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SUMMARY

An experimental investigation was conducted to determine the tensile-strength properties of glass, boron, and graphite composites at 75°, -320°, and -423° F (297°, 77°, and 20° K). Composite tensile-strength and interlaminar-shear-strength tests were made of Naval Ordnance Laboratory (NOL) rings. Tensile strengths of boron filaments were determined at 75° and -320° F (297° and 77° K). Strengths of glass composites were about 29 percent higher at cryogenic temperatures than at ambient temperature. Boron- and graphite-composite strengths were essentially the same in the temperature range investigated. Interlaminar shear strength in glass composites also increased from 75° to -320° F (297° to 77° K); boron and graphite showed no significant change within the temperature range. Filament translation efficiencies ranged from 58 to 69 percent for the materials investigated.

Aluminum-foil liners that were adhesively bonded to the internal surface of glass-filament-wound cylinders withstood pressure cycling to 2.5 percent strain for a cyclic life ranging from 14 to 165 cycles at cryogenic temperatures. Liner failures were associated with buckling of the seam areas.

INTRODUCTION

Filamentary materials are used in applications where high strength-weight ratios are a design criterion. Glass filaments in a resin matrix, for example, are used extensively for pressure vessels, rocket-motor cases, and some aircraft structures. In these applications, the materials are subjected to ambient or moderately elevated temperatures. An urgent need exists for these materials in the cryogenic temperature range for the containment of liquid oxygen and liquid hydrogen. Recently, other filamentary materials with unique properties have become available. Two of these materials, boron and graphite, were chosen for cryogenic evaluation. In tensile strength, these materials can be regarded as being eventually competitive with glass, since they have a strength potential in the range from 500 000 to 1 000 000 psi (345 000 to 689 000 N/cm^2). In addition to their high strengths, the filaments also have tensile moduli which are several times that of glass.

The filaments of glass, boron, and graphite in composites are of particular interest in cryogenic applications. At low temperature, the loss of fracture toughness that plagues most isotropic materials is minimized in composites because their inherent discontinuities inhibit crack propagation between filaments. Their strength properties may also be enhanced at low temperatures.

Utilization of these materials in pressure systems presents a unique problem characteristic of composites. Under stress, the composite becomes porous because of cracking in the resin matrix. This problem exists at normal temperature and is probably more severe at cryogenic temperatures. A "barrier" or liner must be provided on the inner surface of the vessel to contain the pressurizing medium. A preliminary investigation (ref. 1) of the liner problem revealed that polymeric films and metallic foils were limited in performance. However, results of subsequent investigations at Lewis and in reference 2 indicate that plain metallic foil bonded to the composite with selected adhesives is promising as a cryogenic liner.

Presented herein are preliminary results of the evaluation of boron and graphite filaments as possible reinforcements for cryogenic propellant tanks. Also included are data on glass reinforcements that were used in the fabrication of test cylinders for the evaluation of cryogenic liners. Composite tensile-strength characteristics of glass, boron, and graphite were determined at temperatures of 75°, -320°, and -423° F (297°, 77°, and 20° K). Interlaminar shear strength of these composites and single-filament strengths of boron were determined at 75° and -320° F (297° and 77° K). Cyclic tests were performed on cylinders with aluminum-foil liners at temperatures of -320° and -423° F (77° and 20° K) to determine the cyclic life and mode of liner failure.

MATERIALS

The filament-resin materials and specimens evaluated are listed in table I. The glass and graphite filaments in the as-received condition were wound under 0.5-pound (2.22-N) tension into single Naval Ordnance Laboratory (NOL) rings (ref. 3). The boron was cleaned by passing it through a solution of boiling methanol just prior to the resin impregnation. All rings were 0.06 inch (1.52 mm) thick with the outer surface machined. Table II lists the typical properties of the filamentary materials investigated, as published in reference 4.

TABLE I. - COMPOSITE MATERIALS INVESTIGATED

Material	Designation and Description	Epoxy-resin system	Specimen	Curing cycle		
				Time, hr	Temperature	
					^{o}F	^{o}K
Strength tests						
Glass	S/901[a]; single-end roving	[b]ERL2256/ZZL0820	NOL rings	2 3	180 300	356 422
Boron	Boron-halide tungsten substrate	[c]58-68R	Single filament NOL rings	2 2 12	180 350 400	356 450 478
Graphite	Thornel-25[d]	ERL2256/ZZL0820	NOL rings	2 3	180 300	356 422
Liner tests						
Glass	S/901[a] roving and 112 fiberglass cloth	ERL2256/ZZL0820	Cylinder	2 3	180 300	356 422
Aluminum, 3 mil (0.076 mm) thick	1100-O					

[a]Owens-Corning Fiberglas Corp.

[b]Union Carbide epoxy resin, Union Carbide Corp.

[c]Shell Chemical epoxy resin, Shell Chemical Co.

[d]Carbon Products Division, Union Carbide Corp.

TABLE II. - TYPICAL AMBIENT PROPERTIES

OF FILAMENTARY MATERIALS (REF. 4)

Material	Density		Tensile strength		Tensile modulus	
	$lb/in.^3$	g/cm^3	psi	N/cm^2	psi	N/cm^2
S/901 Glass	0.090	2.50	650×10^3	449×10^3	12.5×10^6	8.63×10^6
Boron	.090	2.50	500	345	60.0	41.4
Graphite (Thornel-25)	.054	1.49	200	138	25.0	17.2

APPARATUS AND PROCEDURE

Tensile Tests

NOL rings. - Filament-resin composites in the form of NOL rings were tested at ambient and cryogenic temperatures by the use of a split disk fixture, as described in reference 3. Cryogenic temperatures were established by the submersion of specimens in liquid nitrogen (-320° F (77° K)) or liquid hydrogen (-423° F (20° K)) in special cryostats mounted in a tensile machine. The load was applied at a crosshead rate of 0.1 inch (2.5 mm) per minute.

Cast resin. - Castings, 0.125 inch (3.18 mm) thick of the ERL2256/ZZL0820 (27.5 parts per hundred) resin system cured 2 hours at 180° F (356° K) and 3 hours at 300° F (422° K) were machined into specimens in accordance with ASTM D638-64T-type I. The tensile specimens were tested at ambient temperature and -320° F (77° K) at crosshead speeds of 0.2 inch (5.08 mm) per minute. Strain was measured at both temperature levels by a clamp-on extensometer with a gage length of 1 inch (25.4 mm).

Single filaments. - The tensile strength of single filaments of boron was determined at 75° and -320° F (297° and 77° K). Filaments of 1-inch (25.4-mm) gage length were cemented to metal tabs with room-temperature curing epoxy resin to facilitate loading. The load was applied at 0.1 inch (2.5 mm) per minute.

Interlaminar shear tests. - Interlaminar shear strengths of glass, boron, and graphite composites were determined by the method outlined in reference 3. The specimens were 0.06 inch (1.52 mm) thick by 0.25 inch (6.35 mm) wide and 1 inch (25.4 mm) long. The specimens were loaded in flexure at the midspan of supports with 0.5-inch (12.7-mm) centers.

Lined Filament-Wound-Cylinder Tests

The cylinders used in investigating bonded foil liners were right-circular cylinders, 7.5 inches (19.1 cm) in diameter by 20 inches (50.8 cm) long. The cylinders were fabricated on mandrels of thick-walled aluminum tubing. A slight diametral taper was provided to facilitate removal of the finished cylinder from the mandrel. The details of liner assembly and cylinder construction are presented in the appendix.

The method used for capping the ends of the cylinders is shown in figure 1 and was also used in reference 1. A low-melting-point alloy filled the groove, effectively locking and sealing the end caps to allow pressurization. In cryogenic testing, the cylinders were placed in a cryostat with both inner and outer cylinder surfaces exposed to the cryogen. For the -320° F (77° K) tests, nitrogen gas was used for pressurization. For

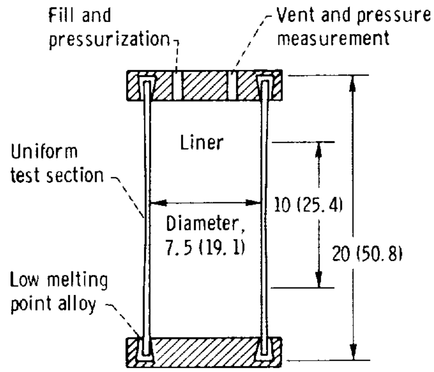


Figure 1. - Schematic diagram of biaxial cylinder with removable end caps used for cyclic tests. (Dimensions are in inches (cm).)

the -423° F (20° K) tests, the cylinder was pressurized with a liquid-hydrogen pump.

Hoop and longitudinal strains were measured by deflection transducers that were instrumented with strain gages. Calibrations were made at ambient and cryogenic temperatures with a screw micrometer as a standard. The hoop strain was determined by circumscribing a 10-mil (0.25-mm) wire about the cylinder at the midpoint of the test section to actuate the transducer. The longitudinal strain was measured similarly between clips secured in the cylinder wall. An installation is shown in figure 2. The strain rate was about 0.02 inch per inch per minute (0.51 mm/(mm)(min)). Nominally, a maximum strain of 2.0 to 2.5 percent was selected for the cyclic endurance (liner failure) tests of the liners.

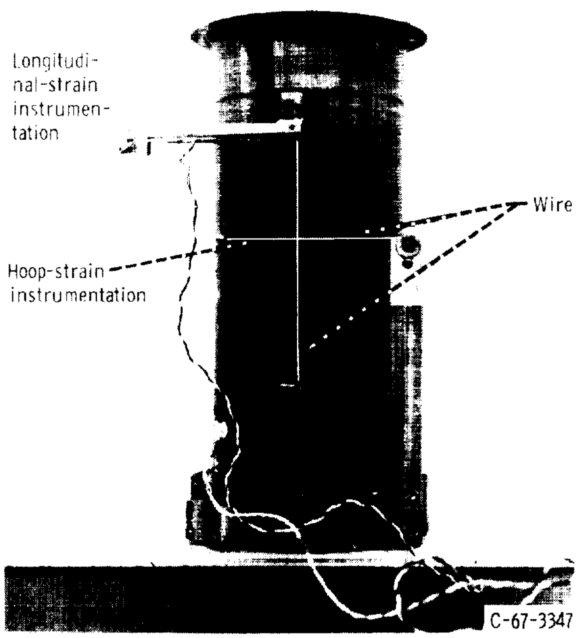


Figure 2. - Test cylinder with instrumentation for measuring longitudinal and hoop strains.

RESULTS AND DISCUSSION

Strength Characteristics of Filament-Resin Composites

The average tensile strengths of filament-resin NOL ring composites are shown in figure 3 as a function of temperature. The number of specimens tested for a given

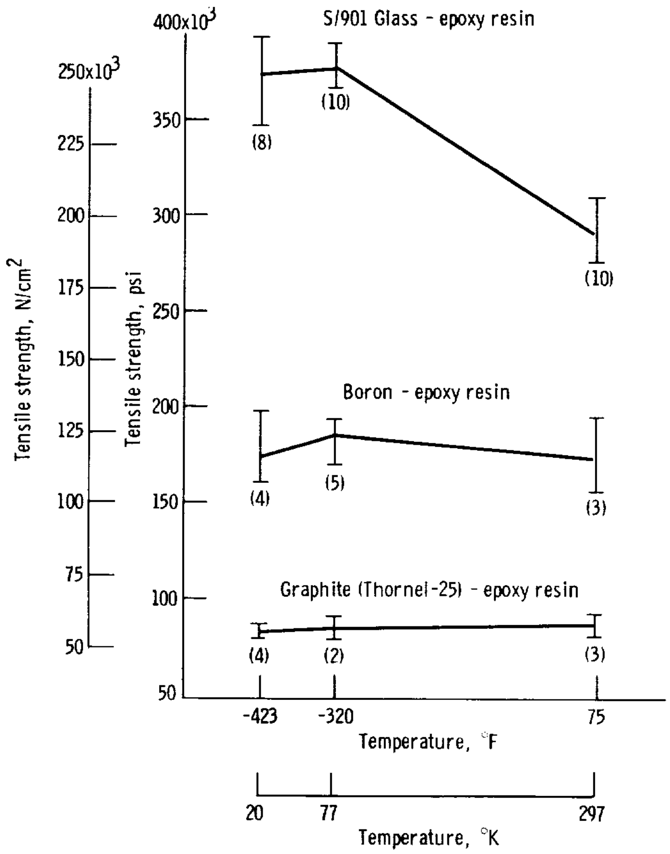


Figure 3. - Composite tensile strength of filament-wound NOL rings as a function of temperature. (Numbers in parentheses indicate number of tests run at each material and temperature.)

material and temperature are indicated, as well as the ranges of the test data. Tensile strength varies with temperature for the materials investigated. For the S/901 glass-epoxy composite, tensile strength increased from 290 000 psi (200 000 N/cm^2) at 75° F (297° K) to about 29 percent higher at cryogenic temperatures, whereas, boron and graphite composites have essentially flat strength characteristics within the temperature range investigated.

Because of their high tensile strengths, glass composites have an advantage over boron and graphite composites for pressure vessel application. This conclusion was also drawn in reference 5, where an analysis showed that glass-reinforced cylinders have a significant weight advantage over boron-reinforced cylinders for internal pressure vessels.

The boron and graphite filaments are of interest where high modulus is a desirable property. The tensile modulus may be a determining factor in pressure-vessel design, to provide rigidity for applications in large boosters or where strain is a limiting factor in liners for cryogenic propellant tanks. These materials possess unique properties that would have to be considered in regard to a particular application.

From a comparison of the tensile strengths of single filaments from table II with the tensile strengths of the composites at normal temperature in figure 3, the composite strengths are less than half of the filament strengths. From this low ratio, it is apparent that the composite strength behavior does not follow the law of mixtures. If full utilization of the filament strength were realized and the strength of the resin were ignored, the strength ratio should be of the order of 60 to 70 percent, which is generally the upper limit of filaments in an efficient composite. In addition to the correction of strength due to resin content, the reduction in filament strength in the composite may be attributed to (1) the inability of the resin to transfer load from filament to filament (interlaminar shear), (2) the strain limitation of the resin inducing cracking and crazing, (3) fabrication and mechanical flaws in filaments, and (4) composite voids. An understanding of the effects of some of these parameters on the composite strength characteristics can be deduced from specific tests. The interlaminar shear strengths are influenced by both temperature and material. As shown in figure 4, the interlaminar shear strength of glass

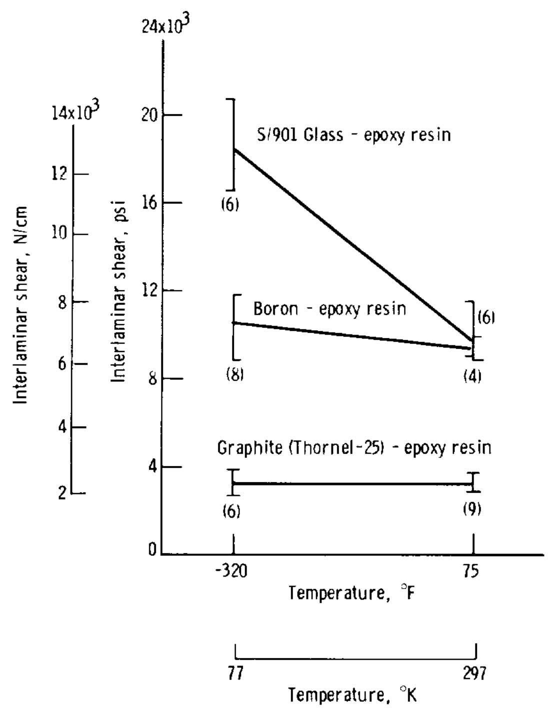


Figure 4. - Interlaminar shear strength of composites as function of temperature. (Numbers in parentheses indicate number of tests run at each material and temperature.)

increased from less than 10 000 psi (6900 N/cm^2) at 75° F (297° K) to about 18 000 psi (12 400 N/cm^2) at -320° F (77° K). The interlaminar shear strengths of boron and graphite composites (10 000 and 3000 psi (6900 and 2070 N/cm^2), respectively) remained essentially constant at the two temperatures. The behavior of interlaminar shear reflects that of tensile strength with temperature (fig. 3). Both the tensile and interlaminar shear strengths of glass composites increased with lower temperature, while boron- and graphite-composite strengths remained essentially constant.

As shown in figure 5, the stress-strain behavior of the ERL2256/ZZL0820 epoxy-

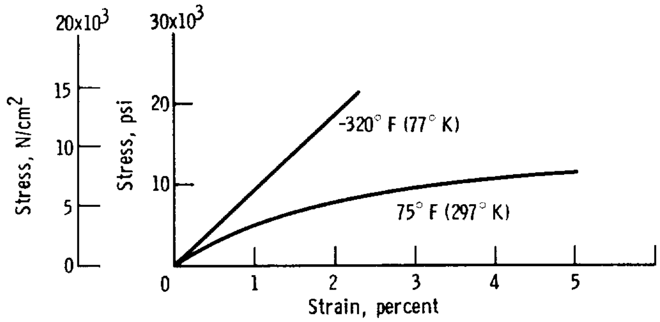


Figure 5. - Stress-strain diagram of ERL2256/ZZL0820 epoxy resin at 75° and -320° F (297° and 77° K). (Curves represent average of three specimens at each temperature.)

resin system changed significantly with temperature. Both the modulus and the tensile strength increased about 100 percent as the temperature was changed from ambient to cryogenic, with an associated strain-to-fracture reduction from 5 percent to about 2 percent at -320° F (77° K). This loss of ductility indicates a possible limitation of the resin matrix in composites where reinforcements are of a low-modulus, high-strength material such as glass. Glass composites may strain as high as 5 percent before fracture at cryogenic temperatures (ref. 1). These results indicate that, at cryogenic temperatures, the glass composite would be degraded above the 2 percent strain level. However, at cryogenic temperatures the composite is not degraded significantly because of the 29 percent strength increase noted in figure 3. Also, the structural integrity of the composite is not destroyed under strain. Therefore, either the low fracture strain of 2 percent is not a true value or the cracking and crazing on a macrostructure level can be tolerated by the composite. Reference 6 shows that the resin matrix is subjected to local strain magnifications of more than 20. This limitation of the resin even at ambient temperatures possibly obscures the embrittlement problem of the resin at cryogenic temperatures.

No determinations of the strength of single filaments of glass and graphite were made in this investigation; however, data at ambient temperature are available in references 4 and 7. From these data and the results of tests of single filaments of boron at 75° and -320° F (297° and 77° K) (fig. 6), it is evident that the composites do not have

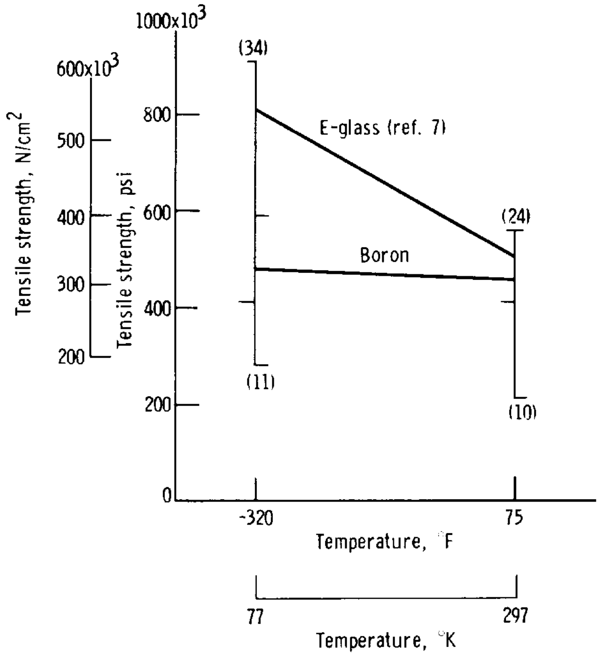


Figure 6. - Single-filament tensile strength as function of temperature. (Numbers in parentheses indicate number of tests run at each material and temperature.)

the high strength that would be expected in translation from filament to composite. Table III lists the filament and composite strengths of the materials presently considered. Also shown is the composite filament strength, which is based only on the total filament area. The assumption was made that the resin carried no load and that the volume of filaments was 65 percent of the composite, exclusive of void content. The filament trans-

TABLE III. - FILAMENT TRANSLATION EFFICIENCY IN NOL-RING COMPOSITES

Filament	Filament strength		Composite strength[a]		Composite filament strength[b]		Filament translation efficiency, percent
	psi	N/cm^2	psi	N/cm^2	psi	N/cm^2	
75° F (297° K)							
S/901 Glass	$650 \cdot 10^3$	$449 \cdot 10^3$	$290 \cdot 10^3$	$200 \cdot 10^3$	$447 \cdot 10^3$	$308 \cdot 10^3$	69
E-glass	507	350	-------	-------	310	214	[c]61
Boron	460	318	174	120	268	185	58
Graphite	200	138	87	60	134	93	67
-320° F (77° K)							
E-glass	$814 \cdot 10^3$	$562 \cdot 10^3$	-------	-------	$510 \cdot 10^3$	$352 \cdot 10^3$	[c]63
Boron	483	333	186	128	286	197	59

[a]Based on total area.

[b]Based on filament area.

[c]Ref. 7.

lation efficiency is defined as the ratio of the average composite filament strength to the average monofilament strength (strength determined from single filaments). On this basis, the filament translation efficiency ranges from 58 percent for boron to 69 percent for S/901 glass at 75° and -320° F (297° and 77° K). Data from reference 7 for E-glass are included because of the cryogenic data, which are also shown in figure 6. The efficiencies for the E-glass were 61 and 63 percent at 75° and -320° F (297° and 77° K), respectively, which is within the range of the materials presently investigated. These results substantiate the argument that the composites are not degraded by low temperature.

Cyclic Characteristics of Liners for Glass-Filament-Wound Cylinders

The requirements for impervious liners to be used in filament-wound cylinders are presented in reference 1. In the present investigation, metallic liners of aluminum foil were adhesively bonded to the inner surface of glass-filament-wound cylinders. No metals have elastic strain behavior comparable to glass. However, it appeared feasible to strain 1100-O aluminum plastically in tension and compression, if an adequate adhesive bond could be maintained. Reference 8 shows that 1100-O aluminum can withstand high uniaxial plastic strain (3 percent for 1000 cycles) before fatigue failure results. Figure 7 shows the probable stress-strain relation experienced by the glass-resin composite cylinder and the aluminum liner under a single cycle at 2.5 percent strain. The cylinder behavior is essentially elastic within the strain range, whereas the aluminum

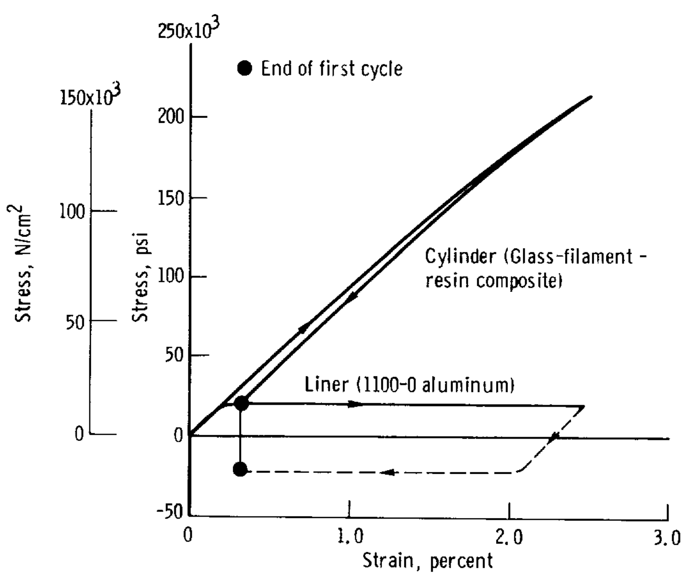


Figure 7. - Stress-strain relation of cylinder and liner.

liner undergoes most of the strain plastically in both tension and compression. At the end of the cycle, the cylinder and liner retain some residual strain because of the compressive restraining force of the aluminum. After subsequent cycling, the stress-strain relation of the cylinder and liner may change because of the progressive cyclic damage to the composite and the cyclic effects on the aluminum. The high degree of elastic strain incompatibility between the aluminum liner and the low-modulus glass-filament - resin composite indicates a distinct advantage in the use of high-modulus filaments, such as boron and graphite, in pressure-vessel applications.

During the present investigation, a number of cylinders were fabricated with plain liners of 1100-O aluminum to establish the feasibility of the system as a permeation barrier. The optimization of variables, such as liner materials, thickness, surface preparation, adhesives, and fabrication techniques, was limited to establishing the capability of strain cycling the lined composite cylinders at cryogenic temperatures.

Plain liners must be bonded to the cylinder to prevent buckling under strain cycling (ref. 1). The G207 adhesive was chosen because of its high strength at cryogenic temperatures and its convenience in handling. The adhesive is a thermoplastic polyester that is fusible at the curing temperature of the epoxy. This capability allowed the liner to be adequately coated with adhesive before the glass-filament winding was applied. The initial winding was applied dry with the composite resin added on the second layer. The blending of the polyester adhesive and the epoxy resin was thus accomplished within a layer of glass. The fabrication as outlined in the appendix resulted in an approximate 1-to-1 hoop- to longitudinal-strain ratio at 2.5 percent strain and an internal pressure of about 400 psi (276 N/cm^2).

Results of tests on cylinders lined with 3-mil (0.076-mm) aluminum foil are given in table IV and show the variables of surface preparation, adhesive selection, test temperature, and strain level. Some of the liners were capable of over 100 cycles at cryogenic temperatures before failure occurred. Chemical surface preparation with Oakite 33 resulted in higher average cyclic life than did sandblasting. Although the liquid-hydrogen tests were limited, performance of the adhesively bonded aluminum liner appears to be comparable at both cryogenic temperatures. The cylinder whose liner was attached with Cybond 4000 adhesive showed good performance. Therefore, there may be other adhesives that will be useful for this application.

All liner failures caused by leaks were located at the lap seam. Generally, the seam contained numerous areas where buckling had occurred. A typical seam-area buckling is shown in figure 8. Some cylinders (table IV) had small areas of buckling not located in the seam; however, none of these areas produced leaks.

TABLE IV. - CYCLIC TESTS OF ALUMINUM-FOIL-LINED CYLINDERS

[Liner thickness, 3 mil (0.076 mm).]

Cylinder	Surface treatment agent	Adhesive	Test temperature		Strain, percent	Cycles	
			°F	°K		No failure	Liner failure
1	50-Micron-grit sand	[a]G-207	-320	77	1.9		46
2					2.5		16
3					2.1		[b]40
4					2.5		14
5			-423	20	2.5		65
6			-320	77	2.4		[b]24
7					2.3		15
8							23
9	[c]Chromic acid solution						26
10	Oakite 33	[d]Cybond 4000					78
11		G-207					107
12			-320	77	2.1	22	
			-423	20	2.1	100	
			-423	20	2.3	10	
			-320	77	2.3	[b]33	165
13					2.3		[e]40
14					2.2		[b]41
15					2.4		103

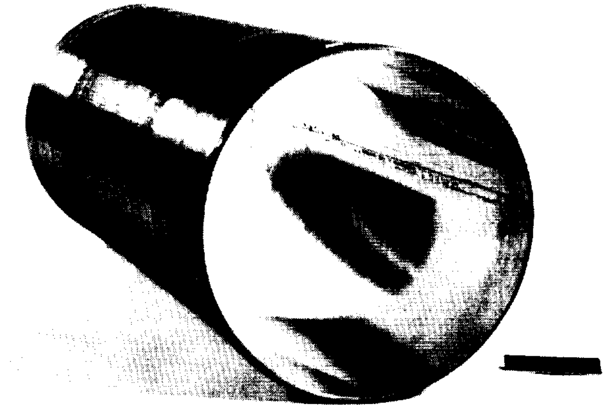
[a]Supplied by Goodyear Aerospace Corp.

[b]Small areas outside of seam buckled.

[c]Parts by weight, sodium dichromate (10), 95-percent sulfuric acid (30), distilled water (100).

[d]Supplied by American Cyanamid Co.

[e]Cylinder burst on pressure cycling.



C-67-1049

Figure 8. - Seam failure of aluminum liner after 103 cycles to 2.4 percent strain in liquid nitrogen.

SUMMARY OF RESULTS

The following results were obtained from an investigation of composites of resin and glass, boron, and graphite filaments at ambient and cryogenic temperatures:

1. The average composite tensile strength of Naval Ordnance Laboratory (NOL) rings of S/901 glass was 290 000 psi (200 000 N/cm^2) at 75° F (297° K), with an increase of 29 percent at cryogenic temperatures.

2. The composite tensile strengths of NOL rings of boron and graphite were 174 000 and 87 000 psi (120 000 and 60 000 N/cm^2), respectively, at 75° F (297° K). Both materials showed no significant increase at cryogenic temperatures.

3. A filament-winding resin (ERL2256/ZZL0820) required about 5 percent strain to fracture at 75° F (297° K). At -320° F (77° K), the fracture strain was reduced to about 2 percent.

4. The interlaminar shear strengths of S/901 glass and boron composites at 75° F (297° K) were both approximately the same (10 000 psi (6900 N/cm^2)). At -320° F (77° K), glass shear strength increased to about 18 000 psi (12 400 N/cm^2), while the boron shear strength increased only to about 11 000 psi (7600 N/cm^2). The interlaminar shear strength of graphite was about 3000 psi (2070 N/cm^2) at both 75° and -320° F (297° and 77° K).

5. Filaments of glass, boron, and graphite in composites showed filament translation efficiencies in a range from 58 to 69 percent at both 75° and -320° F (297° and 77° K), with no apparent degradation at -320° F (77° K).

6. Pressure vessels with adhesively bonded aluminum-foil liners with longitudinal

lap seams were pressure cycled to about 2.5 percent strain for over 100 cycles at -320° and -423° F (77° and 20° K) before failure occurred. The lap seam was the source of all liner failures.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, October 13, 1967,
124-08-08-15-22.

APPENDIX

LINER-CYLINDER FABRICATION

The aluminum mandrel was coated with a Teflon parting agent. The aluminum-foil liner surface was prepared by one of the following methods (see table IV):

(1) Degrease with toluene, dry, place on mandrel, and abrade with 50-micron-grit sand.

(2) Degrease with toluene, dry, clean in chromic acid solution, rinse with tap water, and dry before placing on mandrel.

(3) Clean surface in Oakite 33 (1 part per 10 parts tap water), rinse with tap water, and dry.

The aluminum-foil liner was placed on the mandrel; the lap seam was formed and was held with G207 adhesive (in parts by weight, G207B(100), toluene (63), methyl ethyl ketone (27), and G207C(4)). The liner surface was then brush coated with G207 and allowed to dry at ambient temperature for 12 hours.

An S/901 single-end glass roving applied dry at 48 ends per inch (19 ends/cm) with 0.5-pound (2.2-N) tension in the hoop direction. The dry roving was then brush coated with the epoxy-resin system (ERL2256-ZZL0820) (27 parts per hundred). Four layers of 112-Volan A glass cloth in a 20-inch (50.8-cm) width were applied under 10-pound (44.5-N) tension and wet with the ERL2256 resin. A final hoop wrap of single-end roving at 48 ends per inch (19 ends cm) completed the basic cylinder.

Both ends of the cylinder were additionally reinforced with 1542-Volan A glass cloth in the following sequence: Five layers, 1 inch (2.54 cm) wide; one layer, 2 inches (5.08 cm) wide; one layer, 3 inches (7.62 cm) wide; one layer, 4 inches (10.16 cm) wide; and a final end overwrap with a single-end roving, 5 inches (12.70 cm) from each end. Partial curing was achieved in the winding machine for 2 hours at 180° F (356° K). A final cure was made in the oven at 300° F (422° K) for 3 hours. The cylinder was removed from the liquid-nitrogen-cooled mandrel over the tapered end.

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