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Environmentally Assisted Crack Growth in Glass

G. M. Sinclair and S. P. Withrow

Ballistic Branch
Mechanics Division

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ABSTRACT

Glass exhibits the properties of high compressive strength and low density. However, the surface of unprotected glass contains flaws which grow under low stress in moist conditions to critical size for failure.

Various methods were used to investigate and increase the reliability of glass under stress. Data were obtained on failure of unprotected glass plates subjected to biaxial tension at about 50-percent relative humidity. Analysis by extreme-value statistics indicated that the failure condition could be represented by a plane surface in a three-dimensional coordinate system composed of extreme-value probability, log stress, and log time. Removal of surface flaws by etching in 5-percent aqueous hydrofluoric acid increased the mean failure strength from approximately 30,000 psi to 145,000 psi, with a value of 300,000 psi biaxial tension being attained in one case.

Increases in lifetimes of one, two, and three orders of magnitude were obtained by protecting the glass from atmospheric moisture by preheating and coating with petrolatum, preheating glass and coating with preheated petrolatum, and experimenting at -30°F, respectively. Slight improvement in minimum time to failure was obtained by eliminating the weaker specimens by proof testing.

PROBLEM STATUS

This is an interim report on one phase of the problem; work on this and other phases is continuing.

AUTHORIZATION

NRL Problem F01-17

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ENVIRONMENTALLY ASSISTED CRACK GROWTH IN GLASS

INTRODUCTION

Glass is subject to static fatigue, a failure phenomenon analogous to stress corrosion in metals. If loaded for a period of time at some tensile stress level below the normal breaking stress, surface flaws can grow to critical size for unstable rapid crack growth by reacting with the moisture in the atmosphere. In a recent study, G. R. Irwin (1) has shown that crack speed can vary by several orders of magnitude between "dry" and "moist" testing conditions. S. M. Wiederhorn (2) found, under wet conditions and over a range of temperatures, that crack velocity depended on temperature and varied approximately as an inverse function of stress to the eighth power. J. O. Outwater and D. J. Gerry (3) studied the dependence of fracture energy on the velocity of fracture over a range of velocities and in several environments. These investigations indicate a strong dependence of static fatigue on stress level, environment, and time.

The surface of most unprotected glass contains a random distribution of flaws or cracks. In the present study, it was assumed that failure could be attributed to the largest of the flaws present in the test surface, since, under steady growth conditions, this flaw would achieve critical size for rapid fracture first. Since we are concerned here with the occurrence of only the largest flaw in each specimen, we would anticipate that a distribution function of the extreme-value type would be best for describing stress at failure for constant time or time to failure at constant stress.

Analysis in this report is based on the Weibull distribution (4). With this distribution the cumulative probability, $P(X)$, for some value of the variable equal to or less than X may be given by

$$P(X) = 1 - \exp\left[-\left(\frac{X - X_u}{X_0}\right)^m\right] \tag{1}$$

where

m = constant,

X_0 = scale or location parameter (constant), and

X_u = cutoff parameter (value of X below which the cumulative probability $P(X) = 0$).

By taking logarithms and rearranging terms, the above relation is put into a form more convenient for graphical analysis, i.e.,

$$ln\, ln\, \frac{1}{1 - P} = m\, ln\, \frac{X - X_u}{X_0} \tag{2}$$

which is simply a straight line with slope m.

Under conditions of fixed environment, the probability of failure P will be a function of the two principal variables, stress σ and time t. For any constant probability of failure P, the stress and time will be related through some inverse function. If the

variables P, σ, and t are measured on three orthogonal axes, the failure condition will be defined by a surface in the P, σ, t space.

Since crack-growth rate, hence life, t, depends upon the moisture content of the environment, it is expected that the failure surface will change with different environments. For all environments, however, the failure surface will in theory intersect the σ-P plane at $t = 0$ in the same curve, the "instantaneous" loading-time curve. If it is assumed that there is some fixed relationship between flaw size and critical stress for rapid fracture ($\sigma\sqrt{\pi a} = K_{Ic}$, for example), the "instantaneous" loading-time curve simply represents a measure of the initial distribution of the largest flaws unaffected by environment or time. Since experimentally we are always dealing with finite rates of loading, this curve cannot be measured directly.

One further observation may be made regarding the failure surface. Since no failure will occur at zero stress. or as Charles and Hillig have postulated (5), below the fatigue limit, the failure surface must become asymptotic to the P-t plane for some low value of σ.

OBJECT AND SCOPE

The object of this study was to explore ways of obtaining increased assurance of long-time survival of massive glass. Various protective environments and a high-stress preload or "proof" stress were used. As a further object it was desired to explore the applicability of extreme-value statistics in describing the stress- and time-dependent failure of glass. Because of time limitations, only high stress levels and relatively short failure times were investigated.

EXPERIMENTAL APPARATUS AND PROCEDURE

The samples used were soda lime glass cover slides, with dimensions 4 x 3-3/8 x 0.050 in. The glass slides were stressed by concentric ring loading in static loading machines (Fig. 1). Metalfilm strain gages were bonded to the shaft of the weigh bar in a four-arm Poisson bridge configuration. Output was read on a continuous plotting recorder, with the chart speed either at 0.5 mm/sec or 0.25 mm/sec. Each bridge circuit and corresponding recorder was calibrated on an Instron testing machine. Load was applied to the glass samples by means of the weigh-bar mechanism at a rate of approximately 3000 psi/sec. Felt was used between the glass and the rings to distribute the load. Since the test device was of a constant-displacement type, relaxation in the felt resulted in some fall-off of load with time, and this effect was corrected for during the tests. The felt was vacuumed after each test to remove any glass slivers remaining.

Tests were conducted on unprotected glass in the laboratory environment and in some protective environments, as described later. If failure did not occur within one hour the tests were terminated. A sample size of 24 slides was used in each series of tests on unprotected glass. In some exploratory tests a smaller sample size was used.

RESULTS AND DISCUSSION

Unprotected Glass

The tests on unprotected glass were made under laboratory conditions in the temperature range 72° ± 8°F and with the relative humidity 45 to 65 percent. Results for stress at failure in continuous loading-rate tests are plotted in Weibull probability form in Fig. 2. The data at the loading rate of 3000 psi/sec were taken from tests using the

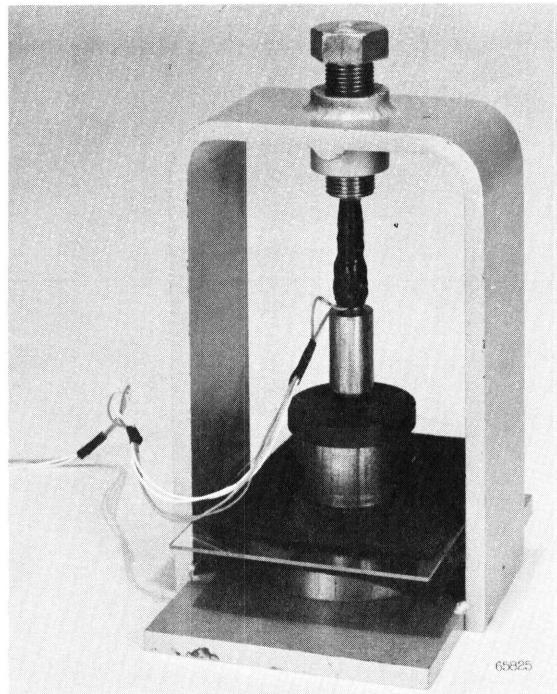

Fig. 1 - Concentric ring static loading machine used to stress glass samples

static loader, while the tests at the loading rate of 2170 psi/sec were made on an Instron. The difference between the means of the 3000 psi/sec and 2170 psi/sec tests was not statistically significant.

Samples could not be loaded to a specific stress instantaneously to obtain a probability versus stress at failure distribution for $t = 0$. Since the data points for long failure times were relatively unaffected by time required to apply the load, these data were extrapolated back to ln (time) = 0, ($t = 1$) in Fig. 3, and a stress at failure probability distribution for $t = 1$ was obtained. These data points obtained by extrapolation result in a straight-line relation, as shown in Fig. 2. The slope m of this line is 3.0.

Figure 3 shows a Weibull probability — time to failure graph for unprotected slides at various constant stress levels. The slope of the lines, n, is the same for the different stresses applied and has a value of 0.249.

From Figs. 2 and 3 the failure surface for unprotected glass can be determined. From Fig. 3, because n, the slope of the probability versus time graph, is a constant at the various stresses used in this investigation, and from Fig. 2, because the P-vs-σ curve for $t = 1$ is a straight line, the failure surface can be represented by a plane in $ln\, ln\ 1/(1-P)$ (Weibull probability), $ln\ \sigma$, $ln\ t$ space. From Eq. (2), the surface has the form

$$ln\, ln\, \frac{1}{1-P} = m\ ln\, \frac{\sigma - \sigma_u}{\sigma_0} + n\ ln\, \frac{t - t_u}{t_0}\ . \tag{3}$$

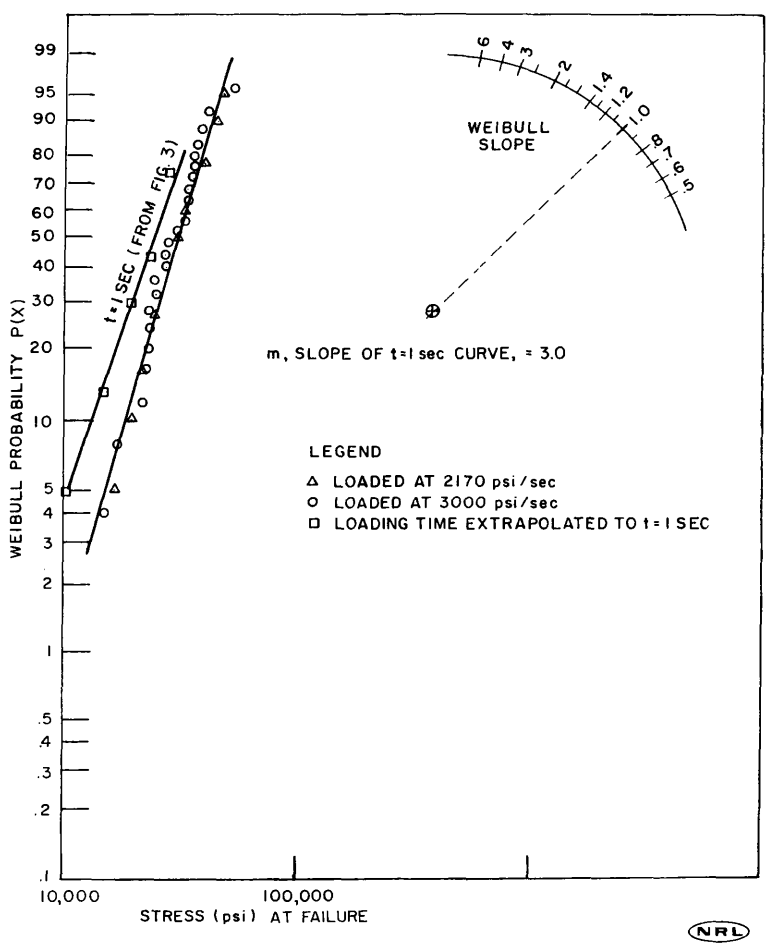

Fig. 2 - Percent failed versus stress at failure for continuous loading

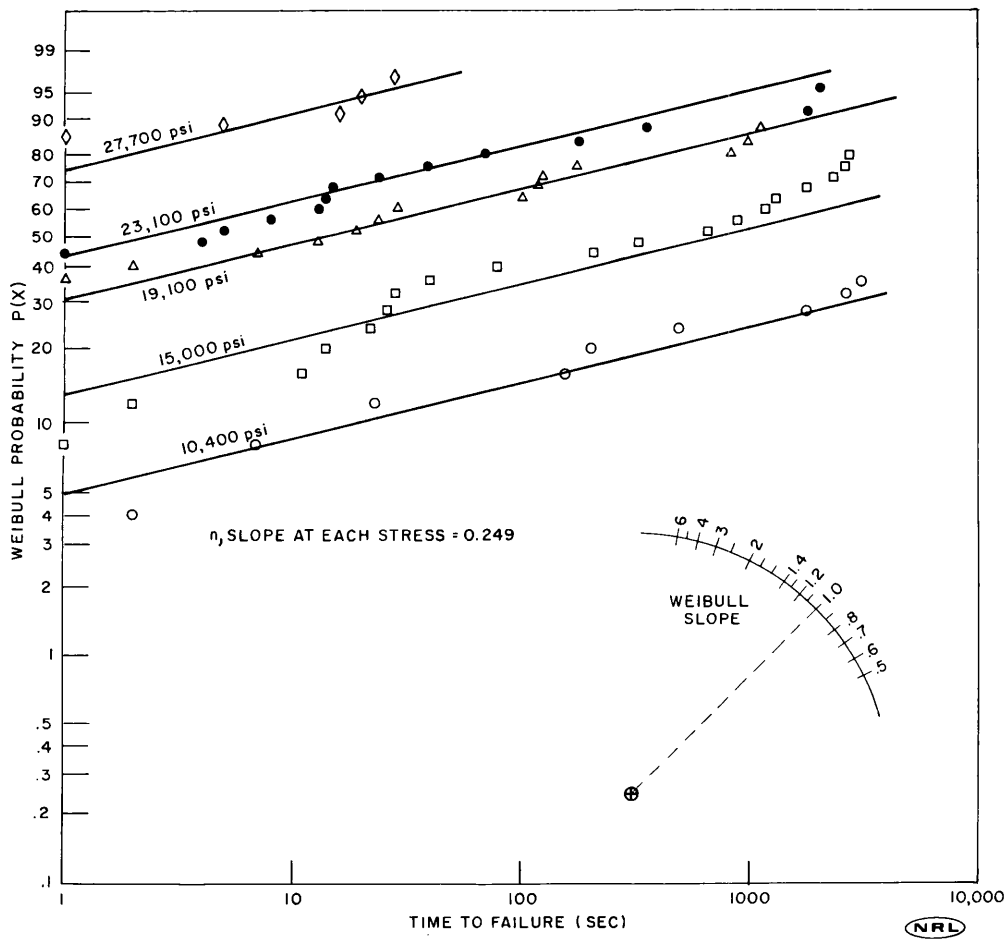

Fig. 3 - Probability of failure versus time to failure for several high constant stress levels. The $t = 1$ sec data points are plotted in Fig. 2.

As previously mentioned, this surface must eventually become asymptotic to the P-t plane at low stresses; however, it appears from the data that at stresses above 10,000 psi a plane surface provides a good representation of the failure condition. The straight-line relationships of Figs. 2 and 3 imply (by extrapolating back to $P(X) = 0$) that the cut-off parameters σ_u and t_u are small compared to the stresses and times considered here and may be neglected. The failure plane then has the form $ln\ ln\ [1/(1-P)] - m\,ln\ \sigma - n\ ln\ t =$ constant. When t equals 1 sec, $n\ ln\ t$ goes to zero, and the constant can be evaluated. If we take $P = 0.63$, the first term goes to zero and the constant is simply $(-m\,ln\,\sigma)$, where σ is the stress associated with $t = 1$, $P = 0.63$ from Fig. 2, i.e., 19,500 psi. The equation of the failure plane for unprotected glass is, for $\sigma > 10{,}000$ psi

$$ln\ ln\ \frac{1}{1-P} - 3.0\ ln\ \sigma - 0.249\ ln\ t = -29.6\ . \tag{4}$$

This plane is shown in Fig. 4.

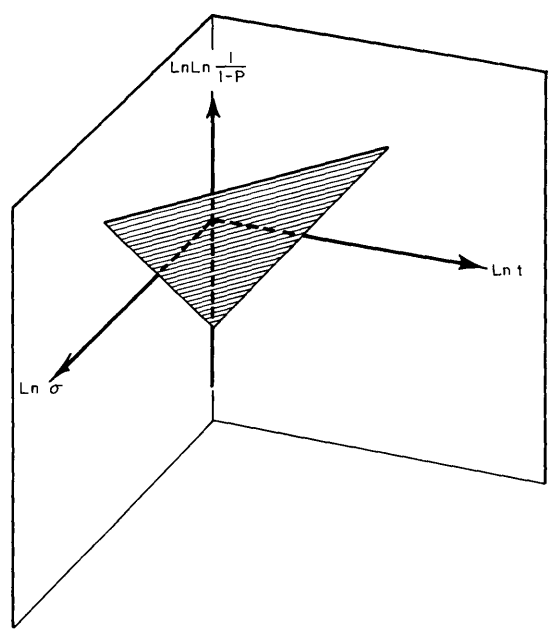

Fig. 4 - Failure surface obtained from unprotected glass

Proof Testing of Unprotected Glass

One means of increasing the expected life of unprotected glass samples at some constant stress is to eliminate the weakest specimens in the population by prestressing or "proof" testing at some higher stress. The survivors have only small flaws, which require more time at the low stress to grow to critical size for fast fracture. Figure 5 presents some results obtained on a sample of survivors of a 27,500-psi proof test.* The three groups were cyclically loaded in an Instron machine for 15 or 60 second cyclic "hold" times at maximum stresses of 17,200 or 13,250 psi, as indicated. Time at maximum load was summed up for purposes of plotting the data; time spent in cyclic loading and unloading was not included.

*NRL data obtained by J. A. Kies from research in progress.

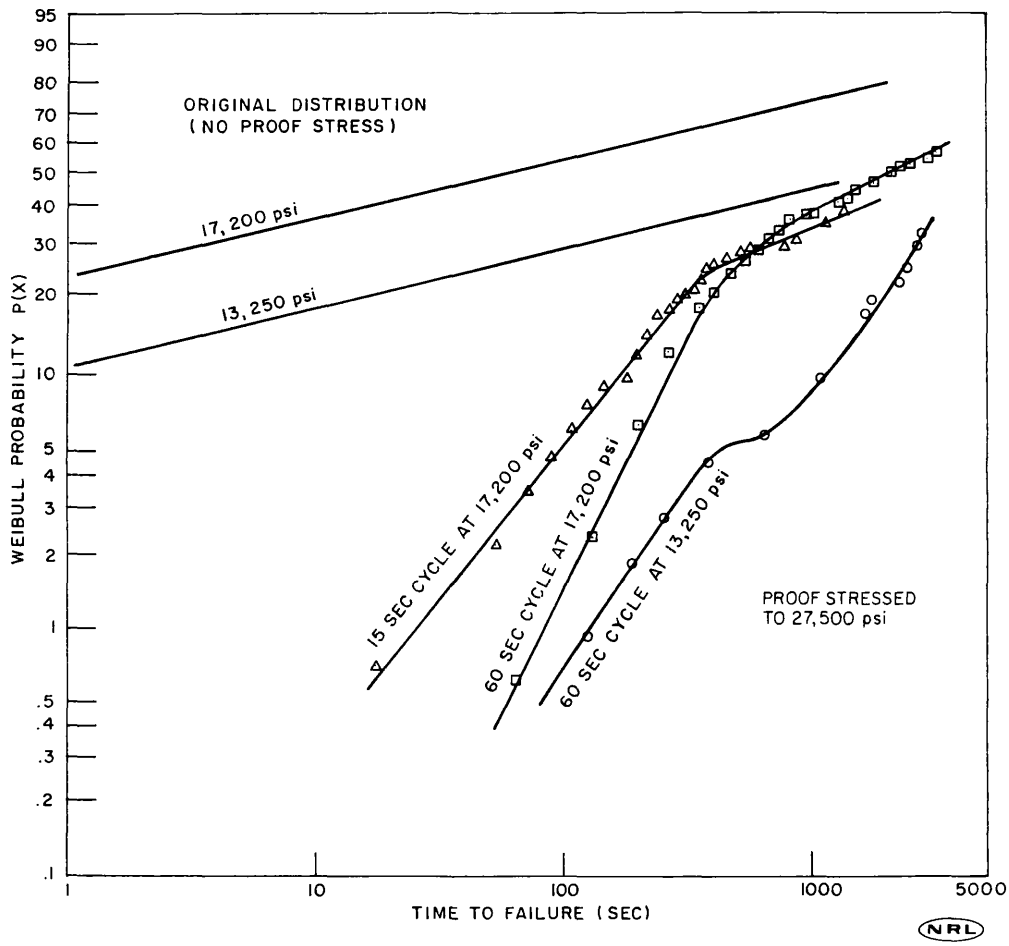

Fig. 5 - Comparison of time to failure of unprotected glass samples and proof-stressed samples. Changes in slope of proof-tested graphs may indicate failures after proof stresses are due to a complex distribution of largest crack sizes.

By comparing the P-t curves for the original population with those obtained from samples which survived the proof test, it can be seen that the proof test did insure some increase in life at a subsequent lower stress. However, the crack-growth rate under the test conditions imposed was sufficiently rapid that the increase in life at the probability of failure of one percent, for example, is still only on the order of 10 to 100 seconds. This result was obtained in spite of the fact that the proof test eliminated approximately half of the specimens in the original sample. After proof testing the graphs have a bend, indicating a complex distribution of failure times. This phenomenon will be studied in a future report.

Estimates of minimum expected life for unprotected glass at some lower stress following survival of a proof test can be made with Eq. (4). The procedure is illustrated schematically in Fig. 6. If a specimen is loaded for proof test at stress σ_1 for time t_1 and survives, the largest crack present must be smaller than that associated with P for failure, where P can be calculated from Eq. (4), since σ_1 and t_1 are given. If the specimen is then loaded instantaneously to stress level σ_2, the minimum time that will be taken up in crack growth to produce rapid fracture at stress σ_2 is given by $t_2 - t_1$, where t_2 can be calculated from Eq. (4) for σ_2 and setting P equal to the same constant.

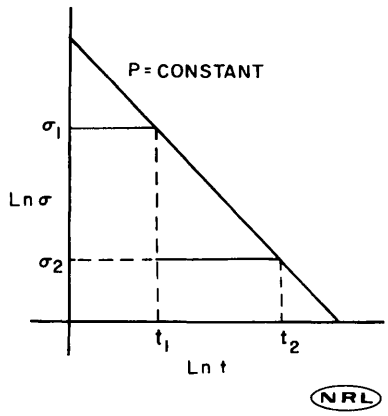

Fig. 6 - Schematic of theoretical estimate of minimum life ($t_2 - t_1$) at some stress σ_2 following survival of a proof test at some higher stress σ_1

Strength of Etched Glass

Strength levels achieved by the original glass loaded at 3000 psi/sec ranged from 15,000 to 50,000 psi, implying that the surface flaws present were several thousandths of an inch in depth. To obtain supplemental data on strength levels attainable in this glass when surface flaws are minimized, a hydrofluoric acid etch treatment was utilized. Nine samples were etched in a 5-percent aqueous solution of hydrofluoric acid for 15 minutes each at 72°F and subsequently loaded to failure at 3000 psi/sec. The reduction in severity of surface flaws due to etching resulted in an increase in mean failure strength from approximately 30,000 psi to 145,000 psi, with the strongest specimen achieving a strength of 300,000 psi in biaxial tension. A comparison between the strength of the etched-glass samples and that of the original untreated glass is made in Fig. 7. Much branching or "forking" of the cracks occurred on failure at these high stress levels in the etched glass.

Protective Coatings and Environments

Since atmospheric moisture is primarily responsible for surface crack growth in this glass (1), some exploratory experiments were carried out to determine the relative effectiveness of various protective coatings and environments. The resulting data are summarized in Fig. 8.

In one series of tests the original glass was heated to 300°F for 15 minutes in an electric furnace and then immediately coated on both sides with petrolatum and one layer of aluminum foil 0.0007 in. thick. The protected samples were then allowed to return to room temperature before being tested. Comparison of the life of the protected samples with that of the original unprotected glass indicates that this treatment was only moderately effective in increasing the life.

In a later test, the petrolatum was heated at 280°F for 25 minutes to evaporate some of the water from it and then used for protection. The resulting lifetimes were about two orders of magnitude greater than those for the unprotected glass.

A second series of tests was made on samples of the original glass enclosed in a dry atmosphere of liquid nitrogen vapor maintained at -30°F. In this environment an increase in life of three orders of magnitude over that of the unprotected glass at the same stress was obtained.

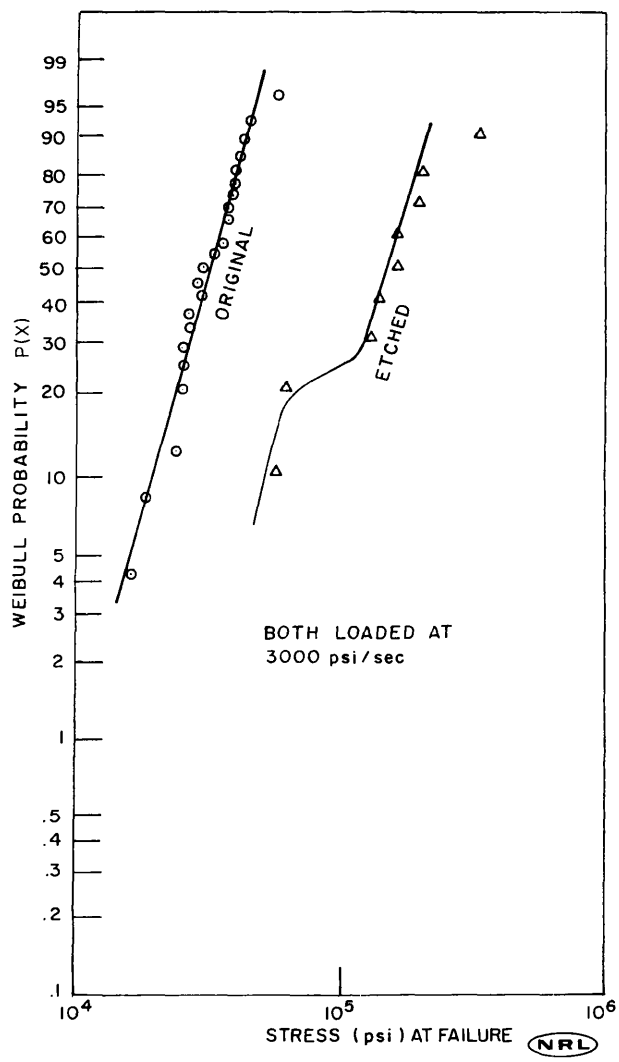

Fig. 7 - Comparison of strength of etched glass samples and that of the unprotected glass samples

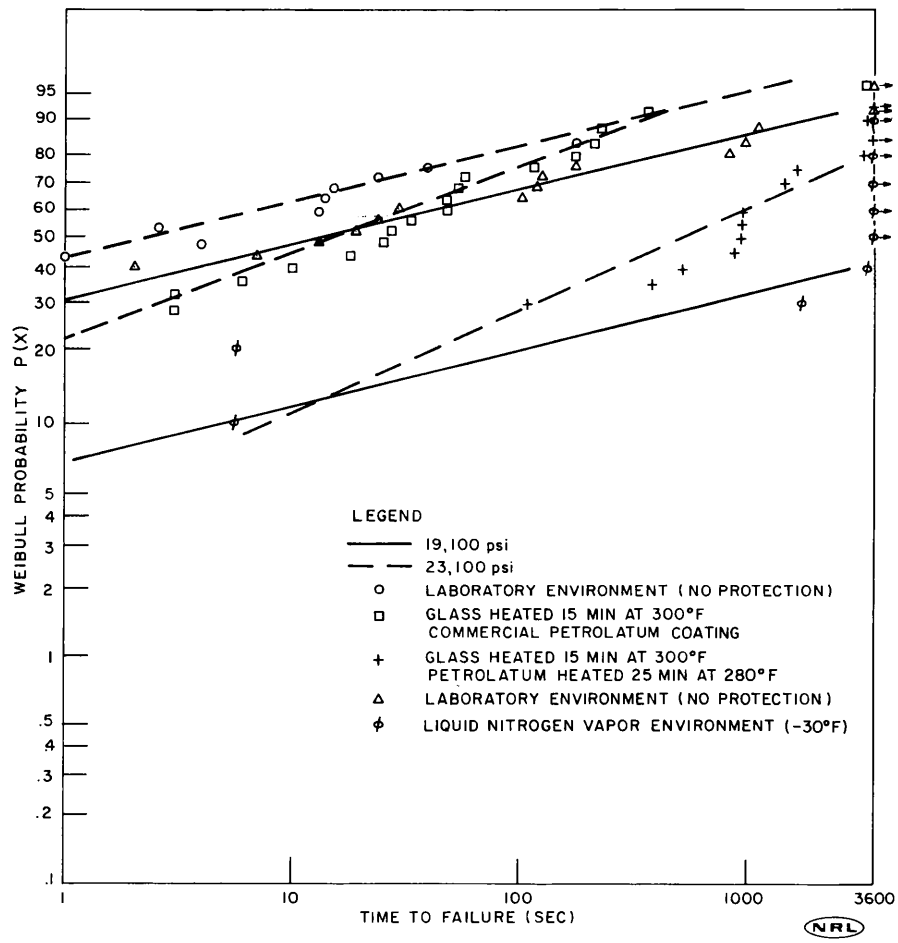

Fig. 8 - Comparison of results for laboratory environment (no protection) and several protective environments

CONCLUSIONS

Within the limits of the brief experiments described here, the following conclusions appear reasonable.

1. The Weibull distribution function provides a good description of failure time at fixed stress or strength at fixed time in static fatigue of glass.

2. In the high-stress, short-time region the failure condition for unprotected glass can be approximated by a plane surface in probability of failure, ln stress, ln time space.

3. The mean strength of this glass was increased from 30,000 psi to 145,000 psi as a result of etching in hydrofluoric acid.

4. A moderate improvement in life was obtained when the original glass surface was protected from moisture by being dried in an oven and coated with petrolatum.

5. An improvement of three orders of magnitude in life was obtained when the measurements were made in an atmosphere of liquid nitrogen vapor at -30°F.

6. Some improvement in minimum life to failure can be obtained by eliminating the weaker specimens in a sample population by proof testing. In unprotected glass the crack-growth rate is such that the improvement is not of practical significance.

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Various methods were used to investigate and increase the reliability of glass under stress. Data were obtained on failure of unprotected glass plates subjected to biaxial tension at about 50-percent relative humidity. Analysis by extreme-value statistics indicated that the failure condition could be represented by a plane surface in a three-dimensional coordinate system composed of extreme-value probability, log stress, and log time. Removal of surface flaws by etching in 5-percent aqueous hydrofluoric acid increased the mean failure strength from approximately 30,000 psi to 145,000 psi, with a value of 300,000 psi biaxial tension being attained in one case.

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Reliability						

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