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ERRATA SHEETS

FOR

HIGH STRENGTH GLASS FIBERS DEVELOPMENT PROGRAM

January 20, 1963

Department of the Navy
Director, Special Projects (SPN)

Contract NOW61-0641-c (FBM)

Task 2

Fourth Bi-Monthly Progress Report

Project Engineers

D. L. Hollinger
H. T. Plant

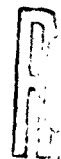
Project Manager

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General Electric Company
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Report Submitted by:

Flight Propulsion Laboratory Department
General Electric Company
Evendale, Ohio

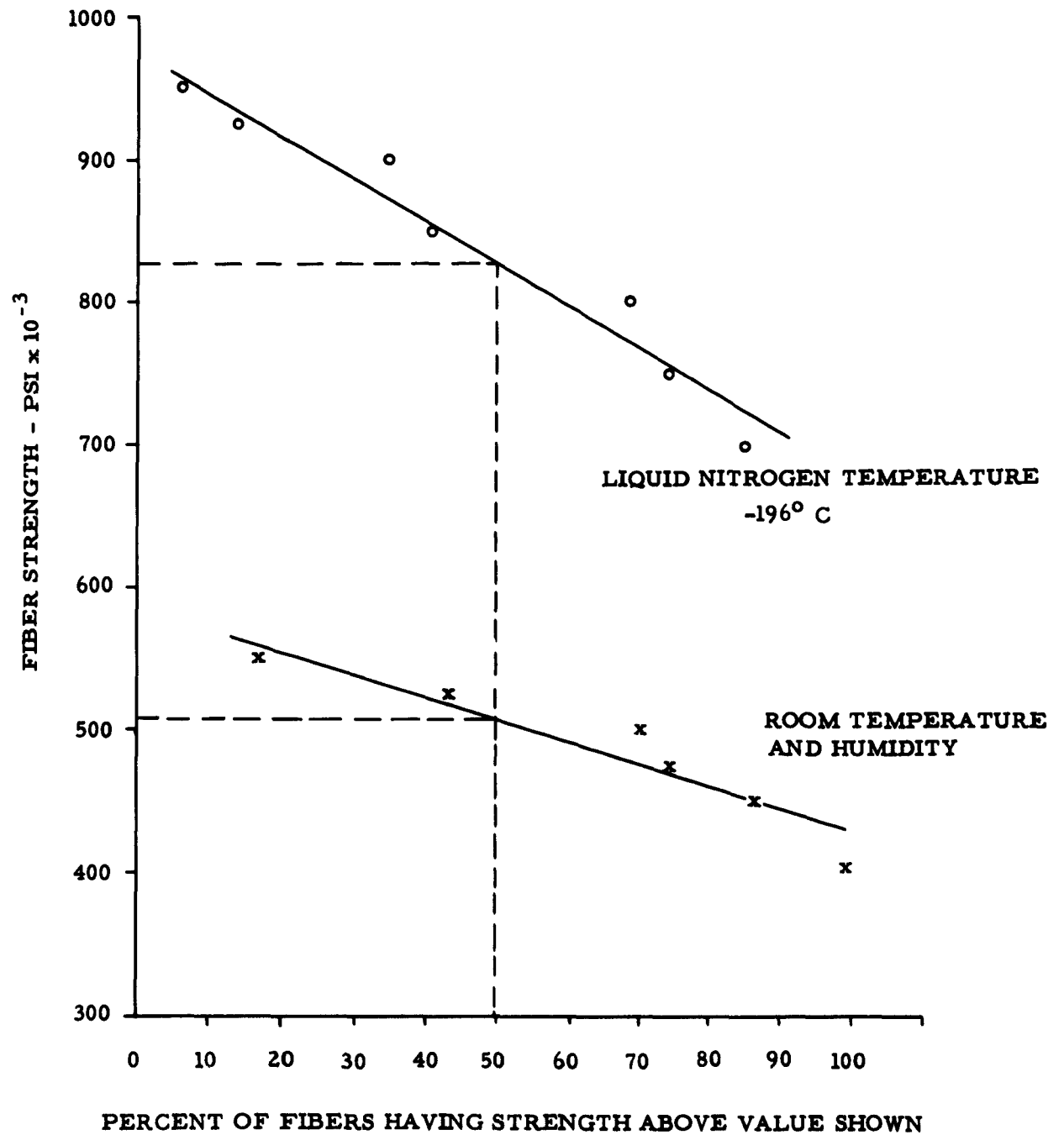


ERRATA

1. The enclosed Figure 8, "E-Glass Fiber Tensile Strengths at Room Temperature and Liquid Nitrogen Temperature", should be included in the subject report immediately following the page containing Figures 6 and 7.
2. Figure 8 in the report as issued, "Temperature Dependence of the Delayed Failure Process", should be labeled Figure 9.
3. Page 16 - Reference to Figure 8 should be changed to read Figure 9.

Figure 8

E-Glass Fiber Tensile Strength at Room
Temperature and Liquid Nitrogen Temperature



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TABLE OF CONTENTS

	<u>Page</u>
I SUMMARY	1
II INTRODUCTION	2
III EXPERIMENTAL PROCEDURE FOR SINGLE FIBER TESTING	4
A. Fiber Production	4
B. Fiber Gathering and Storage	5
C. Method of Mounting Fibers for Tensile Test	6
D. Setup for Testing at Liquid Nitrogen Temperature	7
E. Measurement of Fiber Diameter	8
IV TEST RESULTS	10
A. Single Fiber Tensile Strength	10
B. Composite Rings Tested in Liquid Nitrogen	11
V DISCUSSION	13
VI CONCLUSIONS	18
VII FUTURE WORK	19

Figures

1. Forks for Gathering Fiber During Drawing Operations
2. Stainless Steel Blocks for Potting Ends of Glass Fibers
3. Mounting Fibers in Sealing Wax Grips for Tensile Test
4. Five Tensile Test Specimens on Transfer Rack
5. Transfer of Test Specimen to Stirrups in Instron Machine
6. Fiber in Position for Test Before Adding Liquid N₂
7. Fiber Enclosed in Liquid Nitrogen Container
8. Temperature Dependence of the Delayed Failure Process

Tables

1. Single Fiber Tensile Strength - E-Glass
2. E-Glass and Epoxy Composite Rings Tested in Liquid Nitrogen

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HIGH STRENGTH GLASS FIBER DEVELOPMENT PROGRAM - TASK 2
FOURTH BI-MONTHLY REPORT -- JANUARY 20, 1963

I. SUMMARY

Work during this period has demonstrated that the tensile strength of E-glass fibers approaches 1,000,000 psi when measured at liquid nitrogen temperature (77°K). These same fibers showed strengths averaging 500,000 psi when tested at normal room temperature and humidity. The higher measured strength at low temperature is believed to be the true instantaneous strength of the fiber when chemical reaction with its environment is suppressed.

Composite rings of E-glass monofilament and epoxy resin exhibit the same percentage increase over room temperature strength when tested at liquid nitrogen temperature as do the bare fibers from which they are made. Calculated glass stress in rings made under comparable conditions is 500,000 psi at -196°C and 310,000 psi at room temperature. However, apparent glass stress in rings, calculated simply from the applied load and the supporting cross-sectional area of glass, remains lower than the tensile strength of the fiber at the same temperature, reflecting either inefficient load distribution among all fibers in rings or the presence of stress concentrating effects and stress modes other than pure tension.

II. INTRODUCTION

Previous reports in this series have noted the effects of moisture on the strength of glass-epoxy rings tested at room temperature. In the second bi-monthly progress report, dated September 20, 1962, a series of experiments was reported in which exposure of the glass to moisture was controlled during drawing of the fiber from the melt, during storage of the cured composite rings and during actual testing of the rings in tension. From these experiments it was concluded that the maintenance of dry conditions throughout the total history of the fiber did allow the glass to withstand higher stress. It was not clear, however, at what specific points moisture was harmful, nor whether there might be some circumstances in which it did not degrade the fiber strength.

It appeared likely that the answer to the questions raised above would be most logically found by working with the glass fiber alone rather than in a composite with organic resin and by testing under pure tensile loading instead of the complex loading which is suspected to exist in a split ring test.

On the other hand, we could expect that any increase in tensile strength obtained on single filaments should be reflected in the strength of composite rings to the extent that the fiber is actually stressed in tension in such rings.

Since any effect of water on a single glass fiber would most likely occur via a chemical reaction, one way to reduce the effect would be to slow down the rate of such reactions. A convenient way to do this is to lower the temperature. Another way might be to reduce the concentration of reactants. However, we did not know in what way the rate might depend upon the concentration of water, and it would be very difficult to exclude all traces of water from the glass surface. Therefore, the former method of reducing chemical reaction rates - by reducing temperature - was chosen.

Considerable evidence was available, from prior work in the General Electric Company Research Laboratory and from the open literature, to suggest that the most degrading effect of water on glass fibers might be stress corrosion, i.e., a corrosion reaction which is accelerated by mechanical stress. If such were the case, a most significant effect should be noted by reducing the temperature during the actual tensile test. We therefore began our study of moisture effects on single fibers by comparing tensile strengths of fibers tested at liquid nitrogen temperature with those tested at room temperature when all were made and handled under normal atmospheric conditions.

III. EXPERIMENTAL PROCEDURE FOR SINGLE FIBER TESTING

A. Fiber Production

All E-glass fibers tested in this program were produced from our single hole platinum crucible. The glass batch was heated to 2400°F and held at or slightly above this temperature for at least two hours before fibers for test were collected. The actual drawing temperature, that is, the temperature at the orifice tip, was lower than the temperature in the body of the liquid, but was not determined with great accuracy. Optical pyrometer readings on the tip during drawing of fiber ranged from 2220°F to 2270°F.

Drawing speed was in the range of 1100 - 1300 feet per minute, which resulted in fiber diameters around .00040 inch. This is approximately the size of filaments in commercially produced E-glass for reinforced plastics.

A pyrex glass tube, two inches in diameter and six inches long, enclosed the fiber as it issued from the bottom of the furnace. This acted primarily to prevent drafts from moving the fiber out of alignment and was useful mainly during the start of winding.

When no composite rings were being made concurrently with the gathering of single fibers, the fiber was wound on a 12-inch diameter drum. When rings were also being made from the fiber, the winding apparatus was the 4-inch diameter

split ring mandrel. In the latter case, fibers for test were always gathered immediately after the composite ring was completed. No process conditions were changed with the exception of the removal of the resin coater.

B. Fiber Gathering and Storage

A sample of fiber approximately three feet long was obtained by first clamping the filament just above the winding drum to break it free, then severing it at a point about 10-inches below the crucible orifice. The upper end was weighted with a piece of cellophane tape, then the two ends were cautiously inverted so that the weighted end hung free and the fiber was straight and vertical. It was then a relatively simple matter to wind the fiber onto a fork, such as that shown in Figure 1, with any desired spacing between the segments of the continuous fiber. Double sided adhesive tape on the arms of the fork helped prevent slipping of the fiber segments.

Once wound, the forks were stored in closed containers in such a way that the fibers did not contact any object.

C. Method of Mounting Fibers for Tensile Test

For tensile tests at either room or low temperature the fiber must be held in some sort of grips. It has become customary in this work to use sealing wax to fasten the fiber ends to the pulling mechanism. We were afraid that adhesion of the wax to the fixture and to the glass fiber might be poor at liquid nitrogen temperature. Therefore, we designed small stainless steel blocks to contain the wax and to provide gripping by wedge action in the event of adhesion failure. Figure 2 is a drawing of these blocks. The method of potting the fibers in wax by using the blocks, is indicated in the photograph of Figure 3.

As the wax congealed in a set of blocks with a fiber in place, the fiber was severed from the gathering fork, and the assembly of two blocks with their suspended fiber was slid onto the arms of a transfer rack. As many as ten fibers can be accommodated on one rack. Figure 4 shows five fibers in place on a transfer rack after potting with sealing wax.

For both room temperature and low temperature tests, the potted fibers were transferred one at a time from the rack to special fixtures, called stirrups, in the Instron test machine. Two prongs on each stirrup fit into holes in the blocks containing the fiber. Figure 5 illustrates the method of performing

the transfer. This step required considerable care to avoid overstressing the fiber when the upper block became free to swing at the end of the long wire hook.

In the case of a room temperature test, the next, and last, step was simply to start the cross-head motion of the machine and observe the breaking load on the fiber. Strain gage load cells and machine settings capable of registering full scale deflection with loads of 50 or 100 grams were used in all single fiber work, as the breaking loads were in the range of 25 - 70 grams. Cross-head speed of 0.05-inch per minute was employed and, since our gage length between wax grips was 1-inch, the nominal strain rate was 0.05-inch/inch-minute.

D. Setup for Testing at Liquid Nitrogen Temperature

For tests of single fibers at liquid nitrogen temperature (-196°C), the procedure was identical to that described above for room temperature tests, with one added feature. An insulated tank to contain the liquid nitrogen was lowered over the fiber sample after it was suspended in the Instron machine. This tank was gasketed and bolted to the bottom cross-head member as pictured in Figures 6 and 7.

The lower mounting fixtures were designed so that the level of liquid nitrogen within the tank could be maintained just below the fiber sample by feeding the coolant in at the bottom of the tank. Temperature was monitored in the vapor phase somewhat above the upper block of the test specimen. We found that the temperature at this point would always reach the boiling point of nitrogen (-196°C) by the time the liquid level had been brought to the bottom of the lower block. The glass fiber and its two sealing wax grips were therefore in the relatively quiescent vapor phase while still at the -196°C temperature. This was advantageous, since we found that submergence in the boiling liquid phase caused excessive buffeting of the delicate sample. Once the proper temperature had been established, about a five-minute soak period was allowed to insure equilibrium, and then the cross-head motion was begun.

E. Measurement of Fiber Diameter

In order to calculate the breaking stress in the fiber, one must have an accurate measure of the cross-section area. Ideally, this measurement should be made in the same portion of fiber which is tested in order to eliminate inaccuracies due to possible variations in diameter along the length of

the fiber sample. To achieve the same accuracy without subjecting the test samples to the handling required when making diameter measurements, we measured the fibers on one side of a gathering fork and tested those from the other side. By averaging the measurements obtained on several segments from a given fork, we obtained a good value for the diameter of the test samples from that fork.

Diameter measurements were made by optical microscope using a magnification of about 500 X. Immersion oil surrounded the fiber sample to reduce refractive effects at the edges of the image. A calibrated filar micrometer eyepiece was used, and at least two measurements, approaching the fiber from each side, were made on each segment from a fork. Generally, four segments from each fork were measured.

Because there is no absolute reference measurement to establish the exact focal point for this type of measurement, a small, constant deviation of the measured diameters from the true values is conceivable. However, all measurements have been made by the same operator, using the same equipment and technique. Thus, we feel confident that all of our data may be justly compared to each other. The fact that the average room temperature strength determined for our fibers

is the same as that reported by other investigators for E-glass, makes it appear that very little actual error exists in the diameter measurements.

IV. TEST RESULTS

A. Single Fiber Tensile Strength

Figure 8 summarizes the data obtained on single fiber strength at both room temperature and liquid nitrogen temperature. This particular comparison shows several interesting things about the test results. Obviously, a large difference in strength under the two conditions is indicated. The median strength is 60% higher at -196°C than at room temperature. It is also notable that at least one-fourth of the fibers show strengths in excess of 900,000 psi at low temperature. The difference in slopes of the two lines indicates a somewhat lower dispersion of strengths when measurements are made at room temperature, even though the fibers are weaker in this environment.

The individual test values, used in preparing the relationships of Figure 8, are shown in Table I. The highest single tensile strength measured thus far has been 974,000 psi.

B. Composite Rings Tested in Liquid Nitrogen

It is well known that the large difference in coefficients of thermal expansion for glass and epoxy resin could cause problems when composites of these two materials are subjected to large changes in temperature. One might expect, therefore, that filament-wound rings cured at 150°C and then tested at -196°C might suffer some loss in strength as a result of resin cracking, glass fiber surface spalling, de-bonding, etc. On the other hand, having found that the glass fiber alone showed so much improved tensile strength at liquid nitrogen temperature, we were anxious to see how much carry-over of this improvement could be obtained in a composite ring.

A set of four rings was made by our established procedure with E-glass monofilament and epoxy resin. The resin used was again Union Carbide ERL-2256 with hardener ZZL-0820. Immediately after completion of winding each of the first three rings, a fork of uncoated fibers was collected. This was done without altering drawing speed or changing other process conditions, except to remove the resin coater the moment before the fiber was taken. These fibers, then, were representative of the material in the rings with which they were associated. For purposes of identification, they were designated (and appear in Table I) as follows:

9R1 = Fork No. 9 associated with Ring No. 1

10R2 = Fork No. 10 associated with Ring No. 2

11R3 = Fork No. 11 associated with Ring No. 3

No provision was made to reduce humidity during drawing and ring winding operations for this series. After curing, the four rings were stored in a dessicator over silica gel. The actual testing of these rings was conducted with the entire ring and split steel mandrel submerged in liquid nitrogen. A soaking period of about five minutes was allowed after complete submergence before cross-head motion was begun.

In prior work, rings made and stored under similar conditions showed glass stress values ranging from 300,000 to 320,000 psi when tested in the normal laboratory atmosphere at room temperature. In the four rings tested in liquid nitrogen, glass stresses were 495,000 to 535,000 psi. These data are shown in Table 2.

It is especially interesting to note that the improvement in glass stress in the composite rings at low temperature was the same percentage as the improvement in tensile strength of the fiber from which they were made: approximately 60%. In view of this fact, it is hard to imagine that any significant

damage to the glass occurred because of shrinkage stresses in the bonding resin, despite the drop in temperature from curing to test of some 350°C. Neither is there any apparent loss of efficiency in load distribution among the many fibers, which depends upon the maintenance of some sort of bond, either chemical or mechanical, between the resin and glass.

V. DISCUSSION

The strength of E-glass fibers which have not been mechanically damaged is generally reported in the 500,000 - 550,000 psi range when tested at room temperatures. Recognizing that this is at least a 50% reduction from theoretical strength, most investigators have acknowledged the existence of flaws, generally considered to be surface cracks, as being responsible for the lowered strength. According to the well-known Inglis and Griffith theories, there is a critical flaw size associated with a given failure stress in a brittle material such as glass. The familiar Griffith equation expresses this relationship:

$$\sigma_{cr} = \sqrt{\frac{4ES}{\pi c}}$$

where σ_{cr} = applied failure stress
E = Young's Modulus
S = surface energy
c = crack depth

Any existing flaw larger than this critical size would be expected to propagate spontaneously and cause failure when the stress level

at its tip reached the corresponding critical value. This picture, then, in the absence of further interpretative information, would lead one to the supposition that the measured strength of E-glass fibers was limited by the size of cracks already in place in the sample when the test began.

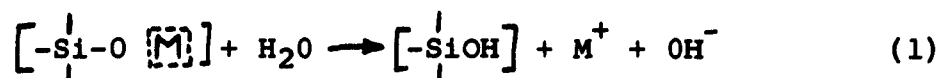
This naive viewpoint becomes quickly confounded by data which have been reported from many sources indicating strong dependence of the strength of glass upon its environment and also upon the duration of the load application. Various referred to as static fatigue or delayed failure, these effects have generally been ascribed to a destructive reaction at the tip of surface flaws, involving water vapor and components of the glass.

The dependence of strength upon time under load implies a rate controlled failure process of some kind. If this rate is sharply reduced, as by lowering the temperature, then the measured strength, under the same load duration, should be increased significantly. This is precisely the effect shown by our data on fibers at liquid nitrogen temperature and room temperature.

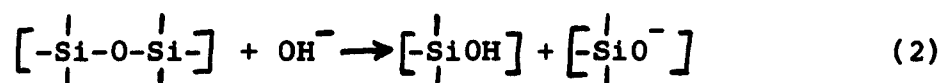
The role of moisture in this failure process with various glasses and fused silica has been broadly reviewed by R.J. Charles⁽¹⁾ of the General Electric Company Research Laboratory. In particular,

(1) Charles, R.J., Journal of Applied Physics, Vol. 29, No. 11, p., 1554-1560 (1958).

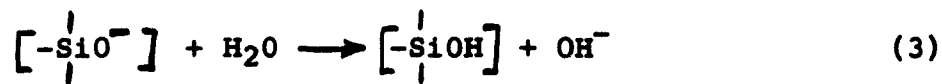
for glasses such as soda-lime glass which contain a silica network structure plus alkali ions which are associated to the silica network as terminal structures, the corrosion reaction mechanism is described as follows:



This first step is a typical hydrolysis reaction of the salt of a weak acid, where [M] represents an alkali metal ion, e.g. sodium in soda-lime glass. This reaction in itself would not significantly degrade the strength since the backbone network of covalent bonded silicon and oxygen atoms is not involved. However, free hydroxyl ion is formed in the process and the second important step may proceed:



In this step, which can proceed only if the first step has already been taken, the very strong Si-O-Si bond is broken. Furthermore, one product of this second reaction is an end structure capable of dissociating another water molecule, viz.,



Thus, excess hydroxyl ion is formed in equivalent amount to each alkali metal ion that is no longer associated with silica. This increase in pH within the corrosion layer tends to make the

glass dissolution process autocatalytic and acceleration occurs. Reaction 1 is a triggering reaction for alkali-silica glass dissolution in water vapor and should be dependent on the ease of migration or diffusion of the alkali metal ion through the glass.

One can imagine a situation in which the rate of the corrosion reaction is sensitive to the state of stress in the glass structure in such a way that the fastest corrosion occurs in the region of highest tensile stress. In such a case, stress concentrating cracks in the glass surface would grow and perhaps even sharpen in the presence of applied stress and a corrosive environment. Evidence for this sort of behavior in alkali silicate glasses was found by Charles in measurements of delayed failure times versus applied stress at various temperatures. The temperature dependence of the delayed failure process at a particular stress level is shown in Figure 8. The linear relationship over the temperature range from -50°C to $+50^{\circ}\text{C}$ allows calculation of an apparent activation energy for the failure process which equals 18.8 kcal/mole. This value agrees closely with the activation energy for Na^+ ion diffusion in the same glass calculated from electrical conductivity measurements (19.4 kcal/mole).

That the corrosion reaction is indeed faster when the glass structure is expanded than when compacted was also shown by Charles in experiments in which fibers of the same glass were exposed to

steam in the "as drawn" or expanded condition and after annealing to compact the structure. The depth of the corrosion layer was nearly three times as great after a two-hour exposure period in the "as drawn" fibers as in the annealed samples. Charles states, "It seems entirely feasible that expansion of the glass structure by large triaxial tensile stresses of mechanical origin would result in a condition, as far as the migration and diffusion of Na^+ ions are concerned, very similar to the expansion of the structure by a high fictive temperature."

With this background of information pointing to stress corrosion by water as the principal cause of low strength in many glasses, our own experiments were designed to investigate this phenomenon in E-glass fibers. Both the large percentage increase in strength and the approach to theoretical strength achieved by a significant proportion of the fibers at liquid nitrogen temperature supports the view that at room temperature, stress corrosion is responsible for the relatively low fiber strength.

It might be argued that one expects the strength of materials to increase as temperature is lowered, even in the absence of chemical reactions. It is true that most theories of mechanical strength including the Griffith relationship for the effect of cracks, do indicate a dependence of strength upon the elastic modulus. However, actual measurements on silica-based glasses show only a

minor change in modulus, in the neighborhood of 5%, in going from room temperature down to liquid nitrogen temperature. In contrast to this, the 60% improvement in measured strength of E-glass fiber at low temperature can best be explained by the great reduction in rate of stress corrosion reactions involving water vapor.

VI. CONCLUSIONS

1. It has been demonstrated that the instantaneous strength of E-glass fibers, produced in a normal atmosphere, can be nearly 1,000,000 psi and that if the rate of stress corrosion reactions with water vapor is suppressed by conducting the tests at liquid nitrogen temperature, an average strength of over 800,000 psi is readily achieved. This represents a 60% increase in strength over the average room temperature strength for these fibers of 500,000 psi.

2. An equivalent percentage increase in glass stress within composite ring structures is obtained by testing the rings at liquid nitrogen temperature, the comparable values being 500,000 psi at -196°C and 310,000 psi at room temperature.

VII. FUTURE WORK

The obvious implication of this work is that if a way can be found to prevent water from contacting the glass fibers while they are under stress, it should be possible to utilize at room temperature the same high strengths found at liquid nitrogen temperature. A coating material, impervious to water vapor and applied while the glass is bone dry, is a logical approach. It is known that most organic resins, while they can slow down the transfer of moisture, are not perfect barriers. Films of higher density, such as metals and a few other materials, are required to do the job.

It is our intention, during the next period, to make preliminary attempts to coat our fibers with materials which can act as a true barrier to moisture vapor and allow room temperature strengths to approach those obtained at liquid nitrogen temperature. Work on the coating of glassy materials recently carried out at the General Electric Company's Research Laboratory has shown considerable promise of providing effective barriers without degrading substrate properties. This is expected to be of help in our own development of a moisture impermeable coating for E-glass fibers.

In order to further substantiate the theory that stress corrosion from water is the source of the strength loss in E-glass at room temperature, we shall run tensile tests on bare fibers at room

temperature in the presence of a very efficient dessicant. This dessicant will be a solution of lithium aluminum hydride in ether. It is hoped that this will be a more direct proof of the role of water vapor, even though it is doubtful that any dessicant can equal the effectiveness of low temperature in preventing some corrosion effects.

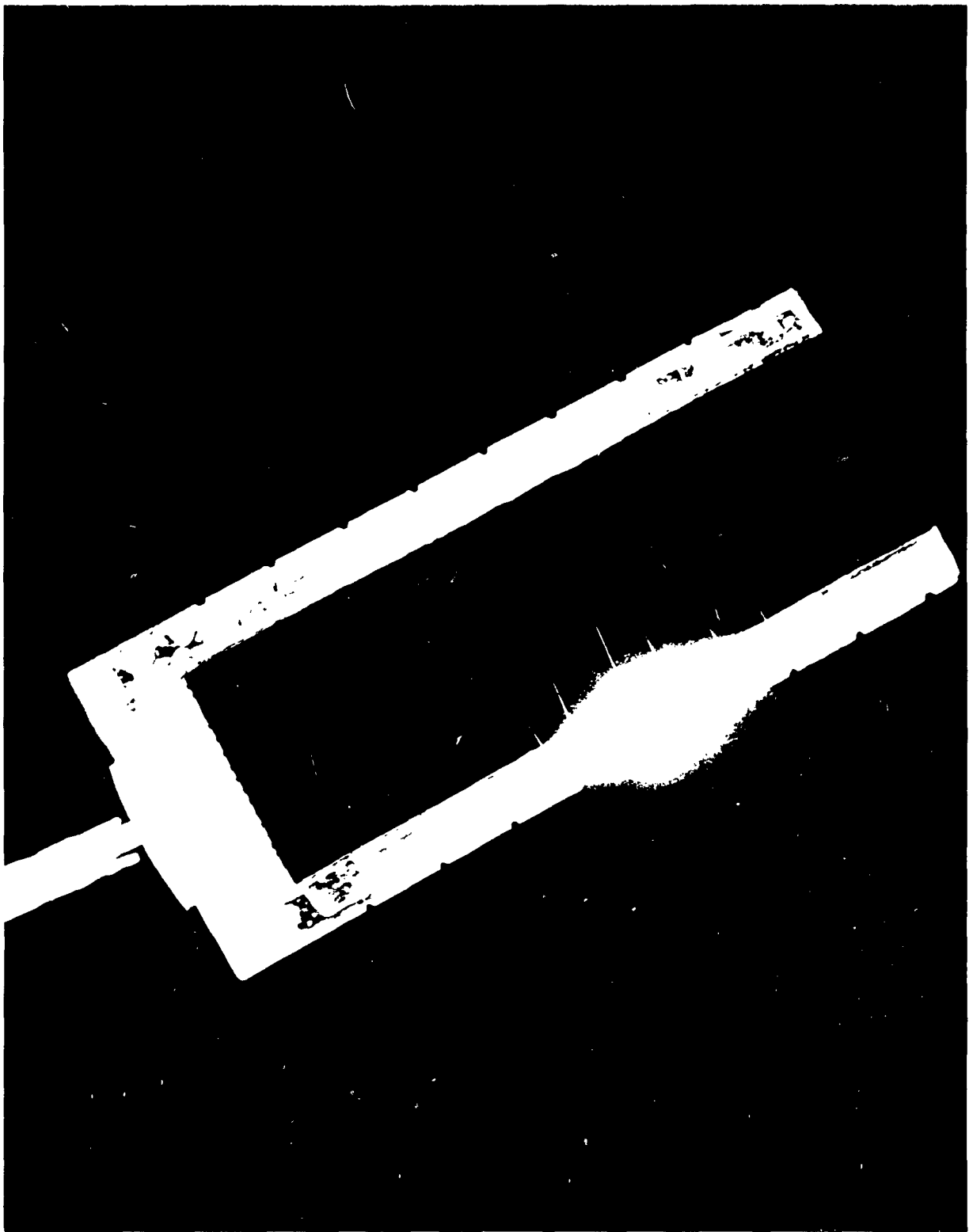


Figure 1

Fork for gathering fiber during drawing operation -

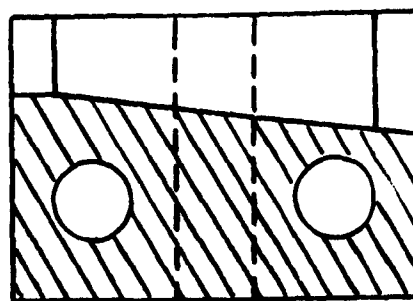
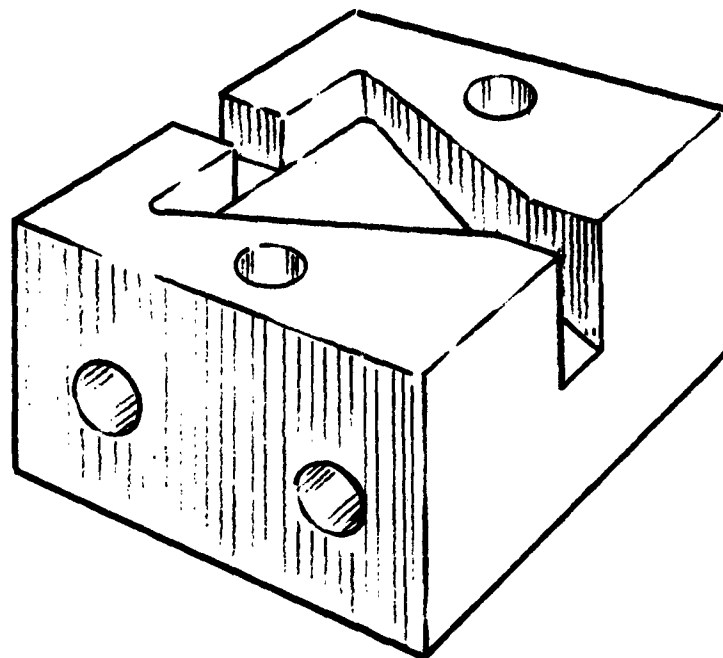


Figure 2

Stainless steel blocks for potting ends of glass fibers

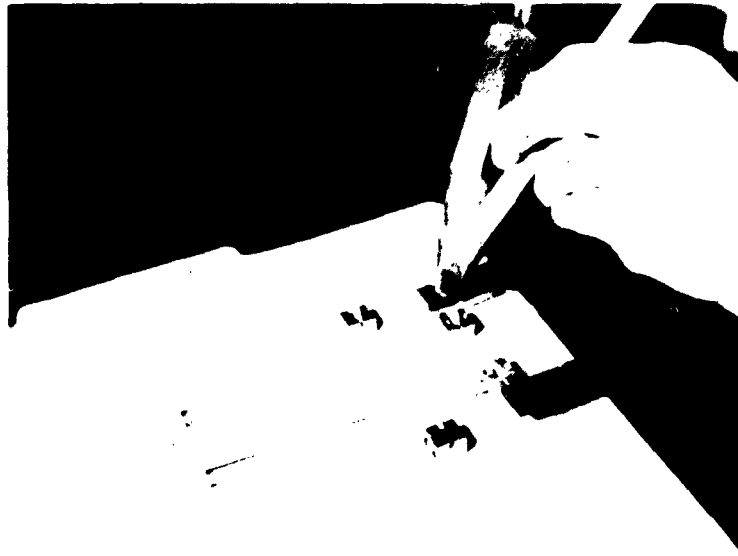


Figure 3

Mounting fiber in sealing wax grips for tensile test

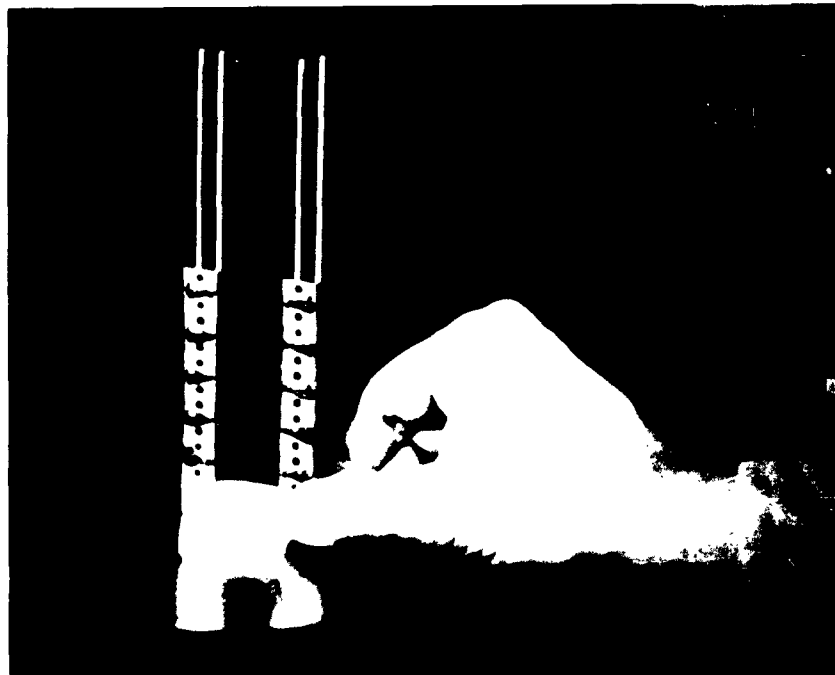


Figure 4

Fiber tensile test specimens on transfer rack

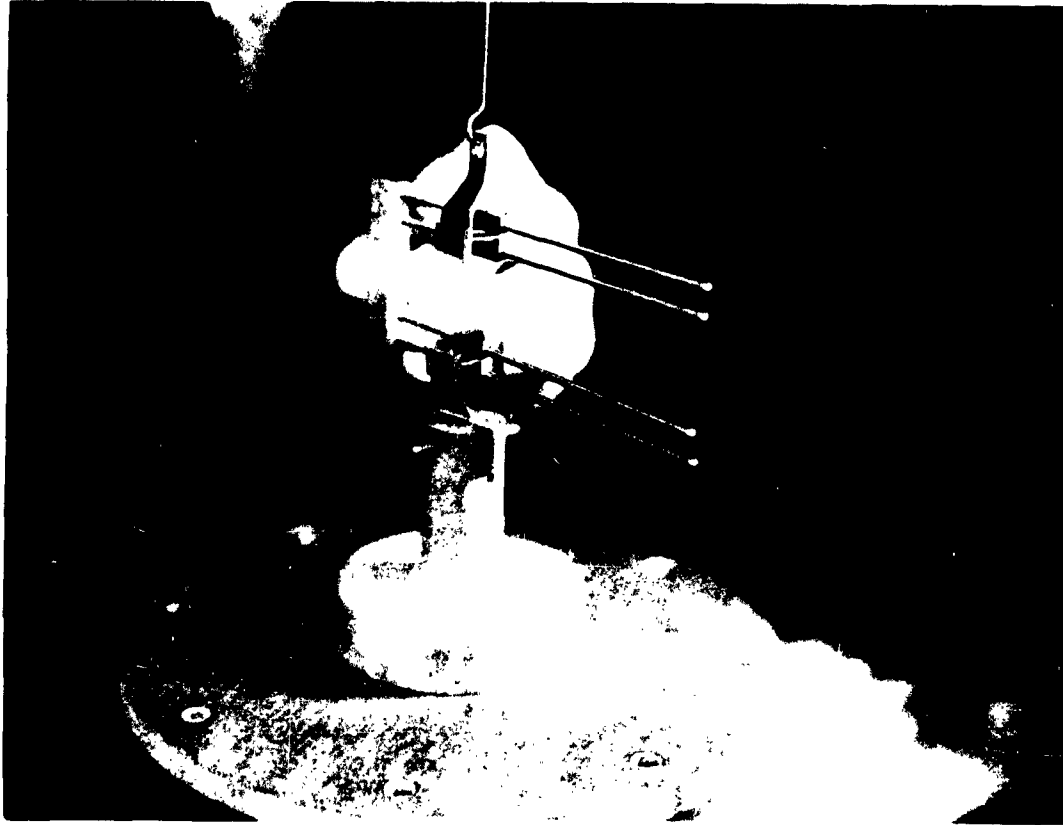


Figure 5

Transfer of test specimen to stirrups in Instron machine

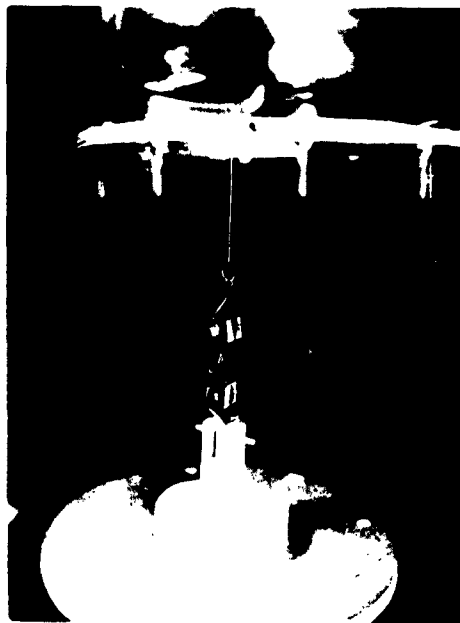


Figure 6

Fiber in position for test before adding liquid nitrogen



Figure 7

Fiber enclosed in liquid nitrogen container

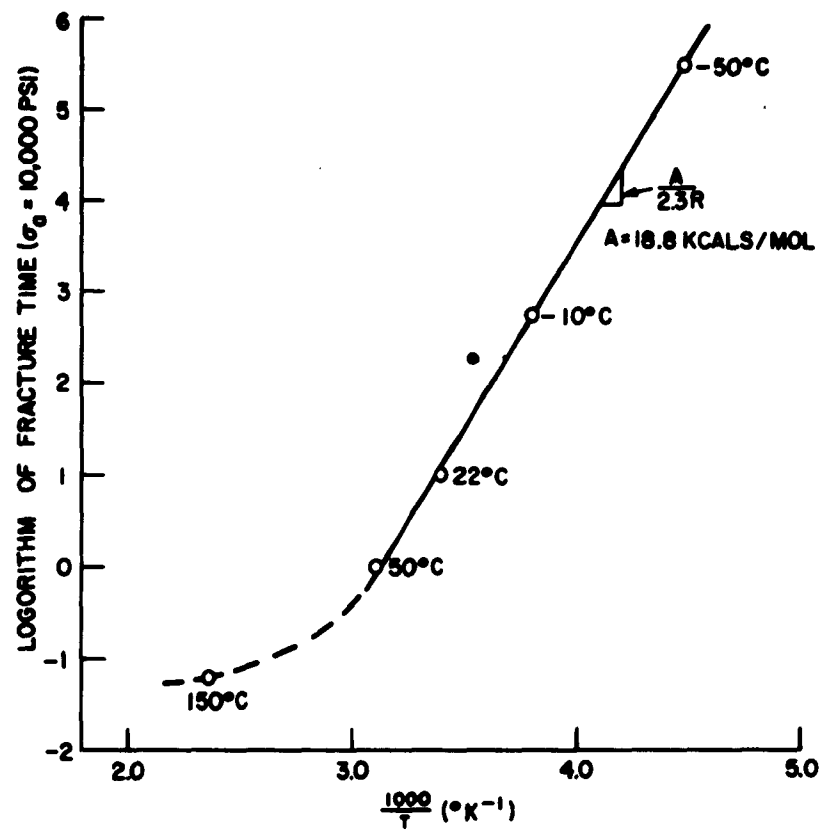


Fig. 8 Temperature dependence of the delayed failure process.

TABLE I
SINGLE FIBER TEST DATA - E GLASS

Fiber No.	Segment No.	Fiber Diam.- Mils	Tensile Strength-PSI Room Temp.	-196°C	Remarks
1 -	1	.47	464,000		
1 -	2		502,000		
1 -	3		417,000		
1 -	4		520,000		
1 -	5		-		Slipped out of wax.
1 -	6		416,000		
2 -	1	.43	519,000		
2 -	2		520,000		
2 -	3		-		Broken in handling.
2 -	4		-		" " "
2 -	5		475,000		
3 -	1	.41	-		Slipped out of wax.
3 -	2			588,000	
3 -	3			411,000	
3 -	4			743,000	
3 -	5			815,000	
3 -	6			724,000	
4 -	1	.46	> 438,000	833,000	Not broken at R.T. Retested at -196°C.
4 -	2			847,000	
4 -	3			795,000	
5 -	1	.42		-	Damaged in liquid N ₂ bath.
5 -	2			749,000	
5 -	3			916,000	
5 -	4			671,000	
5 -	5			800,000	
5 -	6			835,000	
5 -	7		465,000		
5 -	8		450,000		
5 -	9		445,000		
6 -	1	.40	525,000		
6 -	2		525,000		
6 -	3			895,000	
6 -	4			578,000	(Fibers cooled down under compression-
6 -	5			578,000	sharply bent.
6 -	6			847,000	
6 -	7			842,000	
6 -	8			827,000	
6 -	9			900,000	
6 -	10			754,000	
7 -	1	.39	524,000		
7 -	2		535,000		
7 -	3			701,000	
7 -	4			974,000	
7 -	5			912,000	
9R1 -	1	.42	554,000		
9R1 -	2		560,000		
9R1 -	3		548,000		
9R1 -	4			930,000	
9R1 -	5			960,000	
9R1 -	6			913,000	
11R3 -	1	.45		936,000	
11R3 -	2			905,000	
11R3 -	3			> 876,000	
11R3 -	4		551,000		
11R3 -	5		548,000		
10R2 -	1	.44	512,000		
10R2 -	3		540,000		
10R2 -	4		557,000		
10R2 -	5			920,000	
10R2 -	6			915,000	
10R2 -	7			828,000	
10R2 -	8			944,000	
AVERAGE			507,000	814,000	

TABLE 2
E-GLASS AND EPOXY COMPOSITE RINGS
TESTED IN LIQUID NITROGEN

<u>Ring No.</u>	<u>Load to Break - Lbs.</u>	<u>Wt. of Glass in Ring - Gms.</u>	<u>Glass Stress PSI</u>
1	2525	1.3716	495,000
2	2525	1.3565	502,000
3	2970	1.4937	535,000
4	2360	1.2830	495,000

NOTES:

1. Rings are 4 inch diameter x .070 inch wide x .030 inch thick.
2. Cross-head speed is 0.10 inch/min.