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INFLUENCE OF STRESS CORROSION ON
STRENGTH OF GLASS FIBERS

(Unclassified)

Fourth Bi-Monthly Progress Report

November 30, 1964

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Project Manager:

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Manufacturing Engineering Service
General Electric Company
Schenectady, New York

Report submitted by: Advanced Engine & Technology Dept.
General Electric Company
Evendale, Ohio

This work was performed under Contract No. Nonr 4486(00) (X)
for the U.S. Naval Research Laboratory.

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I. SUMMARY

Static fatigue tests at liquid nitrogen temperature have been completed on virgin E-glass single filaments. In these tests, fibers approximately 0.0005-inch diameter were dead-loaded in tension while at -196°C and maintained at that temperature for at least 1.7×10^5 seconds. Loads were varied within the high stress region from 400,000 to 650,000 psi. No static fatigue failures were observed under these conditions, even though the stress range was high enough to cause immediate failure of some fibers upon load application. This is in distinct contrast to the behavior observed at room temperature in normal humidity where delayed failures occurred over several decades of time with stress level ranging from 200,000 to 400,000 psi.

II. INTRODUCTION

Tensile strength measurements on glass fibers, as on many other materials, are normally made with a fixed rate of cross-head motion. When the material exhibits Hookean elasticity essentially to the failure stress, as do glass fibers at room temperature, and the maximum elongation is not large, then the rate of stress increase is approximately proportional to the cross-head rate. In the absence of any time-dependent effects on material properties, the measured ultimate strength would be independent of rate of stress application. However, if the material is subject to any time and stress-dependent structural changes while under test, then the strength property being measured may be changing throughout the duration of the test itself. This is precisely the effect which has been thought to limit the measured strength of glass fibers at room temperature and in normal humidity.

In effect, the ordinary tensile test is a fatigue test of relatively short duration and employing a continuously increasing load. Failure occurs when the integrated stress - time function exceeds some value which is characteristic of the particular sample in the particular environment of the test. The actual function

of applied stress and time which determines failure is not known, and in fact may be different for different samples of the same material because of variations in flaw distributions. A destructive test such as the ordinary tensile strength test gives no information about this function because one cannot know what would have happened to a given fiber if the stress-time sequence had been something different from that actually used to destroy the sample. Static fatigue tests do offer a somewhat better opportunity to observe statistically the effects of time and applied stress as independent variables. Hopefully, this separation of time and stress will yield a clearer insight into the degradation process, more information about the distribution of flaws which govern the ultimate strength of fibers, and, finally, some clues as to the best direction for research aimed at improving usable fiber strength.

III. CURRENT EXPERIMENTAL WORK

Static Fatigue Tests at Liquid Nitrogen Temperature

During the present reporting period, a series of static fatigue tensile tests at liquid nitrogen temperature on single filaments of E-glass was completed. The monofilament was produced in this laboratory, using equipment and techniques described in detail in reports on a preceding Navy contract.⁽¹⁾ The method of applying and maintaining static tensile loads on fiber samples is explained in the second bi-monthly report on the present contract.⁽²⁾

Briefly, single fiber samples with a gage length of one-inch and diameter about 0.00050-inch were subjected to dead weight loading after being cooled to liquid nitrogen temperature (-196 °C), and were maintained at that temperature in the nitrogen gas phase for periods of 1.7×10^5 seconds (48 hours) or longer. The test procedure was identical with that previously used for tests at room temperature and normal humidity, except that an insulated, double-walled tank was brought up to surround the mounted fiber samples.

* * * * *

- (1) Final report, High Strength Glass Fibers Development Program, May 1963, Contract Now61-0641c(FBM).
- (2) Second Bi-Monthly Progress Report, July 20, 1964. Influence of Stress Corrosion on Strength of Glass Fibers.

Figure 1 is an overall view of the test area showing the plastic enclosure and liquid nitrogen supply tanks.

Liquid nitrogen was admitted into the space between the two copper tanks and maintained at a predetermined level by an automatic controller. The latter was composed simply of a level sensor employing thermistors, a suitable relay circuit and a solenoid valve in the liquid nitrogen supply line. Boil-off of nitrogen gas was permitted to escape through holes above the liquid level into the inner tank. When the temperature had been lowered to -196°C and control of the liquid level established, the consumption rate of liquid nitrogen was about 7 liters per hour. This generated enough gas to maintain a slight positive pressure inside a plastic tent which was constructed over the entire test setup and thus helped to keep the enclosed space at a low humidity level. A room dehumidifier also assisted in this effort. It had been found that low humidity was necessary to avoid excessive ice accumulations around the suspension cords where they exit from the cold chamber.

Tests were carried out at each of the following stress levels: 325,000 psi, 400,000 psi, 500,000 psi, 550,000 psi, 600,000 psi and 650,000 psi. The test at 325,000 psi was

made primarily to establish the stress level at which no fibers would fail immediately upon load application. It was not extended for as long a time period as the other fatigue tests.

Results of all fatigue tests at liquid nitrogen temperature are presented in Figures 2 and 3 where they are compared to previously reported data⁽¹⁾ for similar tests at room temperature. Whereas Figure 2 shows the fatigue failure time for each individual fiber at room temperature, it may be easier to visualize the weighted distribution of failure times from the bar graphs of Figure 3. Figure 3 shows, in addition, the percentages of fibers which failed immediately upon application of the dead weight loads. These are indicated by the solid bars on each graph where such failures occurred. The relationship between stress level and the percentage of immediate failures is shown again in Figure 4, where these data for liquid nitrogen and room temperature static loadings are compared. In addition, Figure 4 also shows the distribution of failure stresses determined at the two temperatures when loads are applied gradually in the standard tensile strength test using an Instron machine.

(1) Ibid (2), p. 4

The generally lower level of failure stress at both temperatures in the fatigue test has been attributed to the effect of sudden loading which imposes, momentarily, stresses considerably higher than the nominal applied stress. The significance of the various groups of data is discussed in the next section.

IV. DISCUSSION

Relationship of Delayed Failure to Stress

The most significant feature of the static fatigue results is, of course, the lack of delayed failure at liquid nitrogen temperature. Whereas at room temperature and normal humidity E-glass fibers exhibited static fatigue failures over a very broad range of time under load, at -196°C they did not show any indication of fatigue, at least for 48 hours or more.

The stress levels used at liquid nitrogen temperature ranged much higher than the limit which could be supported, even briefly, at room temperature. For example, with a stress of 400,000 psi at room temperature, 84% of the test fibers failed immediately upon load application, and the remaining 16% had all failed by the end of 6-1/2 hours. At -196°C , at the same stress level, only 33% failed immediately and the rest endured

beyond 75 hours when the test was terminated. At the end of the 75 hour period in this particular test, the nitrogen tank was carefully lowered away from the loaded fibers without disturbing them and all fibers were observed to fail within the first five minutes after exposure to the laboratory atmosphere. Unfortunately, not enough samples were given this latter treatment to allow us to judge whether or not some loss of room temperature strength had occurred during the low temperature cycle.

It is quite clear that a very important difference in behavior of glass fibers under stress exists between room temperature and -196°C . The lack of static fatigue at low temperature found in the present work employing very high stress levels parallels results on bulk glass and small rods reported by other investigators⁽³⁾ where stresses lower by factors of 10-50 were used. Similarly, the presence of delayed failure at room temperature in our experiments on fine fibers may be compared with that observed by Charles with soda-lime glass rods over a range

- (3) R.J. Charles, Static Fatigue of Glass, General Electric Research Lab Reports Nos. 58-RL-1966 and 58-RL-1967 September 1958.

of temperature from -50°C to 150°C . Charles' experiments were carried out in saturated atmosphere, whereas ours were at normal laboratory humidity of perhaps 40-60% r.h. Assuming that the rate of the corrosion reaction which propagates the flaws and leads to delayed failure conforms to an arbitrary power function of stress,

$$v_x \Big|_T = k' (\sigma_m)^n + k$$

where v_x = penetration velocity of crack tip in the x direction.

T = temperature.

k' = constant.

σ_m = tensile stress at crack tip.

n = constant.

k = corrosion rate of the material under zero stress.

it has been shown by Charles that this leads to the following approximation for the relationship between delayed failure time, t, and applied stress, σ_a :

$$\log t \approx n \log \frac{1}{\sigma_a} - \log k'''$$

where k''' = a constant at any given temperature.

Charles showed that his data indicated a value of 16 for the exponential, n. Our data, in lower humidity and with a glass presumably less susceptible to corrosion from water vapor, seem to yield a value of $n \approx 27$. This is illustrated in Figure 5, which is a plot of the most probable

delayed failure time as a function of applied stress. A high value for n , i.e. a strong dependance of failure time upon applied stress would result if the assumed exponential relationship of corrosion rate upon stress at the crack tip is correct. Under these conditions, ordinary corrosion, which would tend to round out the crack tip and reduce its degrading effect is overshadowed by stress-activated corrosion which sharpens the crack and promotes its propagation.

Relationship of Immediate Failure to Stress

In discussing the room temperature static fatigue results, it was pointed out in the Second and Third Bi-Monthly Progress Reports that two relationships between applied stress and the number of immediate failures were observed. First, the number of immediate failures decreased quite uniformly with stress until at 250,000 psi and below there were none. Delayed failures, however, still occurred at the lower stresses over a broad range of times.

The data presented in Figure 4 show that a similar dependance of immediate failures on stress occurred at liquid nitrogen temperatures, although the scatter of points was greater. Thus, a broad range of strengths, even in the absence of stress corrosion, was again demonstrated. In fact, a greater range of strength values was

obtained at low temperature, and this effect also was found previously from the Instron tests.

Secondly, at room temperature the number of fibers failing immediately upon load application in the static fatigue tests was always much higher than would be predicted from previous experience with much lower strain rate tests in an Instron machine. It was concluded that a condition of sudden loading existed in the static fatigue experiments which imposed a transient stress level considerably higher than the nominal dead weight. A maximum momentary stress of twice the nominal was envisioned as a limit.

Since it is presumed that no stress corrosion affects strength at -196°C , a crude estimate of the sudden loading effect can be obtained by comparing the stress to cause 50% failures at liquid nitrogen temperature for the Instron tests with that for the static fatigue loading. The values are approximately 828,000 psi and 500,000 psi, respectively, or a ratio of 1.65 between the two loading methods. The same comparison for the two room temperature test series gives values of 507,000 psi and 350,000 psi, respectively, and a ratio of 1.45. At room temperature, the comparison

is complicated by the effects of stress corrosion, and one should probably expect a somewhat lower ratio than at liquid nitrogen temperature.

V. FUTURE WORK

Static Fatigue Tests in Desiccants

Preparations are nearly complete for conducting static fatigue tests at room temperature with the fibers submerged in a powerful desiccant solution. These experiments are intended to determine how much benefit, in terms of reduction or elimination of stress corrosion effects, may be obtained by removal of surface moisture and testing in anhydrous surroundings. Attempts to achieve complete removal of water from the surface of glass have usually involved evacuation and heating. Difficulty arises in the case of fine fibers, since heating to temperatures necessary for complete removal of surface moisture alters the unique properties of the fibers which contribute to their high tensile strength. Often, the result has been a loss in strength rather than the hoped-for improvement.

In our planned experiments, fibers from the same batch used in all previous static fatigue tests will be submerged in an organic solvent solution of a compound

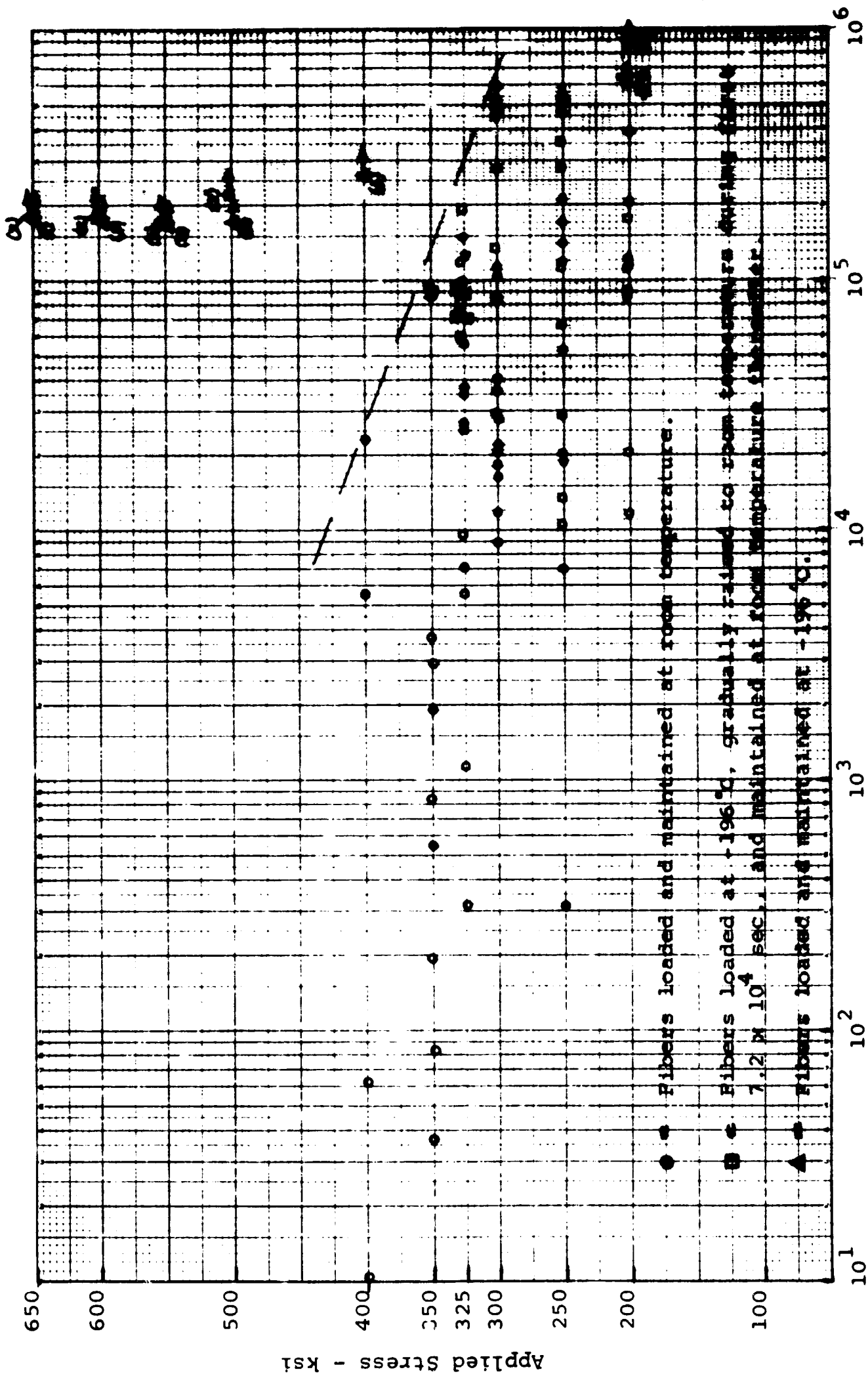
which is extremely reactive to water. Examples of such solutions are triethylaluminum in heptane or aluminum hydride in diethyl ether. In each case, contact with water brings about an irreversible reaction yielding a gaseous product and aluminum hydroxide. The limitation of this type of desiccant is not any finite equilibrium water content, but only the ability of the solution and the active compound to reach the water molecules.

The test apparatus is constructed so that the samples and desiccant solution are totally enclosed. The chamber is purged of air and moisture with dry nitrogen before the desiccant is admitted. Loads are applied to the samples by the same system used in previous tests, except that in the present case the supporting cords pass out of the chamber through a mercury seal. Also, the buoyancy effect from the liquid solution must be taken into account in adjusting the dead weight loads, and an epoxy potting compound must be used in place of the usual sealing wax for gripping the fibers in order to resist the heptane solution.



Figure 1 - General View of Low Temperature Static Fatigue Test Apparatus

Virgin E-Glass Fibers
 $L = 1.0 \text{ in.}$ $D = 0.5 \times 10^{-3} \text{ in.}$



Time Under Load - Seconds

Figure 2

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 Nov. 27, 1964

STATIC FATIGUE OF E-GLASS FIBERS
DISTRIBUTION OF FAILURE TIMES

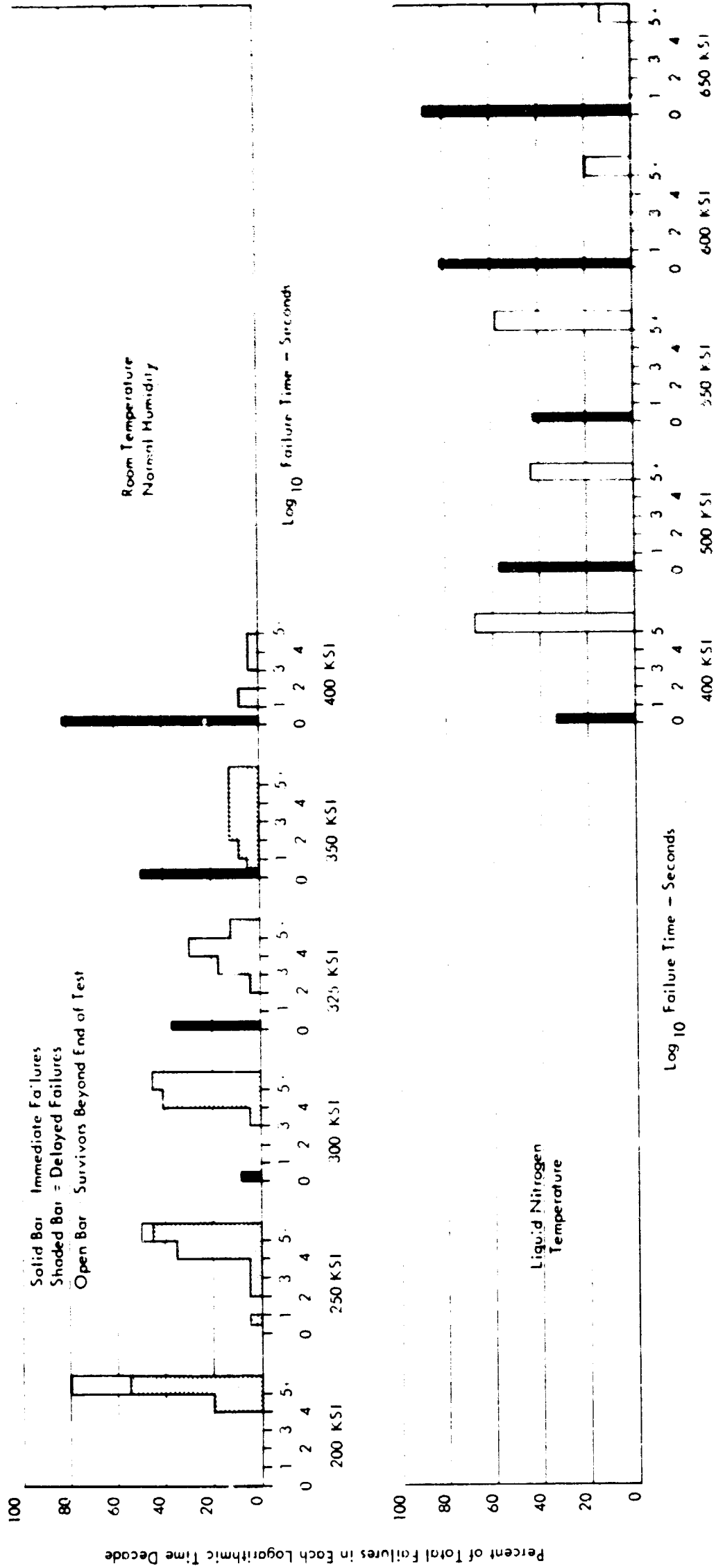


FIGURE 3

FIGURE 4

IMMEDIATE FAILURES UPON LOAD APPLICATION

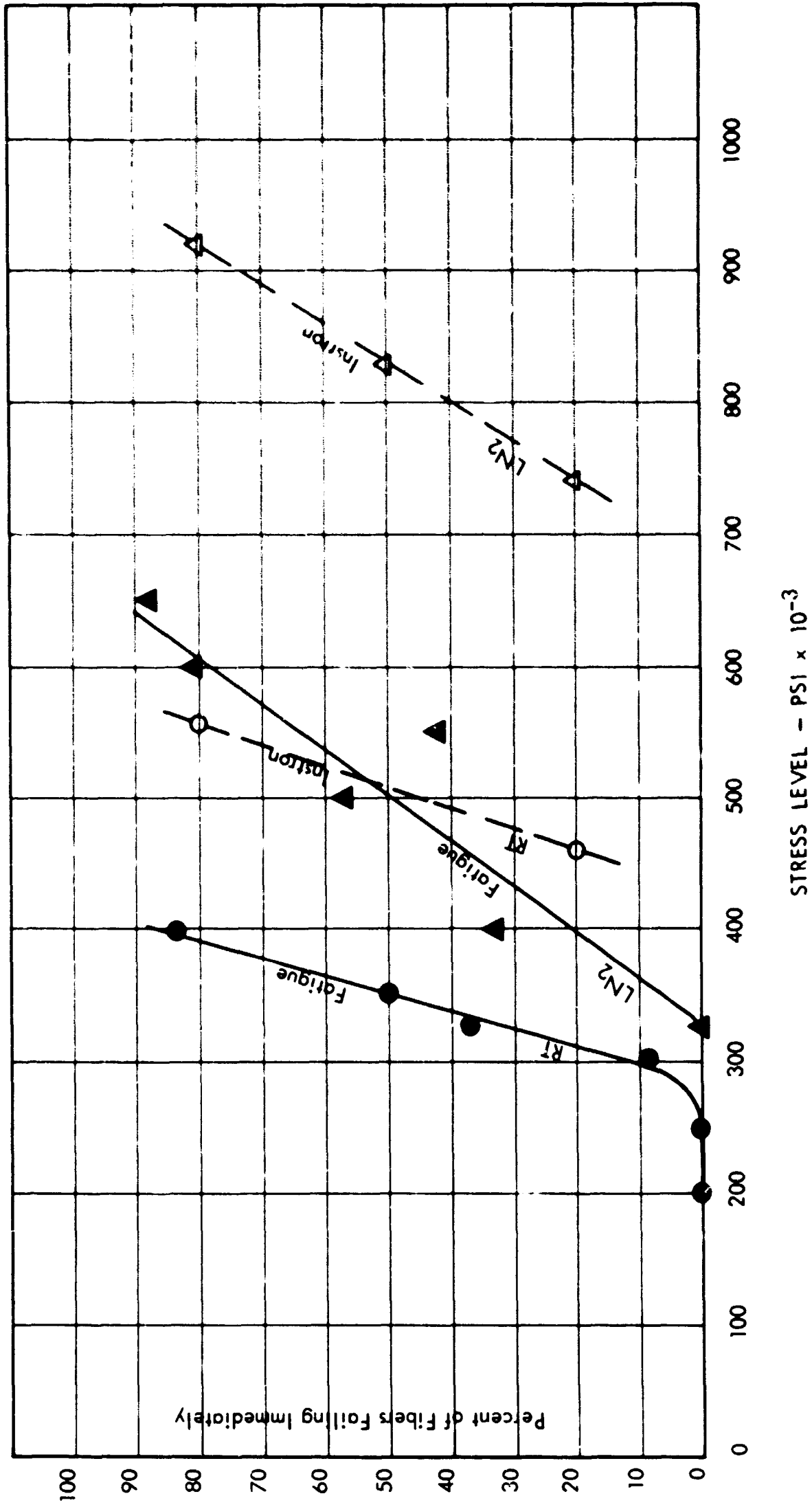


FIGURE 5

DELAYED FAILURE AS A FUNCTION OF APPLIED STRESS

FIG. 5

