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# The Strength of Glass Fibers and the Failure of Filament Wound Pressure Vessels

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From: U.S. Naval Research Laboratory  
To: Distribution list for NRL Report 6034  
Subject: Correction on Table 1B

In Table 1B on page 2 of NRL Report 6034, "The Strength of Glass Fibers and the Failure of Filament Wound Pressure Vessels," by J. A. Kies, Feb. 1964, the compositions of the S and E glasses are exchanged. The correct table is:

Table 1B  
Properties of S Glass and E Glass

Glass	Composition (wt-%)					Young's Modulus (psi)	Density (lb/in. <sup>3</sup> )
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	B <sub>2</sub> O <sub>3</sub>		
S	65	25	-	10	-	$12.2 \times 10^6$	0.90
E	54	14	17.5	4.5	10	$10.5 \times 10^6$	0.92

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## The Strength of Glass Fibers and the Failure of Filament Wound Pressure Vessels

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In glass-reinforced-plastic rockets such as Polaris A3 and third stage Minuteman, the current strength/weight advantage over metals has been achieved in a major degree by utilizing a new glass designated S or S-994. Examination of what is meant by the strength of glass filaments has led to a new method of characterizing the strength, especially for the purpose of exploring new glasses. Since the tensile strength is influenced by flaws and by environmental effects, the strength is therefore strongly dependent on the gage length tested and the time under load. In considering the statistical distribution of fiber strengths more than one population of flaws has been detected. The amount of damage to the fibers in such handling as the making of roving is rather severe, judged by the size or length effect on the strength of filaments. On the other hand with good design this mechanical damage is not reflected in a correspondingly large size effect in the strength of pressure vessels. About the same percentage of the initial virgin fiber strength is achieved in rockets made of E and S glasses, and the rather large discrepancy between virgin filament strength and strength in structure should not be regarded as due to fiber degradation but rather associated with unequal tensioning limitations due to resin, surface finishes, and design factors not yet optimized. It is indicated that a 60-percent increase in the strength of glass fibers and presumably of glass-reinforced plastic could be obtained by suppressing the effects of the moisture apparently always present. It is not reasonable to expect, however, that in large structures the glass stress at failure will ever be the same as the technical upper limiting strength of virgin glass filaments in tested short lengths.

### INTRODUCTION

The present day margin of superiority of filament wound rocket cases over metals on a strength/weight basis on a quantitative scale is in a large measure a reflection of the improvement in glass fiber strengths achieved since early 1962, during which time the production of high-tensile-strength coated fibers designated S glass or S-994 has been brought under close control. It will be shown why the intrinsic strength of the filament as it comes from the bushing does not completely determine the strength of a pressure vessel. This is not unexpected, and the mechanisms of progressive failure in the composite are as worthy of study as is the initial strength of the filaments. Environmental influences have a profound effect, but in practice is seldom noticed because the environments are always apparently about equally bad.

### LIMITING STRENGTH OF GLASS FILAMENTS

Based on atomic forces, a very rough estimate of the limiting strength of a solid, sometimes called theoretical strength, is often quoted as approximately

$$\sigma_t = \sqrt{\frac{E\gamma}{a}} \approx \frac{E}{10} \quad (1)$$

where  $E$  is Young's modulus,  $\gamma$  is the surface energy for the two surfaces, and  $a$  is the lattice spacing. The  $E/10$  value is somewhat arbitrary in that it is assumed that a 10-percent strain will break a lattice bond. Thus the limiting strength expected for glass is between 1 and 2 million psi. A rather concise review of six different suggested theoretical formulas for the strength of solids is to be found in the first chapter of Ref. 1. The various theoretical expressions differ by a factor of as much as 2 in their predictions. Let us consider a more realistic case and assume that a small surface crack larger than the lattice size exists in a glass specimen and that the energy per unit area for a pair of "dry" fracture surfaces at room

Note: This report was the basis for a talk presented at a special meeting of TTCP, Subgroup P, October 16, 1963.

NRL Problem R05-19, U.S. Navy Special Projects Office Task Assignment 71402. This is an interim report on the problem. Work on this and other phases is continuing. Manuscript submitted October 24, 1963.

temperature is given by  $\bar{G}_c = 0.08$  in.-lb/in.<sup>2</sup>, as measured at NRL (2). The "semielliptical" formula provided by Irwin (3) states that

$$E\bar{G}_c = \frac{1.2 \pi \sigma^2 a}{(\varphi^2 - p)} \quad (2)$$

where  $p$  is a plasticity correction not needed here and

$$\varphi = \int_0^{\pi/2} \sqrt{1 - k^2 \sin^2 \varphi} d\varphi$$

in which

$$k^2 = 1 - \left(\frac{a}{c}\right)^2.$$

Here,  $a$  is the crack depth (whereas  $a$  was the lattice spacing in Eq. (1)) and  $2c$  is the crack length open on the surface of the specimen. If the crack is semicircular, as is often the case,  $\varphi^2 = \pi/2$ . If  $2c \gg a$ , then  $\varphi = 1$ . A valid assumption is  $E = 11 \times 10^6$  lb/in.<sup>2</sup>.

Thus the expected strength of moderately dry glass with a semicircular crack open to the surface is (based on  $\bar{G}_c = 0.08$  in.-lb/in.<sup>2</sup>)

$$\sigma_c \approx \frac{6.1 \times 10^2}{\sqrt{a}} \quad (3)$$

At the 500,000 lb/in.<sup>2</sup> strength level, the flaw depth  $a$  would then be at the moment of fast rupture, and including any slow growth during application of the load,  $a = 1.46 \times 10^{-6}$  in. The apparent main role of moisture is to promote such slow growth as "stress corrosion."

This  $a$  is too small for easy seeing, especially on a curved surface; therefore ordinary fracture mechanics formulas are not applied to individually measured flaws in fibers, and we must resort to statistical treatment. Recently we have made significant progress at least experimentally in characterizing the strength of glass filaments.

#### STRENGTH OF FIBERS AS COMPARED WITH GLASS STRENGTHS IN GLASS-REINFORCED-PLASTIC STRUCTURES

Before describing the progress in characterizing the strength of glass filaments, it seems

appropriate to present in Tables 1a and 1b what the glass fiber and rocket industries consider to be the strength of current production fibers. Note that single values are quoted without reference to statistics or scatter. By way of comparison the glass stress at burst is given in Table 1 for other typical composite specimens. A forecast of future performances is given in Appendix A.

TABLE 1A  
Strength of Fibers in Current Production

Fibers	Strength (psi)	
	S Glass	E Glass
Virgin*	700,000	500,000
Strand (roving)	450,000	340,000
4-in. Bottle	400,000	310,000
Motor Case (Polaris 1st Stage)	270,000	225,000

\*Tested in 6-cm lengths.

TABLE 1B  
Properties of S Glass and E Glass

Glass	Composition (wt-%)					Young's Modulus (psi)	Density (lb/in. <sup>3</sup> )
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	B <sub>2</sub> O <sub>3</sub>		
S	54	14	17.5	4.5	10	12.2 × 10 <sup>6</sup>	0.90
E	65	25	-	10	-	10.5 × 10 <sup>6</sup>	0.92

At the moment we shall examine briefly what is meant by the size effect on strength. Many investigators have already reported on the statistical nature of the strength of glass in massive as well as in fiber specimens. For details of such studies there are provided several references at the end of this paper (4-10). It is convenient now to mention only one of the many equations suggested in the past, because within reasonable limits it readily permits us to estimate the size effect and that is one of the most obvious differences between a short length of filament and a useful engineering structure such as a Polaris first stage rocket case containing about 400 lb of glass fibers.

Weibull (10) proposed that the cumulative probability of rupture for an applied stress rising from zero to failure could be expressed as

$$S = 1 - \exp\left(-\int_0^v B_0 dv\right) \quad (4)$$

where  $v$  indicates volume and

$$B_0 = \left( \frac{\sigma}{\sigma_0} \right)^m$$

in which  $\sigma$  is the applied tensile stress and  $m$  is the Weibull coefficient. Here  $\sigma_0$ , although originally intended to be related to upper limiting strength, becomes rather useless operationally because for  $S \rightarrow 1$ ,  $\sigma \rightarrow \infty$  in Eq. (4). In a previous paper (5), the author suggested using

$$B_0 = \left( \frac{\sigma - \sigma_u}{\sigma_0 - \sigma} \right)^\alpha \quad (5)$$

and it was shown that the size effect data then in the literature could be better fitted for constant  $\alpha$ , because all known data indicated that a finite upper  $\sigma$  limit was being approached as the specimen size decreased. In Eq. (5),  $\sigma_u$ , the lower limit, is practically zero. This formula (5) is however inconvenient for purposes of calculation. The Weibull formula of Eq. (4) was shown by Irwin (8) to provide us with the convenient aids to calculation given as

$$\frac{\bar{\sigma}_1}{\bar{\sigma}_2} = \left( \frac{V_2}{V_1} \right)^{1/m} \quad (6)$$

where  $\bar{\sigma}_1$  is the average strength of specimen of linear size 1,  $V_1$  is the specimen volume, and the Weibull coefficient is

$$m = \sqrt{1.5/\eta} \quad (7)$$

in which  $\eta$  is the relative standard deviation. Volume distribution of flaws is assumed; if, as in glass in tension, only surface flaws are important, the volume is replaced by surface area in the equations. For relatively undamaged virgin glass  $m \approx 50$ ; for moderate damage  $m \approx 6$ , as it is for natural minerals such as coal and limestone. For nonuniform stress Eq. (6) is much more complicated, as shown in Ref. 5.

For example, for the case of a circular-cross-section beam in three-point loading the effective area  $A_{eff}$  for surface flaws is (for radius  $r$  and span  $L$  of the cylinder)

$$A_{eff} = \frac{2 r L}{m+1} \frac{[2 \cdot 4 \cdot 6 \cdots (m-1)]}{(1 \cdot 3 \cdot 5 \cdots m)}, \quad (8)$$

if  $m$  is an odd integer

and

$$A_{eff} = \frac{\pi r L}{m+1} \frac{[1 \cdot 3 \cdot 5 \cdots (m-1)]}{(2 \cdot 4 \cdot 6 \cdots m)},$$

if  $m$  is an even integer. (9)

Again,  $m$  is the Weibull coefficient. For volume distributed flaws the effective volume for a cylindrical beam in three-point loading is

$$V_{eff} = \frac{2 r^2 L}{(m+1)(m+2)} \frac{[2 \cdot 4 \cdot 6 \cdots (m-1)]}{(1 \cdot 3 \cdot 5 \cdots m)}, \quad \text{for } m \text{ odd} \quad (10)$$

and

$$V_{eff} = \frac{2 r^2 L}{(m+1)(m+2)} \left\{ \frac{\pi [1 \cdot 3 \cdot 5 \cdots (m-1)]}{2 (2 \cdot 4 \cdot 6 \cdots m)} \right\}, \quad \text{for } m \text{ even.} \quad (11)$$

For a rectangular parallelepiped in three-point loading as a beam the effective area is

$$A_{eff} = \frac{L}{m+1} \left( b + \frac{D}{m+1} \right) \quad (12)$$

where  $L$  is the span,  $b$  is the width, and  $D$  is the depth of the beam.

Let us now refer back to Table 1 and see how Eq. (6) can be used as a means of evaluating the performance of glass in the tests. In order to do this, Fig. 1 was prepared, in which the average stress in the glass filaments at failure was plotted against the weight of the glass. In going from the single filament 6 cm long to the Polaris first stage, we cover about 13 decades of weight on the log scale. The breaking stress is also plotted on a log scale, and the slope gives the Weibull  $m$  value according to Eq. (6). It can be seen to our satisfaction that the  $m$  value for the small specimens is in the range 25 to 50, which is characteristic of high quality material without obvious manufacturing and design defects. Between the 4-inch bottle and the Polaris first stage,  $m$  is about 20, which is much greater than 6 and therefore much better than if we were scaling up the defects in proportion to the specimen size. In other words defects in Polaris have been scaled down from those in the 4-inch bottle. It should be emphasized that when  $m = 6$  the linear size of the

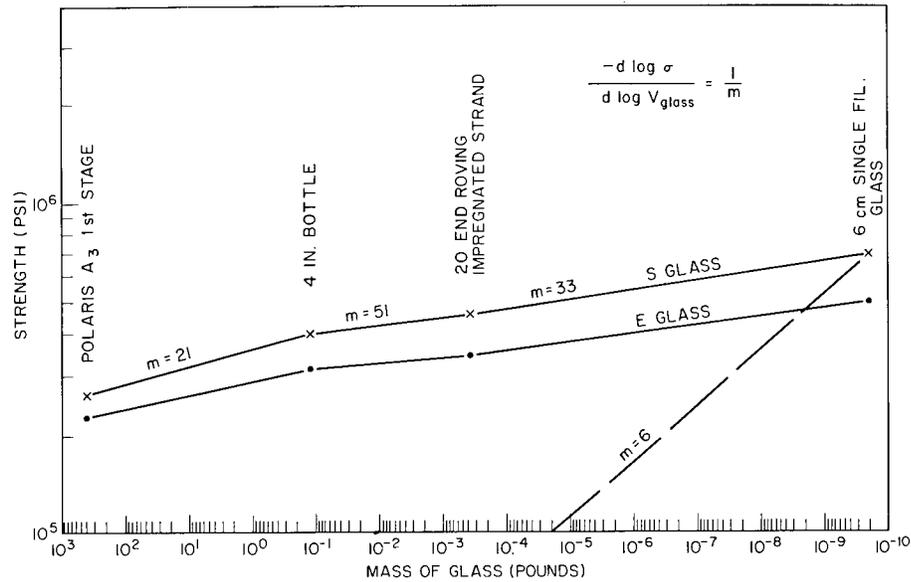


Fig. 1 — The size effect showing the glass stress at failure vs weight of glass fibers for a wide range of sizes of specimen

worst defect is proportional to the linear size of the test piece. In Fig. 1 we have shown a dashed line for  $m = 6$  connected to the point representing the 6-cm filament of S glass. For S glass which has been made into roving and then single filaments removed for test the mechanical damage results in  $m \approx 6$ . We conclude from this that the mechanical damage to the glass during processing is far less deleterious in a composite structure than it is to the average strength of the filament itself. Reasons for the easy tolerance for fiber damage lie in the structure redundancy of the material and the fact that the resin effectively transmits the stress around the breaks if there are not too many bunched in one place.

It is contrary to our experience with high-quality structural materials to expect  $m$  to exceed 50; therefore it seems possible that the glass stress in Polaris might reach 340,000 psi as an upper limit through extreme refinement in design and manufacturing and that 290,000 psi (for  $m = 25$ ) would be a more reasonable goal.

The sober warning to be drawn from all this is that we cannot go to indefinitely larger structures of glass-reinforced plastic and expect the average strength of 700,000 psi as measured in a single filament. The size effects just discussed have never been explicitly incorporated in design studies by structural engineers; however, it is worth

our while to consider them as a lesson taught by experience.

Results like those of Fig. 1 are sometimes interpreted to mean that we are not utilizing the full strength of the glass (say 700,000 psi) in a structure with breaks at a "glass stress" of 250,000 psi. This erroneous interpretation results from not distinguishing between nominal average stress and the local stress at the origin of failure, which could approach very close to the upper value. Nonuniform tensioning, built-in stress raisers, and bad interlaminar shear conditioning are largely responsible for the discrepancy between virgin filament strengths and the nominal strengths at failure in the structures. We see in Fig. 1 that S glass gives greater strengths by about the same percentage for all the different test specimens. We should conclude that future increases in the strength of glass would be reflected to a degree in the strength of structures, and in that sense we are using and will use the full strength of the glass.

#### RECENT EXPERIMENTS ON THE SIZE EFFECT IN GLASS FIBERS

In the foregoing no attempt has been made to be thorough, since extensive literature on the statistics of strength already exists. We shall now

consider new experiments, because we have evidence to indicate that all statistical formulas used for curve fitting in the past do not seem adequate for the exploration of new glasses in fiber form. Recent progress has depended on the development of refined techniques for testing a wider range of filament lengths, especially shorter ones than have been reported on in previous literature. Bear in mind that the immediate purpose is to explore and compare new glasses.

On a contract to NRL (Nonr-3654(00)(X)), G. Schmitz has developed a tensile testing device in which the ends of two vertical steel cylinders about 0.5 inch in diameter are capped with a special wax.\* The filament is laid across the two caps of melted wax, and after cooling, the filament is pulled at 0.06 in./in./min. The gage length

is taken as the free uncoated length of filament between the pulling heads. The equipment is shown in Figs. 2 and 3.

In previous publications, with one known exception, rather long lengths were always tested so that a population of gross flaws was always represented by some of its members in every test specimen. This seemed to fit not too badly into formulas which assumed a single population such as a Gaussian or Weibull distribution. Note however in Fig. 4 what happens when we accumulate a respectable amount of data on virgin E glass only 0.25 cm long. Here, probability of failure vs stress is shown. Remember that the standard tables give data for 6 cm. In Fig. 4 we believe that two distributions are indicated, each of which could be Gaussian. Note also that the upper strengths measured in population B are substantially better than the ones advertized, *e.g.*, in Table 1 of this paper. Further experiments were

\*Wax 3066 composition by High Test Chemical Corp., 722 64th St., Brooklyn.

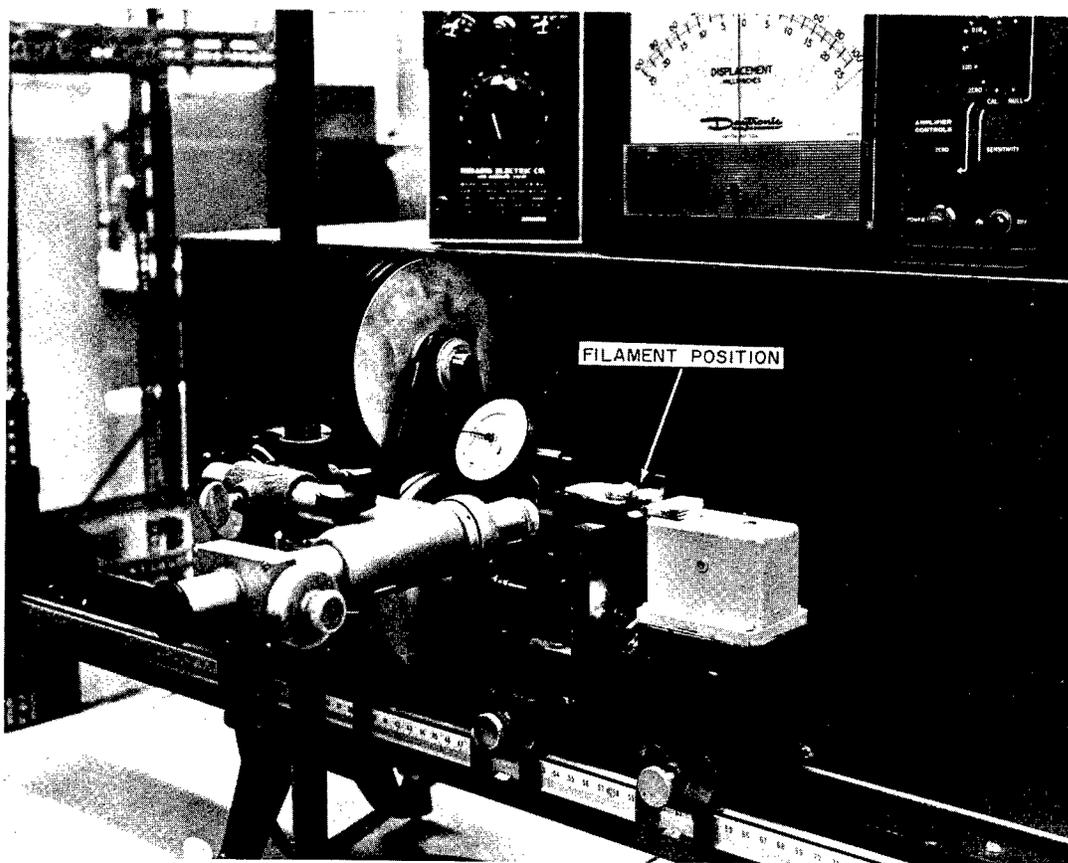


Fig. 2 — Single filament tester developed by Solar for a wide range of filament lengths including very short ones

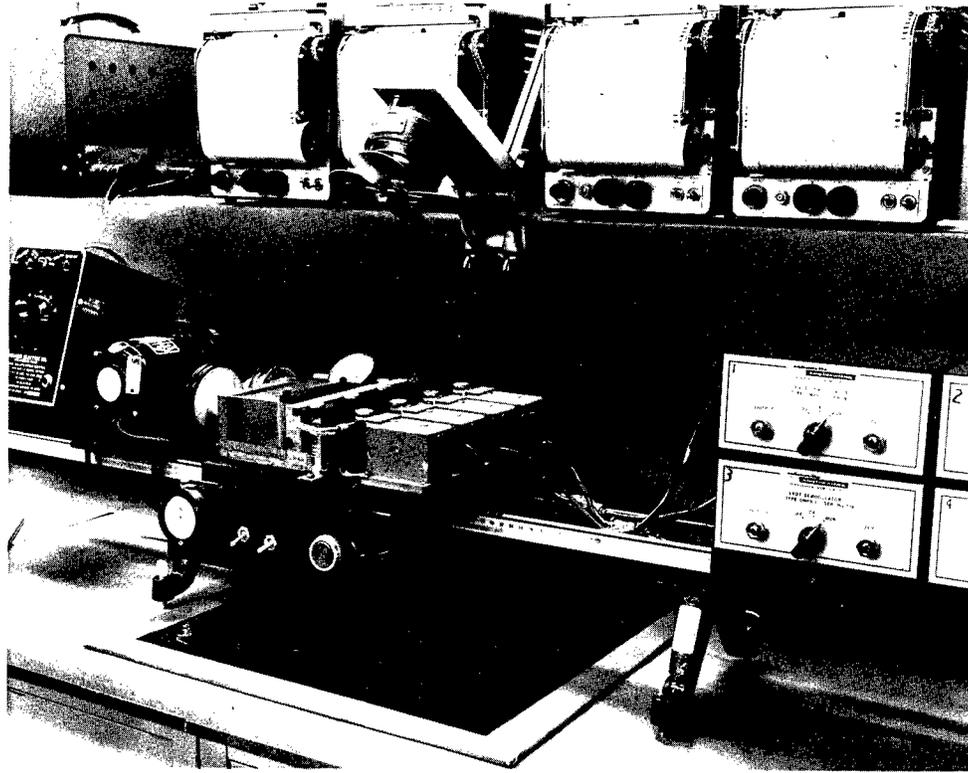


Fig. 3 — Multiple filament test unit for testing very short fibers

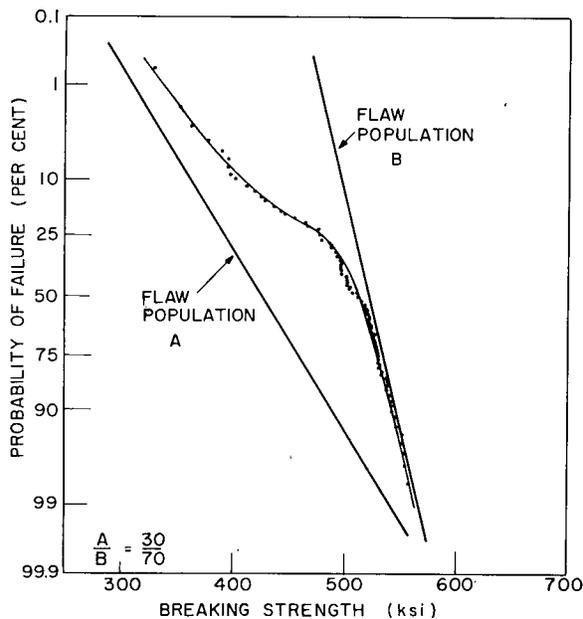
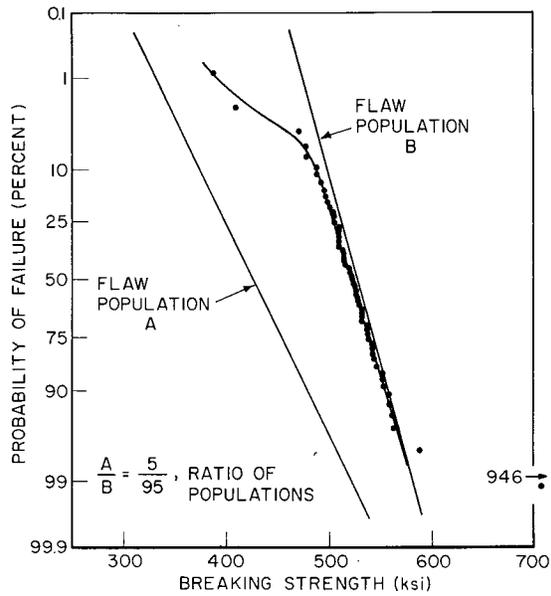


Fig. 4 — Probability plot for E glass fibers 0.25 cm long. A bimodal distribution is suggested.

done on filaments only 0.05 cm long as shown in Fig. 5. Here it is seen that most of the tests are now dominated by population B and that a few specimens are too strong to qualify for membership in either A or B.

The situation for S glass is shown in Figs. 6-8. Figure 6 gives the tensile strengths of 8 cm long specimens about the same as were used for Table 1. It seems that in Table 1 population A has been ignored. In Fig. 7 giving results for 1-cm specimens more of the results shift to the B curve, and in Fig. 8 for 0.05 cm a still further shift to the B population is seen with some evidence for a few extra strong nonconforming members. The size effect and scatter are shown directly for S glass in Fig. 9. Note the sharp break in slope at 1 cm length. Figure 9 provides an example in which the Weibull coefficient  $m = 8$  applies for lengths greater than 1 cm, whereas for shorter lengths almost no size effect can be detected; thus  $m$  would have to be very large, say 50, to represent the short test lengths.



ANALYSIS OF BI-MODAL FAILURE DISTRIBUTION

Fig. 5 — Probability plot for E glass fibers 0.05 cm long. A bimodal distribution is suggested.

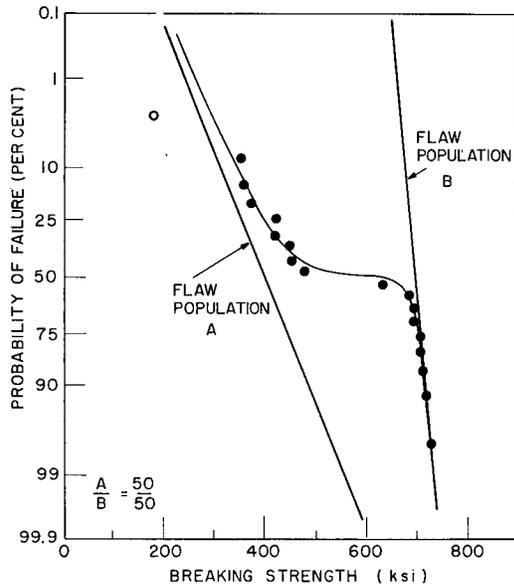
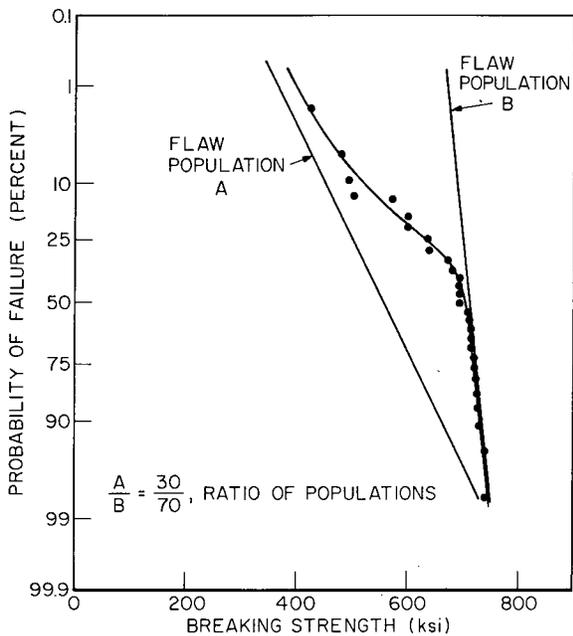
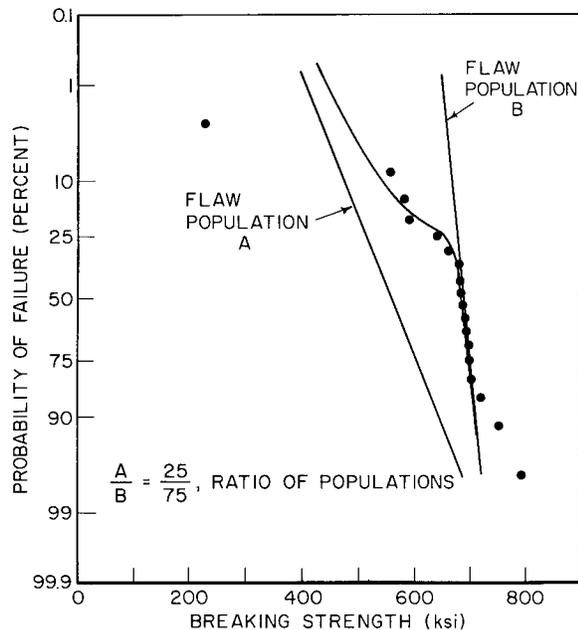


Fig. 6 — Probability plot for S glass fibers 8 cm long



ANALYSIS OF BI-MODAL FAILURE DISTRIBUTION

Fig. 7 — Probability plot for S glass fibers 1 cm long



ANALYSIS OF BI-MODAL FAILURE DISTRIBUTION

Fig. 8 — Probability plot for S glass fibers 0.05 cm long

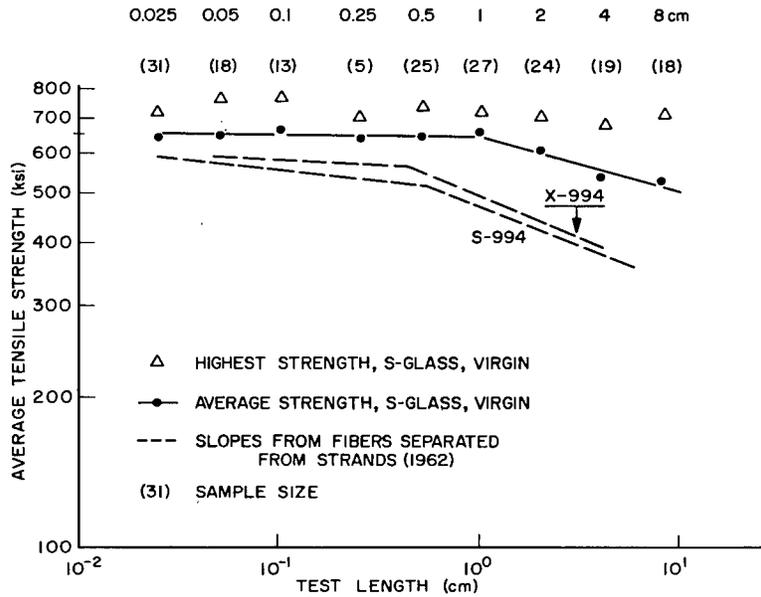


Fig. 9 — The size effect and scatter for S glass fibers

We are beginning a study of how mechanical and moisture damages affect the different flaw populations in the Solar tests (11,12). It will be the latter part of 1964 before much data will be ready for release on this subject; however, if the different flaw populations turn out to be physically different in kind as well as degree, then we may have an improved method of determining, for example, how moisture or surface finishes affect or preserve the strength of glass. A bimodal distribution was found by Cornelissen and others for rods (4), but no apparent use was made of this discovery other than to connect it with its cause in manufacturing the glass. Cornelissen found that a certain step in the process introduced a separate flaw population.

At present we can at least say that bare E glass has in one sense been shown stronger in air than it is advertized to be, whereas for bare S glass the advertized strength in air is close to the upper values obtained for the stronger members of the population B. Preliminary but unreported results indicate that the act of making a single end roving (of high-tensile-strength coated fibers and removing single fibers for test) degrades the strength of the fibers considerably below the virgin strengths. This degradation of average strength has been found at Solar to be the same percentage for S and E glasses. The degradation is a measure of

the abrasion and the protection or lack of it provided by the high-tensile-strength finish. It is suggested here that the lubricating value of a finish be judged in accordance with the  $m$  value for abraded fibers.

The main emphasis in the study we are beginning will be on new high modulus fibers and on the effects of moisture exposure before and during testing. At this time the question of how to make an engineering application of these test results to the prediction of the strength of structures is deferred.

#### EFFECT OF MOISTURE ON SHEAR FRACTURE IN THE RESIN OF GLASS-REINFORCED PLASTIC

We have already mentioned that moisture can degrade the strength of glass, probably by a stress corrosion effect in which slow growth of surface cracks occurs during time under load if moisture can penetrate the cracks. The puzzling thing about this to most people is that a bad effect of moisture on the tensile strength of laminates is not easy to demonstrate without a drastic treatment such as by boiling. There are at least two possible reasons for this difficulty: first, some moisture is always present in ordinary test samples, and secondly, failure of a laminate is partly a

resin failure and partly a resin glass debonding failure. The resin and the resin glass bonding are not necessarily adversely affected by water, at least in short time tests.

For example, at NRL  $G_{III}$  type shear fractures made using slotted hoops of E high-tensile-strength glass in Epon 828 CL indicated (Table 2a) with practically 100-percent confidence that the stiffness for shear was increased by an immersion for 3-1/2 months and that the fracture strength in shear was not degraded significantly from those specimens stored in a dessicator for the same time. On the other hand storing dry for 3-1/2 months degraded the shear fracture strength to a 94-percent confidence level as compared with only 11 days dry storage. Those stored dry for 3-1/2 months were weaker in shear fracture resistance than those stored in water but with only an 88-percent confidence level. Thus if we omit consideration of the glass strength we have shown in preliminary tests that a moist environment does not generally impair the shear fracture properties in the resin of the composite. In Table 2b the specimens of Table 2a were slightly modified by sawing only one slot. The results are somewhat contradictory and without obvious reason. This illustrates the need for further study. Such discrepancies are typical of the published literature

on the effects of moisture. The treatments and confidence levels of the test results are shown in Tables 2a and 2b. This is new data using non-standard tests. Further more refined explorations will soon follow in our BuShips program.

The fact that moisture can have a drastic effect on the strength of glass is demonstrated in Fig. 10. These data obtained by General Electric on BuWeps contract Now61-0641-c are for bare E glass filaments drawn in air at room temperature tested in ordinary air and tested in nitrogen vapor at liquid nitrogen temperature. Here we see a dramatic effect of the suppression of moisture effects on the strength. In fact, the strength was 60 percent higher at lower temperature, -196°C. In pursuing this idea the G.E. group tested 4-inch-diameter epoxy composite rings at the same two temperatures and arrived at modal strengths of 500,000 psi and 310,000 psi respectively for the glass stress at failure at -196°C and room temperature. This is the same percentage difference as for the filaments. Since we can assume that stress corrosion by moisture was suppressed by the low temperature to the same extent in the filaments and composite rings, then we must attribute the difference between filament and ring strengths to mechanical damage and nonuniformity of tensioning. The nonuniformity in

TABLE 2A  
Results of Statistical Analysis of Double Slot Beam Tests, Shear  $G_{III}$

Lot No.	Description	$\bar{P}_{max}$ , Strength	$\Delta\bar{P}/\Delta\delta$ , Stiffness						
1	11 days at 100°F and 20% RH	27.3 lb	424.6 lb/in.						
2	11 days at 110°F and 98% RH, cycled to 20% RH, 3 days apart	26.5 lb	426.5 lb/in.						
4	Stored dry 3-1/2 months	23.8 lb	424.2 lb/in.						
5	Stored in water 3-1/2 months; air dried 24 hr before test	26.6 lb	441.1 lb/in.						
Level of Significance Between Means									
		$\bar{P}_{max}$ , Strength				$\Delta\bar{P}/\Delta\delta$ , Stiffness			
		1	2	4	5	1	2	4	5
1		-	0.55	<b>0.94</b>	0.45	-	0.58	0.10	<b>0.99</b>
2		0.55	-	0.88	0.07	0.58	-	0.50	<b>0.94</b>
4		<b>0.94</b>	0.88	-	0.88	0.10	0.50	-	<b>1.00</b>
5		0.45	0.07	0.88	-	<b>0.99</b>	<b>0.94</b>	<b>1.00</b>	-

TABLE 2B  
Results of Statistical Analysis of Single Slot Beams ( $\bar{Q}_{III}$ )  
(All specimens loaded on the slotted half only)

Lot No.	Description	$\bar{P}_{max}$ , Strength				$\Delta\bar{P}/\Delta\xi$ , Stiffness			
3	Tested immediately (control group)	33.4 lb				514 lb/in.			
6	Stored dry 3-1/2 months	37.3 lb				545 lb/in.			
7	Stored in water 3-1/2 months, air dried 24 hr before testing	38.0 lb				527 lb/in.			
8	Stored in water 3-1/2 months, tested wet	31.8 lb				511 lb/in.			
Level of Significance Between Means									
		$\bar{P}_{max}$ , Strength				$\Delta\bar{P}/\Delta\xi$ , Stiffness			
		3	6	7	8	3	6	7	8
3		—	<b>0.95</b>	<b>0.98</b>	0.62	—	<b>0.99+</b>	0.84	0.15
6		<b>0.95</b>	—	0.22	0.98	<b>0.99+</b>	—	0.89	0.96
7		<b>0.98</b>	0.22	—	<b>0.99</b>	0.84	0.89	—	0.69
8		0.62	<b>0.98</b>	<b>0.99</b>	—	0.15	<b>0.96</b>	0.69	—

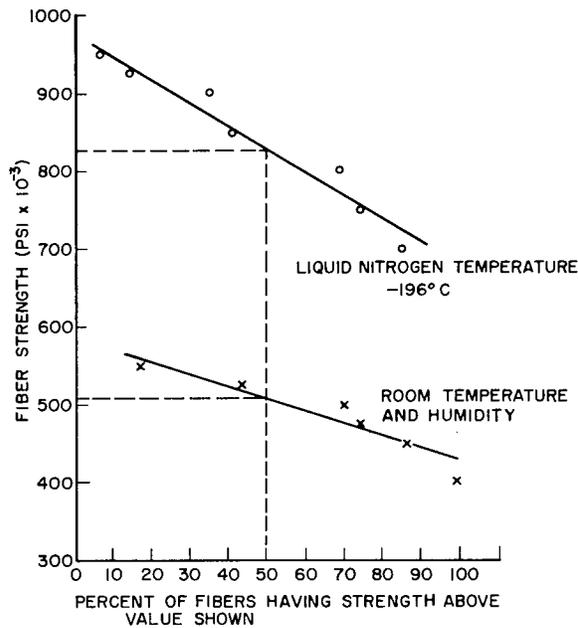


Fig. 10 — The suppression by low temperatures of moisture effects on the tensile strength of E glass fibers

tensioning does not all come from imperfection in the roving materials and manufacture of the rings; also, mechanical damage to the glass can develop as the mechanical test proceeds. Fixtures

for testing rings do not usually achieve a perfectly uniform tension on the rings. For example, we need to make a correction for bending in the split D ring test and for some damage to the ring by shear cracking during test.

It is clear that to get the moisture low enough in engineering structures to realize the 60-percent improvement implied by the G.E. work, rather heroic measures would have to be adopted. It is not clear that this is practically possible. Metallic coatings may yet be the answer, although to date they have been of little value for glass. A review of a considerable body of reports on metalized coatings has recently been issued (13). Any new research to be undertaken on metal coatings should somehow be different in approach, and accomplishing new research different in approach will not be easy.

### THE LOOP TEST

Although the testing of glass filaments by pulling a loop is not new, good instrumentation for performing the test has only recently been provided. Figure 11 shows schematically the apparatus developed by R. Trimble of the University of North Carolina on NRL contract Nonr-3605(00).

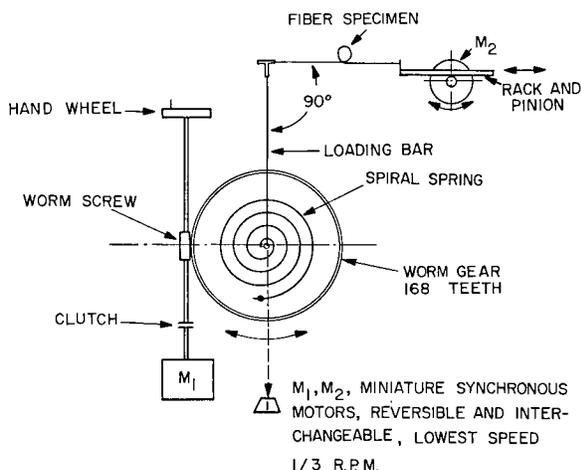


Fig. 11 - Loop test apparatus

The purpose is to measure the strengths of extremely short gage lengths in bending and to avoid the uncertainty in gage length due to possible slippage in the grips. At the present, we have no test results to report. In making the loop test we have our choice between measuring the diameter of the loop at fracture or of measuring the tension in the fiber. The maximum stress  $\sigma_m$  calculated for the top of the loop is given by Irwin (14) as

$$\sigma_{max} = \frac{4}{r} \sqrt{\frac{ET}{\pi}} \quad (13)$$

and the effective length is given as

$$L_{eff} = \frac{r^2}{2m} \sqrt{\frac{\pi E}{T}} \quad (14)$$

where  $r$  is the radius of the filament,  $E$  is Young's Modulus for the glass,  $T$  is the tensile force on the filament, and  $m$  is the Weibull coefficient, which may be estimated from the standard deviation formula (Eq. (7)).

The equations for the elastica (natural shapes rods when loaded) were also previously solved by McFadden of NRL in an unpublished paper, and  $\sigma_m$  was expressed by him as a function of the loop diameter. Experimentally this has been unsatisfactory in that an accurate knowledge of the diameter at failure was not easy to obtain. Measurement of  $T$  is recommended. The effective

length of filament in the loop test is approximately 0.01 cm.

### FAILURES IN FILAMENT WOUND PRESSURE VESSELS

We have more than an academic interest in the strength of glass fibers. We really want to know to what extent their phenomenal strength is usable in a composite structure. This calls for some knowledge of how things fail, and that is not a simple matter of tensile failure in filaments. Before discussing failure modes let us consider further where we stand in structural efficiencies of rockets.

Table 3 compares various large solid rockets on the basis of proof pressure times volume divided by weight of the motor case. This is not an indication of burst pressure. In this table we see that for the Polaris A2 second stage the structural efficiency of the glass-reinforced plastic was not particularly high.  $E$  glass and a conservative design were in use. For the Minuteman third stage the PV/W index is almost the same as for the two metal stages and better than the Polaris A2 second stage. This table represents current achievement with  $S$  glass. Polaris A3 in both stages uses improved designs and  $S$  glass. Here we can note that in Polaris the glass-reinforced plastic outperforms all the other rockets listed. This does not represent an optimization but rather an advanced practical solution that met a deadline.

TABLE 3  
Comparison of Motor Cases  
of Large Solid Rockets

Motor Case	Material	PV/W (in.)
Polaris A1 2nd	Steel	$0.12 \times 10^6$
A2 2nd	GRP*	0.14
Typhon	Steel	0.18
Pershing 1st	Steel	0.18
2nd	Steel	0.20
Minuteman 3rd	GRP	0.23
2nd	Ti	0.25
1st	Steel	0.25
Polaris A3 2nd	GRP	0.29†
A3 1	GRP	0.33†

\*Glass-reinforced plastic.  
†S glass.

In considering failures we should realize that there are terminating ends of glass in a filament winding, nonuniformities in tensioning, and a variety of irregularities in manufacturing so that stress raisers exist. In addition, we have the certainty of some degradation in strength of the glass itself due to handling and processing of the motor case. The possible effect of moisture on the strength of glass-reinforced plastic has been extremely elusive to determine. Much conflicting data exist, and work is continuing at NRL. Thus far it appears that for rocket cases no appreciable degradation on storage for several years is anticipated.

As an illustration of progressive failure, Fig. 12 gives a photo taken early during a 3-minute hold at constant pressure of a 6-inch-diameter pressure vessel. The outer windings are in the hoop direction. During this hold, more and more small strands broke or came loose by unwinding, so that the wall was gradually losing material and presumably strength during the hold time. After 3 minutes of such continuing unwinding the pressure was raised to produce sudden failure. This type of unwinding failure has been described elsewhere (15-17) and the glass stress for its beginning depends on the number of fibers broken in a bundle and on the resistance to the propagation of a shear crack in the resin. A typical value of  $Q_{IIc}$  for such shear cracking is 5 to 8 in.-lb/in.<sup>2</sup> for current epoxy and glass combinations.

Filament wound pressure vessels are vulnerable to cuts or scratches and the residual strength is not determined by the strength of glass so much as it is by the toughness of the resin and the winding pattern or lay-up. This has been discussed in a previous paper (15). At present consider Fig. 13 in which the pressures for the beginning of unwinding failure and the burst pressures are shown for bottles containing surface cuts. In the undamaged state both kinds of bottles had the same strength, but in the cut condition this was not the case.

The effect of time under load is important and especially drastic if there is a cut. Figure 14 shows how the burst strength of layered (the superior kind) bottles is affected by time under load. The curve marked fast test is for bottles pressurized at such a rate as to cause burst in 30 seconds for undamaged ones. Figure 15 shows typical burst bottles in this series. In a numerical analysis it

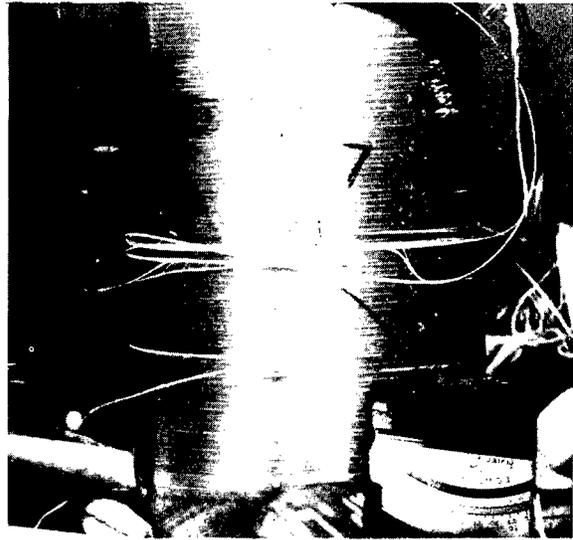


Fig. 12 — One step in the progressive failure of a 6-inch chamber under internal pressure

was shown that a law as follows would fit the results:

Assume that the burst strength  $\sigma$  is proportional to the square root of a damage zone size  $a$ , and that during loading the time rate of increase of  $a$  is

$$\frac{da}{dt} = k \sigma^n a. \quad (15)$$

When  $n$  was set at 2, all of the differences between upper and lower curves due to time under load could be fitted satisfactorily. This was not the case however with tests by Outwater (18) on NRL contract Nonr-3219(01)(X). Outwater used isotensoid geodesic ovaloid bottles without cuts and found the burst strength to be expressed as

$$\sigma_b^2 = \frac{k_2}{a_0 + k_1 \int_0^t \sigma_1^n dt} \quad (16)$$

where  $t$  is time and  $\sigma_1$  is stress during hold prior to burst. Outwater found  $n$  to be about 25, too large to be considered a fracture mechanics effect but rather a chemical or stress corrosion effect. Such a law was previously found with  $n=17$  by Charles (19) of G.E. for glass rods. Figure 16 shows Outwater's results. We are thus confronted with evidence which tells us that failure mechanisms are not simply defined and cataloged for glass-reinforced plastic and that the strength of

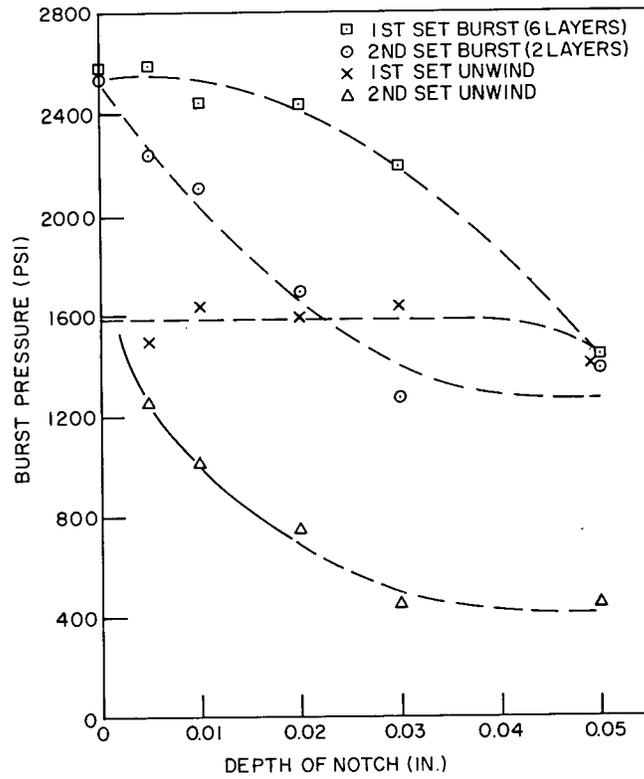


Fig. 13 - Pressure vs cut depth for the beginning of visible failure and for final burst in 6-inch bottles

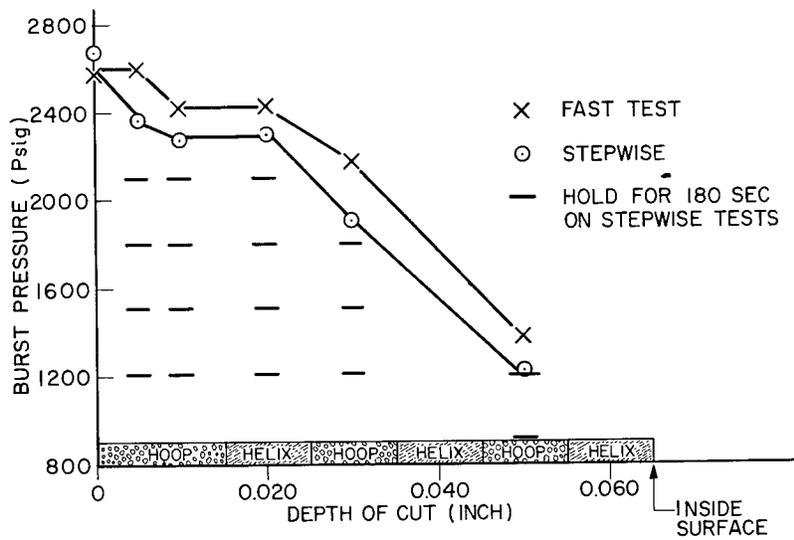


Fig. 14 - The effect of time on burst strengths of six 6-inch-diameter bottles with layered windings containing cuts



Fig. 15 — Appearance of bursts in cut bottles

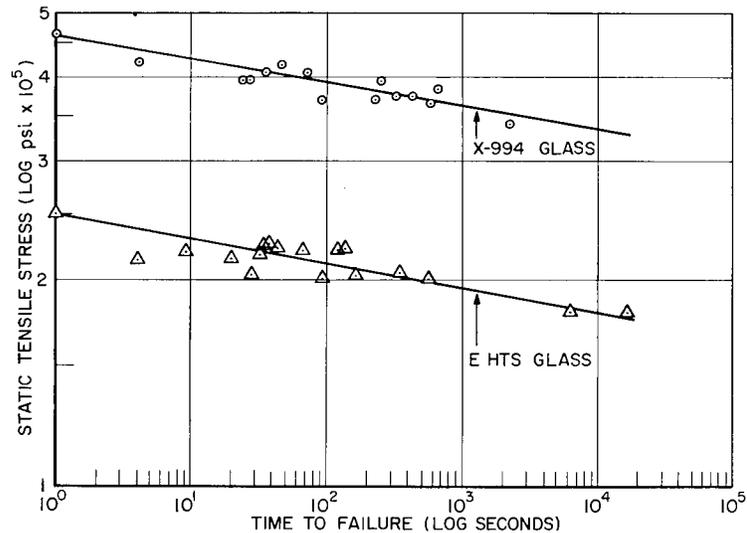


Fig. 16 — Effect of time under load on the strength of Outwater geodesic isotensoids

the glass is only one of many factors determining the strength of a pressure vessel. In this presentation no mention has been made of the role of the resin. This is an extremely important part of NRL research, especially research for the Bureau of Ships. After hydrotest every Polaris contains millions of cracks in the resin. An example is shown in Fig. 17. We have reason to believe that such cracks lay open the glass to environmental attack, especially by moisture.\* The strain magnification in the resin is very large and has been described

\*See Appendix A.

in a previous paper (20). The contribution of the resin strength to the mechanical strength and stability of dimensions of the structure are of great importance. We are only beginning to build from the ground up our knowledge of the role the resin plays in progressive failure.

In conclusion let us emphasize the fact that the words "strength of glass filaments" have a different meaning depending on the test size, testing speed, and environment. Almost nothing is known about how to relate the tensile strength of filaments directly to the strength of a large glass-reinforced-plastic structure except by

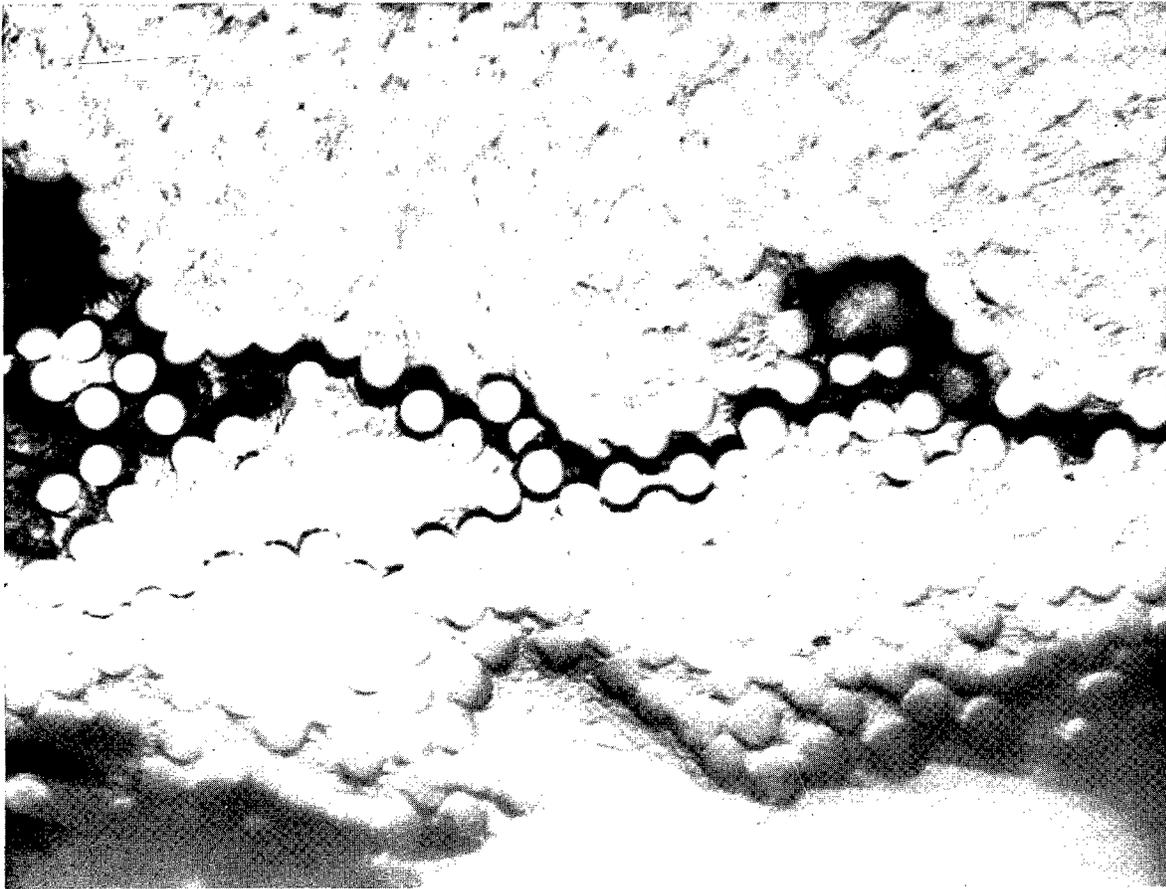


Fig. 17 — Resin cracks greatly magnified. The glass rods are about 0.0004 in. in diameter.

empirical methods. We believe, however, that recent improvements in the strength of filaments have almost all carried over into the strength of solid rockets. In the next year or two we are led to expect that U.S. industry will be able to produce glass fibers above 800,000 psi in strength and that even higher strength will come later. Mechanical damage to the fibers is less important than uneven tensioning, etc. In a word of caution, however, let us realize that to fully utilize such high strengths with current values of elastic modulus or stiffness of the glass the strains in service would have to be too high for compatibility with metal parts and dimensional tolerances dictated by present operations. It is therefore suggested that the emphasis on near future glass fiber research should be mainly on obtaining higher modulus glass without beryllium, and that problems of design and testing for structural compatibility be kept in mind by those planning

materials research. In our brief reference to mechanisms of failure in glass-reinforced-plastic structures we have omitted several important failure mechanisms and causes of failure which have been discussed in previous papers (21-23). These have been the result of errors, flaws, and characteristic inherent structural weaknesses not directly associated with the tensile strength of the glass.

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## APPENDIX A

## FORECAST OF FUTURE PERFORMANCES

## AN INDUSTRIAL FORECAST

As a guide in deciding what areas of materials research and development will be most rewarding, a copy of an industrial forecast is given as Fig. A1. In this figure the highest usable strength/density ratio is given for the material. For filament winding the numbers refer to filament reinforced plastic, not to the filaments alone. It is seen that glass-reinforced plastics are regarded with great optimism. Cost has not been considered.

## OTHER STRONG FIBERS\*

Metal filaments now producible in continuous form are listed in Table A1. Here the strength values are quoted from various sources. The test results have not been given with adequate detail for purposes of fully describing what uncertainties may exist.

\*H. Bernstein, "Materials for Advanced Solid Propellant Motor Cases," Interagency Solid Propellant Meeting, Seattle, Washington, Sponsored by Johns Hopkins Applied Physics Lab., July 1963

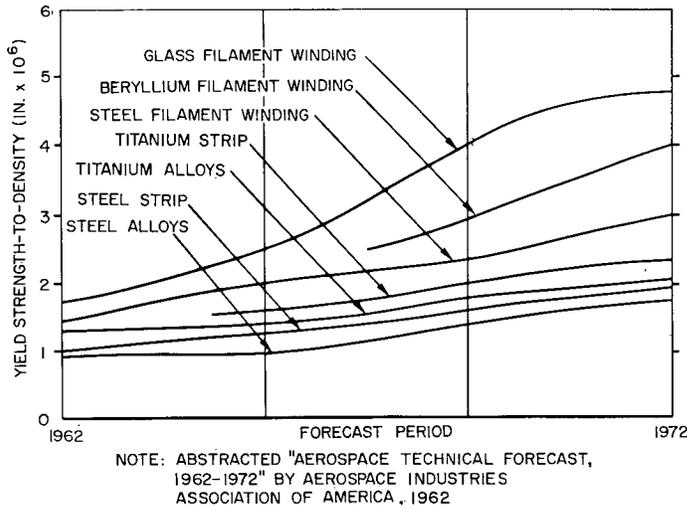


Fig. A1 - Industrial forecast of the performance of glass reinforced plastics and other materials for rocket cases

TABLE A1  
Strengths of Continuous Metal Filaments

Metal	Tensile Strength (psi)	Density (lb/cu in.)	Strength/Density (in.)	Modulus (psi)
Boron	400,000	0.094	$4.3 \times 10^6$	$55 \times 10^6$
Beryllium	190,000	0.067	$2.7 \times 10^6$	$44 \times 10^6$
Steel	600,000	0.282	$2.1 \times 10^6$	$30 \times 10^6$
Titanium (B120VCA)	270,000	0.170	$1.6 \times 10^6$	$15 \times 10^6$
Tungsten	700,000	0.695	$1.0 \times 10^6$	$50 \times 10^6$

**EFFECT OF MOIST ENVIRONMENT ON PRESTRAINED 4-INCH-DIAMETER PRESSURE BOTTLES**

It has not been easy to find clear evidence of the degradation of filament wound glass-epoxy specimens due to exposure to moisture. Following, however, are unpublished data obtained by Dr. S. Brelant and Mr. I. Petker of the Aerojet-General Corp. at Azusa, California. The purpose of the 80 percent of ultimate prestress was to produce a large number of resin cracks as exemplified in Fig. 17 of this report. According to the data of Table A2, exposure to moisture did degrade the bottles after prestrain but did not degrade the strength without the prestrain. Also,

TABLE A2  
Effect of Environment Upon Prestressed 4-in.-Diameter Bottle

Prestress (% ultimate)	Exposure	Actual Burst Pressure (psi)
0	None	2900, 2950
80	None	3025, 2900
0	10 days, 95% R.H.	2725, 2940
80	10 days, 95% R.H.	2325, 2230

the prestrain did not by itself degrade the strength. Only two specimens were tested for each condition, and so no significance test was applied.

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**EFFECT OF MOIST ENVIRONMENT  
ON PRESTRAINED  
GEODESIC ISOTENSOID BOTTLES**

In the work of Brelant and Petker the specimens were 4-inch cylinders with semielliptical ends. Some shear strain and change of shape were experienced during pressurization. On the other hand, in Outwater designed specimens the shape tends to remain constant. Tables A3-A5 are taken from Outwater's Report\* and refer to test specimens made from the same roll. Since the number of specimens was too small for good testing of significance, this data must stand as tentative.

Work on this subject is in progress at NRL by Dr. I. Wolock and Mr. H. Ewing.

TABLE A3  
Effects of Moisture on the Strength of  
Outwater Bottles not Prestressed

Vessel	Failure Load in Air (lb/end)	Vessel	Failure Load in Under-Water Burst (lb/end)
SB-1	6.52	SB-4	6.07
SC-1	6.84	SC-7	6.54
SD-1	6.67	SD-4	6.48
SF-4	6.21	—	—
SG-4	6.92	SG-3	6.92
Mean	6.63	Mean	6.50

Conclusion: The slight difference shown is probably not significant.

\*J. Outwater and W. J. Seibert, "The Effects of Water on the Strength of Laminated Pressure Vessels," Tech. Memo. 194 on Project 62R05-19A, Contract Nonr-3219(01)(X), Univ. of Vermont, Oct. 5, 1962

TABLE A4  
Effect of Submergence in Water for 7 Days  
With and Without 80% Preload

Vessel	Failure Load with 80% Preload (lb/end)	Vessel	Failure Load Without Preload (lb/end)
SC-2	6.38	SC-6	7.02
SB-6	6.39	SB-2	6.69
SD-6	6.71	SG-2	7.57
SF-6	6.14	SF-1	7.03
Mean	6.40	Mean	7.08

Conclusion: Submergence in water for one week is deleterious only if the vessel has been preloaded. This conclusion is tentative.

TABLE A5  
Effect of Exposure to 100% Relative Humidity  
for 7 Days After Preload of 80%

Vessel	Failure Load Without Exposure (lb/end)	Vessel	Failure Load After 7 Days Exposure to 100% R.H. (lb/end)
SF-7	7.06	SB-5	6.80
SF-2	7.13	SC-5	6.65
SB-3	7.20	SF-3	7.06
SG-1	6.98	SF-9	6.39
SC-3	7.36	SC-8	6.97
Mean	7.15	Mean	6.77

Conclusion: The effect of exposure to 100% relative humidity for 7 days is to reduce the strength of the vessels which have been craze cracked by prestressing prior to exposure. This is a tentative statement.