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FILAMENT-WOUND COMPOSITE VESSEL MATERIALS TECHNOLOGY

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ABSTRACT

This paper reviews a number of programs that were conducted to establish a technology base for applying advanced fibers or resins to high performance filament-wound pressure vessels for containment of cryogenics and high pressure gases. Materials evaluated included boron, graphite, PRD 49-I and III/epoxy and S-glass/polyimide composites. Closed-end cylindrical, and oblate spheroid-shaped vessels were fabricated in 4- and 8-inch diameter sizes. Vessels were subjected to single-cycle burst, low-cycle fatigue, and sustained loading tests over a -423° F to room temperature range for epoxy composites and a -423° to 500° F temperature range for the polyimide composites. Vessels tested at cryogenic and/or 500° F had thin (3 to 20 mils) metallic liners whereas vessels tested at room temperature had elastomeric liners. Correlations between acoustic emissions and burst and cyclic properties of PRD 49-I filament-wound vessels are discussed. Vessels were rated on the basis of a pressure vessel performance factor, $P_b V/W$, where P_b is the burst pressure, V is the volume, and W is the weight of the vessel. Values of $P_b V/W$ (excluding liner weight) for the single-cycle burst tests ranged from 0.6×10^6 inches for boron/epoxy to 1.8×10^6 inches for PRD 49-III/epoxy vessels.

INTRODUCTION

The high-strength-to-density and modulus-to-density ratios of advanced fiber composites has prompted interest in their potential applica-

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tion to construction of filament-wound (FW) pressure vessels for containment of cryogenics and high pressure gases. Currently, metallic pressure vessels made from the stainless steel alloys, aluminum, Inconel, or titanium are used for these applications. The performance efficiencies of the metallic tanks, based on a pressure vessel performance factor ($P_b V/W$), where P_b is the burst pressure, V is the volume, and W is the weight of the vessel, range from 0.3 to 0.6×10^6 inches. FW composite tanks made from advanced fiber/resin composites equipped with thin metallic liners attached to metallic bosses have the capability of yielding $P_b V/W$ values in the range of 1.0 to 1.8×10^6 inches (including liner weight). Therefore FW composite pressure vessels can provide a significant decrease in weight, in comparison with metallic tanks of similar volume and pressure.

This paper presents a summary of past and current NASA-Lewis Research Center programs for the development of materials technology for FW composite vessels. The intended application for these vessels is for the containment of cryogenics and high pressure gases over a cryogenic to high temperature range (-423° to 500° F). The primary objectives of the programs were to establish the feasibility of using advanced fiber/resin composites for the construction of high performance vessels and also to generate FW composite vessel performance data.

The report describes the basic requirements for a FW vessel design subjected to internal pressure. The mechanical performance data for each vessel design and comparisons of their $P_b V/W$ values are discussed.

BACKGROUND

In order to achieve structurally efficient FW vessels, for high per-

formance applications, the vessels must be designed for operation at high fiber stress levels. High fiber stress levels can, however, result in significant elongation of FW glass composite structures (up to 2% for glass/epoxy). At this elongation matrix cracking occurs and a liner is needed to prevent leakage of the fluid contained in the vessel. Because polymeric materials, in general, have little extensibility at cryogenic temperatures, polymeric films cannot be used as liners for FW glass-epoxy vessels at -423° F. A NASA technology program (ref. 1) for the development of polymeric film liners indicated that Kapton film was the best material for use as a liner for glass/epoxy composite cylinders at -320° F. However, difficulties in processing Kapton film into the shape of a typical vessel and the material's inherent permeability to helium and hydrogen gases have delayed further work with this material indefinitely. Emphasis accordingly was shifted to the development of metallic liners for FW glass/epoxy vessel service. Two liner types evolved from this work, i.e., the thin-liner and the load-bearing liner concepts. In the thin-liner concept, the majority of the load is carried by the composite overwrap while in the load-bearing liner concept a relatively thick metallic liner is overwrapped with reinforcing filaments and results in load-sharing between the liner and the composite overwrap.

Regardless of recent advancements in the development of a "hinge-boss" liner-to-boss attachment design, the thin-metal liner concept has been found to have limited utility for application to FW glass/epoxy vessels. Although bonding of the liner to the inside wall of the vessel prevented liner buckling during depressurization, the liners eventually failed at a relatively small number of vessel-depressurization cycles.

The load-bearing liner concept has proven to be more successful and, although weight savings are not as high as could be attained by a thin-liner, the concept has provided an interim solution to the liner problem for FW glass/epoxy vessels.

The recent availability of advanced reinforcing fibers with a wide range of high strengths and moduli prompted NASA interest in the use of these fibers for the construction of FW composite vessels. The strength-to-density versus the modulus-to-density ratios for a number of advanced fibers are shown on figure 1. These values were derived from tensile strength tests made on epoxy resin-impregnated strand specimens. Also shown are typical values for metals used for construction of high performance vessels.

Because of higher moduli FW composite vessels made of the advanced fibers would experience significantly lower operating strains than glass/epoxy vessels and would provide a more favorable strain-matching condition for thin metallic liners. In table I are listed the calculated extensibilities of materials (in the form of vessel specimens) subjected to typical operating stresses. The data clearly illustrate that advanced composites would provide a better strain compatibility with thin metallic liners than would glass fiber composites.

A series of contractual programs, summarized in this report, have been sponsored by NASA to investigate the feasibility of using advanced fibers such as boron (ref. 2), graphite (refs. 3, 11, 12, and 13), and PRD 49-I and III fibers (ref. 4) for construction of lightweight, structurally efficient composite vessels for the generation of mechanical property data.

VESSEL DESIGN AND FABRICATION CONSIDERATIONS

The approach used for each of the programs generally involved the determination of the mechanical properties of fiber/resin composites in the form of strand and NOL ring specimens. These specimens were tested for strength properties over a cryogenic (-320° and -423° F) to room temperature range and provided a basis for the design and fabrication of filament-wound vessel specimens. This preliminary work also provided an opportunity to assess the uniformity of the candidate fibers and their processing characteristics.

Because no prior experience base was available for the design of the advanced composite FW vessels, the design and fabrication methods utilized for glass/epoxy vessels were specified. The initial designs were derived using a netting analysis technique. As data were generated during the programs, it was found that a combined netting analysis and finite element design technique resulted in vessels having greatly improved structural efficiencies. The combined design technique showed that local reinforcement of the dome and cylinder-to-dome transition areas was required. Significant improvements in $P_b V/W$ values were attained using the combined technique. In one program (ref. 5) highly stressed areas in a vessel specimen were identified using holographic interferometry. These areas were then locally reinforced on subsequent vessels with additional FW composite material. A re-examination of the vessels by holographic interferometry confirmed that the stress patterns of the modified vessels were now more uniform. Subsequent burst tests of the vessels resulted in significantly improved values of $P_b V/W$.

Vessels fabricated in all of the programs were equipped with elasto-

meric liners for room temperature and with thin-metallic liners for testing at cryogenic and 500° F temperatures. Because the programs were funded to emphasize the development of material property data, conventional state-of-the-art thin-metal liners, developed for earlier glass/epoxy composite vessel programs (ref. 6) were utilized without modifications for weight reduction or elimination of high liner-to-boss attachment stresses. Figure 2 shows a typical vessel/thin-metallic liner assembly. Generally, the thin-metallic liner design described above proved to be satisfactory for both cryogenic and room temperature single-cycle burst and cyclic testing of boron/epoxy vessels. During cyclic testing at operating pressures the liners experienced some plastic deformation. The integrity of the adhesive bond between the liner and the inside wall of the composite vessel prevented buckling failure of the liners during the depressurizing portion of the cycle.

However, for vessels made of the lower modulus graphite and PRD 49-III fibers, the use of conventional thin-metallic liners for development of cyclic data would be impractical because of the higher strains incurred, especially in localized areas such as the liner-to-boss attachment. Current programs for the generation of cryogenic cyclic data for graphite and PRD 49-III vessels are using "hinge boss" thin-metallic liners developed under a related glass/epoxy vessel program (ref. 7). The differences between the conventional and "hinge boss" liner-to-boss attachment design can be seen in figure 3. The "hinge boss" liner-to-boss attachment design (fig. 3(b)) allows the liner to strain over the boss area during vessel pressurization. In the conventional liner-to-boss attachment design (fig. 3(a)), the high stress concentration imposed

on the liner-to-boss weld results in early liner failure.

The effect of other FW parameters such as various fiber types and resins, fiber content, winding tension, fiber impregnation method, mandrel types, and winding techniques on vessel performance are discussed in the VESSEL TESTING section of this report.

VESSEL TESTING

Test Matrix

Table II lists the types of vessels tested in the programs summarized in this report along with the types of tests and test temperatures. Vessels were instrumented for internal pressure, temperature, strains, and for acoustic emission signals (PRD 49-I and III/epoxy vessels only). Preliminary correlations of acoustic emission signals with the mechanical properties of PRD 49-I/epoxy vessels are discussed in the Cyclic Fatigue Tests of PRD 49-I/Epoxy Vessels section.

Vessel Test Results

The results of the single-cycle burst tests of the composite vessels are summarized in Table III. The $P_b V/W$ values are compared on the basis of the weight of the composite and do not include the weight of the liner or bosses. The $P_b V/W$ values were obtained at the peak of the contractors' "learning curve" and also represent values attained using the highest strength fibers that were commercially available for fabrication of the vessels. Pertinent details concerning each of the vessel types are given below.

Boron/epoxy. - The boron/epoxy vessels were filament-wound using 1/8" wide boron/epoxy tape (made by collimating 29 single boron monofilaments having a minimum tensile strength of 400 KSI). The epoxy resin

used was 58-68R. Essentially conventional glass FW procedures were used for vessel fabrication. Vessels were in-plane wrapped over a 6 mil stainless steel liner made by roll-resistance welding hydroformed heads to a cylindrical section. The liner was adhesively bonded to the inside wall of the vessel by using a cryogenic adhesive impregnated nylon scrim (ref. 2).

The single-cycle burst strength of the boron vessels at -423° F (252 KSI fiber hoop stress) exhibited an increase of 15 percent compared to the room temperature value (220 KSI fiber hoop stress). Results from cyclic testing of boron/epoxy vessels appeared to be independent of test temperature. The fatigue life characteristics were similar to that of S-glass FW vessels. One vessel was cycled 100 times to 55 percent of its ultimate strength (based on vessels tested at -423° F for single-cycle burst strength) at -423° F and showed an 84 percent retention of its cryogenic burst strength when subsequently pressurized to failure. Vessels pressurized at 70 percent of their ultimate strength for periods up to 90 days (70° F) showed no strength loss when subsequently pressurized to failure. Strains at burst ranged from 0.4 to 0.5 percent. These values are approximately 1/10 of the values for glass FW vessels. No vessel failures could be attributed to liner failures. $P_b V/W$ values attained throughout the program reflected improvements in both tape quality and fabrication experience. Details of this program are described in reference 2.

S-glass/polyimide. - The S-glass/polyimide vessels were fabricated using S-glass roving impregnated with a high temperature-resistant (500° F) polyimide matrix resin (Gemon L). The vessels were in-plane

wrapped over a 6 mil stainless steel liner.

Modified S-glass filament-winding vessel fabrication procedures were developed for winding of the prepreg roving and subsequent curing. The single-cycle $P_b V/W$ values (1.0×10^6 in.) attained are nearly equal to values for S-glass/epoxy vessels of identical structural design.

Vessels subjected to thermal aging for 100 and 500 hours at 500° F and then pressurized to failure at 70° F retained 80 and 50 percent of their initial room temperature strength, respectively. Details of this program are described in reference 8. A program to develop higher temperature resistant FW polyimide resins is being conducted in Contract NAS3-16760 (ref. 9).

PRD 49-I and III/epoxy. - The PRD 49/epoxy vessels in the first PRD 49 fiber program (ref. 4) were fabricated from PRD 49-I fibers using an in-plane winding pattern and were equipped with elastomeric liners. Prior to vessel fabrication, a preliminary winding study was conducted to determine the effect of various winding and composite parameters (e.g., fiber content) on the mechanical properties of NOL ring specimens. The resulting data were then used to guide the fabrication of vessel specimens. It was found that the uniformity of these fibers was excellent and filament winding with the fibers presented no difficulties. The details of this program are described in reference 4. The results of cyclic testing of vessels in this program are discussed in the Cyclic Fatigue Tests of PRD 49-I/Epoxy Vessels section. The objective of a follow-on program (Contract NAS3-15480, ref. 10) is to develop the mechanical properties of thin-metallic lined PRD 49-III composite vessels that will permit testing at cryogenic temperatures.

The objective of another related program (NASA-AEC Purchase Request C-13980C, ref. 5) is to improve design and fabrication techniques for PRD 49-III composite vessels. A $P_b V/W$ value of 1.8×10^6 inches obtained in this program for both 4- and 8-inch diameter vessels is the highest known value reported and represents a fiber efficiency of over 90 percent in the vessel's circumferential wraps.

Graphite Modmor II/epoxy. - The objective of this program (NASA-Defense Purchase Request C-10360B, ref. 3) was to generate mechanical property data for graphite/epoxy composites made from commercially available graphite fibers. The data resulting from this effort were utilized for the fabrication of composite vessels for cryogenic service. A $P_b V/W$ value of 0.5×10^6 inches was reported in reference 11. Vessels were in-plane wrapped using the first available continuous graphite fiber (Modmor II tow), impregnated with an epoxy resin using a conventional wet winding process. The relatively low $P_b V/W$ value was attributed to a combination of variability of fiber quality and high resin content in the composite. This was the first known program for filament winding closed-end graphite composite vessels equipped with thin-metal liners for cryogenic service. An important finding from this program was that conventional S-glass/epoxy vessel design and fabrication techniques could not be used for graphite vessels. It was concluded that the tow was susceptible to damage from high winding tensions and that the resin content of the cured vessel was difficult to control when a wet winding process was used.

Graphite T-400/epoxy (oblate spheroid). - On the basis of data generated in the program described above, a program was conducted under Contract NAS3-13305 to develop optimum graphite vessel filament-winding

parameters and to generate vessel performance data. An extensive parametric study of vessel fabrication parameters was conducted using strand, NOL ring, and vessel specimens (4- and 8-in. diameter). The effect of winding parameters on the mechanical properties of the specimens was, however, generally obscured by nonuniform qualities of the Modmor II graphite tow (then available) used in the majority of the program. The low $P_v V/W$ values for vessels made from this fiber were attributed to poor fiber quality and an inappropriate vessel design which did not provide for the local reinforcement of highly stressed areas near the vessel domes.

HTS and T-400 graphite fiber types were also evaluated in the program and an oblate spheroid shaped vessel made of the latter fiber type attained a $P_b V/W$ value of 0.9×10^6 inches. The details of this program are described in reference 13.

Graphite HTS/epoxy, graphite T-400/epoxy. - The HTS and T-400 graphite/epoxy vessels, tested in NASA-Defense Purchase Request C-10360B, were cylindrical in shape with an L/D ratio of 1.5. The $P_b V/W$ values for these vessels averaged 0.8 and 0.9×10^6 inches, for the HTS and T-400 vessels, respectively. The high $P_b V/W$ values for this vessel shape and L/D ratio (cylindrical composite vessels with an L/D ratio of 1.5 are less efficient, structurally, than an oblate spheroid vessel shape) are attributed to the following: (1) an efficient finite element design technique, (2) consistent high fiber quality, and (3) a modified resin impregnation technique (for the HTS vessels) that accurately controlled resin content and inhibited fiber handling damage. The results of this program are described in reference 13.

Cyclic Fatigue Tests of Graphite Composite Cylinders

The potential for constructing high cyclic life vessels from graphite fibers was first indicated by in-house tests made on unidirectional graphite/epoxy composites. It was also found that these composites exhibited extremely low creep and high creep rupture strength characteristics. The results from further in-house studies on the fabrication and cyclic testing of 6-inch diameter open-ended FW cylinders, made of Thornel 50/epoxy and equipped with thin polymeric and metallic liners, are summarized on figure 4. The details of this program are described in reference 14. The findings from this pioneer research effort were responsible for initiation of the contractual efforts for the development of graphite vessels.

Cyclic Fatigue Tests of PRD 49-I/Epoxy Vessels

The results of the cyclic testing (at room temperature) of PRD 49-I/epoxy vessels are shown on table IV. The vessels were initially subjected to 1000 cycles at 60 percent of ultimate strength and were then pressurized to failure on the next cycle. The cycled vessels failed at a higher pressure than vessels of identical design subjected to single-cycle burst tests. It is believed that the strength enhancement of the cycled vessels is due to the progressive fracture of fibers at relatively low stress levels rather than at high stress levels as in vessels subjected to single-cycle burst tests. In the latter case, the fiber fractures which occur at higher stress levels results in sufficient energy release to initiate and propagate failure of adjacent fibers causing a lower overall composite strength. It is also possible that a more uniform fiber loading condition was provided in the cycled vessels by the viscoelastic flow of the matrix during the vessel cycling process.

The performance of the vessels subjected to cyclic testing at 90 percent of ultimate strength indicates that extensive fiber damage was occurring at this stress level. The number of cycles-to-failure, for one of the vessels exhibiting the lowest cycles-to-failure value (34 cycles) in the test series, is greater by a factor of about 10 compared to the performance of S-glass vessels of similar design.

The vessels in this program were equipped with transducers for recording acoustic emission signals emitted during testing. The objective of the study was to determine the feasibility of using acoustic emission signals as a tool for quality control, and fatigue life prediction or incipient failure detection.

Analysis of the acoustic emission signatures obtained during the single-cycle tests of these vessels indicated that the total (cumulative) count parameter (number of signal events whose amplitude exceed a given level) may be an exponential function of applied pressure or vessel strain. Results of the acoustic emission signals emitted during the fatigue cycling of the vessels appear to indicate that the total counts-to-failure is a constant for a given vessel design regardless of whether the vessel failed in a single-cycle or cyclic process.

It needs to be pointed out that a small number of vessels were acoustically monitored. Further work is required to be done to establish the validity of the acoustic emission/mechanical properties correlations.

RESULTS AND CONCLUSIONS

The in-house and contractual filament-wound vessel programs that have been conducted have contributed significantly to the development of advanced fiber/resin composite vessel technology. Design and fabrica-

tion procedures have been developed for small diameter (4- and 8-in. diameter) closed-end vessels equipped with thin elastomeric or thin metallic liners. Specifically the following results and conclusions are given.

1. The feasibility has been established for constructing FW vessels for containment of cryogenics and high pressure gases from PRD 49/epoxy, graphite/epoxy, boron/epoxy, and S-glass/polyimide composites that are lighter than metallic vessels of comparable designs.

2. Static burst tests of cycled PRD 49-I vessels which were previously subjected to 1000 cycles at 60 percent of ultimate strength (based on single-cycle data) showed a 25 percent increase in vessel efficiency over vessels subjected to single-cycle burst tests only. The results indicate that such vessels potentially have fatigue lives greater than 1000 cycles at the above stress level.

3. Based on single-cycle burst data, vessels fabricated from PRD 49-III fibers are 40 percent lighter and stiffer by a factor of two than S-glass vessels of similar design.

4. On the basis of single-cycle burst data, the average composite hoop strains of T-400/epoxy vessels ranged from 0.9 to 1.1 percent and are less by a factor of about three, than hoop strains in S-glass/epoxy vessels (at similar fiber stress levels). Lower values of composite strain would provide a more favorable strain matching condition with thin-metallic liners and would result in a high cyclic life vessel design.

5. Cyclic fatigue test data from filament-wound Thornel 50 graphite fiber composite cylinders indicate that graphite FW cylinders have very high cyclic fatigue characteristics compared to S-glass cylinders of

similar design.

Based on the results summarized in this report, the application of advanced composites for the construction of structurally efficient FW vessels is considered practical and sound. It is believed that the pay-off from the use of composite vessels warrants the continued development of FW pressure vessel technology. It is recommended that larger sized vessels equipped with efficient thin-metal liners be fabricated and tested for mechanical properties and qualification testing.

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TABLE I. - EXTENSIBILITY OF VESSEL AND LINER MATERIALS

Materials	Operating strain, percent	
	75° F	-423° F
S-glass/epoxy 65 percent of US*	2.3	2.5
PRD 49-III/epoxy 75 percent of US	1.9	---
65 percent of US	1.6	---
T-400/epoxy 75 percent of US	1.1	---
65 percent of US	.9	---
HTS/epoxy 75 percent of US	0.7	---
65 percent of US	.6	---
Boron/epoxy (based on 500 KSI fiber strength) 75 percent of US	0.6	---
65 percent of US	0.5	---
Elastomers (maximum elastic strain)	700	0
Mylar (maximum elastic strain)	2.0	1.3
Stainless steel (maximum elastic strain)	1.3	1.6
Aluminum (maximum elastic strain)	0.07	0.1
Inconel X (maximum elastic strain)	0.4	0.47

*Ultimate strength.

TABLE II. - VESSEL TESTING MATRIX

Vessel type	Single-cycle burst	Tests/test temperature	
		Low-cycle fatigue	Sustained loading
Boron/epoxy	70 ^o , -320 ^o , -423 ^o F	70 ^o , -320 ^o , -423 ^o F	70 ^o F
Graphite/epoxy	70 ^o , -320 ^o , -423 ^o F	-----	-----
PRD 49-I/epoxy	70 ^o F	70 ^o F	-----
PRD 49-III/epoxy	70 ^o F	-----	-----
S-glass/epoxy	70 ^o , -320 ^o , 500 ^o F	-----	-----

TABLE III. - FW VESSEL SINGLE-CYCLE BURST TESTING (70° F)

Composite	Vessel diameter, in.	$\frac{P_b V}{W}$ $\times 10^6$ in.	Liner type	Vessel fabricator	Report number
Boron/epoxy	8	0.6 (9)	6 mil stainless	Aerojet	NASA CR-72899
S-glass/PI (Gemon L)	4	1.0 (20)	6 mil stainless	Structural Composites	NASA CR-121139
PRD 49-I/epoxy	4	0.8 (14)	Elastomeric	Boeing	NASA CR-120835
PRD 49-III/epoxy	4	1.4 (1)	Elastomeric	Boeing	NASA CR-120835
PRD 49-III/epoxy	4,8	1.8 (110)	Elastomeric, 20 mil alum.	Lawrence Livermore	(Program in progress)
Graphite Modmor II/epoxy	8	0.5 (12)	6 mil stainless	Aerojet	NASA CR-72652
Graphite T-400/epoxy	8 (major)	0.9 (16)	Elastomeric	Martin-Marietta	NASA CR-120951 (oblate spheroid vessel shape)
Graphite HTS/epoxy	8	0.8 (4)	Elastomeric	Hercules, Inc.	NASA CR-121138
Graphite T-400/epoxy	8	0.9 (4)	Elastomeric	Hercules, Inc.	NASA CR-121138

^aValues based on composite weight only.

All vessels, except where noted, were cylindrical in shape with an L/D of 1.5.

The numbers in the parenthesis are the total number of vessels tested in the given program.

TABLE IV. - CYCLIC TESTING OF FW PRD 49-I/EPOXY VESSELS (70° F)

Vessel specimen design

FW 4-inch diameter, closed-end cylindrical (1.5 L/D), in-plane wrap pattern

Average, single-cycle burst pressure, psig

2155 (5 replicates)

Average, single-cycle $P_b V/W \times 10^6$ inches

0.8

Average, fiber stress, circumferential wrap, ksi

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Vessel number	Cyclic level, % of U.S.	Number of cycles	Burst pressure after cycling, psig	$P_b V/W \times 10^6$ in. after cycling	Fiber stress in circ. wraps, ksi
5	60	^a 1000	2537	1.01	120
6	↓	↓	2792	1.03	121
7	↓	↓	2673	1.11	124
8	↓	↓	2669	.98	129
9	90	1002	----	----	192
10	↓	^a 1000	2122	.81	186
11	↓	38	----	----	195
12	↓	544	----	----	192
15	↓	34	----	----	180

^aTest terminated, vessel pressurized to failure.

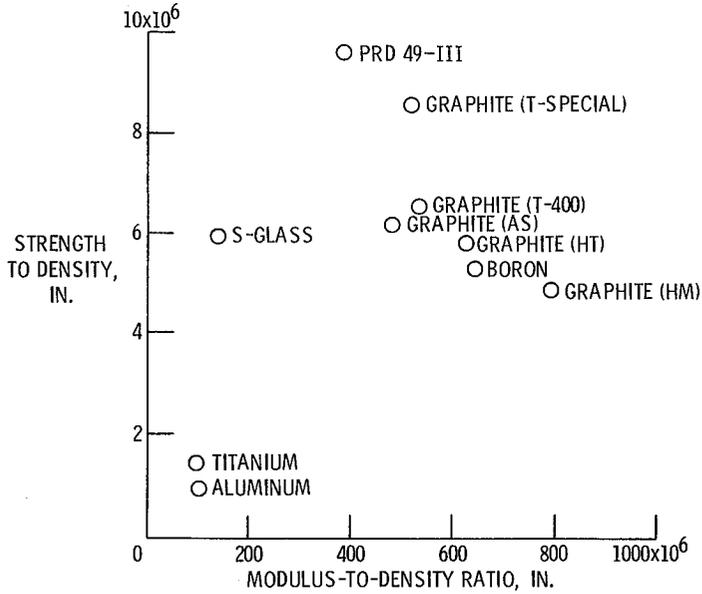


Figure 1. - Strength-to-density and modulus-to-density ratios of fibers based on strand properties.

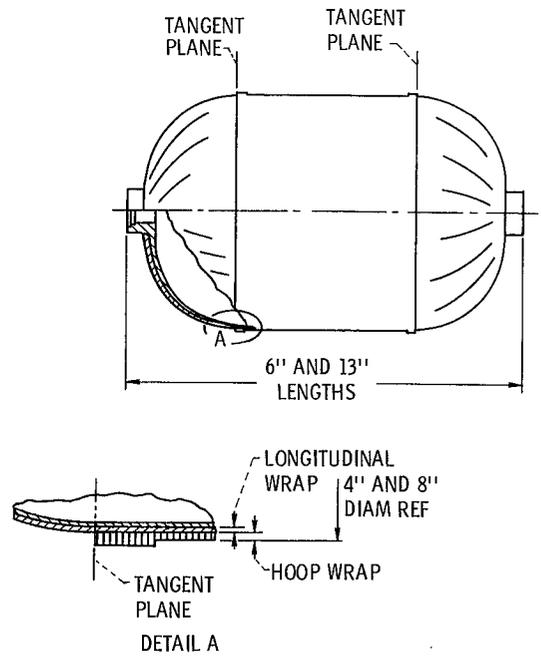


Figure 2. - Schematic of FW cylindrical vessel specimens.

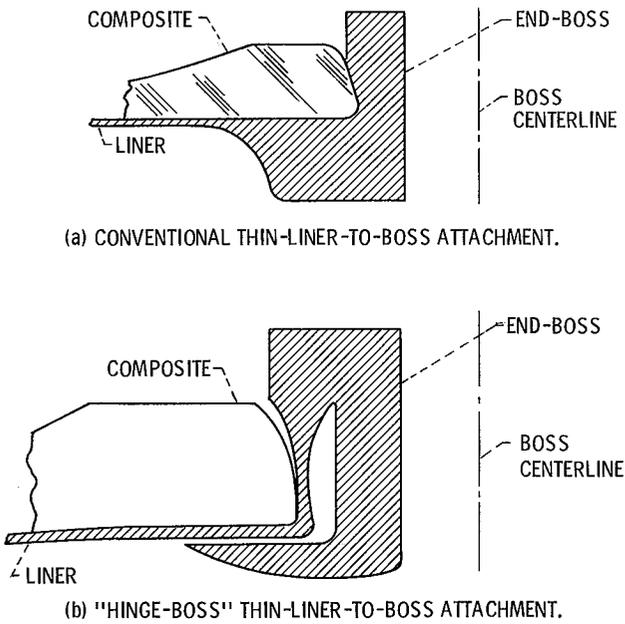


Figure 3. - Sketches of liner-to-boss attachment.

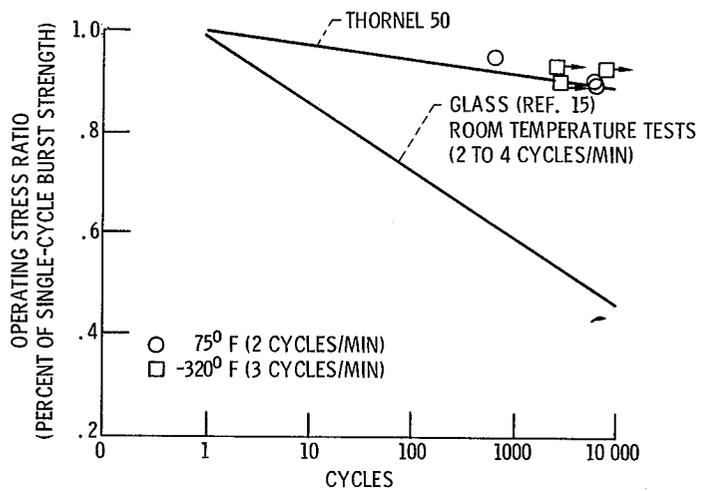


Figure 4. - Comparison of cyclic fatigue of performance glass and graphite FW cylinders.

