	137.0
2.0					9.3	8.4	130.0	152.0
20.0					5.0	3.9	70.0	88.0
1 000.0	6.45	6.53	13.30	14.00	6.45	6.53	71.50	58.75
10 000.0	5.32	4.65	14.35	12.67	11.55*	11.50*	47.00*	37.50*
55-Percent Crystallinity Sample								
0.02					7.0	7.7	180.0	139.0
0.2					4.6	4.5	189.0	90.0
2.0					9.2	8.8	214.0	92.0
20.0					6.5	3.8	59.0	53.0
1 000.0	6.90	6.62	15.35	14.70	6.90	6.62	63.30	42.75
10 000.0	5.30	4.30	11.13	11.00	12.25*	10.27*	23.30*	24.00*

*Failure appeared to be a two-step process

TABLE VIII

POLYMER LINER BEHAVIOR AT 20°K

Mechanical Property	Tedlar BG-30-WH	H-Film	Mylar A	Polyurethane Seilon UR29E
Contraction of Liner, 10^{-3} in./in.	9.16	5.26	3.86	16.31
Chilldown Differential, percent (difference in contraction between liner and fiberglass cylinder)	0.73	0.34	0.20	1.44
Average Ultimate Elongation of Liner, percent	2.16	1.69	0.82	2.20
Residual Elongation of Liner, percent	1.43	1.3	0.62	0.76
Predicted Liner Failure, psi percent of cylinder ultimate	786 48	742 45	345 21	418 25
Actual Liner Failure, psi percent of cylinder ultimate	184 11	355 to 388 21 to 24	360 22	Not tested

TABLE IX

AVERAGE PERMEATION (ρ) RATES (std cm^3-cm/cm^2-sec-cm Hg)

Material	No. of Samples	Room Temperature*	Liquid Nitrogen Temperature*	Liquid Hydrogen Temperature*
H-Film, 1 mil thick	3	$\rho < 1.2 \times 10^{-10}$	$\rho < 1.8 \times 10^{-13}$	$\rho < 1.8 \times 10^{-13}$
Mylar Film, 1 mil thick	3	$\rho < 3.9 \times 10^{-11}$	$\rho < 1.1 \times 10^{-13}$	$\rho < 1.1 \times 10^{-13}$
Aluminized Mylar, 1 mil thick	3	$\rho < 1.6 \times 10^{-12}$	$\rho < 1.3 \times 10^{-13}$	$\rho < 1.3 \times 10^{-13}$
181EE-Glass Cloth, 828/Z, 43.9% Resin, 0.011 in. thick	4	$\rho < 1.7 \times 10^{-12}$	$\rho < 1.9 \times 10^{-12}$	$\rho < 1.5 \times 10^{-12}$
181 Quartz Cloth, 828/Z, 43.1% Resin, 0.012 in. thick	3	$\rho < 1.6 \times 10^{-12}$	$\rho < 1.6 \times 10^{-12}$	$\rho < 1.5 \times 10^{-12}$
S-Glass Cloth, 828/Z, 46.7% Resin, 0.012 in. thick	3	$\rho < 7 \times 10^{-13}$	$\rho < 6.1 \times 10^{-13}$	$\rho < 6 \times 10^{-13}$

* < Denotes that the permeability was at or below the least readable count of the output meter of the mass spectrometer detector, and hence lies at or below the noted permeability values.

TABLE X

THIN FILMS*

Material Type	Test Temperature	Modulus, x 10-7 psi	Ultimate Stress, x 10³ psi	Maximum Strain, $\Delta L/L$	Average Thickness, x 10³ in.
Plain Mylar (M–Series)	Room	0.80 (3.8%)	17.5 (21%)	0.160 (8.8%)	0.70 ±0.05
	LN₂	1.47 (4.1%)	43.5 (8%)	0.038 (10%)	0.50 ±0.05
	LH₂	1.40 (12%)	34.3 (6.7%)	0.030 (23%)	0.60 ±0.05
Aluminized Mylar (R–Series)	Room	0.86 (4.7%)	23.6 (27%)	0.140 (29%)	0.45 ±0.05
	LN₂	1.60 (2.5%)	38.7 (8.0%)	0.034 (17.7%)	0.50 ±0.05
	LH₂	1.57 (5.7%)	29.1 (8.6%)	0.020 (15%)	0.50 ±0.05
H–Film (H–Series)	Room	0.48 (7.1%)	16.5 (20%)	0.110 (19%)	0.90 ±0.05
	LN₂	1.09 (4.6%)	30.7 (3.3%)	0.033 (3.0%)	0.80 ±0.05
	LH₂	0.96 (4.4%)	27.3 (16%)	0.033 (27%)	0.90 ±0.05

*Percentage values are the maximum deviation from average of three tests.

TABLE XI

EPOXY IMPREGNATED QUARTZ CLOTH PROPERTIES IN THREE DIRECTIONS

(1 Ply # 181)*

Direction of Pull	Test Temperature	Initial Modulus, x 10-6 psi	Ultimate Stress, x 10³ psi	Maximum Strain, $\Delta L/L$	Secondary Modulus, x 10-6 psi	Transition Stress, x 10-3 psi
Warp (A–Series)	Room	2.71 (3.3%)	44.6 (3.6%)	0.037 (3.0%)	2.06 (6.8%)	13.0 (13%)
	LN₂	2.78 (8.6%)	73.3 (2.3%)	0.040 (10%)	1.85 (1.6%)	11.2 (15.2%)
	LH₂	3.87 (14.5%)	71.4 (8.0%)	0.034 (8.8%)	1.97 (6.6%)	11.1 (12.6%)
Woof (C–Series)	Room	2.52 (4.4%)	45.5 (9.0%)	0.023 (8.7%)	1.89 (4.8%)	9.7 (1.0%)
	LN₂	2.82 (2.1%)	57.2 (4.5%)	0.038 (10.5%)	1.48 (5.4%)	6.3 (17.5%)
	LH₂	2.99 (6.4%)	68.5 (16.8%)	0.041 (5.0%)	1.59 (12.6%)	6.6 (16.7%)
45 Degrees to Warp (B–Series)	Room	1.62 (14.0%)	15.0 (4.7%)	0.029 (15%)	----	----
	LN₂	2.51 (9.2%)	21.2 (30%)	0.019 (88%)	----	----
	LH₂	2.88 (11.8%)	19.2 (2.1%)	0.014 (28.6%)	----	----

*Percentage values are the maximum deviation from average of three tests.

TABLE XII

EPOXY–IMPREGNATED CLOTHS

(1 Ply # 181)*

Material Type	Test Temperature	Initial Modulus, x 10-6 psi	Ultimate Stress, x 10-3 psi	Maximum Strain, $\Delta L/L$	Secondary Modulus, x 10-6 psi	Transition Stress, x 10-3 psi
Fiberglass (E–Series)	Room	3.14 (6.4%)	31.4 (7.0%)	0.013 (17.0%)	2.10 (11.0%)	10.1 (21%)
	LN₂	4.25 (3.5%)	51.0 (1.6%)	0.024 (8.3%)	1.78 (9.5%)	11.3 (7.1%)
	LH₂	5.02 (11.6%)	52.4 (4.6%)	0.023 (8.7%)	1.77 (13.0%)	8.0 (10%)
S–Glass (D–Series)	Room	2.83 (4.2%)	58.9 (12.0%)	0.12* (8.5%)	2.25 (9.3%)	17.7 (13%)
	LN₂	3.92 (8.2%)	92.0 (1.4%)	0.045 (8.9%)	1.97 (6.6%)	16.0 (13.1%)
	LH₂	3.60 (4.7%)	85.6 (1.9%)	0.052 (77.7%)	1.73 (6.4%)	12.6 (12.7%)
Quartz (A–Series)	Room	2.71 (3.3%)	44.6 (3.6%)	0.037 (3.0%)	2.06 (6.8%)	13.0 (13.0%)
	LN₂	2.78 (8.6%)	73.3 (2.3%)	0.040 (10.0%)	1.85 (1.6%)	11.2 (15.2%)
	LH₂	3.87 (14.5%)	71.4 (8.0%)	0.034 (8.0%)	1.97 (8.8%)	11.1 (12.6%)

*Averages of three tests along with maximum deviation from average expressed as percent.
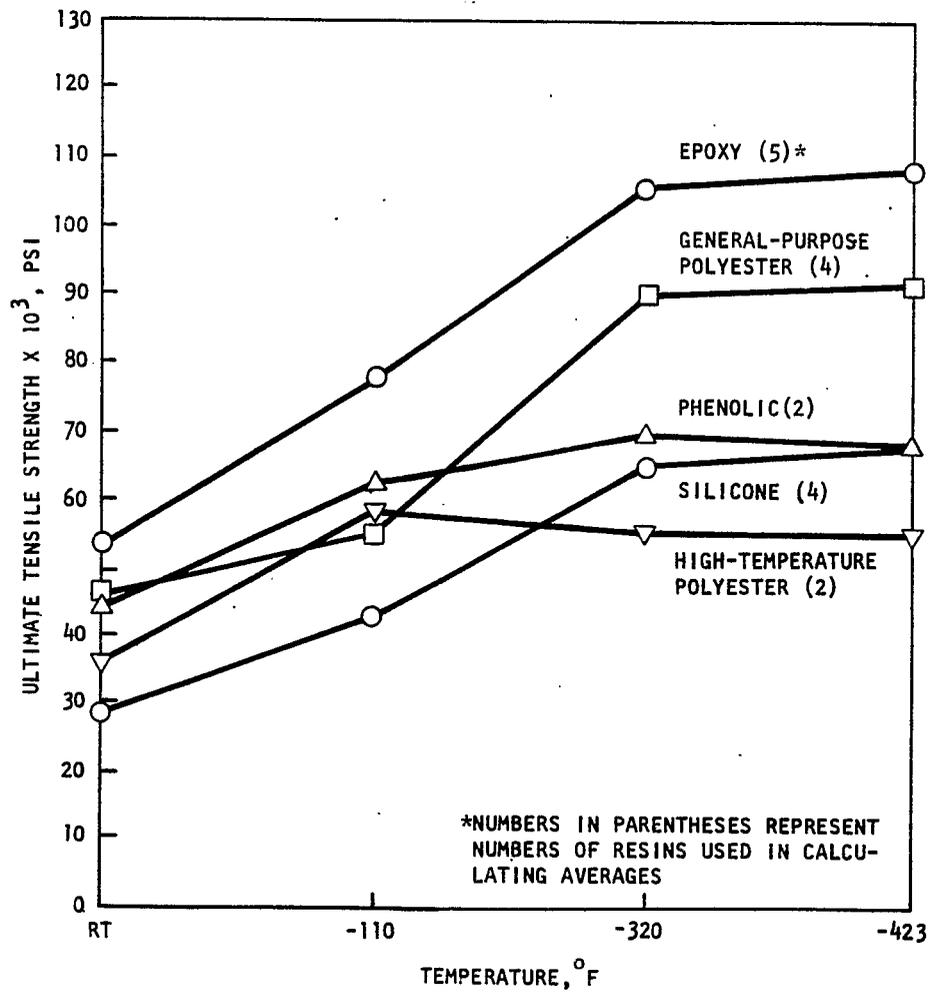
Fig. 1.   Average tensile strength of glass-reinforced plastic
          laminates laminated with 181 glass cloth.

ULTIMATE TENSILE STRENGTH, PSI X 10³ (WIDE BARS)
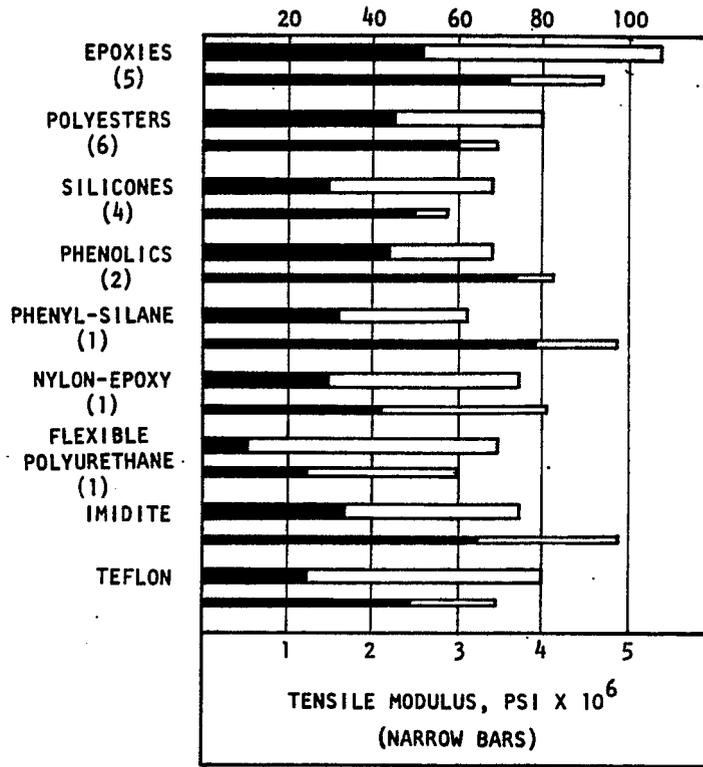
Fig. 2.   Average ultimate tensile strength and modulus of laminates
at room temperature (shaded bars) and at 20°K.

ULTIMATE COMPRESSIVE STRENGTH, PSI X 10³ (WIDE BARS)
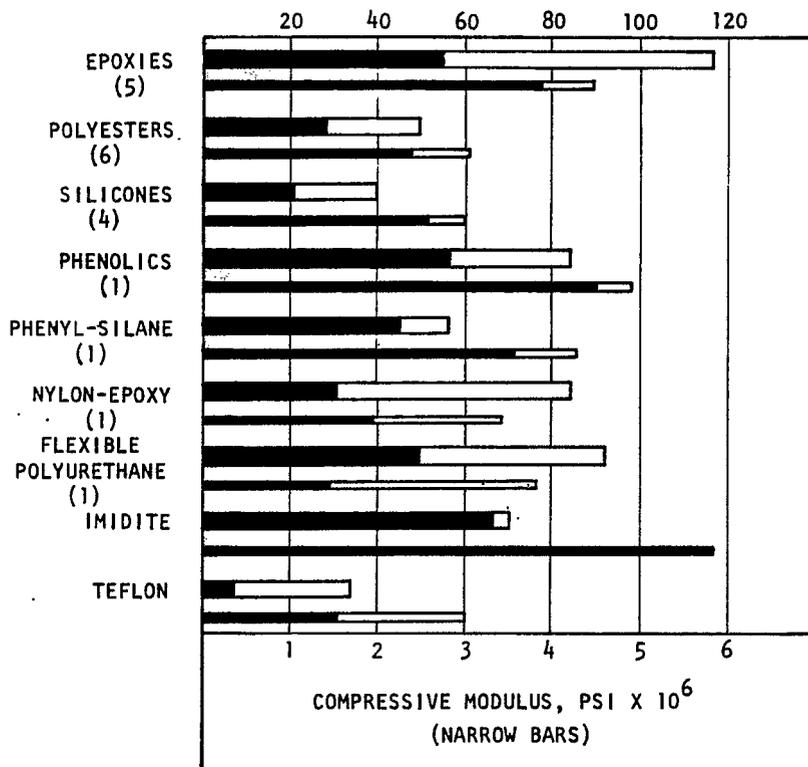
Fig. 3.   Average ultimate compressive strength and modulus at
room temperature (shaded bars) and at 20°K.
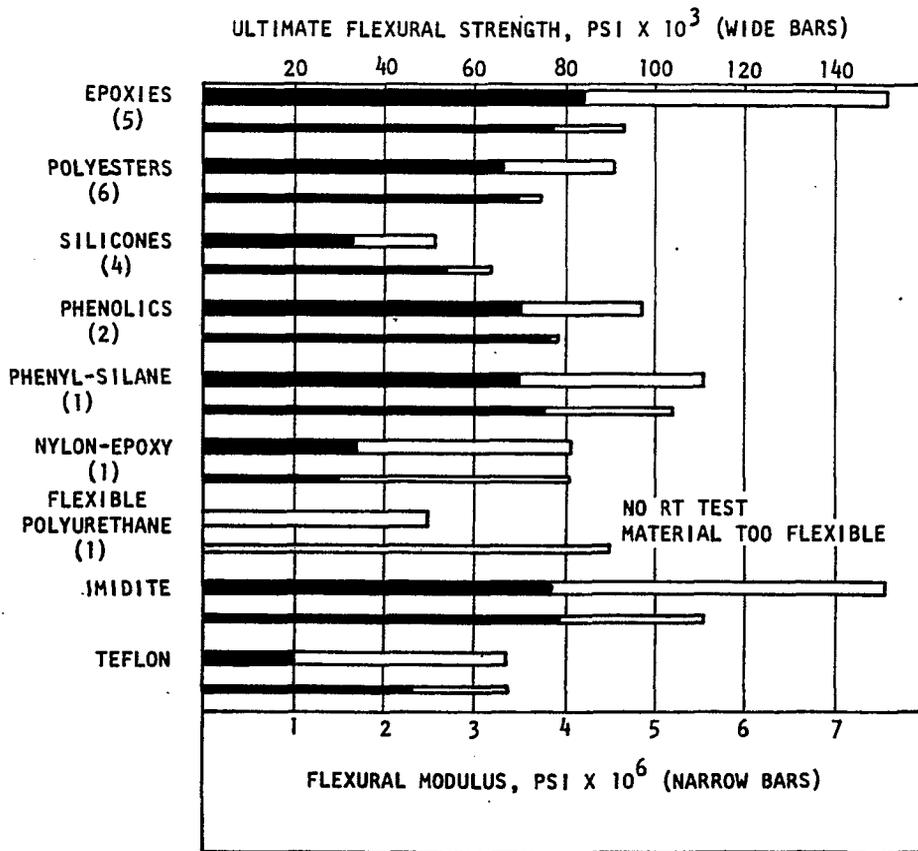
Fig. 4. Average flexural strength and modulus at room temperature (shaded bars) and at 20°K.

AVERAGE BEARING STRENGTH, PSI X 10^3
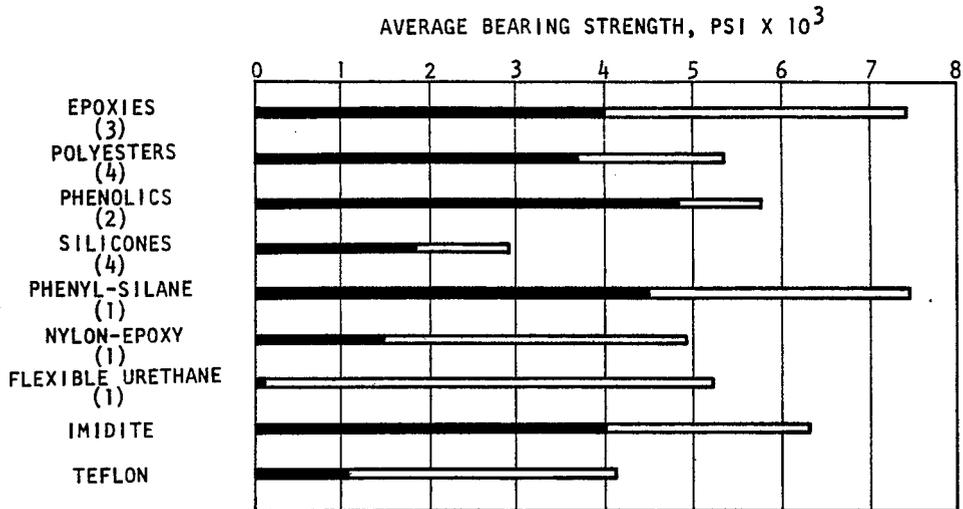
Fig. 5.   Average bearing strength of 1/8 in. thick laminates on a
3/16 in. diameter pin at room temperature (shaded bars)
and at 77°K.
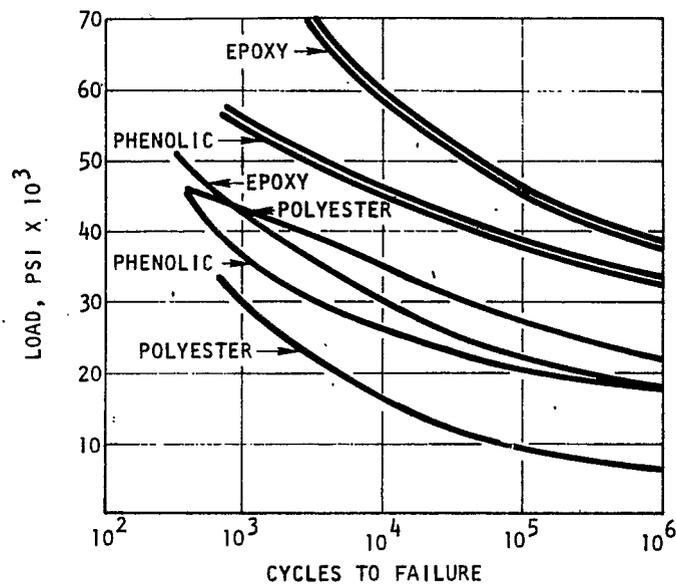
Fig. 6.   Composite S-N curves derived from average
fatigue strength values at room temperature
(lower in each case) and at 20°K.
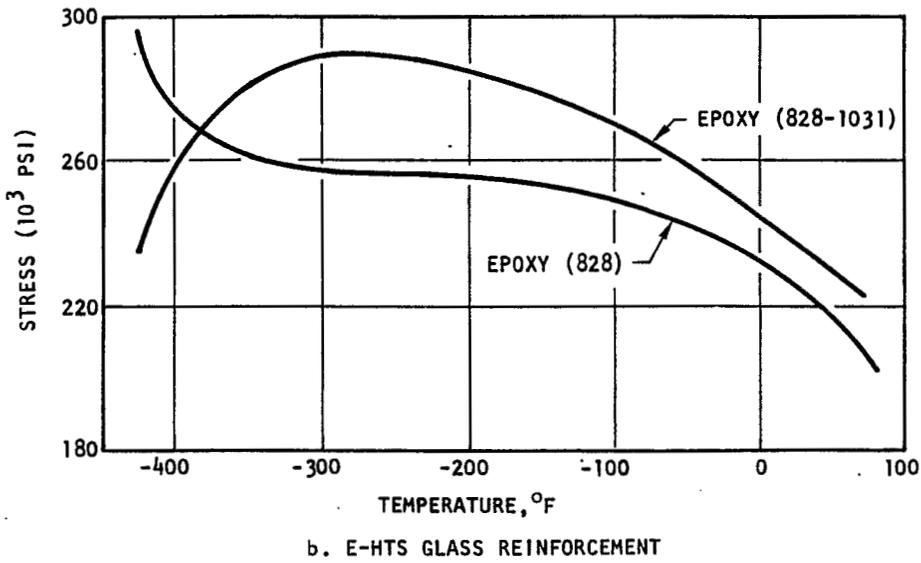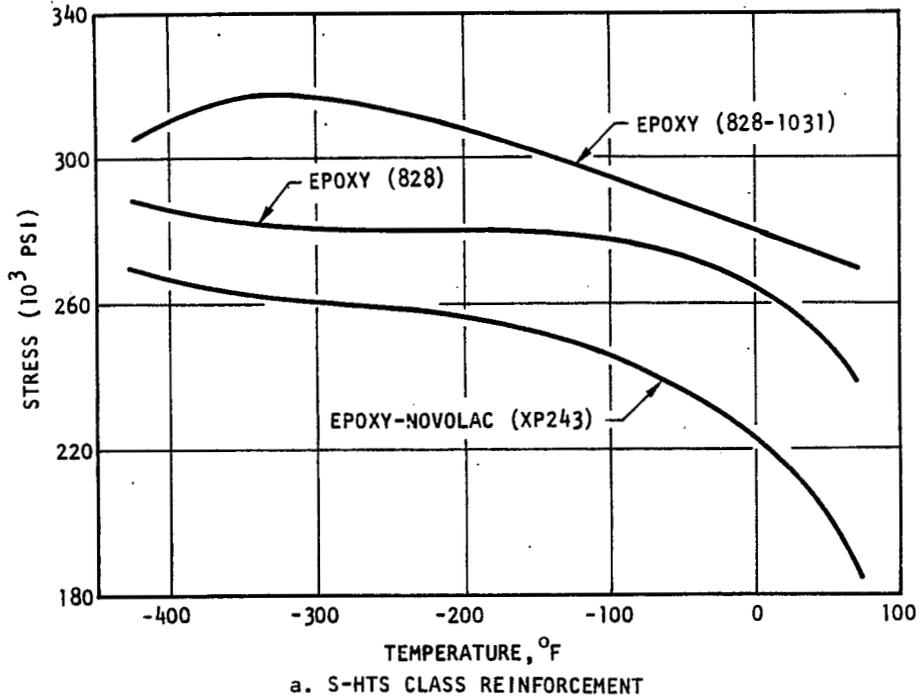
Fig. 7.   Strength of epoxy filament-wound rings at cryogenic temperatures.
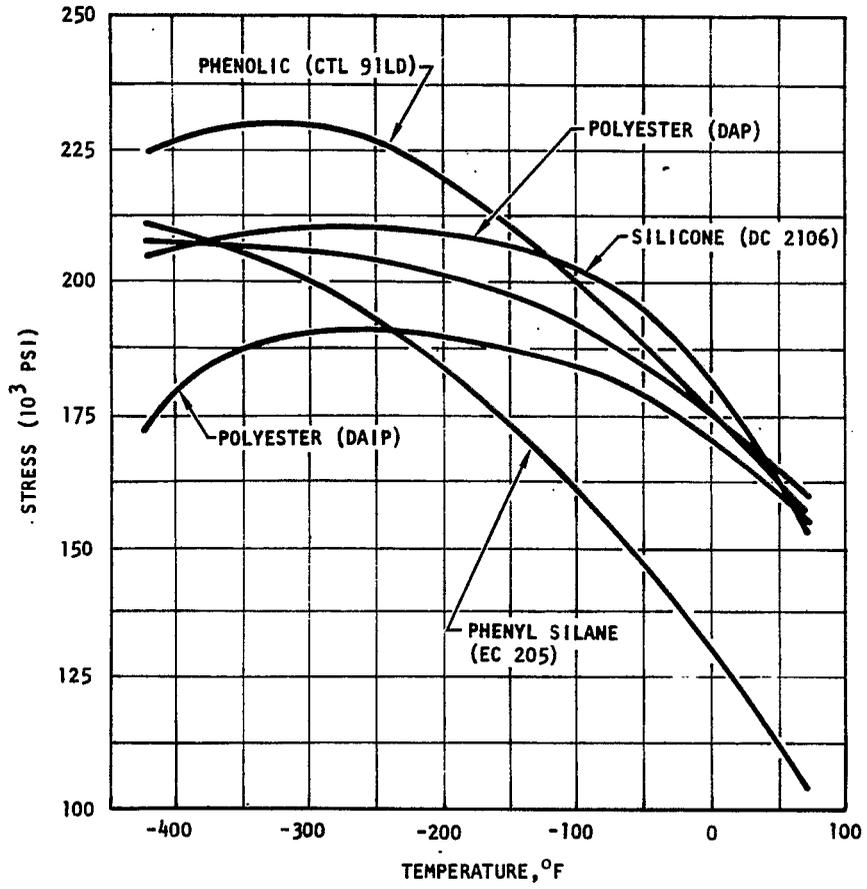
Fig. 8.   Strength of filament-wound rings at cryogenic temperatures.
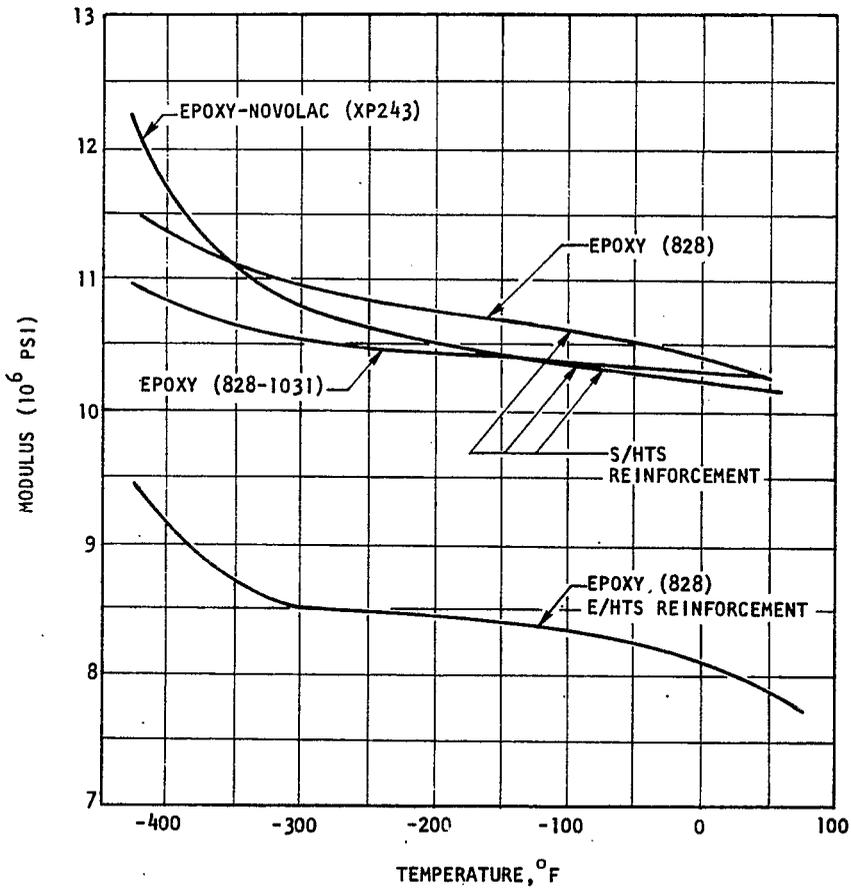
Fig. 9.   Modulus of elasticity of epoxy filament-wound rings at cryogenic temperatures.
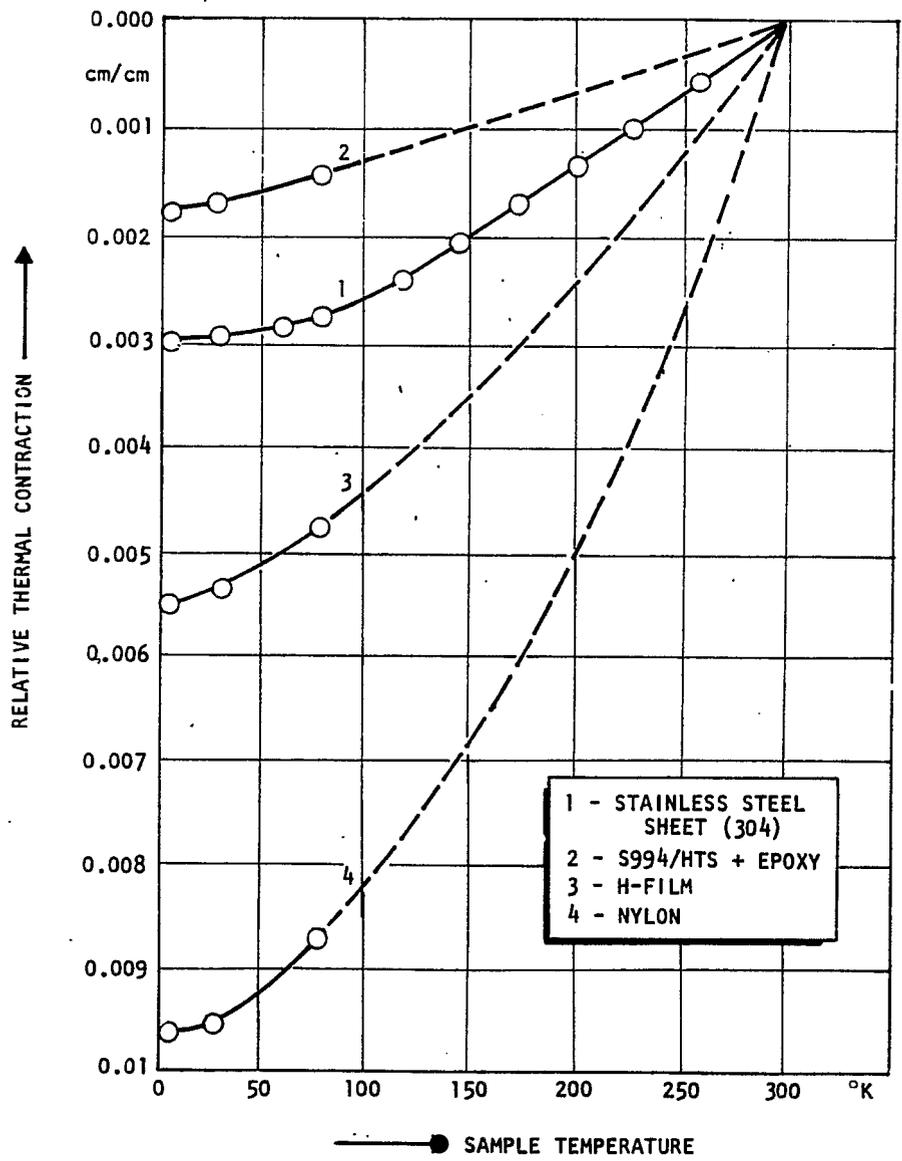
Fig. 10. Linear thermal contraction vs bath temperature.

Fig. 11. Tensile shear strength at -320°F of existing adhesive types with metal-to-metal adherends.
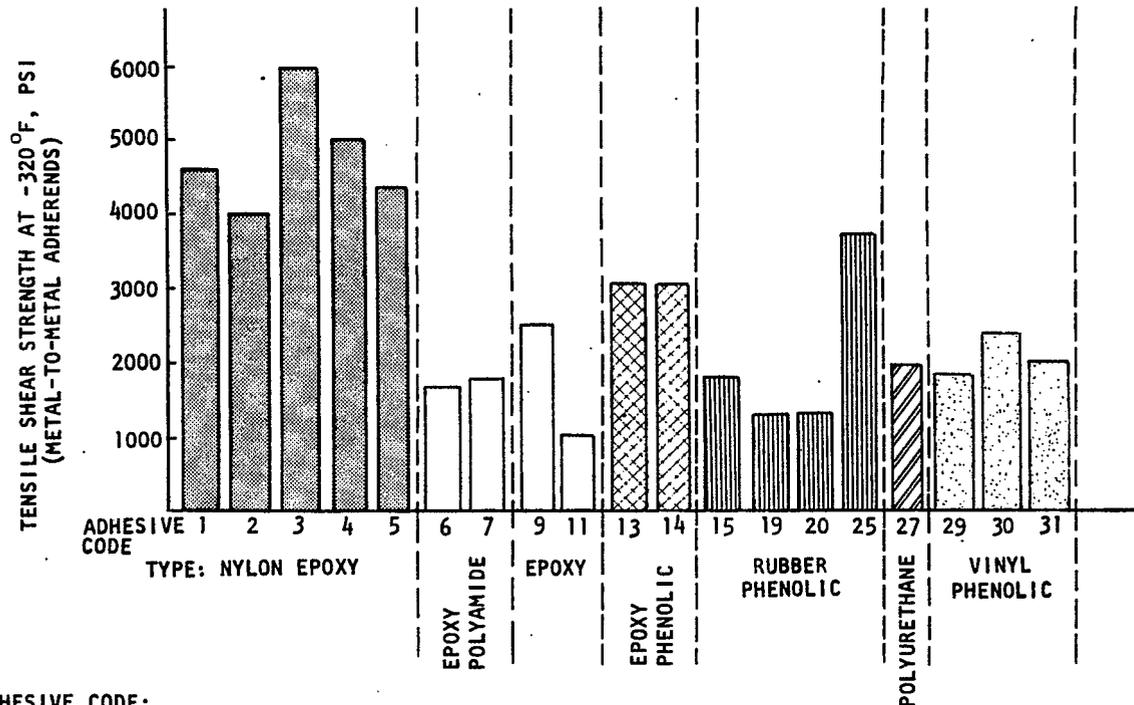
ADHESIVE CODE:

1. METLBOND 406	6. RESIN 3135 AND CURING AGENT	13. METLBOND 302	25. METLBOND 303(MN3C)
2. METLBOND 408	7. EC/1933B/A	14. EPON 422J	27. APCO-1261
3. AF-40	9. EC-1469	15. METLBOND 4021	29. SWEDLOW 371W
4. AF-41	11. ALUMINA-FILLED EPOXY	19. AF-5930	30. FM-47
5. FM-1000		20. BONDMASTER M24B	31. REDUX 775
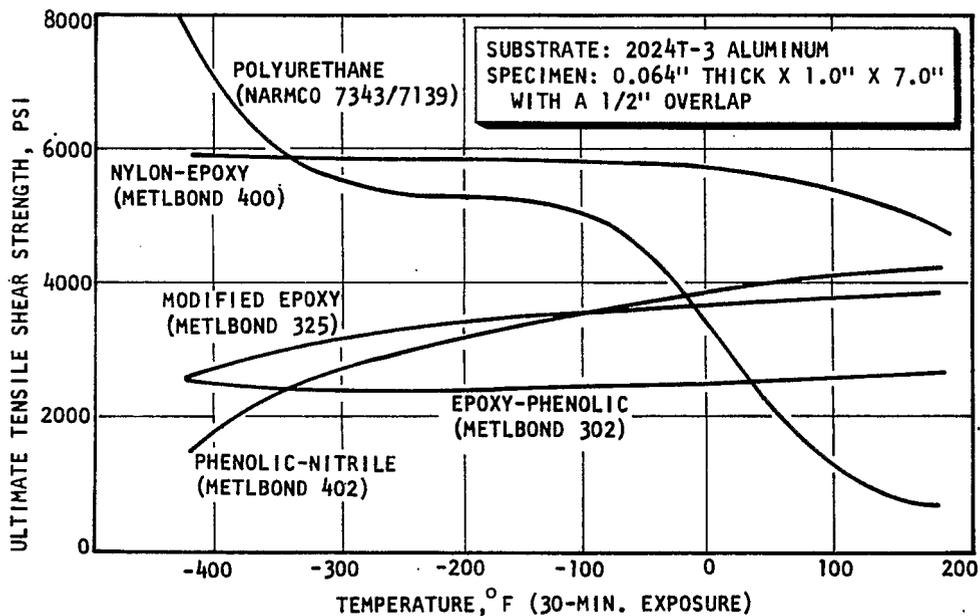
Fig. 12. Effect of temperature on ultimate tensile shear stress of different classes of structural adhesives.

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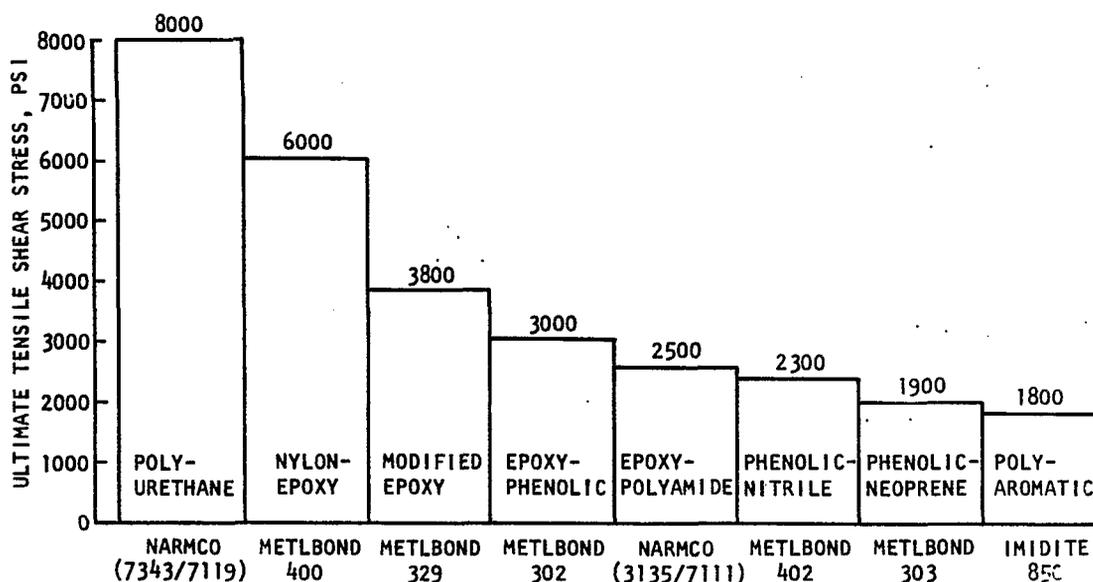
Fig. 13. Effect of liquid hydrogen temperature (-423°F) on the tensile shear strength of several different classes of structural adhesives.
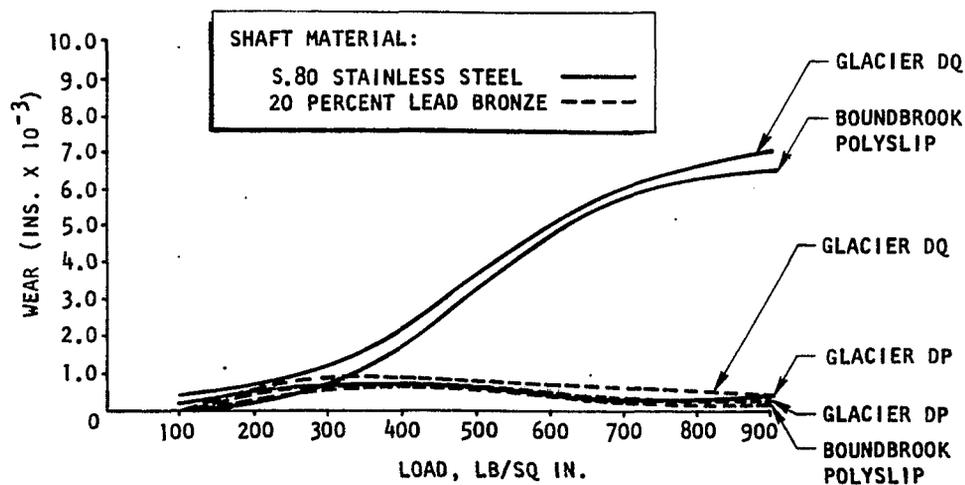
Fig. 14. PTFE-containing bearings in liquid oxygen.
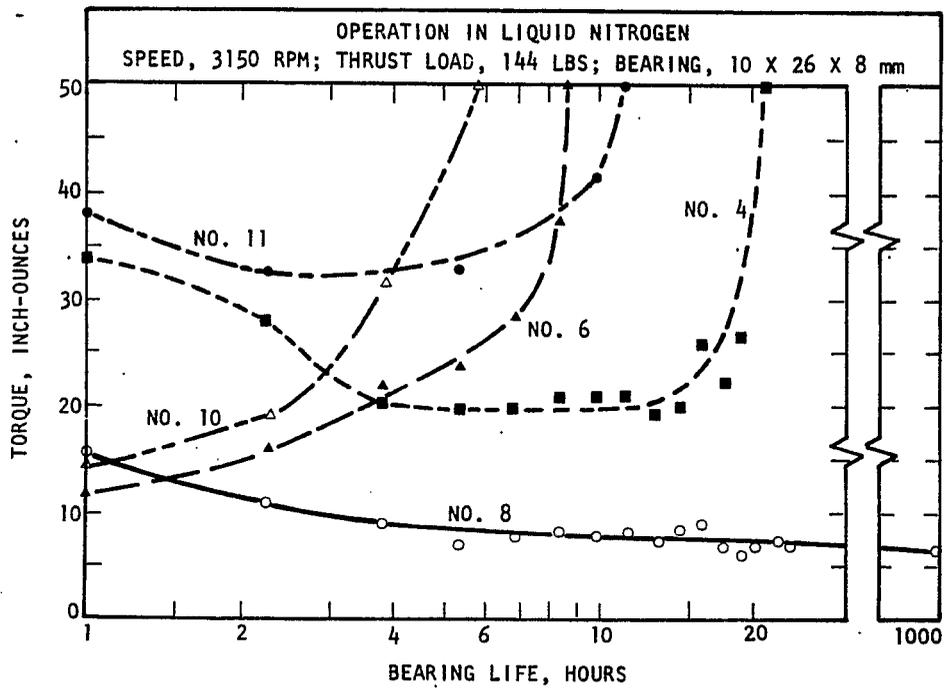
Fig. 15. Bearing torque history.
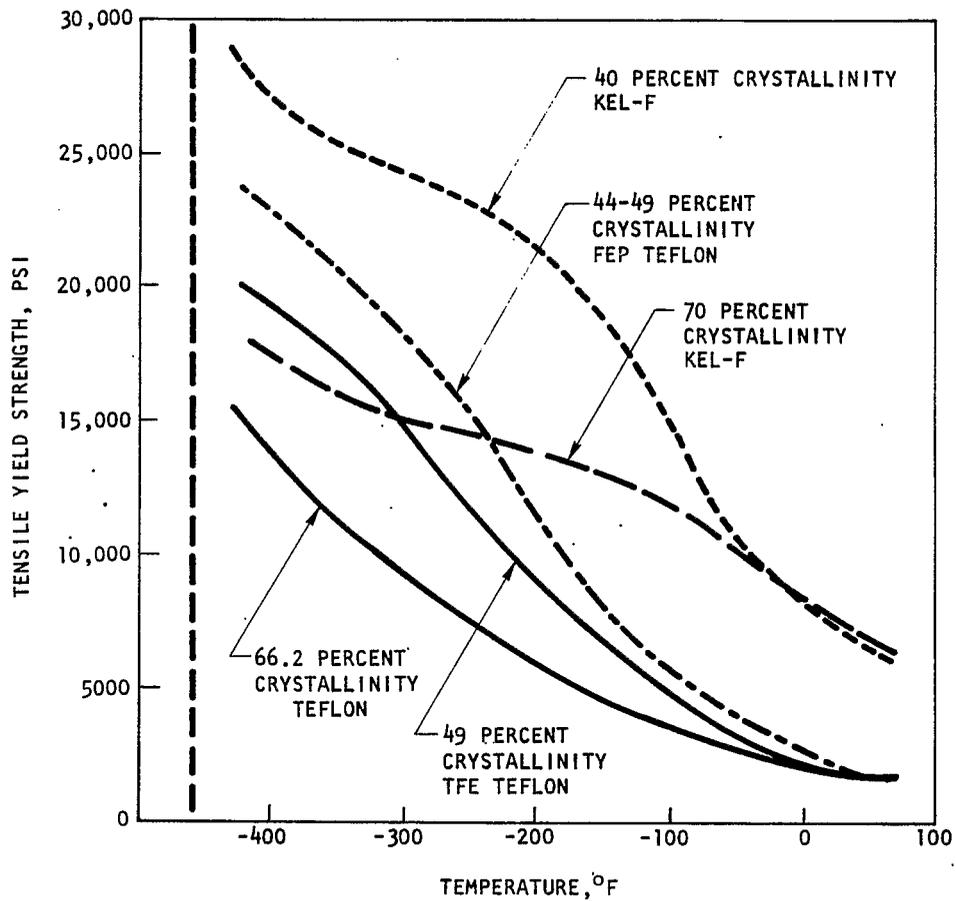
Fig. 16. Effect of temperature on the tensile yield strength of
fluorocarbon plastics of different crystallinities.

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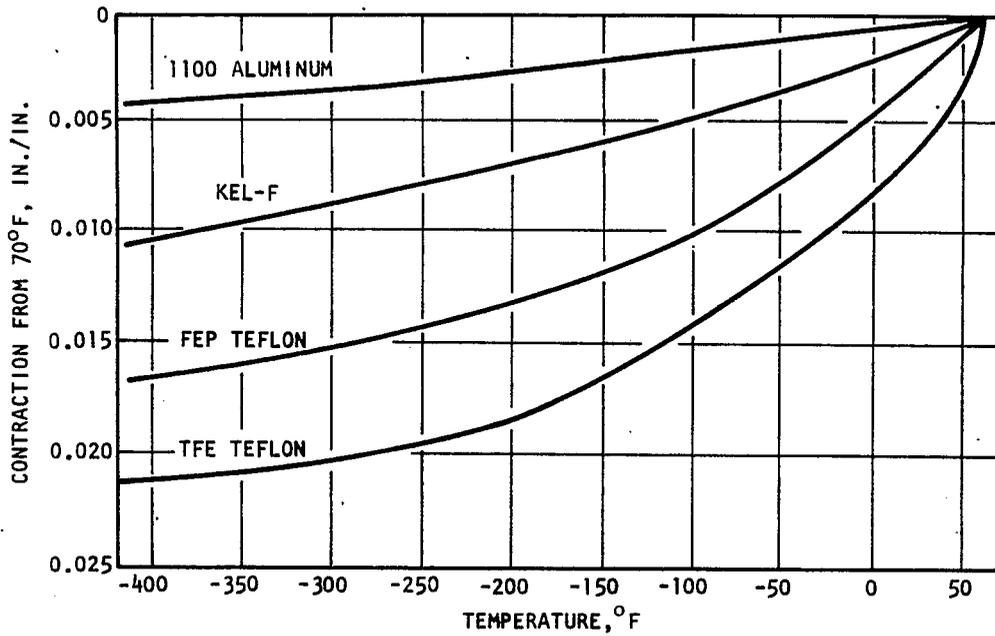
Fig. 17. Total contraction of Kel-F, FEP Teflon, TFE Teflon
and aluminum from 70 to -423°F.
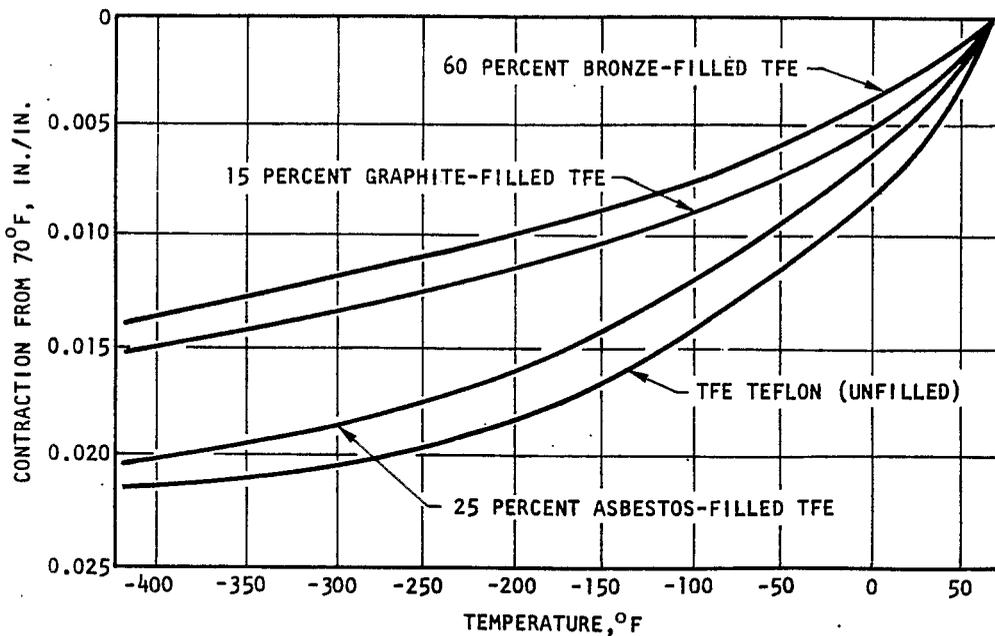
Fig. 18. Total contraction of powder-filled and unfilled
TFE Teflons from 70 to -423°F.
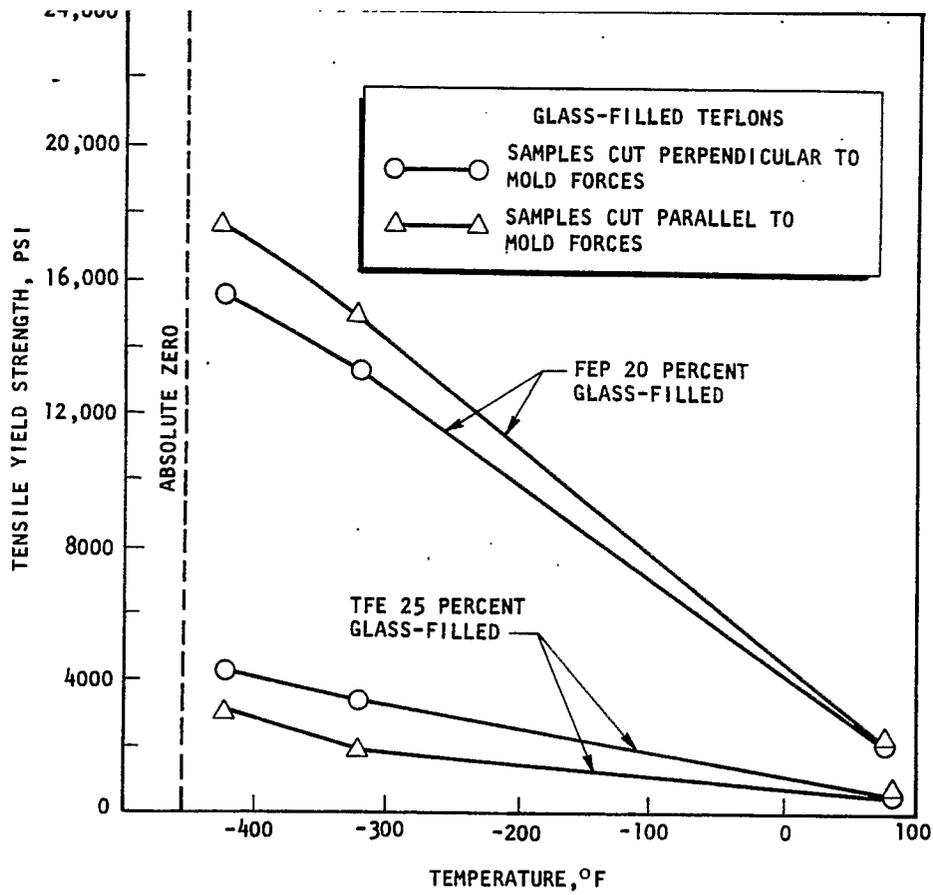
Fig. 19. Effect of temperature on the tensile yield strength
of glass-fiber-filled Teflons.
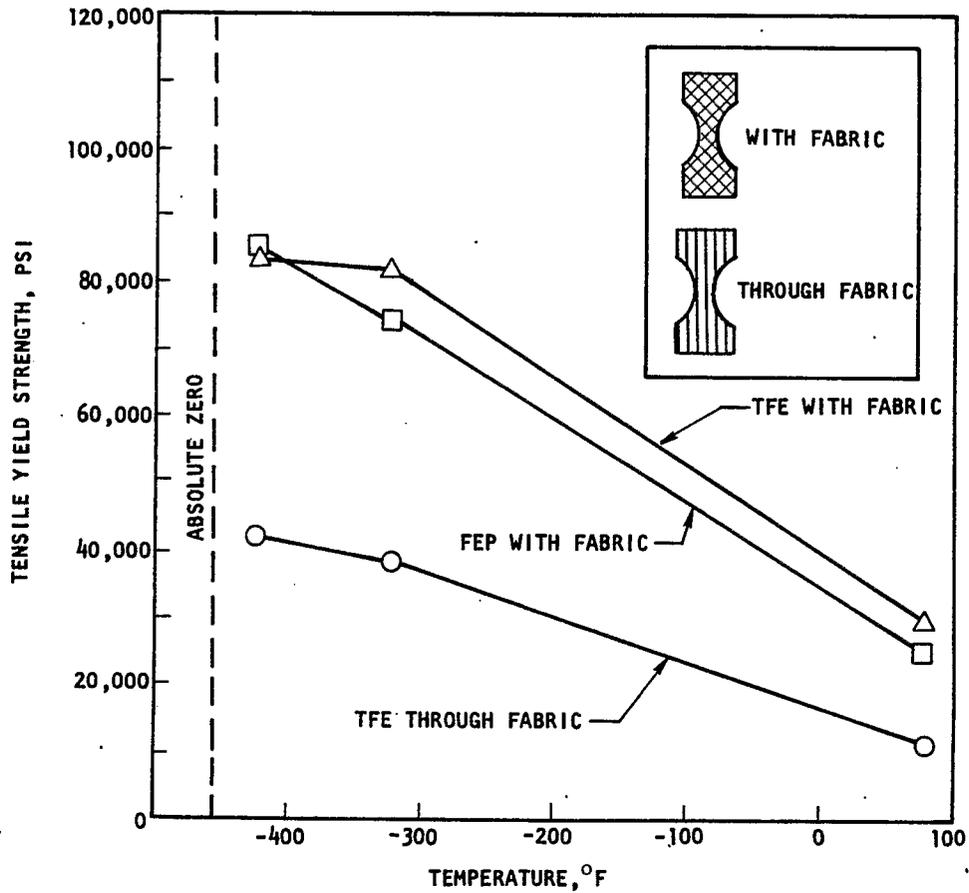
Fig. 20. Effect of temperature on the tensile yield strength of
glass-fabric-reinforced Teflons.
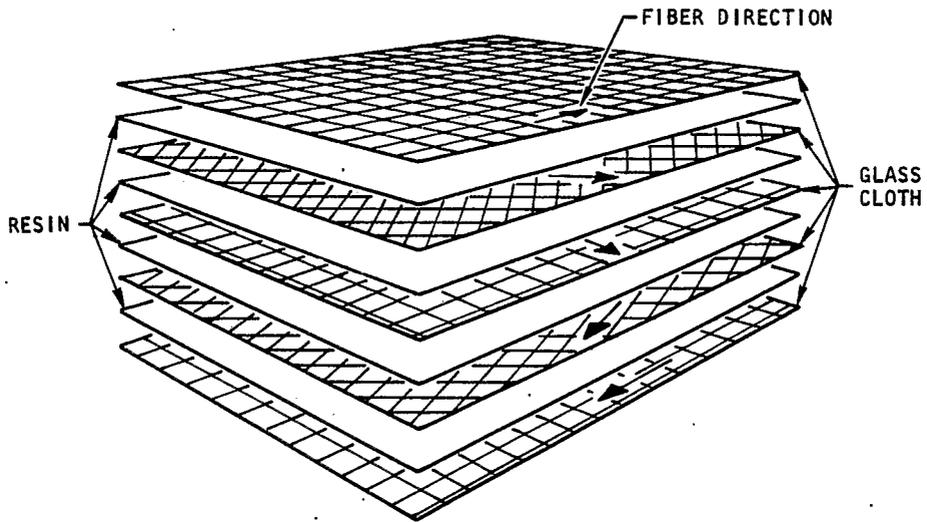
Fig. 21. Laminate lay-up.
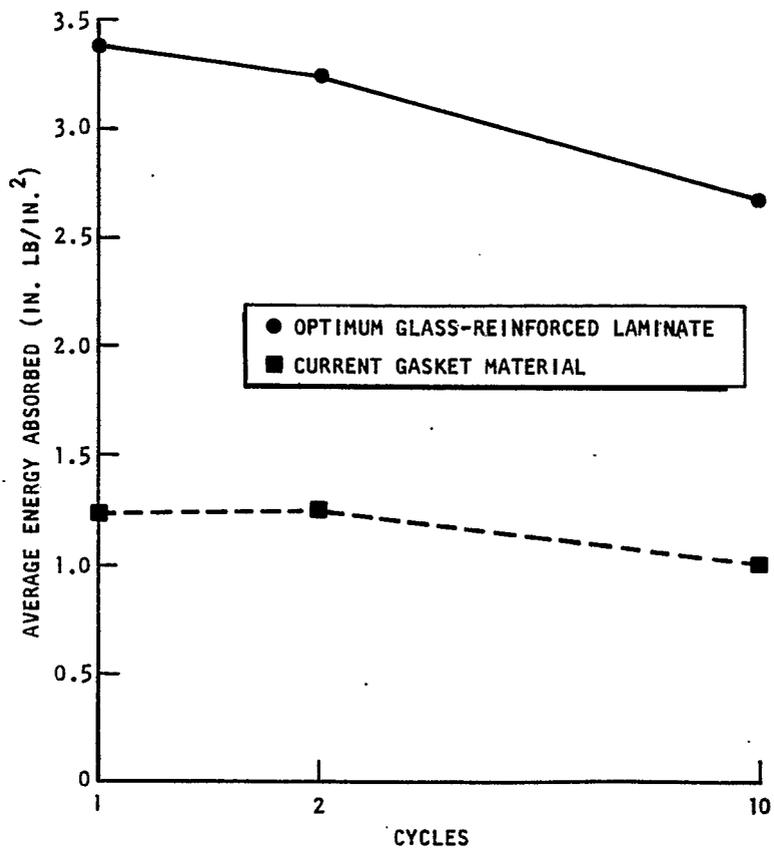
Fig. 22. Energy absorption of glass-reinforced laminate
          and current gasket material.
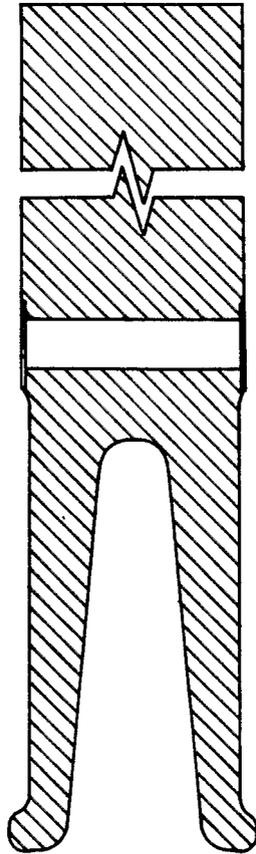
Fig. 23.   Cross section of Naflex seal.
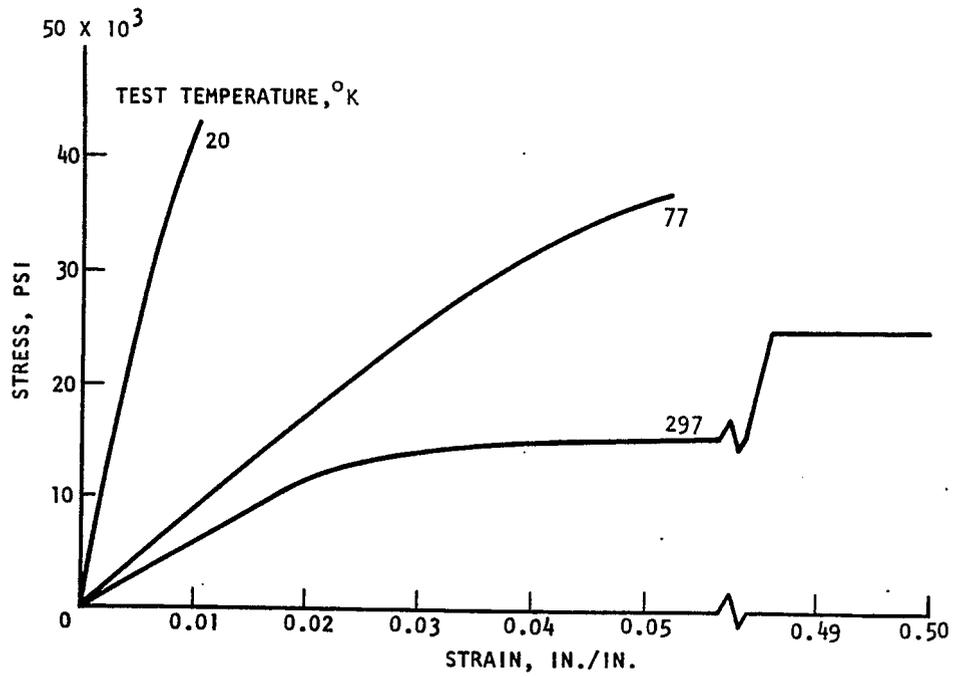
Fig. 24.   Temperature-strength behavior — Mylar A film.
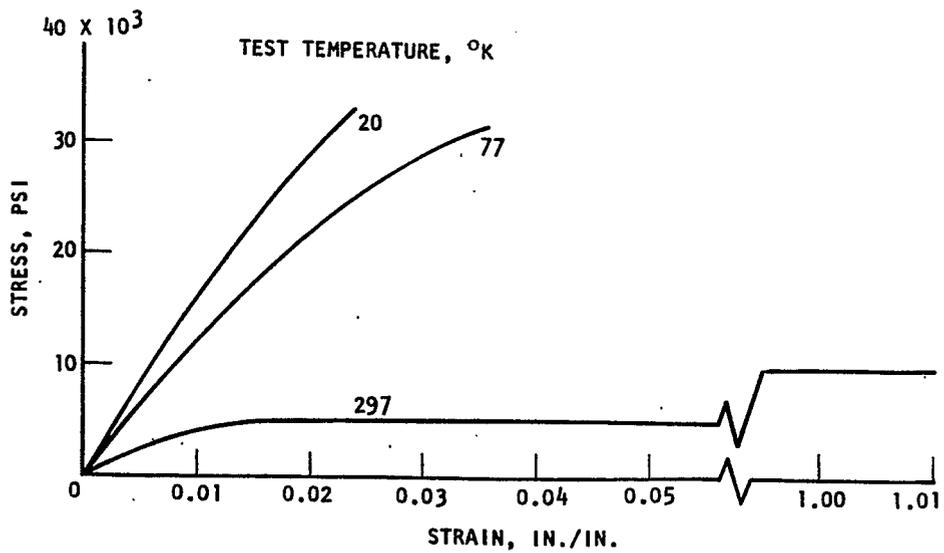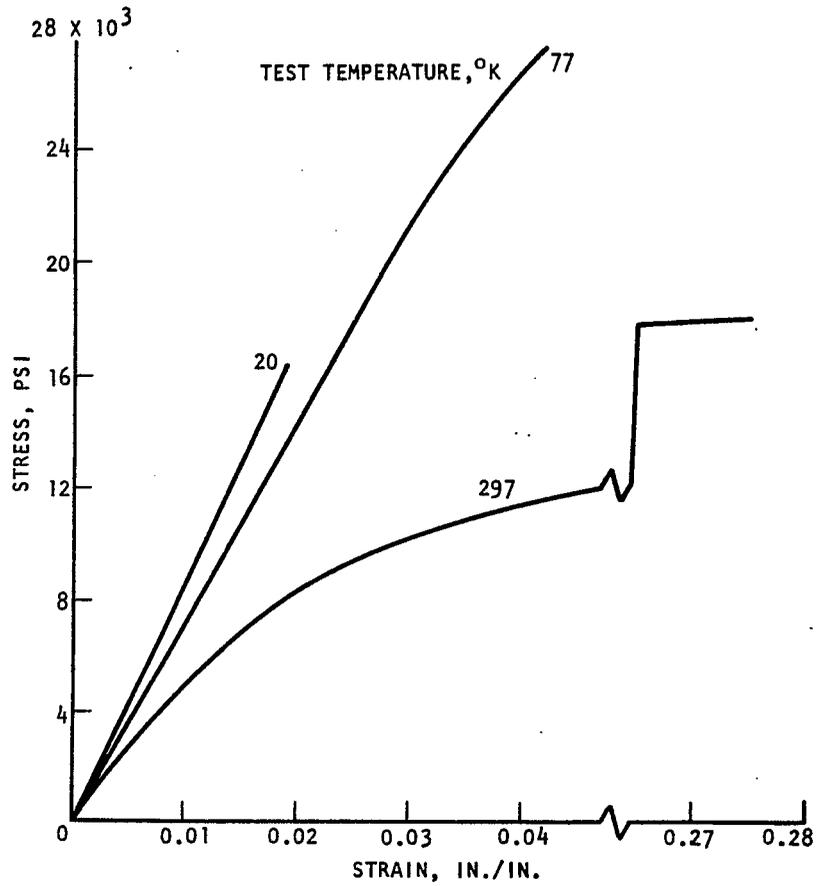

Fig. 25.   Temperature-strength behavior — Tedlar BG-30-WH film.

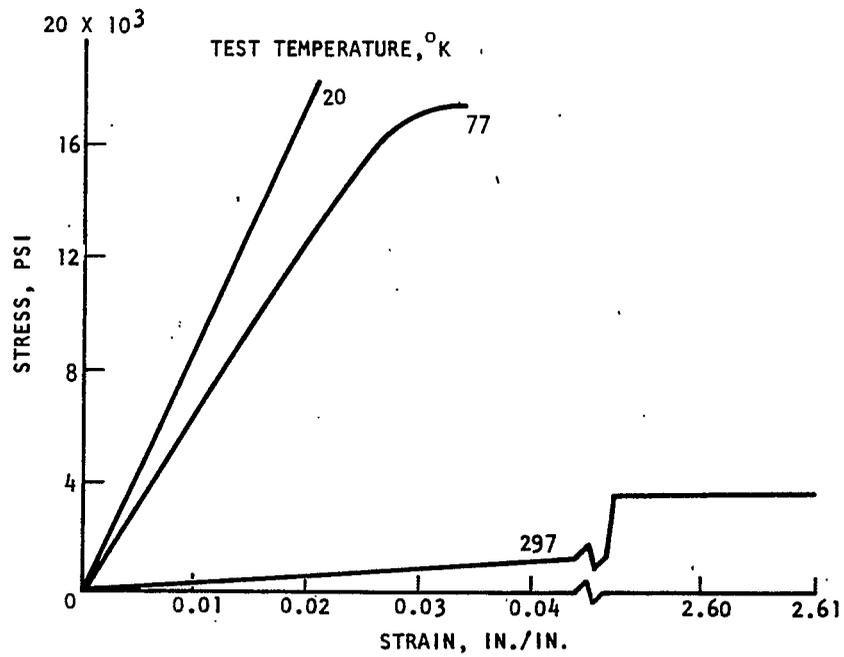Fig. 26.   Temperature-strength behavior — H film.

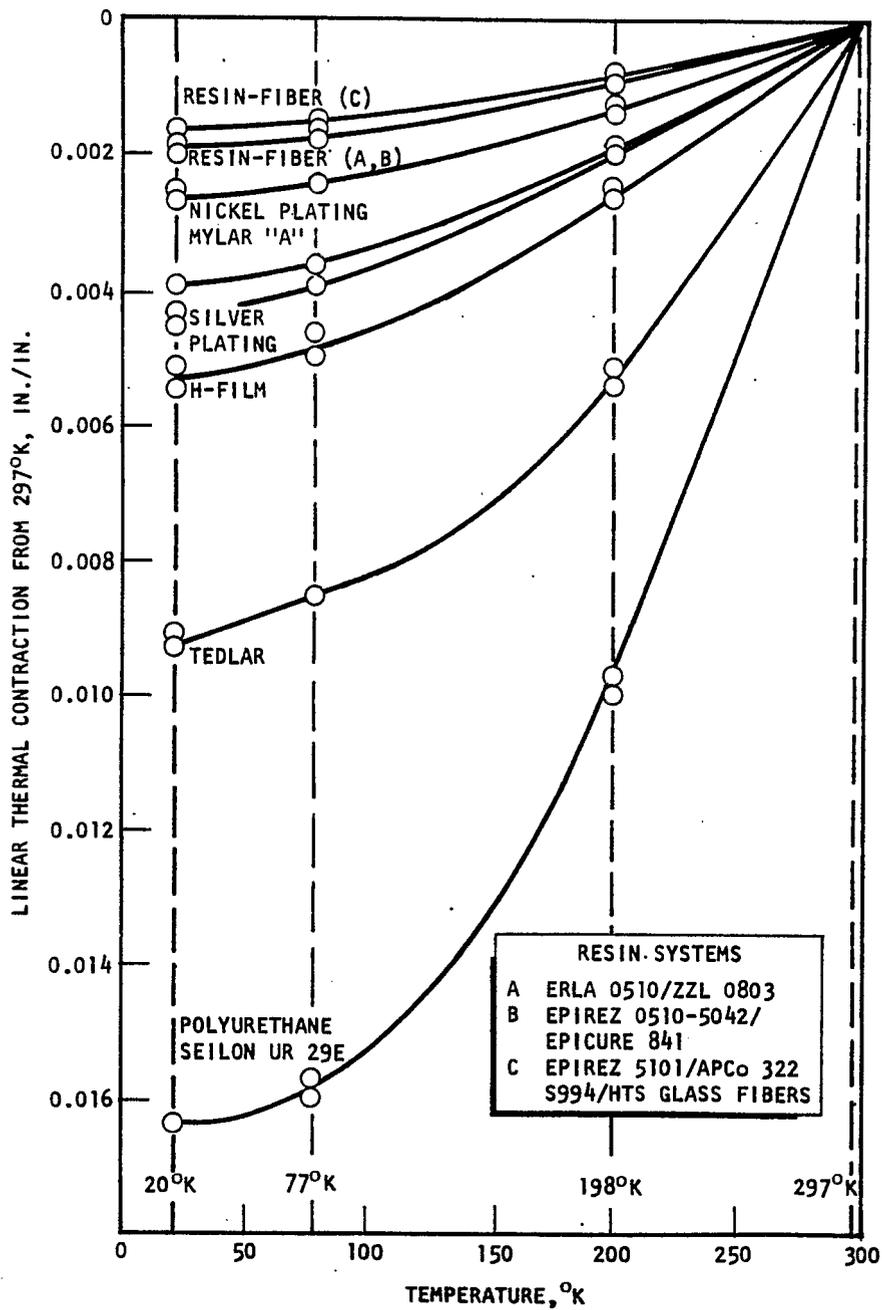Fig. 27.   Temperature-strength behavior — polyurethane Seilon UR29E.
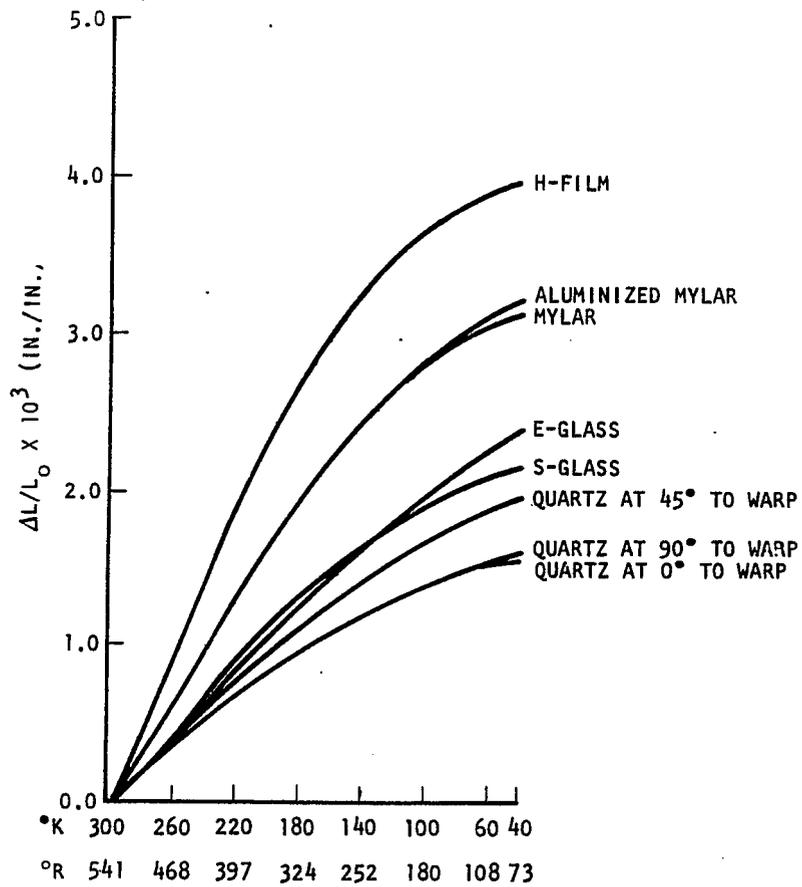
Fig. 28. Contraction curves of candidate liner materials.

Fig. 29.   Thermal contraction of films and laminates.