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SALT WATER RESISTANCE OF SURFACE-COMPRESSION STRENGTHENED GLASS

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NAVAL ORDNANCE LABORATORY, WHITE OAK, SILVER SPRING, MARYLAND

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SALT WATER RESISTANCE OF SURFACE-COMPRESSION  
STRENGTHENED GLASS

Prepared by:

Roger E. Wilson, H. A. Perry, F. J. Koubek

**ABSTRACT:** Three types of ion-exchange surface-compression strengthened (SCS) glass, with ultimate flexural strengths of 38,000 to 66,800 psi, were abraded and maintained in circulating salt water for periods up to three years under constant flexure at stress levels of 60-70% of their ultimate strengths. Tempered Pyrex with strength of 11,700 psi also was abraded and maintained for like periods at the 50% stress level in salt water. There were no long-term fatigue failures. Residual strains after unloading seemed to be asymptotic to zero. Surface regression rates were not greater than  $4.5 \times 10^{-5}$  cm/year. Their average flexural strengths gained 5-11%, with the strongest increasing to 77,800 psi. Their coefficients of variation decreased from 3.4-10.5% down to 2.2-5.3%. Their conservative ( $\bar{x}-3S$ ) strengths increased and became 6.2-49.0% higher. There were no differences between wet and dry flexural strength tests or between directions of static loading prior to testing. The important effect of time under stress and salt water was to make SCS glass stronger. Surface-compression strengthened glass is attractive for use as a naval structural material.

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Salt Water Resistance of Surface-Compression Strengthened Glass

The work performed in this test program on the stress and salt water resistances of four types of surface-compression strengthened glasses was supported in the beginning under NOL Task RRMA 03-043/212-1/R007-04-01 and was completed under NOL Task: 63702/54624/SHIP 12327 300 23A. This report is intended for use in the selection of naval structural materials for weapons, ships and other marine applications, and for use in planning additional research, development, testing and evaluation programs on naval structural materials.

Many of the materials discussed in this report were obtained from commercial sources. Their use by the Laboratory in no way implies Navy endorsement for naval applications. Neither is this consideration of a material by the Navy to be used for promotion purposes. There is no implication intended that other materials might not have performed as well as those selected for these studies.

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### SUMMARY

Surface-compression strengthened (SCS) glasses of four types in flat strips were loaded in flexure and maintained under constant stress in circulating salt water at 23°C for periods up to three years. The stress levels set during exposure ranged from 5,900 psi for tempered Pyrex to 46,800 psi for an ion-exchanged glass. These static stresses were fractions of the original mean dry strengths of the SCS glasses, which ranged from 11,700 to 66,800 psi. There were no fatigue failures. After exposure and unloading, the mean dry strengths of all SCS glasses were found to be higher than their original strengths. The original coefficients of variation of strengths of the four types were between 10.5% and 3.4%. In general, the stronger SCS glasses had lower variabilities. After exposure and unloading, all coefficients of variation were found to have decreased to between 5.3% and 2.2%. Calculated estimates of the conservative strengths ( $\bar{x}-3S$ ) of all types were increased by exposure and became 6.2% to 49% higher. Residual strains after unloading seemed asymptotic to zero. Weight losses after three years ranged from 0.112 to 0.315 mg/cm<sup>2</sup>. Two SCS glasses were less soluble than Pyrex. The highest average surface regression rate was estimated to be  $4.5 \times 10^{-5}$  cm/year. There were no significant differences in SCS glasses between wet and dry ultimate strengths or between directions of static loading prior to ultimate testing.

In general, the exposure of four types of abraded SCS glasses for three years to salt water and/or constant stress makes for a more uniform and predictable product. It is concluded that SCS glasses are attractive for use as a naval structural material.

### INTRODUCTION

A need exists for naval structural materials that will survive in hostile environments for ten to twenty years between refurbishment opportunities with minimal deterioration [1].

In recent years, SCS glass [2] has become increasingly attractive as a naval structural material because of its very high compressive strength, low density, relative insolubility, creep resistance, and transparency. With the development of improved surface-compression strengthening processes, the flexural strengths of silicate glasses have been increased to the 50,000 to 100,000 psi region. Weight-for-weight, and with these strengths, SCS glass corresponds to aluminum alloys with yield strengths of 57,000 to 115,000 psi, or to titanium alloys with yield strengths of 90,000 to 180,000 psi, or to steels with yield strengths of 160,000 to 320,000 psi, making SCS glasses serious contenders for use in high-performance, load-bearing naval structures.

Structures intended for use in ocean environments, e.g., pilings, navigation buoys, hulls, and weapons casings, must withstand the

long-time effects of salt water and marine organisms as well as the combined and simultaneous stresses imposed by wave action, external pressure, temperature changes, impacts, and flexural and torsional loadings. When SCS glasses were first proposed as materials for naval structures, especially for hulls for deep ocean vehicles, these long-time environmental effects became a matter for concern. Since there was insufficient previous experience upon which to predict the performance of SCS glasses under stress and strain in the marine environment, two long-term studies were launched.

The first of these was underwater exposure investigation of SCS glasses, locked in constant strain, that was conducted over a 16-month period at three sites in the sea. The results of this study are covered in another report [3]. It was concluded that no significant deterioration occurred during these tests.

The second study was a laboratory investigation in which glass specimens were exposed to salt water at 23°C under static flexural loads for periods up to three years. The details of this investigation are described in this report.

#### BACKGROUND

The older method of locking compression stresses in surfaces to strengthen a glass is to quench it from a temperature above its transformation temperature. So long as the locked surface-compression stress is not overcome, the glass remains resistant to applied forces. The strength of tempered glass is proportional to the intensity of the locked compression stress. The highest locked stresses and strengths that are attainable by tempering are about 30,000 psi, Figure 1.

An improved method of producing SCS glass was announced in 1962. This is by ion exchange, e.g., by soaking an alkali glass in a molten ionic salt while the glass is at an elevated temperature below its transformation temperature. If the salt bath alkali ions are larger than the ions residing in the glass then, by ion exchange, the larger ions stuff and swell the surface, while the interior glass remains unchanged, and a compression stress is locked in all surfaces. Intense stresses can be developed. In some combinations of glass and salt, these stresses exceed 120,000 psi (reference 2), with correspondingly high strengths. Ion-exchange strengthened glass can be at least four times stronger than tempered glass.

Other differences exist between the tempering and ion exchange processes. It is difficult to maintain close tolerances in articles during tempering, because it is necessary to heat the article above its transformation temperature where its viscosity is less than  $10^{12}$  poises. Gravity and handling forces can then deform the article. In contrast, during ion exchange at below the transformation temperature the silicate network remains nearly intact and the article is still elastic. Gravity and handling forces do not warp the article.

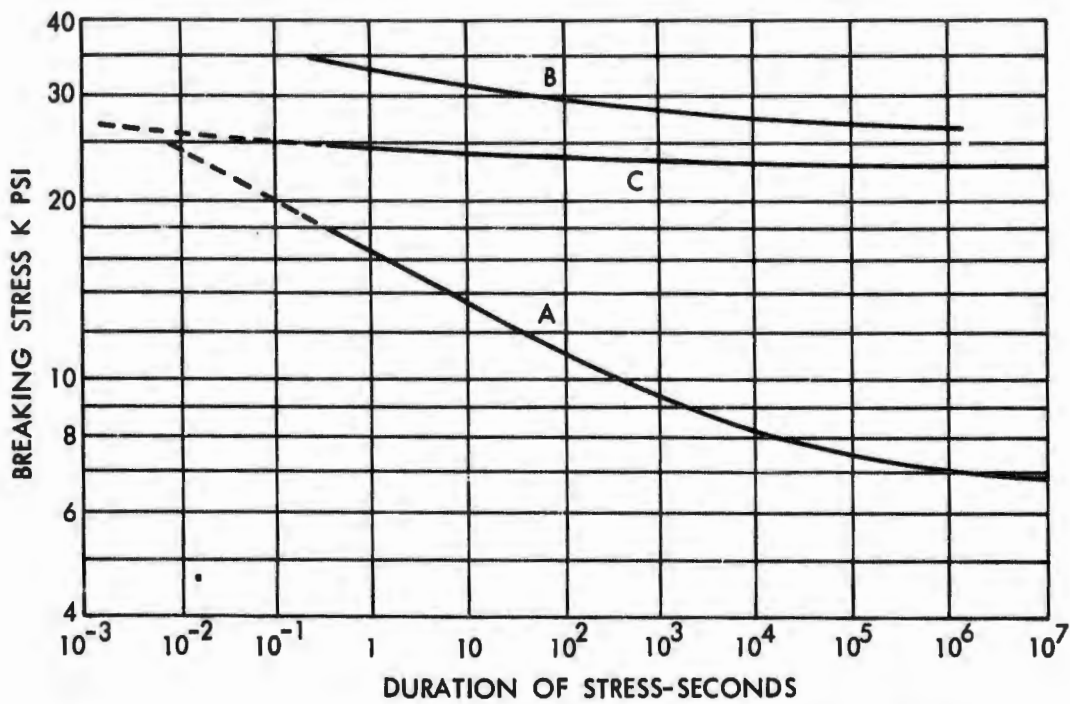


FIG. 1 STRESS-TIME CHARACTERISTICS OF GLASS BROKEN IN FLEXURE TESTS AT ROOM TEMPERATURE. COMPOSITE CURVES. A - ANNEALED GLASS, TESTED IN AIR. B - TEMPERED GLASS, TESTED IN AIR. C - ANNEALED GLASS, TESTED IN VACUUM. SHAND, E. B., GLASS ENGINEERING HANDBOOK, P. 51.

Chill tempering produces a profile of compression stress that tapers through zero deep in the article. The stress-reversal depth is always about 1/3 the total thickness of the article. In thick sections the compressed layer is of substantial thickness. Ion exchange also produces a tapered compression stress profile. However, in contrast with the physics of quenching [4], ion diffusivities limit the stress-reversal depth severely. In 1964 when this study began, attainable stress-reversal depths did not exceed 0.020 inches, independent of section thickness, and were usually in the range of 0.005-0.010 inches. It was feared that such thin compressed layers, although strong to begin with, might be extremely susceptible to abrasion and corrosion, might lose strength rapidly, and might be unsatisfactory. Conversely, if found to be durable, these lightweight SCS glasses might be attractive and possibly become the fourth-generation naval structural material in the series: woods; alloys; composites; SCS glasses.

Both of the strengthening processes to attain high strengths require that the alkali-ion content of the glass be high. For a high temper, the thermal expansion coefficient must be high, which can only be attained by adding alkali. For a high-strength ion exchange, a substantial alkali content is again required. And, of course, high alkali glasses are less resistant to water, acids, and bases than low-alkali glasses. Figure 1 shows that tempered glass is relatively durable under stress in wet conditions. Rothermel [5] showed later on that ion-exchange strengthened glasses are far less susceptible to solution by hot water and hydrochloric acid than non-stressed glasses having the same chemical composition, Figures 2 and 3. But when this program began, no data existed on the durability of the ion-exchange strengthened products. Experiments to determine the resistances of tempered and ion-exchange strengthened glasses to abrasion sustained mechanical loads and wet environments are described below.

#### EXPERIMENTAL

STANDARD SALT WATER. The present study was made to gain insight into the strength behavior of SCS glasses under constant stress and in constant contact with sea water over long periods of time. It was appreciated that actual marine environments could not be duplicated in the laboratory. The salt composition and marine organisms in sea water vary to extremes both with geographical location and depth while unknown concentrations of many pollutants foreign to the sea are encountered in the various harbors frequented by the Navy's ships. In view of these obvious difficulties in duplicating conditions at sea, a salt water composition containing many of the salts expected to be corrosive to underwater structural materials, and described by the ASTM specification D-1141-52, was selected for use in our apparatus. Although the salinity was typical, other differences existed. The heavy metal salts normally found in concentrations less than 0.004% in

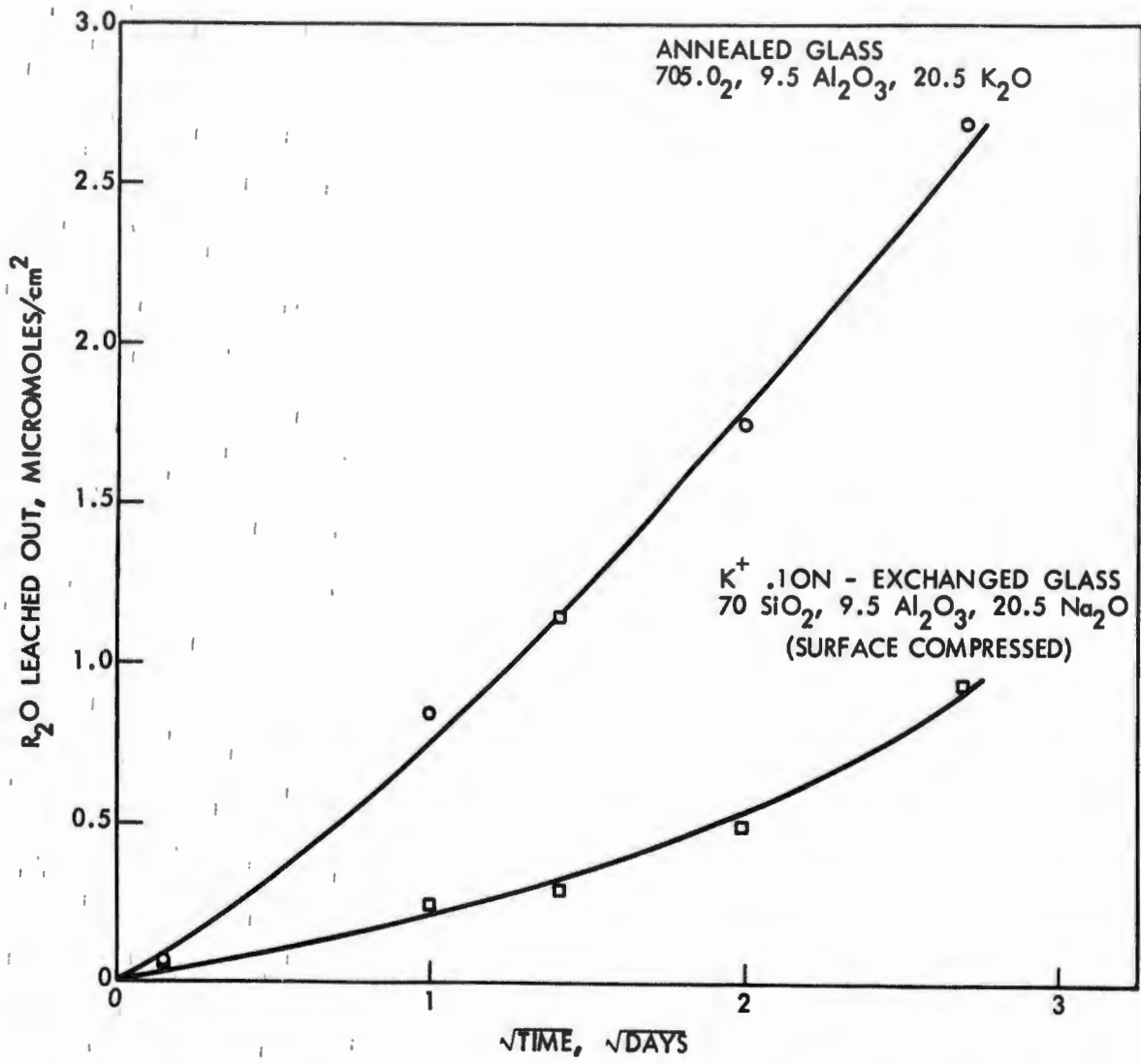


FIG. 2 EFFECT OF SURFACE COMPRESSION ON ALKALI LEACHED BY WATER AT 95°C (AFTER ROTHERMEL, J.AM. CERAM. SOC. VOL. 50 NO. II)

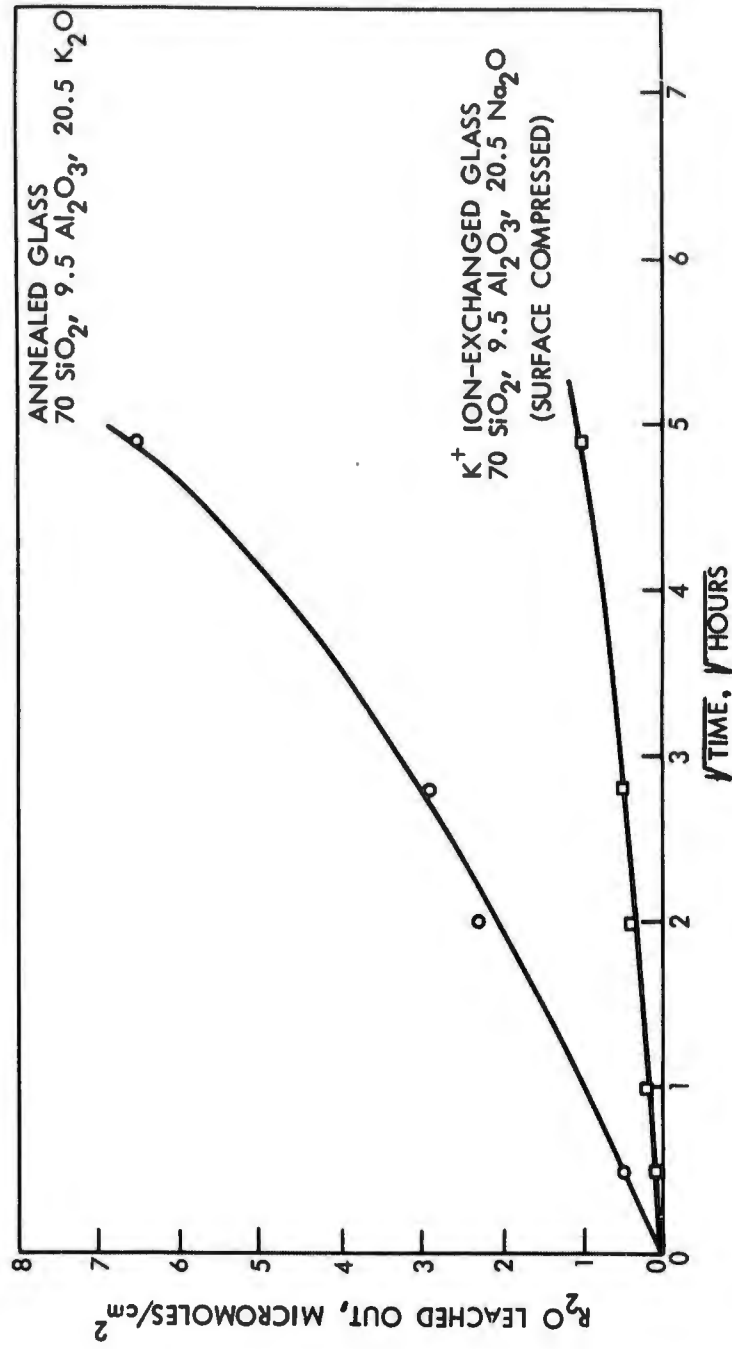


FIG. 3 EFFECT OF SURFACE COMPRESSION ON ALKALI LEACHED BY 5% HCl SOLIN AT 95°C (AFTER ROTHERMEL, J. AM. CERAM. SOC. VOL. 50 NO. II)

ocean waters were omitted. Also particulate matter which is normally present in sea water and capable of inflicting abrasive damage to under-water surfaces was omitted. Instead, each glass specimen was systematically abraded by sandblasting in its most vulnerable spot. Also, the growth of organisms was inhibited, and the oxygen contents were not regulated but reached equilibrium with room air.

In the present study the tendency to refer to the salt water circulated in the apparatus in contact with the specimens as "synthetic" and "artificial" sea water was avoided by referring to it as salt water or standard salt water, SSW.

The validity of laboratory-obtained data in predicting the outcome of actual use of a material in marine environments, e.g., its long-time strength, has yet to be established. Some light should be shed on this question by concurrent tests which were run on similar glasses under constant bending strain (not constant stress) stored for periods up to 16 months at three locations at the bottom of the sea. This work is reported in NOLTR 71-57. However, various technical differences between the experiments and the limited number of specimens that could be stored at sea make it illogical to attempt a rigorous comparison between the strength of those specimens with specimens stored in salt water in the laboratory.

GENERAL EXPERIMENTAL APPROACH. The apparatus used in the laboratory studies consisted of 144 individual fixtures, each capable of exerting a constant 4-point bending load on a flat 10-inch long specimen by a system of weights and levers. The specimens were immersed in salt water in plastic tanks. Figure 4 shows a schematic of this arrangement and Figure 5 is a photograph showing all 144 fixtures. Parallel studies in which flexurally stressed specimens exposed to laboratory air (50% R. H.) as well as specimens stored in air unstressed were conducted. At various time intervals, specimens were withdrawn, wiped, weighed, and then broken in a 4-point flexure fixture (see Figure 10) to determine their modulus of rupture (bending strengths). Observations of changes in strength were of prime interest, but weight changes were also of interest since strength change may be related to solubility.

Apparatus Details. A weight and lever method of applying a static load to the specimen was selected for simplicity and reliability. The lever arrangement made it easier to load a large number of specimens in a confined space, and it also greatly reduced the amount of weight needed to get the desired load. The alternative was to clamp the specimens to a fixed strain point. It also eliminated changes in stress by delayed relaxation or by fixture corrosion at points of contact.

A photograph of the loading system is shown in Figure 6. Parallel stainless steel rods mounted in polyethylene trays serve as bottom



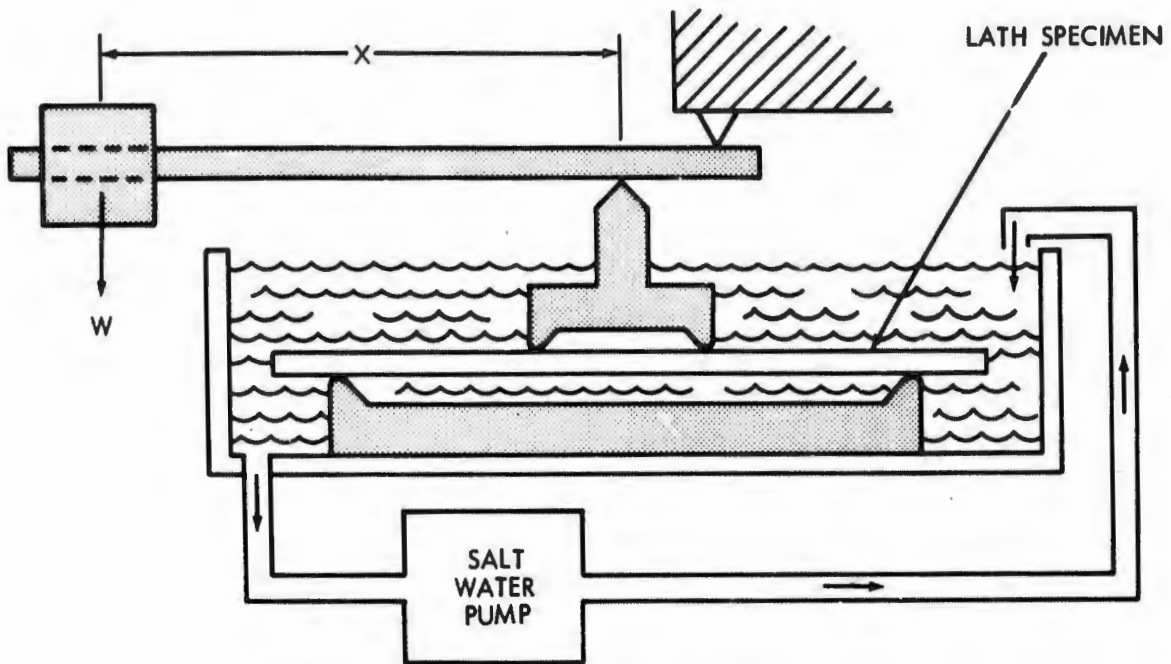


FIG. 4 SCHEMATIC OF A SINGLE SPECIMEN IN THE 144 SPECIMEN FIXTURE UNDER LOAD IN CIRCULATING SALT WATER



FIG. 5 APPARATUS FOR APPLYING VARIOUS BENDING LOADS TO 144 GLASS SPECIMENS  
WHILE IN CONSTANT CONTACT WITH CIRCULATING SALT WATER

NOT REPRODUCIBLE

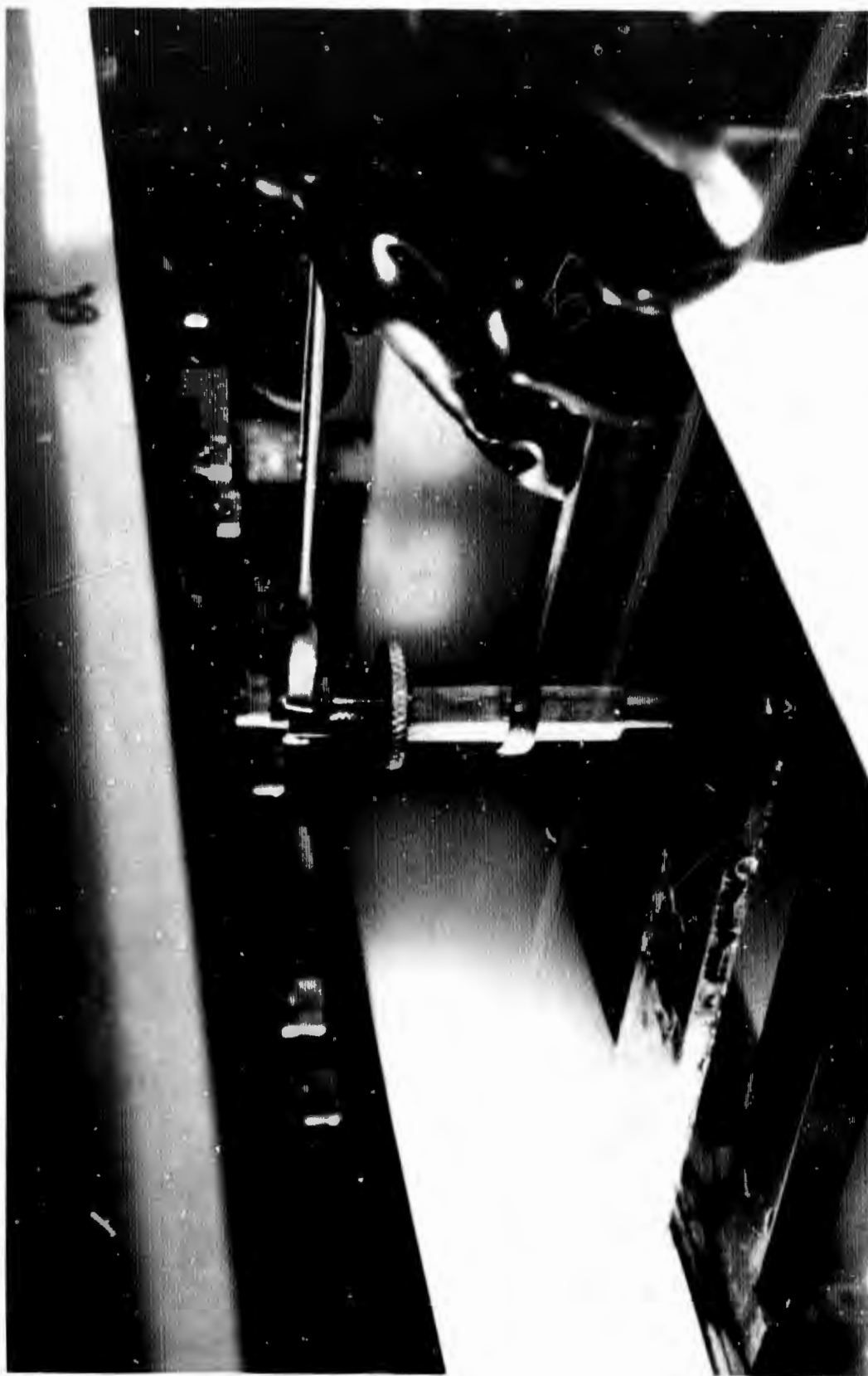


FIG. 6 ADJUSTING LENGTH OF VERTICAL POST TO LEVEL THE LEVER ARM

knife edges supporting the 10-inch long glass laths. Salt water was pumped continuously through the trays and covered the specimens at all times. As shown in Figure 6, the load was transmitted from the lever arm to the specimen by means of a vertical rod and a flat block of stainless steel having rounded ends. The latter made contact with the flexed specimen along parallel lines, which were spaced equally from the support rods. The adjustable length of the vertical rod assured application of the load normal to the specimen by leveling the horizontal lever. A small spirit level was used as a guide. The lever arm was hinged at one end and fitted with sliding weights on its free end. A specially designed fixture (not shown) was used to position each glass specimen so it was normal to the lower-supporting knife edges while centering the top knife edges and aligning them parallel to the lower ones.

Loads were set using a specially calibrated specimen equipped with a strain gage. This was an aluminum lath, identical in size and thickness with the glass laths, which had been calibrated by loading it with a Baldwin-Southwark Universal Test Machine and obtaining strain gage readings vs. loads. After positioning the calibrated specimen in one of the 144 test stations, the weights on the lever arm were moved outwards until the strain gage reading indicated the desired load had been obtained. The calibrated specimen was then replaced with the specimen to be tested, and the process repeated at the next station.

Standard salt water (SSW) was prepared from a commercial formulation made to ASTM specification D-1141-52, Table 1, dissolved in demineralized water. Individual pumps continuously circulated SSW to the top tray of each of the four stacks of trays. It then flowed by gravity, cascading from tray to tray and then back to the pump. Transparent plastic covers on the tray excluded dust and reduced evaporation losses. Weekly specific gravity and pH measurements aided in monitoring and adjusting the composition of the SSW.

Glass Specimens. The five commercial glasses selected for study are listed in Tables 2a and 2b. They included three alkali-alumina silicate glasses strengthened by ion exchange. A borosilicate glass was also included that was supplied in both the annealed and surface-compressed state, the latter being accomplished by chill tempering.

Tables 2a and 2b also list the approximate amounts of constituents found in these glasses as obtained by chemical analyses. All of the ion-exchanged glasses contained large amounts of alkali and the Herculite II had the highest amount along with a fair amount of zinc and phosphorous oxides.

The C-112 and C-113 glasses were of nearly identical composition, which was confirmed by repeated wet analyses. The difference in their strengths must be due to a difference in the ion-exchange processing.

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TABLE 1

COMPOSITION OF SALT WATER

Composition of "SEA-SALT" (1)		Comparison of Test Water and ASTM Standard		
Salt Added	%	Salt	g/liter	
			Test Water (2)	ASTM D-1141-52 (3)
NaCl	58.490	NaCl	28.57	24.53
MgCl·6H <sub>2</sub> O	26.460	MgCl	6.06	5.20
Na <sub>2</sub> SO <sub>4</sub>	9.750	Na <sub>2</sub> SO <sub>4</sub>	4.76	4.09
CaCl <sub>2</sub>	2.765	CaCl <sub>2</sub>	1.35	1.16
KCl	1.645	KCl	0.81	0.695
NaHCO <sub>3</sub>	0.477	NaHCO <sub>3</sub>	0.23	0.201
KBr	0.238	KBr	0.12	0.101
H <sub>3</sub> BO <sub>3</sub>	0.071	H <sub>3</sub> BO <sub>3</sub>	0.034	0.027
SrCl <sub>2</sub> ·6H <sub>2</sub> O	0.095	SrCl <sub>2</sub>	0.027	0.025
NaF	0.007	NaF	0.003	0.003
		Water	Bal	Bal

- (1) Lake Products Co., St. Louis, Mo.
- (2) Prepared by adding sufficient de-ionized water for each 41.953 g of "Sea-Salt" to make one liter
- (3) "Substitute ocean water" containing essential salts but omitting particulates and heavy metals normally present in sea water in concentrations less than 0.004 g per liter

TABLE 2a

Description of Glasses Selected for  
Salt Water Studies

<u>Commercial Designation</u>	<u>C-112</u>	<u>C-113</u>
Type of Glass	Surface Compressed	Surface Compressed
Type of Treatment	Ion Exchanged	Ion Exchanged
Supplier	Corning Glass Works	Corning Glass Works
Nominal Dimensions of Specimens - in.	10 × 1.5 × 0.1	10 × 1.5 × 0.1
Edges ground	yes	yes
Chemical Constituents* in Mole %:		
Silica (SiO <sub>2</sub> )	66.2	66.6
Sodia (Na <sub>2</sub> O)	8.6	8.0
Potassia (K <sub>2</sub> O)	0.1	0.3
Lithia (Li <sub>2</sub> O)	7.0	7.2
Alumina (Al <sub>2</sub> O <sub>3</sub> )	15.8	15.8
Calcia (CaO)	0.0	0.0
Magnesia (MgO)	1.3	1.4
Arsenic Oxide (As <sub>2</sub> O <sub>3</sub> )	0.0	0.0
Antimony Oxide (Sb <sub>2</sub> O <sub>3</sub> )	0.4	0.2
Boron Oxide (B <sub>2</sub> O <sub>3</sub> )	0.6	0.5
Titanium Oxide (TiO <sub>2</sub> )	tr	tr
Phosphorous Oxide (P <sub>2</sub> O <sub>5</sub> )	-	-
Zinc Oxide (ZnO)	-	-
Sulfate (SO <sub>3</sub> )	0.0	0.0
	100.0	100.0

\*Determined by wet analyses

TABLE 2b

Description of Glasses Selected for  
Salt Water Studies

<u>Commercial Designation</u>	<u>Herculite II</u>	<u>7740</u>
Type of Glass	Surface Compressed	Borosilicate
Type of Treatment	Ion Exchanged	a) Annealed b) Chill Tempered
Supplier	Pittsburg Plate Glass	Corning Glass Works
Nominal Dimensions of Specimens - in.	10 × 1.5 × 0.25	10 × 1.5 × 0.25
Edges ground	no	yes
Chemical Constituents* in Mole %:		
Silica (SiO <sub>2</sub> )	53.2	83.1
Sodia (Na <sub>2</sub> O)	13.4	3.8
Potassia (K <sub>2</sub> O)	0.0	0.2
Lithia (Li <sub>2</sub> O)	10.2	-
Alumina (Al <sub>2</sub> O <sub>3</sub> )	13.0	1.4
Calcia (CaO)	0.0	-
Magnesia (MgO)	0.0	-
Arsenic Oxide (As <sub>2</sub> O <sub>3</sub> )	0.1	-
Antimony Oxide (Sb <sub>2</sub> O <sub>3</sub> )	0.0	-
Boron Oxide (B <sub>2</sub> O <sub>3</sub> )	0.2	11.5
Titanium Oxide (TiO <sub>2</sub> )	tr	-
Phosphorous Oxide (P <sub>2</sub> O <sub>5</sub> )	9.3	-
Zinc Oxide (ZnO)	0.6	-
Sulfate (SO <sub>3</sub> )	0.0	-
	100.0	100.0

\*Determined by wet analyses

The C-113 glass may have been treated with an ion of larger radius than that used on the C-112 glass.

After brief preliminary studies of various size specimens (5, 7, and 10 inches in length), a 10-inch lath similar to that described in ASTM test C158-43 was selected as a compromise between a desire for a large stressed area and a large number of replicates. While all specimens used in the tests were laths 10 inches in length and 1 1/2 inches in width, they were obtained in two thicknesses, 1/10 inch and 1/4 inch depending on which size was readily available from the manufacturer. Corning glasses C-112 and C-113 were available only in the 1/10 inch thickness while Herculite II and the borosilicate glasses were 1/4 inch thick. Edge preparation also varied with the C-112 and C-113 having a "pencil" edge and the Pyrex (7740 borosilicate) laths being slightly beveled, while the Herculite II was left sharp cornered and unfinished after cutting, Figure 6.

Processing of Specimens. All of the specimens were weighed and their dimensions measured prior to the investigation. In addition, they were grouped into sets of 12 specimens each using center stress measurements obtained with a Babinet compensator as a guide. This procedure is described in Appendix A.

The average center tensions ( $\sigma_T$ ) as measured in the ion-treated surface-compressed glasses are as follows:

	$\sigma_T$	$\delta$
Herculite II	2,930 psi	0.016 inches
C-112	7,970 psi	0.018 inches
C-113	6,350 psi	0.085 inches

The depth of stress-reversal ( $\delta$ ) in each ion-exchange type of thickness (reference  $d$ ) was estimated by assuming a tapered compression stress profile and by using the relationship:  $\delta = \sigma_T d / (\text{MOR} + 2\sigma_T)$ . These estimates of ion-exchange depths ( $\delta$ ) are also tabulated above. It is evident that the C-113 SCS glass had a shallow treatment compared with the C-112 and Herculite II products.

The chill-tempered Pyrex was assumed to possess a parabolic stress profile and a stress-reversal depth of about one-third of the thickness (1/4 inch) or 0.083 inch on each side.

Prior to determining modulus of rupture or loading and exposure, specimens were roughened by a standard sandblast on the tensile face near the center section of each specimen, Figure 7. A spot, measuring one inch diameter was blasted onto the specimen using the apparatus shown in Figure 8. This was done to reduce the probability of edge failures in determining the flexural strength and to give a more



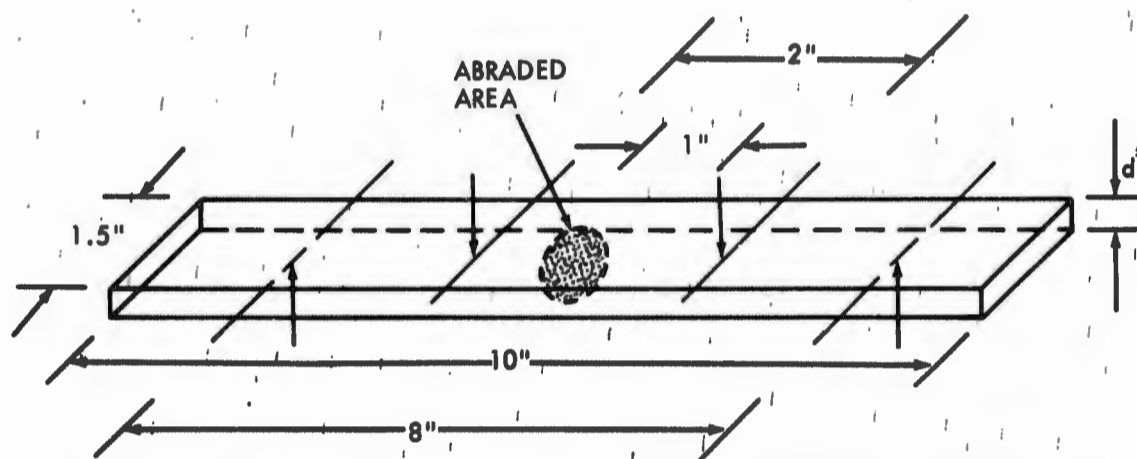


FIG. 7 SHOWING SAND BLASTED AREA AND POINTS OF LOADING DURING MODULUS OF RUPTURE DETERMINATION AND IN STORAGE UNDER FLEXURAL STRESS

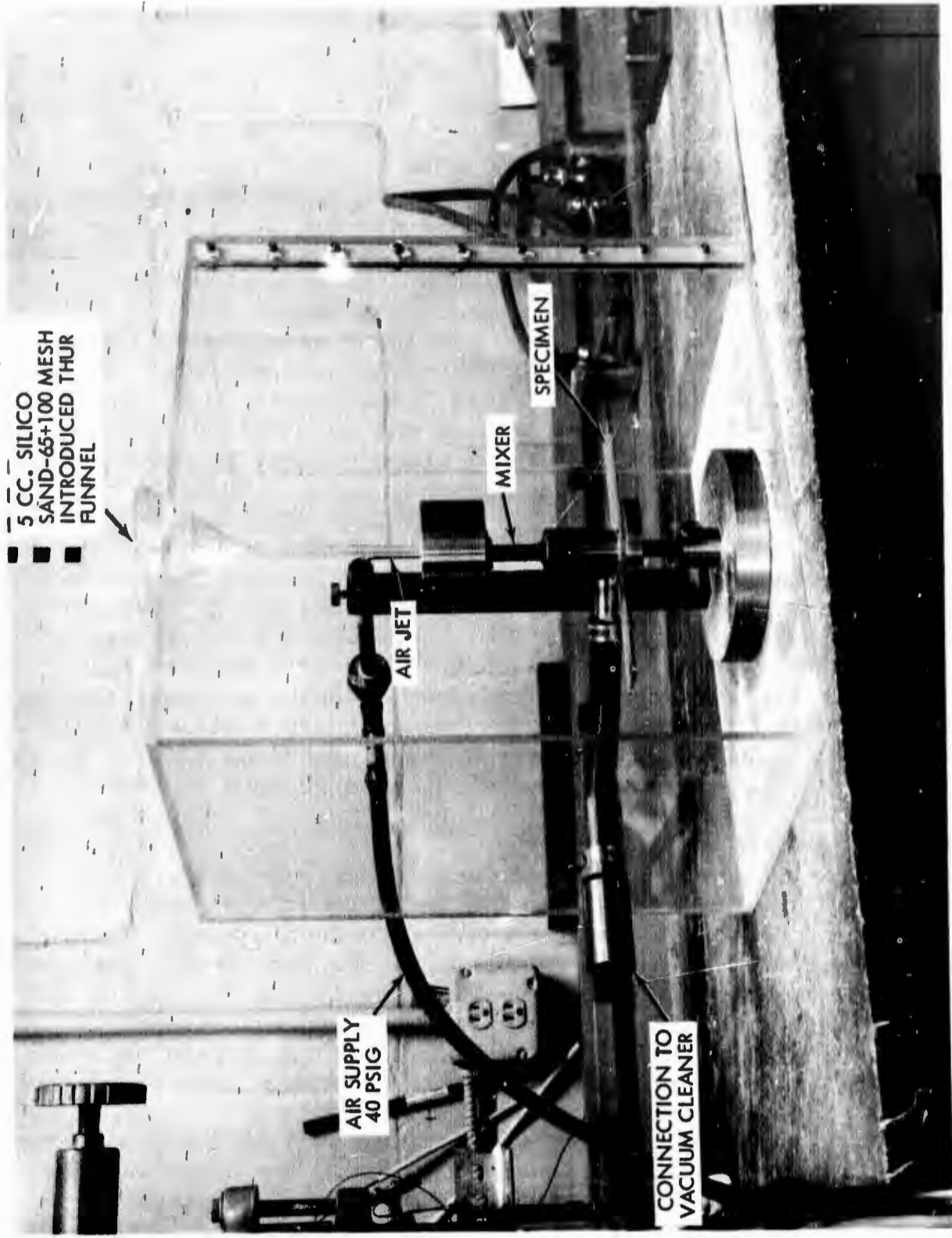


FIG. 8 SAND ABRADER -- APPARATUS FOR INFLECTING STANDARD DAMAGE TO THE CENTER OF THE FACE

representative measure of glass strength after surface damage in use. The sandblasting procedure was adapted from a technique used by the National Bureau of Standards and several glass manufacturers. Essentially the procedure consists of blowing a jet of air supplied at 40 psig onto the surface of the glass specimen and then introducing a volume (5ml) of glass sand into the air stream to affect abrasion. The area abraded is restricted by confining the sand and air inside a one-inch ID cylinder placed against the glass. This cylinder is connected to a vacuum cleaner hose which sucks off the sand after impact with the glass. Care was taken with the Herculite II specimens to abrade the surface of the glass which was not the one scored in breaking it to size before it was surface compressed.

After exposure to the salt water, specimens were scrubbed with water to remove any residual salt and then weighed, measured, and scotch tape applied to both faces before being broken in a 4-point flexural test. The general appearance of the sandblasted laths before and after exposure to salt water during storage under bending stress is shown in Figure 9. A general increase in surface haze and visibility of the sandblasted center spot is indicative of glass solubility which was verified later by weight loss measurements (see Figure 18). The MOR was determined in a Baldwin-Southwark Universal Test machine using an eight-inch span between specimen supports and two inches between loading points, Figure 10. The temperature and humidity of air during all tests throughout the program was 23°C and 50% R.H. The loading rate was sufficient to produce a modulus of rupture stress of 71 Kpsi per minute in the specimen. Preliminary tests on 14 specimens with tape on top only, and 14 specimens with tape top and bottom showed the tape had no effect on the modulus of rupture of the specimens.

### 30-DAY PRELIMINARY LOADING STUDIES

General. Prior to the main three-year exposure program, a preliminary study was made to determine the maximum mean bending stress that each type of glass could withstand for 30-days exposure. This information was needed as a guide in setting the load levels for the long-term studies. We wanted maximum load levels for the latter but not so high that many specimens would fail on loading or shortly thereafter. This proved to be a wise move; and, as it will be shown later, even with the load levels selected, there were some short-term failures in the long-term exposure studies.

There appears to be two factors that contributed to the occurrence of specimen breakage during the loading process. The first was the improbability of arriving at an accurate estimate of the true mean strength and standard deviation of the glass population using only a small number of replicates. Second was the hazard of accidentally overloading the specimens with the weight and lever apparatus while applying the load and leveling the lever. The latter required that

NOT REPRODUCIBLE

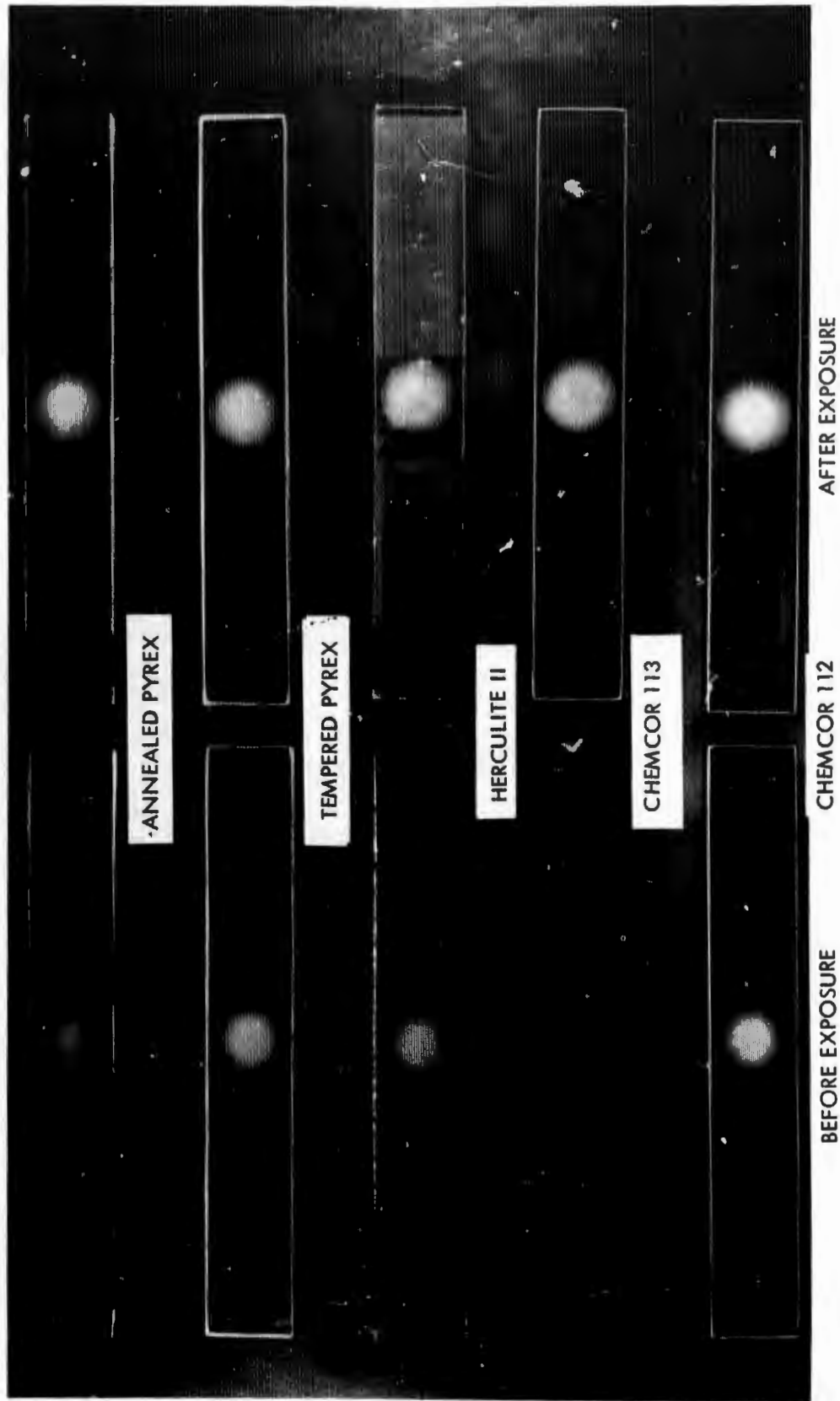


FIG. 9 PHOTOGRAPH OF LATHS OF VARIOUS GLASSES SHOWING EFFECT OF SALT WATER

NOT REPRODUCIBLE

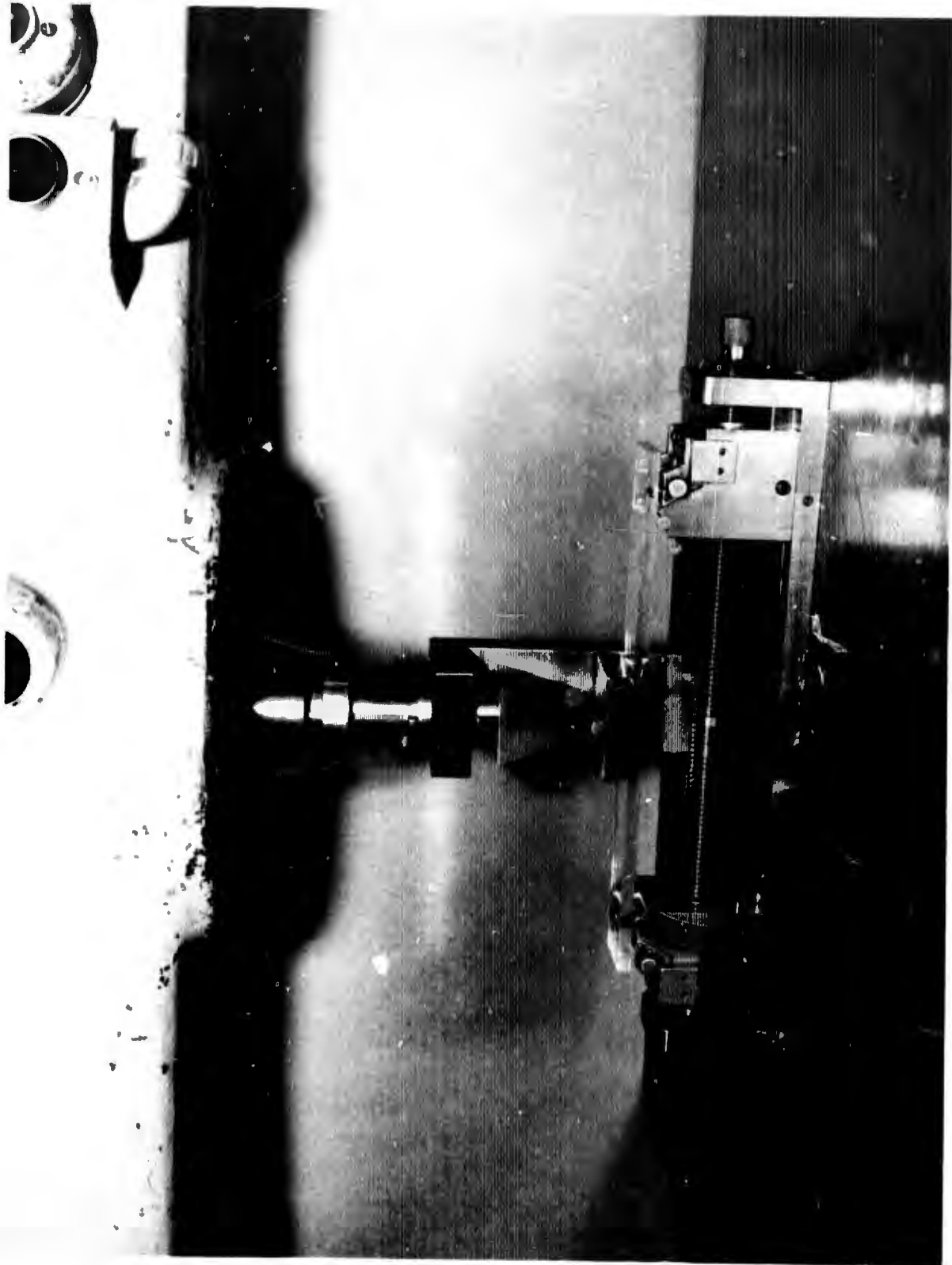


FIG. 10 FOUR-POINT FLEXURE TEST FIXTURE

extreme care be taken by laboratory personnel during adjustments of the mechanical systems.

Procedure Followed. The mean strength for each type of glass was determined by averaging the flexural strength of a set of 12 specimens which had been grouped by the method outlined in Appendix A. Then, beginning with a maximum load level equivalent to 90% of the average strength (60% for non-chemically strengthened glass), loads were applied successively to the specimens until a level was found where 30-day survival could be expected.

Data and Results. A running history of the 30-day survival investigation is given in Table 3. All of the chemically strengthened glasses subjected to 90% of the estimated average dry strengths of these glasses failed during static loading before the standard salt water (SSW) was added. (The general procedure followed was to load all of the specimens in one column of trays and then add the water.) All of the C-113 specimens survived loading and subsequent immersion in SSW for 30 days at both 80% and 70% levels. In contrast, C-112 specimens were not able to survive initial loading until the load was reduced to the 60% level. At this level all specimens survived 30 days immersion under load. Only one-third of the Herculite II specimens survived initial loading at the 80% level, but all specimens survived 30 days immersion under load at the 70% level.

The non-chemically strengthened semi-tempered Pyrex glasses failed to withstand initial loadings of 60% of the estimated average strength. However, some of the semi-tempered Pyrex specimens (3 of 5) did take initial loading and subsequent 30-day immersion. At the 50% level, 4 of 5 performed in a similar manner with one failure on initial loading. All annealed specimens failed until lowering the load to the 30% level. At this level, 5 of 6 specimens failed during 30-day immersion in SSW. At the 20% level, all Pyrex annealed specimens survived 30-day immersion under load.

### 3-YEAR EXPOSURE PROGRAM

General. Based on the 30-day studies, stress levels were selected for the long-term studies; and they are listed at the bottom of Table 3 and in Tables 4, 5, 6, and 8. Table 4 summarizes the program by showing the types of glasses and lengths of exposures for each type of glass.

Loading Procedure and Failures Experienced During Loading. After setting the weights on the lever arms, the strain gauged dynamometer lath was replaced with the SCS glass test specimen to be exposed, and the load was applied by gently lowering the weight and allowing it to come to rest. The lever arm was then adjusted to a horizontal position

TABLE 3  
Running History of 30 Day Load Studies

	Ann. Pyrex	Temp. Pyrex	Herc. II	C-112	C-113
Estimated Mean Strength, $\bar{X}$ , Kpsi	6.3	11.7	41.3	38.0	66.8
Design Allowable Strength, $\bar{X}$ -3s, Kpsi	2.7	9.7	36.1	34.3	46.5
Starting load level, Kpsi	3.8	7.0	37.2	34.2	60.1
Starting load level, % of $\bar{X}$	60	60	90	90	90
Number of survivors/total loaded	0/2*	3/5*	0/2*	0/2*	0/1*
2nd Load level, Kpsi	3.2	5.9	33.0	30.4	53.4
2nd Load level, % of $\bar{X}$	50	50	80	80	80
Number of survivors/total loaded	0/2*	4/5*	2/6*	0/2*	6/6
3rd Load level, Kpsi	2.5	-	28.9	26.6	46.8
3rd Load level, % of $\bar{X}$	40	-	70	70	70
Number of survivors/total loaded	0/2*	-	6/6	0/2*	5/5
4th Load level, Kpsi	1.9	-	-	22.8	-
4th Load level, % of $\bar{X}$	30	-	-	60	-
Number of survivors/total loaded	1/6**	-	-	6/6	-
5th Load level, Kpsi	1.3	-	-	19.0	-
5th Load level, % of $\bar{X}$	20	-	-	50	-
Number of survivors/total loaded	6/6	-	-	6/6	-
Load level selected for long term studies - Kpsi	1.3	5.9	28.9	22.8	46.8
% of $\bar{X}$	20	50	70	60	70

\* all specimen failures occurred during loading  
 \*\* all specimen failures occurred during 30 days exposure in SALT WATER

TABLE 4

Listing of Groups of Specimens as to Storage  
Conditions and Time Periods in the  
3 YEAR EXPOSURE PROGRAM

Exposure Period	<u>Annealed</u>	<u>Tempered</u>	<u>Herculite II</u>	<u>C-112</u>	<u>C-113</u>
	<u>Pyrex</u>	<u>Pyrex</u>			
0 Day Controls*	X	X	X	X	X
176 Days Controls*	X	X	X	X	-
3 Years Controls*	X	X	X	X	-
90 days - ssw	X	X	X	X	X
150 days - ssw	X	X	X	X	-
365 days - ssw	X	X	X	X	-
3 years - ssw	X	X	X	X	X
90 days - Air	-	-	X	-	-
150 days - Air	-	-	X	X	-
365 days - Air	-	-	X	X	-
Static stress level - Kpsi	1.3	5.9	28.9	22.8	46.8

\* No load applied to controls, all others stored under 4 point flexural loads

X Denotes specimens used in this time period - 12 specimens were used per exposure period



by the device shown in Figure 7. The standard salt water (SSW) was not added to the plastic trays until all the specimens were loaded in one column of trays. As seen in Figure 5, each tray held six specimens and six trays comprising each column were serviced by one circulating pump.

Table 5 summarizes specimen failures occurring either during loading and leveling, or within two hours after loading. There are two possible causes for those failures which occurred before the specimens could be immersed in the salt water. The strength of the failed specimen may have been considerably less than the mean previously determined on small lots, or the actual load applied may have accidentally exceeded the intended amount momentarily. The latter was found to occur if the load was left on the specimen while making final leveling adjustments. Turning the adjusting nut on the leveling screw too quickly resulted in greatly magnified loads and immediate failure. To avoid this, the load was lifted from the specimen while adjusting the column length and then gently lowered back onto the specimen.

The frame with loaded specimens was located on a basement slab floor in a busy laboratory. A specimen-failure count was taken daily five days a week. The test laboratory atmosphere was maintained constant at 23° C and 50% R. H.

At the end of their allotted times, specimens were unloaded, removed from the tray, wiped and placed on a flat plate, convex side up under a dial gage, to follow the relaxation of strains, Figure 11. Measurements began within the first minute after unloading and continued in some instances out to forty minutes. Unfortunately, data were not taken beyond this time and had to be extrapolated to make estimates of the principal relaxation times of the glasses tested in this manner.

Failures during Long-Term Exposure. Table 6 summarizes specimen failures occurring one day or longer after loading and while immersed in SSW. Of the eight failures, two were annealed glass, three were tempered glass, and three were ion-exchanged glass. Of the latter, one of the failures was a specimen under load in air. The seven failures in SSW are thus only a very small percentage (3-1/2%) of the 200 specimens immersed under load (2% of the ion-exchanged glasses failed). In addition, there is no observable pattern of these early failures, nor was there any subsequent failures with time. While there is no good explanation for these early failures, it is reasonable to believe that stress corrosion was not the failure mechanism.

Data and Results. Individual strength values are given in Tables B-1 through B-9 in Appendix B. The mean flexural strengths (MOR) for each of the five types of glass after storage periods up to three years (1100 days) are listed in Tables 7 through 11. Also presented in these tables are: the standard deviations, coefficients of

TABLE 5

Summary of Failures  
During Loading of Specimens or  
Shortly Thereafter\*

Exposure Period	<u>Annealed</u>	<u>Tempered</u>	<u>Herculite II</u>	<u>C-112</u>	<u>C-113</u>
	<u>Pyrex</u>	<u>Pyrex</u>			
90 days - ssw	0	0	1	2	4
150 days - ssw	0	0	1	0	N.A.
365 days - ssw	0	1	0	2	N.A.
3 years - ssw	0	0	0	2	4***
90 days - Air	N.A.	N.A.	0	N.A.	N.A.
150 days - Air	N.A.	N.A.	0	1	N.A.
365 days - Air	N.A.	N.A.	0	0	N.A.
Totals	0	1	2	7	8
% Loading **	20	50	70	60	70

N.A. Not applicable, no specimens in this time period

\* Less than 2 hours after loading

\*\* Per cent of estimated mean strength

\*\*\* 80% loading on 2 specimens

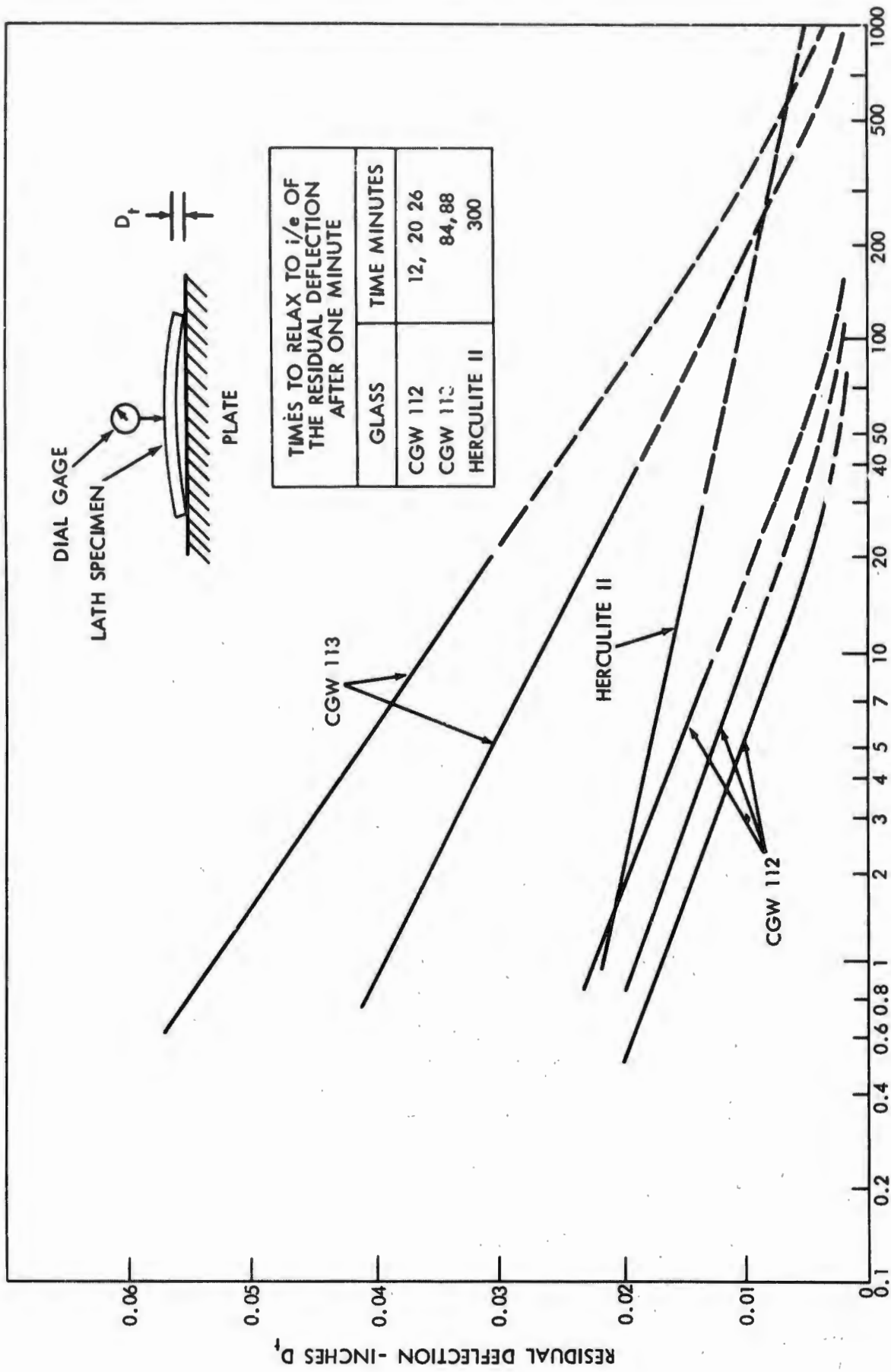


FIG. 11 VISCOELASTIC RECOVERY AFTER 3 - YEARS UNDER LOAD

TABLE 6

Summary of Failures During Immersion  
in Salt Water or Air Under Load

Exposure Period	<u>Annealed</u> <u>Pyrex</u>	<u>Tempered</u> <u>Pyrex</u>	<u>Herculite II</u>	<u>C-112</u>	<u>C-113</u>
90 days - ssw	1-27 days	1-48 days	0	1-6 days	0
150 days - ssw	0	1- 6 days	0	0	N.A.
365 days - ssw	0	1- 3 days	0	0	N.A.
3 years - ssw	1- 0 days	0	0	1-4 days	0
90 days - Air	N.A.	N.A.	0	N.A.	N.A.
150 days - Air	N.A.	N.A.	0	0	N.A.
365 days - Air	N.A.	N.A.	0	1-1 day	N.A.
Totals	2	3	0	3	0
% Loading *	20	50	70	60	70

N.A. Not applicable, no specimens in this time period

\* Per cent of estimated mean strength

TABLE 7

Mean Modulus of Rupture Strength Initially  
and After Various Storage Conditions for  
PYREX ANNEALED

<u>Time-Days</u>	<u>Storage Conditions</u>	<u>% Load</u> <sup>1</sup>	<u>Sample Size</u>	<u>Mean</u> <u><math>\bar{x}</math></u>	<u>s</u> Kpsi	<u>CV</u> <sup>2</sup> %	<u>DA</u> <sup>3</sup> Kpsi
Initial	-	-	12	6.3	1.27	20.3	2.5
30	SSW	20	6	6.7	0.867	13.0	4.1
90	SSW	20	9	5.8	0.734	12.6	3.6
150	SSW	20	12	6.4	0.567	9.0	4.7
365	SSW	20	12	6.7	0.967	14.5	3.8
1100	SSW	20	11	6.2	0.834	13.7	3.7
176	Air	-	12	6.7	0.700	10.6	4.6
1100	Air	-	12	6.4	0.700	11.1	4.3

- (1) percent of initial mean MOR failure stress applied as flexural stress to each lath during storage
- (2) coefficient of variation,  $100S/\bar{x}$
- (3) design allowable stress,  $\bar{x} - 3S$

TABLE 8

Mean Modulus of Rupture Strength Initially  
and After Various Storage Conditions for  
PYREX SEMI-TEMPERED

<u>Time-Days</u>	<u>Storage Conditions</u>	<u>% Load<sup>1</sup></u>	<u>Sample Size</u>	<u>Mean <math>\bar{x}</math> Kpsi</u>	<u>Standard Deviation S Kpsi</u>	<u>CV<sup>2</sup> %</u>	<u>DA<sup>3</sup> Kpsi</u>
Initial	-	-	12	11.7	0.700	6.0	9.6
30	ssw	50	4	13.1	0.793	6.1	10.7
90	ssw	50	11	12.3	0.942	7.7	9.5
150	ssw	50	11	12.2	0.925	7.6	9.4
365	ssw	50	10	12.9	0.766	5.9	10.6
1100	ssw	50	12	13.0	0.694	5.3	10.9
176	Air	-	12	12.2	0.600	5.0	10.4
1100	Air	-	12	11.4	0.300	4.5	9.9

- (1) percent of initial mean MOR failure stress applied as flexural stress to each lath during storage  
 (2) coefficient of variation,  $100 S/\bar{x}$   
 (3) design allowable stress,  $\bar{x} - 3S$

TABLE 9

Mean Modulus of Rupture Strength Initially  
and After Various Storage Conditions for  
HERCULITE II

<u>Time-Days</u>	<u>Storage Conditions</u>	<u>% Load<sup>1</sup></u>	<u>Sample Size</u>	<u>Mean <math>\bar{x}</math> Kpsi</u>	<u>Standard Deviation s Kpsi</u>	<u>CV<sup>2</sup> %</u>	<u>DA<sup>3</sup> Kpsi</u>
Initial	-	-	12	41.3	1.82	4.4	35.8
30	ssw	70	8	43.5	1.46	3.4	39.1
90	ssw	70	11	45.2	0.900	2.2	42.5
150	ssw	70	11	45.4	1.63	3.6	40.5
365	ssw	70	12	47.3	2.00	4.2	41.3
1100	ssw	70	12	45.2	1.00	2.2	42.2
90	Air	70	12	43.3	3.00	6.9	34.3
365	Air	70	12	42.3	2.33	5.5	35.3
176	Air	-	12	42.3	1.93	4.6	36.5
1100	Air	-	12	41.5	1.50	3.6	37.0
150 (4)	ssw	70	11	46.0	2.21	4.8	39.4
150 (4)	Air	70	6	41.3	1.82	4.4	35.8

- (1) percent of initial mean MOR failure stress applied as flexural stress to each lath during storage
- (2) coefficient of variation,  $100 S/\bar{x}$
- (3) design allowable stress,  $\bar{x} - 3S$
- (4) data taken from Appendix B as check on reproducibility of data

TABLE 10

Mean Modulus of Rupture Strength Initially  
and After Various Storage Conditions for  
CHEMCOR C-112

<u>Time-Days</u>	<u>Storage Conditions</u>	<u>% Load<sup>1</sup></u>	<u>Sample Size</u>	<u>Mean <math>\bar{x}</math> Kpsi</u>	<u>Standard Deviation S Kpsi</u>	<u>CV<sup>2</sup> %</u>	<u>DA<sup>3</sup> Kpsi</u>
Initial	-	-	12	38.0	1.3	3.4	34.1
30	ssw	60	12	40.3	1.82	4.5	34.8
90	ssw	60	9	40.0	2.23	5.6	33.3
150	ssw	60	12	41.2	1.37	3.3	37.1
365	ssw	60	10	42.7	2.13	5.0	36.3
1100	ssw	60	8	39.8	1.18	3.0	36.2
150	Air	60	11	39.0	2.27	5.8	32.2
365	Air	60	10	39.6	1.47	3.7	35.2
176	Air	-	11	39.1	1.40	3.6	34.9
1100	Air	-	10	36.4	1.24	3.4	32.7
150 (4)	ssw	60	12	40.9	1.15	2.8	37.5
150 (4)	Air	60	12	37.8	1.76	4.7	32.5

- (1) percent of initial mean MOR failure stress applied as flexural stress to each lath during storage
- (2) coefficient of variation,  $S/\bar{x}$
- (3) design allowable stress,  $\bar{x} - 3S$
- (4) data taken from Appendix B as check on reproducibility of data



TABLE 11

Mean Modulus of Rupture Strength Initially  
and After Various Storage Conditions for  
CHEMCOR C-113

<u>Time-Days</u>	<u>Storage Conditions</u>	<u>% Load<sup>1</sup></u>	<u>Sample Size</u>	<u>Mean <math>\bar{x}</math> Kpsi</u>	<u>Standard Deviation S Kpsi</u>	<u>CV<sup>2</sup> %</u>	<u>DA<sup>3</sup> Kpsi</u>
Initial	-	-	12	66.8	7.00	10.5	45.8
30	SSW	70&80	7	74.6	7.07	9.5	53.4
90	SSW	70	8	73.2	6.50	8.9	53.7
1100	SSW	70	8	75.4	2.40	3.2	68.2

- (1) percent of initial MOR failure stress applied as flexural stress to each lath during storage
- (2) coefficient of variation,  $S/\bar{x}$
- (3) design allowable stress,  $\bar{Y} - 3S$

variation and the design allowable strength calculated for each group of laths tested. For more ready comparison between glasses, these data are summarized in Tables 12 through 15. The mean modulus of rupture data and coefficient of variations for each group of specimens is presented graphically in Figures 12 through 18.

Table 16 presents the weight loss of the glass laths after each storage period listing the weight loss as milligrams per square centimeter of specimen surface area. This is shown graphically in Figure 19.

A striking observation to be made on this data, as seen in Tables 12 and 15, is that all the surface compressed glasses increased in strength when exposed to salt water. Even more important is the 6 to 49% increase in design allowable strength found for glasses stored in salt water under stress, as seen in Table 15.

A noteworthy observation, however, is a drop in modulus of rupture strength for both the Herculite II and Chemcor C-112 during the one year to three year period as seen in Figure 15. The validity of this apparent change in strength and, in fact, the validity of all the strength changes that were observed require statistical treatment. This is described in the Discussion of Results.

Figure 18 presents the data in a graphical summary for easy observation of strength changes with time and comparison of strength after storage in salt water and air.

The relationship between the coefficients of variation (CV) and the average MORS of all sets of data, wet and dry, in the study is shown in Figure 20. Except for the C-113 glass, a regular trend toward lower CVs (i.e., more uniformity) with higher MORS exists.

The C-113 glass seems to be the exception to this general observation. It may be significant that these specimens had a thin (0.008 inch) compressed layer, compared with the other types (0.016 inch-0.018 inch). The air-grit abrasion treatment may have been unduly severe on this thin compressed layer. No tests were conducted on any of the glasses unabraded.

To examine the differences between glasses further, some of their frequency distributions were plotted on probability paper, Figures 21 and 22. On this paper a "normal" gaussian distribution generates a linear slope. The plots of 0, 1, and 3-year strengths (see Appendix B) on tempered Pyrex, Herculite II, and C-112 glasses are normally distributed; but the 0-year C-113 strengths are not. It is remarkable how much effect three years in the salt water had in increasing, normalizing, and equalizing the strengths of the C-113 and Herculite II glasses. A similar picture on C-112 is spoiled by two non-normal data points.

Discussion of Results. The primary objective of this investigation was to determine if SCS glass subjected to constant high stress and in

TABLE 12

Average Strength<sup>1</sup> of Different Glasses Initially and After Exposure to Salt Water or Air Under Load

Exposure Period	Average MOR <sup>1</sup> -Kpsi				
	Annealed	Tempered	Herculite II	C-112	C-113
	<u>Pyrex</u>	<u>Pyrex</u>			
Initial	6.3	11.7	41.3	38.0	66.8
90 days - ssw <sup>2</sup>	5.8	12.3	45.2	40.0	73.2
150 days - ssw	6.4	12.2	45.4	41.2	N.D.
365 days - ssw	6.7	12.9	47.3	42.7	N.D.
3 years - ssw	6.2	13.0	45.2	39.8	75.4
90 days - Air <sup>3</sup>	N.D. <sup>5</sup>	N.D.	43.3	N.D.	N.D.
150 days - Air	N.D.	N.D.	41.7	39.0	N.D.
365 days - Air	N.D.	N.D.	42.3	40.4	N.D.
170 day control <sup>4</sup>	6.7	12.2	42.3	39.9	N.D.
3 year control	6.4	11.4	41.5	36.4	N.D.

1 Four point flexural strength

2 ssw - standard salt water

3 Air - 72 F, 50% relative humidity

4 no load on controls during exposure periods

5 N.D. no data (no specimens in this time period)

TABLE 13

Coefficient of Variation for Different Glasses  
After Exposure in Salt Water or Air Under Load

Exposure Period	<u>Annealed</u>	<u>Tempered</u>	<u>Herculite II</u>	<u>C-112</u>	<u>C-113</u>
	<u>Pyrex</u>	<u>Pyrex</u>			
	%	%	%	%	%
Initial	20.3	6.0	4.4	3.4	10.5
90 days - ssw <sup>1</sup>	12.6	7.7	2.0	5.6	8.9
150 days - ssw	9.0	7.6	3.6	3.3	N.D.
365 days - ssw	14.5	5.9	4.2	5.0	N.D.
3 years - ssw	13.7	5.3	2.2	3.0	3.2
90 days - Air <sup>2</sup>	N.D. <sup>4</sup>	N.D.	6.9	N.D.	N.D.
150 days - Air	N.D.	N.D.	5.2	5.8	N.D.
365 days - Air	N.D.	N.D.	5.5	8.1	N.D.
0 day control <sup>3</sup>	20.3	6.0	4.4	3.4	10.5
176 day control	10.6	5.0	4.6	7.4	N.D.
3 year control	11.1	4.5	3.6	3.4	N.D.

- 1 ssw - standard salt water
- 2 Air - 72°F, 50% relative humidity
- 3 no load on controls during exposure periods
- 4 N.D. No data (no specimens in this time period)

TABLE 14

Design Allowable Strength<sup>1</sup> for Different Glasses  
After Exposure in Salt Water or Air Under Load

Exposure Period	Design Allowable Strength - Kpsi				
	<u>Annealed Pyrex</u>	<u>Tempered Pyrex</u>	<u>Herculite II</u>	<u>C-112</u>	<u>C-113</u>
90 days - ssw <sup>2</sup>	3.6	9.5	42.5	33.3	53.7
150 days - ssw	4.7	9.4	40.5	37.1	N.D.
365 days - ssw	3.8	10.6	41.3	36.3	N.D.
3 years - ssw	3.7	10.9	42.2	36.2	68.2
90 days - Air <sup>3</sup>	N.D. <sup>5</sup>	N.D.	34.3	N.D.	N.D.
150 days - Air	N.D.	N.D.	35.2	32.2	N.D.
365 days - Air	N.D.	N.D.	35.3	30.6	N.D.
0 day control <sup>4</sup>	2.5	9.6	35.8	34.1	45.8
176 day control	4.6	10.4	36.5	31.0	N.D.
3 year control	4.3	9.9	37.0	32.7	N.D.

- 1 Design Allowable Strength =  $\bar{x} - 3S$
- 2 ssw - standard salt water
- 3 Air - 72°F, 50% relative humidity
- 4 no load on controls during exposure periods
- 5 N.D. no data (no specimens in this time period)

TABLE 15

Summary Comparing Initial Flexural  
and Design Allowable Strengths of the Various  
Glasses Tested with their Strengths After 3 Years in  
Air and 3 Years Stressed Under Salt Water

Type of Glass	Flexural Strength MOR - Kpsi			
	<u>Initial</u>	<u>3 Years in Air</u>	<u>3 Years Salt Water</u>	<u>% Increase</u>
Pyrex, annealed	6.3	6.4	6.2	None
Pyrex, semi-temp.	11.7	11.4	13.0	12
Herculite II	41.3	41.5	45.2	9.5
Chemcor C-112	38.0	36.4	39.8	4.7
Chemcor C-113	66.8	N.A.	75.4	12.9

Types of Glass	Design Allowable Strength <sup>1</sup> MOR - Kpsi			
	<u>Initial</u>	<u>3 Years in Air</u>	<u>3 Years Salt Water</u>	<u>% Increase</u>
Pyrex, annealed	2.5	4.3	3.7	48.0
Pyrex, semi-temp.	9.6	9.9	10.9	13.5
Herculite II	35.8	37.0	42.2	17.9
Chemcor C-112	34.1	32.7	36.2	6.2
Chemcor C-113	45.8	N.A. <sup>2</sup>	68.2	49.0

1. Based on a margin of three standard deviations below the sample mean which provides a 99.73% certainty that the true populations mean lies above the figures given.
2. Not Applicable - no specimens available for this test

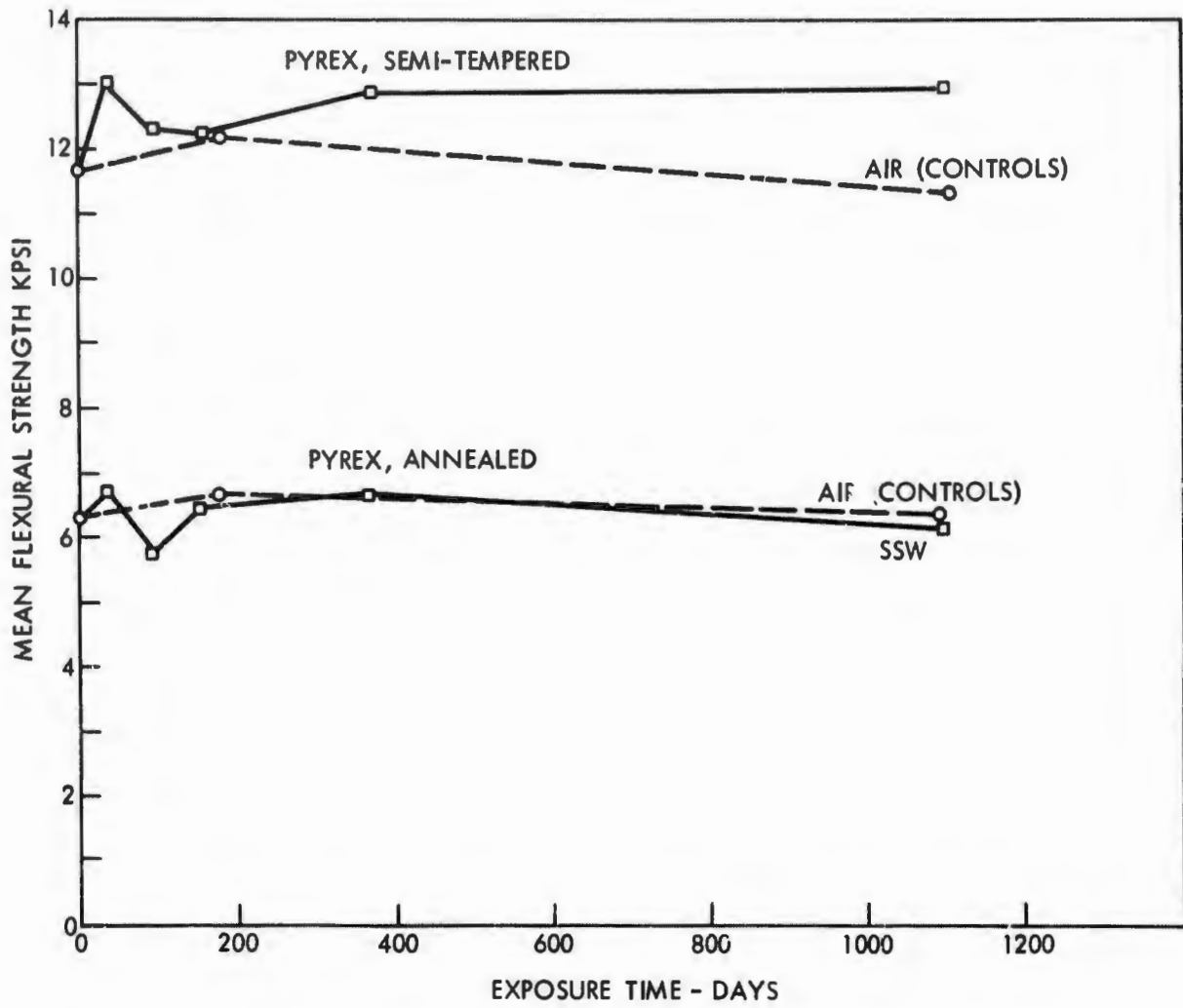


FIG. 12 MOR OF PYREX GLASS LATHS STORED FOR PERIODS UP TO THREE YEARS UNDER FLEXURAL STRESS IN SALT WATER AND UNSTRESSED IN AIR

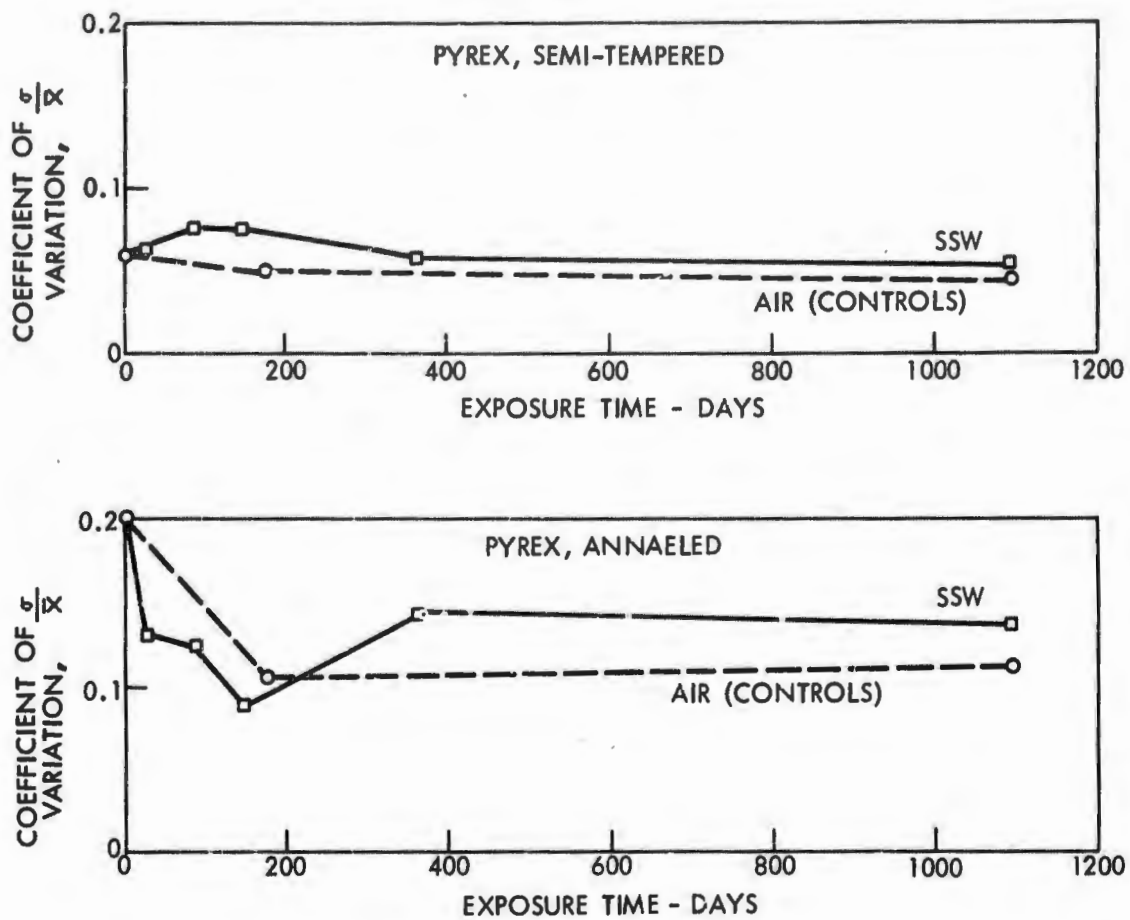


FIG. 13 COEFFICIENTS OF VARIATIONS FOR MOR DATA OF FIG. 10



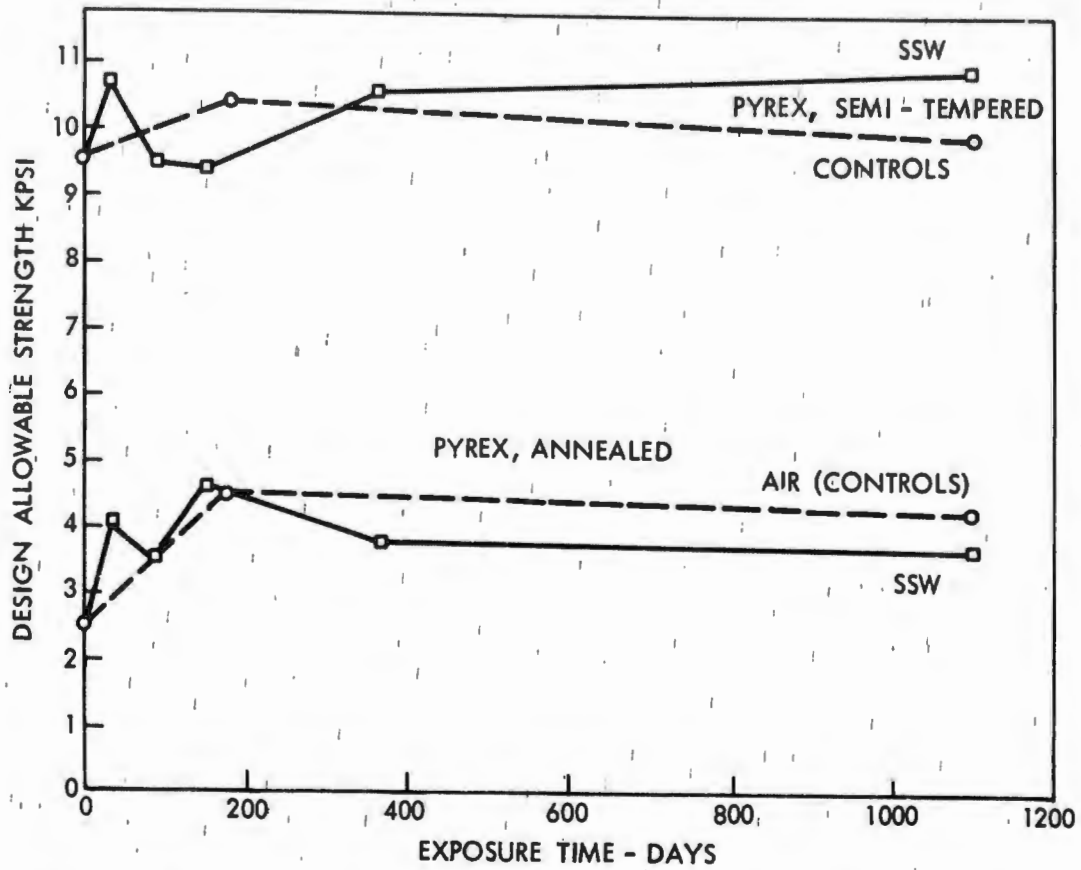


FIG. 14 DESIGN ALLOWABLE STRENGTHS OF PYREX AFTER VARIOUS PERIODS OF TIME IN AIR OR STRESSED IN SALT WATER

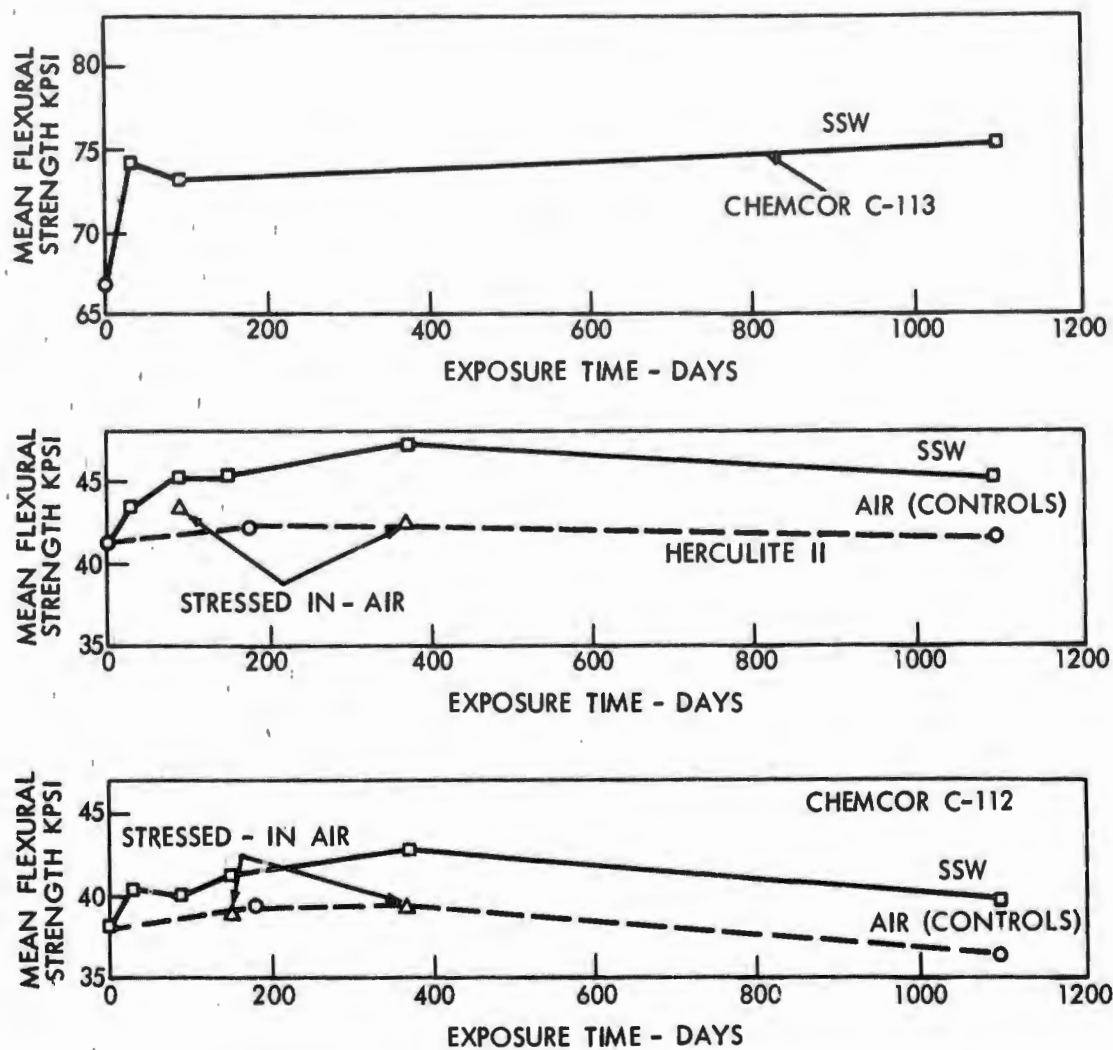


FIG. 15 MOR OF CHEMICALLY STRENGTHENED GLASS LATHS STORED FOR PERIODS UP TO THREE YEARS IN STRESSED AND UN-STRESSED CONDITIONS AND IN CONTACT WITH AIR OR SALT WATER

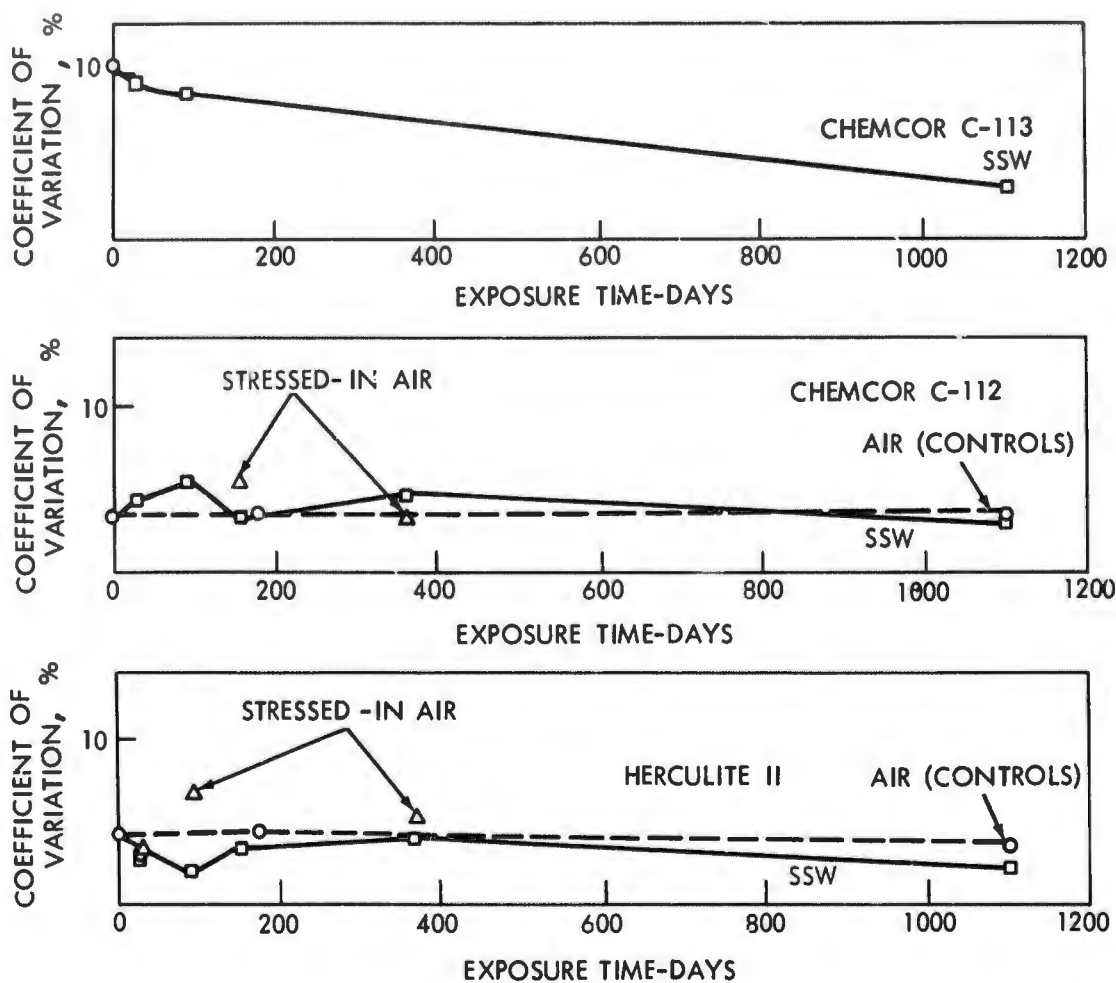


FIG. 16 COEFFICIENTS OF VARIATION FOR MOR DATA OF FIG. 14

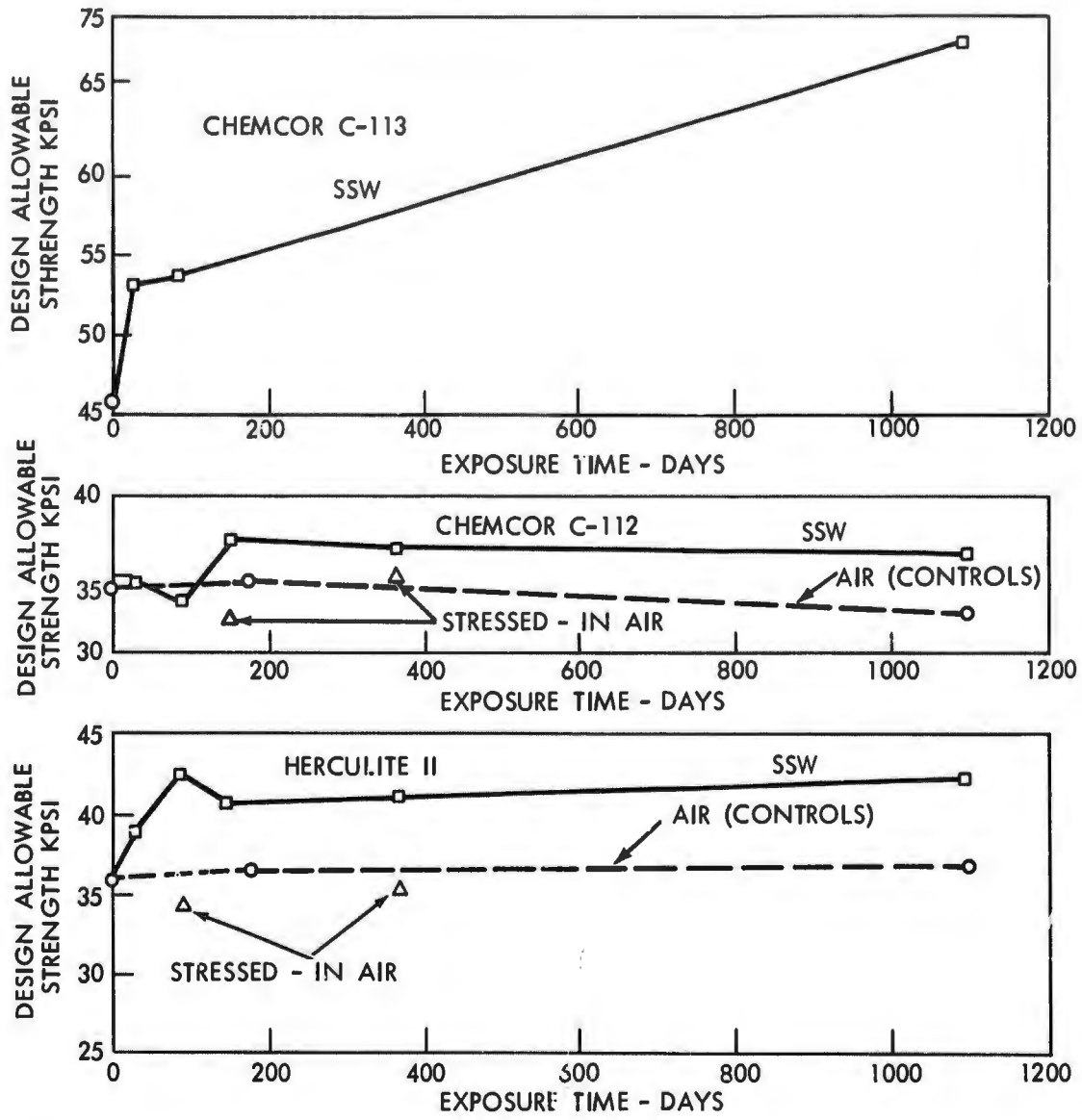


FIG. 17 DESIGN ALLOWABLE STRENGTH OF CHEMICALLY STRENGTHENED GLASSES AFTER VARIOUS PERIODS OF TIME IN AIR OR STRESSED IN SALT WATER

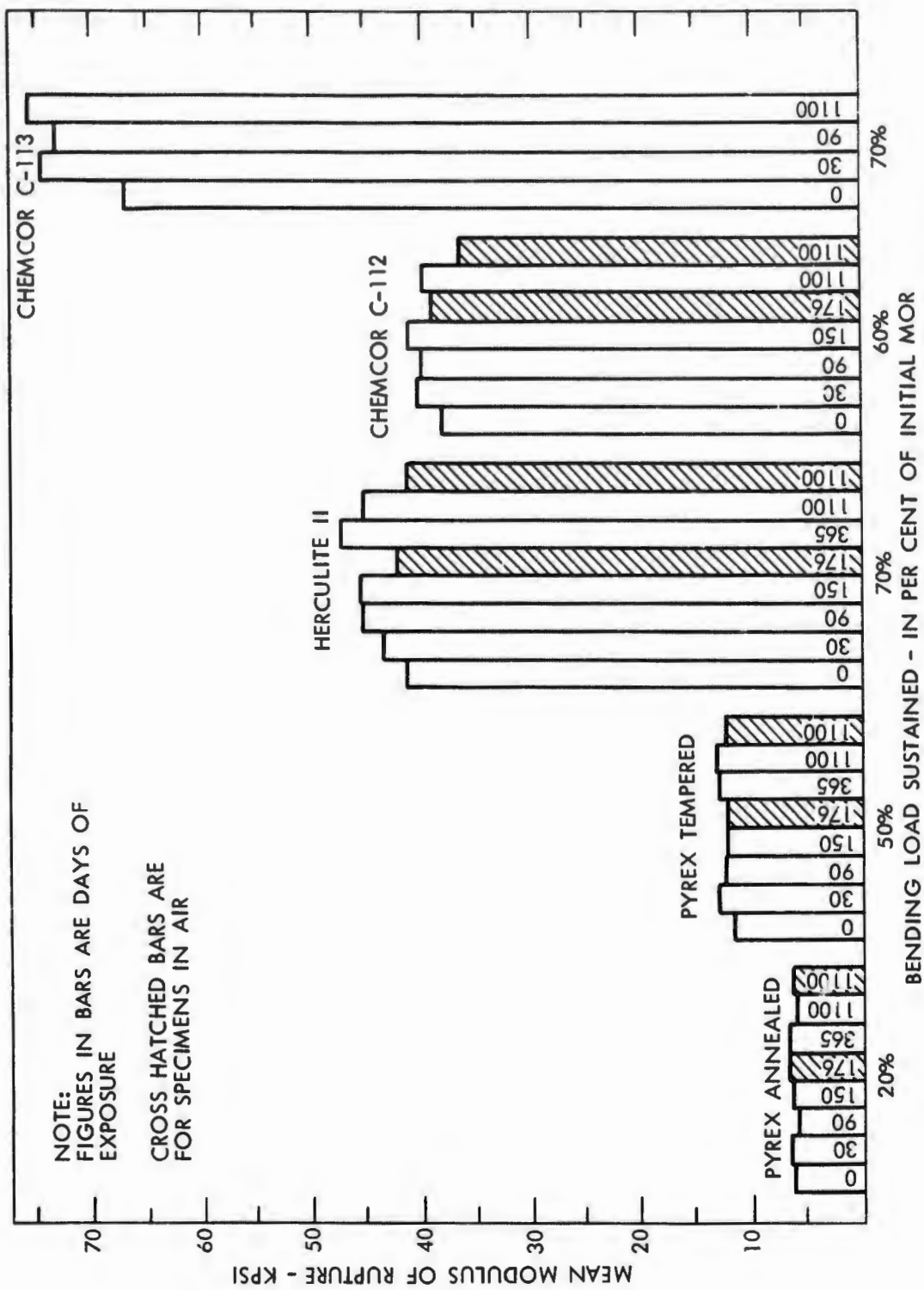


FIG. 18 AVERAGE MOR STRENGTH AFTER EXPOSURE TO SALT WATER UNDER BENDING STRESS OR TO AIR WITHOUT STRESS

TABLE 16

Solubility of Glass Laths Stored for  
 Various Periods in SSW or AIR  
 Weight Loss - mg/cm<sup>2</sup>

<u>Time-Days</u> <u>Storage</u>	<u>Medium</u>	<u>Annealed</u> <u>Pyrex</u>	<u>Tempered</u> <u>Pyrex</u>	<u>Herculite II</u>	<u>C-112</u>	<u>C-113</u>
30	ssw	0.009	-	-	0.027	0.009
90	ssw	0.005	0.003	0.062	0.034	0.010
150	ssw	0.017	0.030	0.107	0.055	-
365	ssw	0.065	0.098	0.126	0.065	-
1100	ssw	0.189	0.167	0.315	0.114	0.112
90	Air	-	-	0.0091	-	-
150	Air	-	-	0.0255	0.009	-
365	Air	-	-	0.0230	-	-
1100	Air	-	-	-	0.016	-

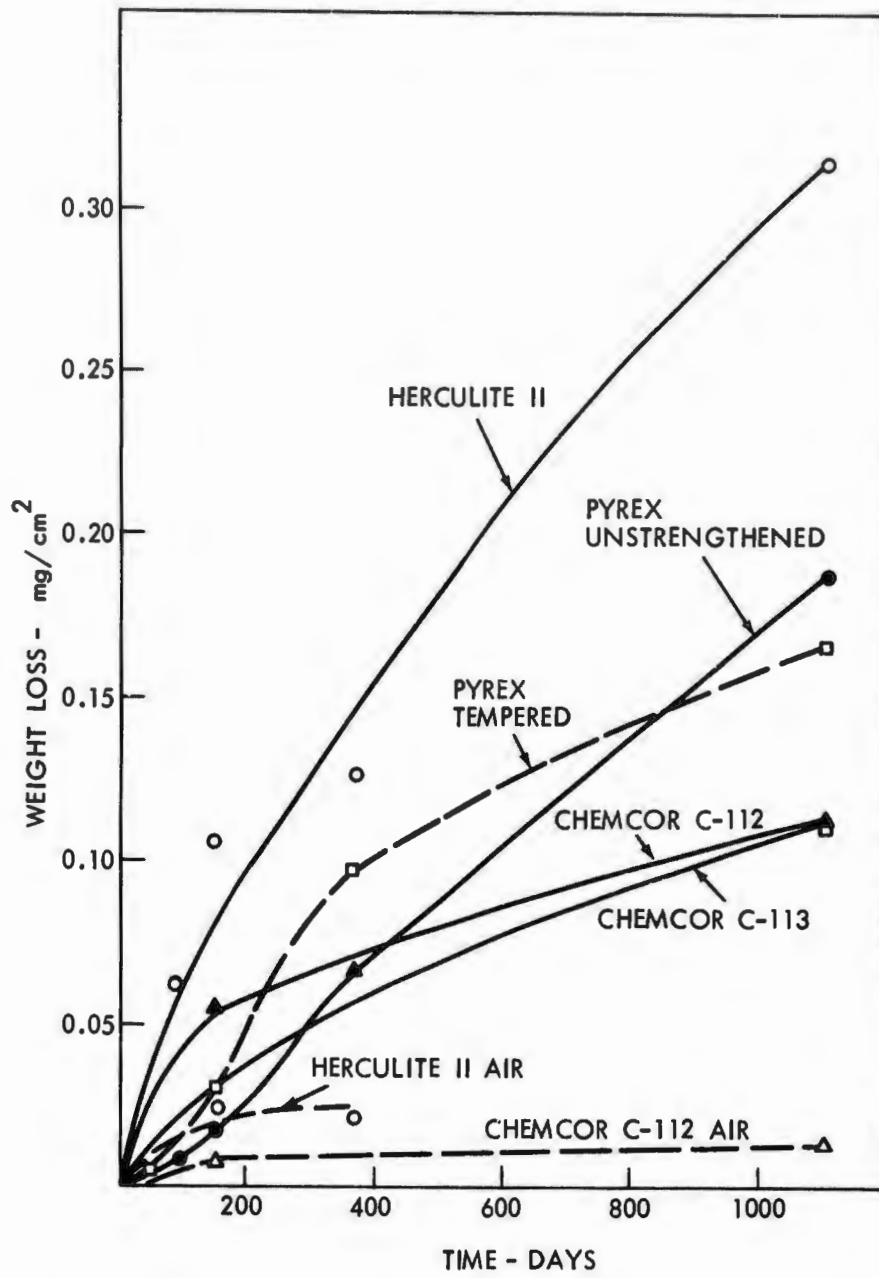


FIG. 19 SOLUBILITY OF GLASSES EXPOSED TO AIR AND SALT WATER UNDER A CONSTANT BENDING STRESS

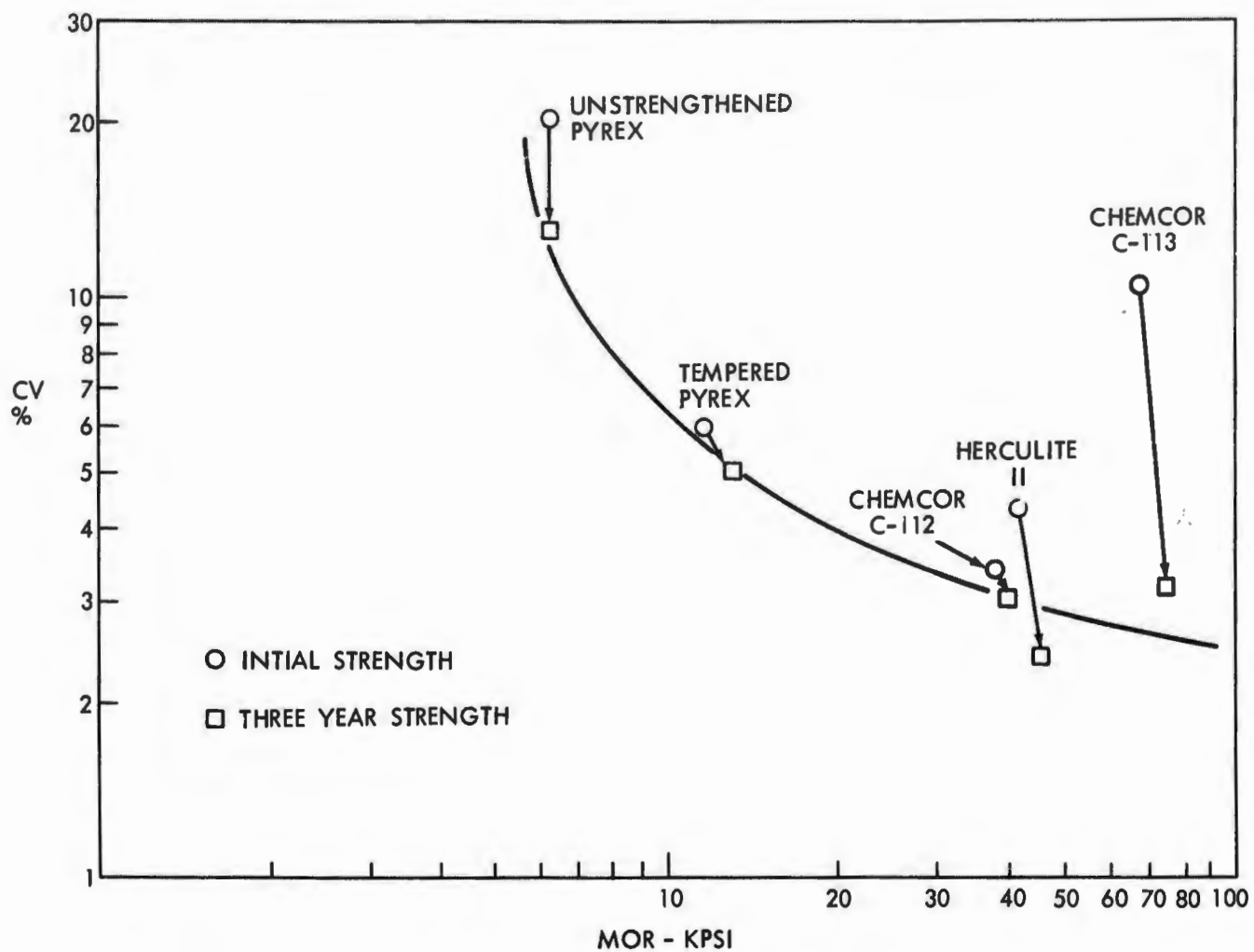


FIG. 20 CHANGES IN THE STRENGTH AND VARIABILITY OF SCS GLASSES DURING THREE YEARS UNDER STRESS IN SALT WATER



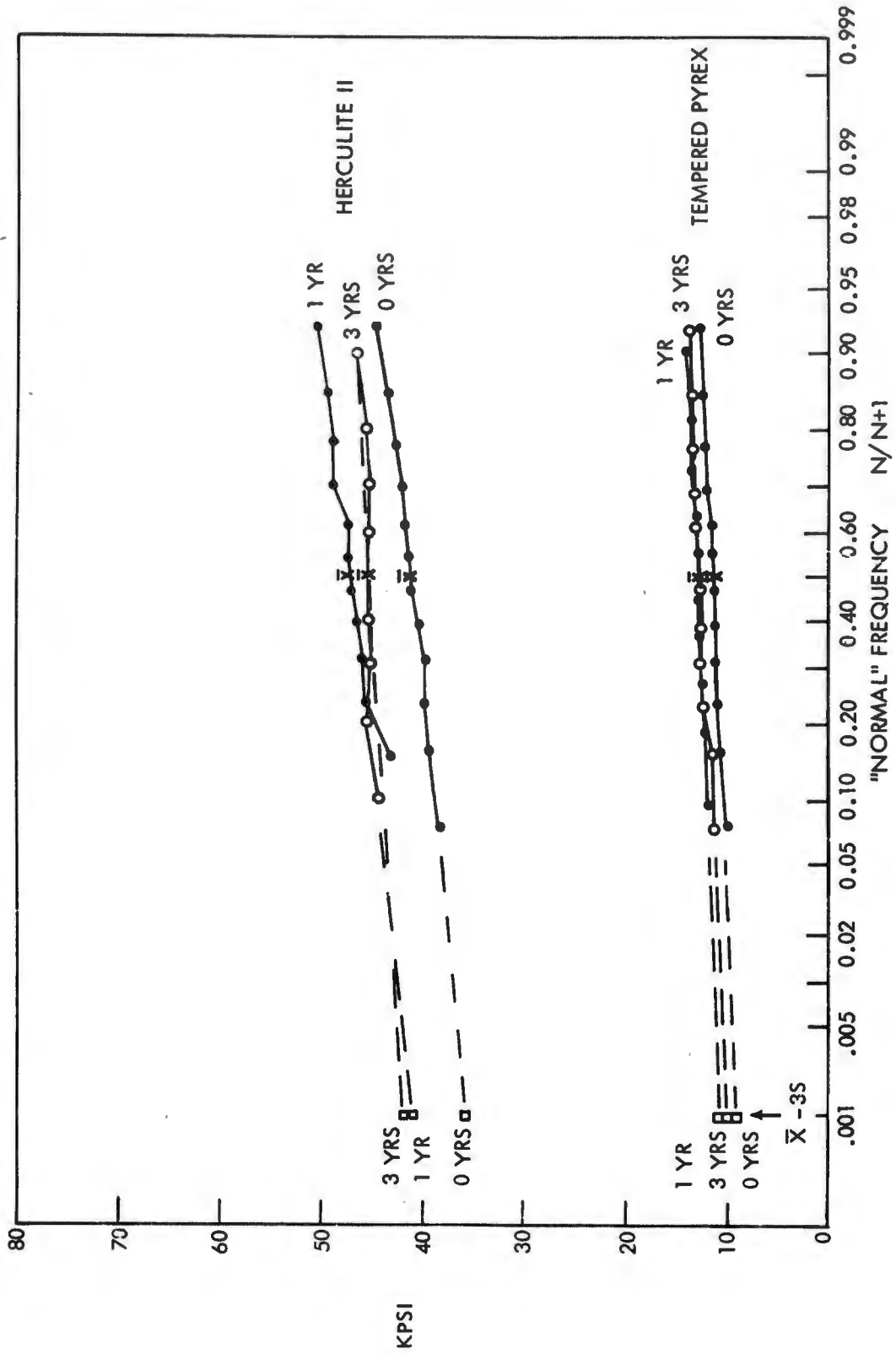


FIG. 21 DISTRIBUTIONS OF STRENGTHS AFTER 0, 1 AND 3 - YEAR EXPOSURE UNDER LOAD

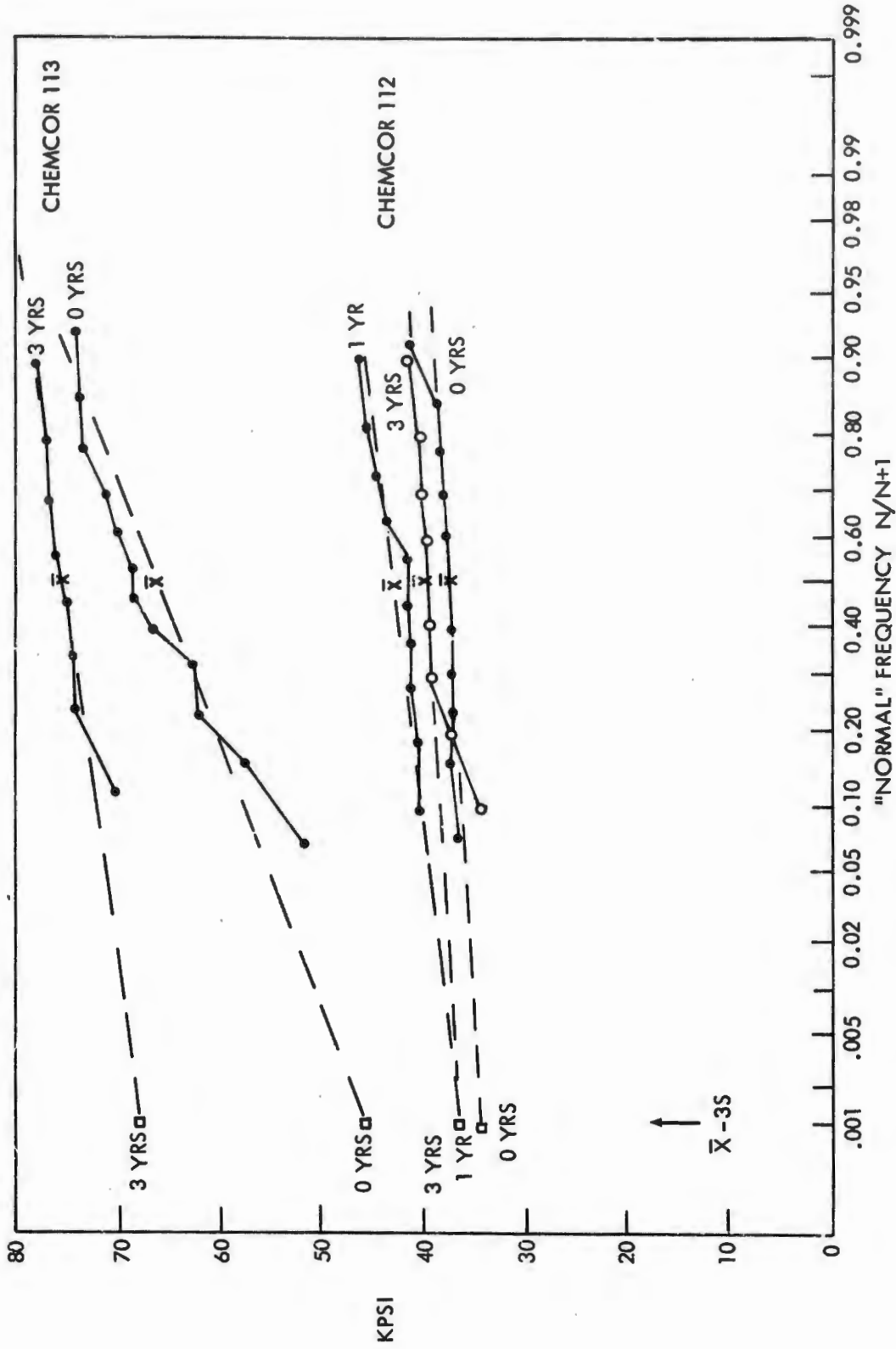


FIG. 22 DISTRIBUTIONS OF STRENGTHS AFTER 0, 1 AND 3 - YEAR EXPOSURE UNDER LOAD

constant contact with flowing salt water over long periods of time would lose any of its initial strength in a manner similar to the stress-corrosion cracking of metals, ceramics, or unstrengthened glass. That stress-corrosion fatigue was not a failure mechanism working on the flexurally stressed SCS glass specimens being held in air, and salt water was implied by the absence of an increase in spontaneous specimen failures with increasing time. The acquisition of a mass of strength data obtained on the glass laths after the various intermediate time periods had the purpose of more closely looking for trends in strength changes with time.

Question of Stress Corrosion Vulnerability or Ion Migration of The Convex Face Vs. The Concave Face of Flexed Glass Laths.

During the course of the test program a challenge arose as to whether:

1. the concave (more compressed) face of a glass lath subjected to flexural stress in salt water might not be more vulnerable to stress corrosion than the convex (less compressed) face, or
2. whether the exchanged ions in the concave (more compressed) face might be forced elsewhere by the added compression stress thus dissipating their effectiveness for strengthening the glass.

Although these possibilities seemed far fetched on theory, a test program comparing the modulus of rupture strength of glass laths stored under flexural stress with the sandblasted face on the concave side (spots up) with strength of similar specimens stored with the sandblasted face on the convex side (spots down) was carried out.

It must be noted that while the surface of the convex face of a flexed specimen is normally thought of as being in tension, this is not necessarily the case for specimens having compressed surface layers. With the exception of the annealed Pyrex laths, the bending loads sustained during storage appear to be insufficient to place the convex face of any of the laths in tension. Both surfaces of the flexed specimens were, therefore, under compressive stress during storage with the top (concave) surface being much more highly compressed than the bottom (convex) surface.

Both Herculite II and Chemcor C-112 were studied in the programs and the effect of both salt water and air during a 150-day period were investigated. The results are given in Table 17 and 18. This study showed an increase in modulus of rupture strength for the laths stored in 50% R.H. air and in salt water for 150 days regardless of whether the sandblasted spots were up or down during storage. Of course, all specimens were broken in the modulus of rupture test with their spots down, i.e. spots on the face subject to tensile stress. Also, the

TABLE 17

Modulus of Rupture and Weight Loss  
of Chemcor C-112 After Being  
Exposed 150 Days to Salt Water and  
Loaded in Flexure to 60% of Initial MOR

	SPOTS-UP		SPOTS-DOWN	
	<u>MOR</u> <u>Kpsi</u>	<u>Weight Loss</u> <u>mg/cm<sup>2</sup></u>	<u>MOR</u> <u>Kpsi</u>	<u>Weight Loss</u> <u>mg/cm<sup>2</sup></u>
	37.6	0.0698	41.5	0.0547
	39.2	0.0628	40.1	0.0509
	37.9	0.0552	40.5	0.0487
	39.3	0.0689	40.3	0.0504
	35.5	0.0472	41.3	0.0487
	41.2	0.0505	42.2	0.0428
	37.5	0.0467	41.1	0.0556
	40.9	0.0487	42.7	0.0575
	38.8	0.0532	38.2	0.0674
	42.1	0.0523	40.9	0.0532
	39.5	0.146	40.2	0.0487
	38.2	0.0613	41.3	0.0604
Mean, $\bar{x}$	39.0	0.0561	40.9	0.0533
Std. Dev., S	1.83	0.0081	1.15	0.0065
$\Sigma(x-\bar{x})^2$	$36.8 \times 10^6$	$7 \times 10^{-4}$	$14.6 \times 10^6$	$4.62 \times 10^{-4}$
Sample Size, N	12	11	12	12
Design Allow., $\bar{x}-3s$	33.5		37.5	

TABLE 18

Modulus of Rupture and Weight Loss  
of Chemcor C-112 After Being  
Exposed 150 Days to Air (50% RH)  
and Loaded in Flexure to 60% of Initial MOR

	SPOTS-UP		SPOTS-DOWN	
	<u>MOR</u> <u>psi</u>	<u>Weight Loss</u> <u>mg/cm<sup>2</sup></u>	<u>MOR</u> <u>psi</u>	<u>Weight Loss</u> <u>mg/cm<sup>2</sup></u>
	46.700	0.0061	35.5	0.0061
	34.900	0.0014	38.3	0.0010
	35.400	0.0028	39.6	0.0038
	47.900	0.0005	37.3	0.0033
	40.000	0.0057	37.9	0.0066
	38.200	0.0001	34.5	0.0052
	38.900	0.0001	36.4	0.0010
		0.0024	39.4	0.0047
	39.000	0.0028	36.8	0.0057
	37.900	0.0038	39.6	0.0047
	38.200	0.0028	39.8	0.0052
			38.9	0.0085
Mean, $\bar{x}$	39.70	0.0026	37.8	0.0047
Std. Dev., S	4.30	0.0021	1.76	0.0022
$\Sigma(x-\bar{x})^2$	16.7	$4.38 \times 10^{-5}$	33.9	$5.16 \times 10^{-5}$
Sample Size, N	10	11	12	12
Design Allow. $\bar{x} - 3S$	27.000		32.5	

modulus of rupture data scatter as indicated by the coefficient of variation was decreased by storage in air and salt water regardless of whether the spots were up or down.

From this study it was concluded that the final modulus of rupture strength of glass laths having one face abraded by a standard sandblasting procedure and stored under flexural stress in 50% R.H. or in salt water is not effected by the state of stress of the sandblasted face during the storage period under flexural load.

Question of Wet Strength Vs. Dry Strength. After the three-year test program was finished an additional question arose as to what the alternative result might have been had the ultimate strengths of the specimens been measured while the specimens were wet with salt water, rather than in equilibrium with 50% R.H. Since a long delay and expense would be required to obtain more lath specimens, use was made of a commercial product made of similar [1.] surface-compression strengthened glass, i.e. one-milliliter COREX pipets. These were purchased by the gross and a three-week ancillary test program was set up. One group of 12 was tested dry. Another set was immersed in salt water and tested immediately. A third set was soaked in salt water for three weeks and tested in salt water. The results are presented in Figure 23, which shows that their average MOR was reduced slightly when determined on wet specimens, while the CV was decreased substantially and the  $\bar{X}$ -3s strength was increased significantly by testing them wet, rather than dry.

The effect, Figure 23, of exposure to water was to reduce the high values and increase the low values of strength, which makes for a more uniform, more predictable product.

This ancillary test program offered no indication that a serious difference would have been observed in the results of the main test program had we tested the SCS glass lath wet, rather than in equilibrium with 50% R.H. air.

Statistical Treatment of Data. The nature of the data obtained and the objective of the study make Student's "t" test which is commonly used (reference 5 and 6) for determining the significance of the difference between two means an extremely valuable tool. This test determines statistically the probability, P, that the difference between two means,  $\bar{X}_1$  and  $\bar{X}_2$ , is accidental. For non-paired data of small and unequal sample sizes the following equation is used to determine "t".

$$"t" = \frac{\bar{X}_1 - \bar{X}_2}{\sqrt{\frac{\sum (x_{i1} - \bar{X}_1)^2 + \sum (x_{i2} - \bar{X}_2)^2}{N_1 + N_2 - 2} \left( \frac{1}{N_1} + \frac{1}{N_2} \right)}}$$

CONDITIONS <sup>(1)</sup>  STORAGE <sup>(2)</sup> TEST <sup>(3)</sup>	BENDING STRENGTH <sup>(6)</sup> - KSI				
	NO. N	AVERAGE $\bar{X}$	STD. DEV $\sigma$	COFF. VAR. %	99.9% RELIABLE $\bar{X}-3\sigma$
AIR <sup>(4)</sup> AIR	12	50.24	4.24	8.45	37.5
AIR WET	12	48.65	2.72	5.58	40.5
WET WET	12	48.69	1.81	3.72	43.3

- (1) ROOM TEMPERATURE
- (2) THREE WEEKS
- (3) 0.05 in./min.
- (4) LABORATORY ATMOSPHERE
- (5) SALTWATER
- (6) FOUR-POINT FLEXURE

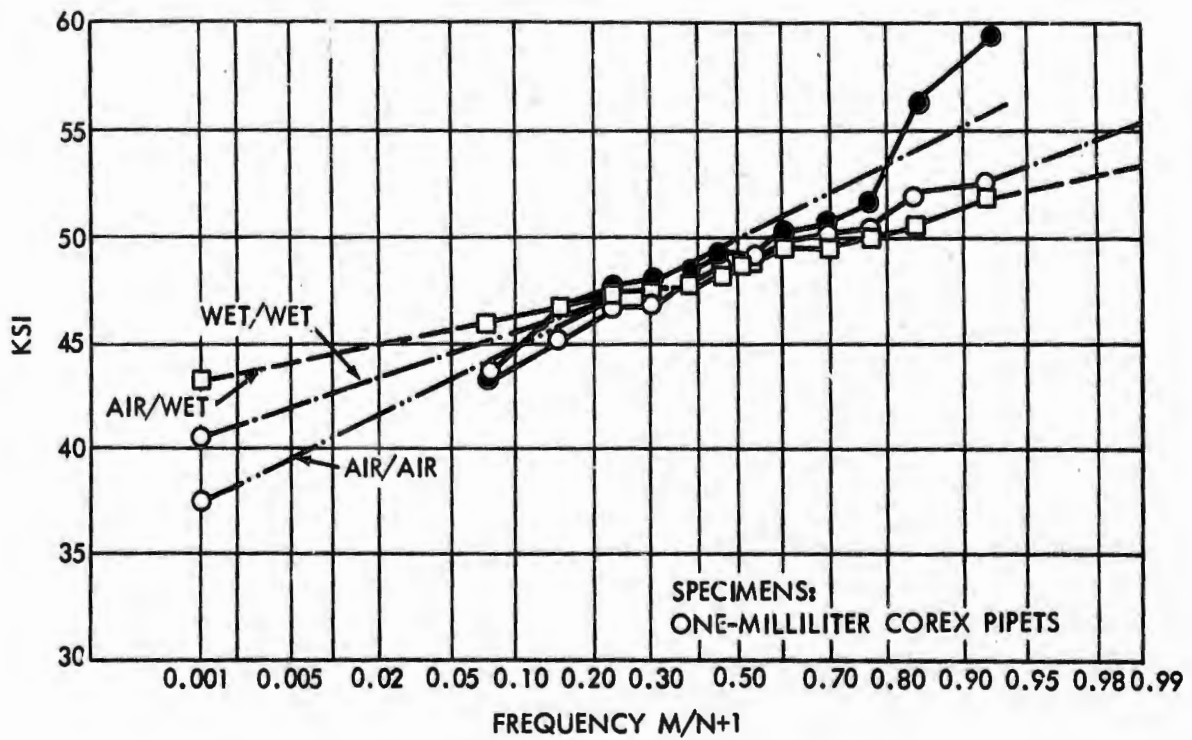


FIG. 23 WET STRENGTH VS DRY STRENGTH OF SCS GLASS

where:  $\bar{X}$  = Group means  
 $X_i$  = Individual values  
 $N$  = Number of values in the group

The probability,  $P$ , is found by entering Fisher's table of "t" values with the "t" calculated and using the row for the proper "number" of degrees of freedom" (defined as  $N_1 + N_2 - 2$ ) finding the correct probability column. More precise values of  $P$  may be determined by interpolating or by graphical solution.

The probabilities that observed differences in strength between exposures occurred by chance are given, by materials in Tables 19 through 23. The smaller the probability of chance, the greater is the significance of the observed difference.

General Observations. Assuming that holding the glass laths under constant stress in contact with air or salt water has an effect on their strength, this effect would be expected to increase with time. Therefore, the change in strength obtained after the longest period of time, three years, should be the most reliable observation of the long-time effect. However, since minimum strength is the controlling factor in designing to avoid failure, strength determinations at intermediate times are important in ruling out anomalous strength behavior. That is, strength could conceivably first decrease and then increase with time. Since the mean strength data presented in Figures 11 through 16 show no such anomalous strength behavior, the data presented in Table 15 reliably summarizes changes in strength and design allowable strength data for periods up to three years. With the exception of annealed Pyrex all the glasses tested showed a higher modulus of rupture strength after storage under stress in salt water for the three years. More important, all glasses without exception showed an increase in the design allowable strength due to the decrease in data scatter as well as to the fact that no glass lost strength during the time period. Contrary to this general three year strength increase, the modulus of rupture data presented graphically in Figure 14 shows the average strength for both Herculite II and Chemcor C-112 to be dropping 4% and 7% respectively in the time interval between one and three years. This is a significant observation. It appears that stress corrosion may be effecting these glasses causing their strength to first increase for roughly one year and then gradually drop off. However, after the three-year period their mean strengths are still above the initial value but heading downwards. Similar action could have been occurring with Chemcor C-113, but, unfortunately, no one-year strength data was taken on C-113.

The solubility data presented in Table 16 and graphically in Figure 18 could be expected to have a bearing on the strength changes



TABLE 19

The Significance of the Differences Between  
 Mean MOR Values Obtained for Groups of  
Pyrex (Annealed) Laths Using Student's "t" Test

Fisher's Probability, P

<u>Time-Days</u>	<u>Initial</u>	<u>30</u>	<u>90</u>	<u>150</u>	<u>365</u>	<u>1100</u>
Initial	-	0.53	0.30	0.81	0.39	0.82
Air 176	0.35	-	-	0.25	-	-
Air 1100	0.80	-	-	-	-	0.51

Degree of Significance

- \*\*\* Extremely significant - Difference at 0.1% level of confidence
- \*\* Highly significant - Difference at 1% level of confidence
- \* Significant - Difference at 5% level of confidence
- No significances

TABLE 20

The Significance of the Differences Between  
 Mean MOR Values Obtained for Groups of  
Pyrex (semi-tempered) Laths Using Student's "t" Test

Fisher's Probability, P

<u>Time-Days</u>	<u>Initial</u>	<u>30</u>	<u>90</u>	<u>150</u>	<u>365</u>	<u>1100</u>
Initial	-	0.007**	0.1	0.25	0.001***	<0.001***
Control 176	0.07	-	-	0.75	-	-
Control 1100	0.23	-	-	-	-	<0.001***

Degree of Significance

- \*\*\* Extremely significant - Difference at 0.1% level of confidence
- \*\* Highly significant - Difference at 1.0% level of confidence
- \* Significant - Difference at 5.0% level of confidence
- No significance

TABLE 21

The Significance of the Differences Between  
Mean MOR Values Obtained for Groups  
of HERCULITE II Laths Using Student's "t" Test

Fisher's Probability, P

<u>Time-Days</u>	<u>Initial</u>	<u>30</u>	<u>90</u>	<u>150</u>	<u>365</u>	<u>1100</u>
Initial	-	0.01**	<0.001**	<0.001**	<0.001**	<0.001**
Air 90	0.06	-	0.06	-	-	-
Air 150	0.65	-	-	<0.001**	-	-
Air 365	0.25	-	-	-	<0.001**	-
Controls 176	0.20	-	-	<0.001**	-	-
Controls 1100	0.75	-	-	-	-	<0.001**
ssw 1100	-	-	-	-	0.006**	-

Degree of Significance

- \*\*\* Extremely significant - Difference at 0.1% level of confidence
- \*\* Highly significant - Difference at 1.0% level of confidence
- \* Significant - Difference at 5.0% level of confidence
- No significant difference

TABLE 22

The Significance of the Differences Between  
 Mean MOR Values Obtained for Groups  
 of CHEMCOR C-112 Laths Using Student's "t" Test

Fisher's Probability, P

<u>Time-Days</u>	<u>Initial</u>	<u>30</u>	<u>90</u>	<u>150</u>	<u>365</u>	<u>1100</u>
Initial	-	0.002 <sup>**</sup>	0.03 <sup>*</sup>	<0.001 <sup>***</sup>	<0.001 <sup>***</sup>	0.008 <sup>**</sup>
Control 176	0.08	-	-	0.002 <sup>**</sup>	-	-
Control 1100	<0.001 <sup>***</sup> (loss)	-	-	-	-	<0.001 <sup>***</sup>
Air 150	0.2	-	-	0.01 <sup>**</sup>	-	-
Air 365	0.02 <sup>*</sup>	-	-	-	0.003 <sup>**</sup>	-
ssw 1100	-	-	-	-	0.005 <sup>**</sup>	-

Degree of Significance

- \*\*\* Extremely significant - Difference at 0.1% level of confidence
- \*\* Highly significant - Difference at 1.0% level of confidence
- \* Significant - Difference at 5% level of confidence
- No significance

TABLE 23

The Significance of the Differences Between  
 Mean MOR Values Obtained for Groups  
 of CHEMCOR C-113 Laths Using Student's "t" Test

Fisher's Probability, P

<u>Time-Days</u>	<u>30</u>	<u>90</u>	<u>1100</u>
Initial	0.04*	0.05*	0.005**

Degree of Significance

- \*\*\* Extremely significant - Difference at 0.1% level of confidence
- \*\* Highly significant - Difference at 1% level of confidence
- \* Significant - Difference at 5% level of confidence
- No significance

with time. It is noteworthy that the two glasses losing strength in the one to three year period, Herculite II and Chemcor 112, are respectively the most soluble and the least soluble of the group but had almost the same depth of treatment. From this it may not be inferred that the strength loss resulted only from the large ions, used to stuff the surface of the glass forcing it into compression, being preferentially dissolved in the salt water. Abrasion crack geometries also changed, as can be seen in Figure 10. Apparently the fissures were being broadened and made visible by contact with salt water.

It is also worth noting that both Chemcor C-112 and C-113 glasses were even less soluble than Pyrex which is considered to be a chemically resistant glass. In spite of its relatively low solubility and highly compressed surface layer, Chemcor C-112 lost average strength between the first and third years of storage in salt water while gaining in its design allowable strength. At the same time the semi-tempered Pyrex which was found to be more soluble in the salt water, but had a very thick compressed layer, did not lose strength in this period. All of this appears to show that while a low solubility of the parent glass may be important for maintaining long-term strength, the depth of compression may also be important.

Significance of Differences. Results of Student's "t" test studies of the data summarized in Tables 19 through 23 give Fisher's probability (P) that the difference between the two means of small sets of data being compared could have occurred by chance. That is, the smaller the figure given in the tables, the greater is the significance of the difference between the means under consideration.

Pyrex, Annealed. As would be surmized from the raw strength data and calculated means, Table 18 shows no significant change in average strength with time for annealed Pyrex. However, as shown in Table 7 the useful or design allowable strength of the annealed Pyrex does increase with exposure to salt water or air, because of a decrease in its initial large coefficient of variation.

Pyrex, Semi-Tempered. Table 18 shows highly significant and extremely significant increases in the average strength of semi-tempered Pyrex exposed to salt water while under stress for all time periods except for 90 and 150 days. Consideration of Table 8, Figure 18 and the raw data of Appendix B shows a relatively large scatter of strength for the semi-tempered Pyrex laths at the end of these two periods. However, storage of the semi-tempered Pyrex glass lath under stress in salt water for periods of 365 and 1100 days very definitely increased its strength while air storage of the glass produced no change in strength during the same period of time.

Herculite II. More data points were obtained for Herculite II than for any of the other types of glass. Table 21 shows there is no significant increase in strength of this glass either stressed or unstressed in air, but there is a decided strength increase for laths stored in salt water for all time periods through 1100 days.

However, as mentioned under results, there is a definite (4%) drop in mean strength during the 365 to 1100 day interval. The "t" test shows this drop to be highly significant, i.e. only a 0.006 chance that the observed difference is purely accidental. In view of the fact that Herculite II owes the greatest part of its strength to the existence of a highly compressed surface layer and the weight measurements show it to be a relatively soluble glass, the development of a peak strength after some point in time followed by a gradual decline in strength appears reasonable to expect. Evidence in the present study indicates this to be the case for the Herculite II specimens tested.

Chemcor C-112. All Chemcor C-112 laths held in bending stress in salt water, Table 22, showed an increase in mean strength over the initial value; but as for the Herculite II laths, there was a highly significant drop (7%) in mean strength in the time period between 365 and 1100 days. While the same explanation as for the Herculite II may hold, Table 22 shows Chemcor C-112 to be only about one third as soluble as the Herculite II and has a slightly thicker compressed layer. The strength change of Chemcor C-112 with time in air is somewhat more puzzling. That is, when stored without an externally applied stress, there was a definite loss in strength at the end of 1100 days; but held under bending stress in air, there was a significant increase.

Chemcor C-113. For Chemcor C-113, the highest strength glass of all those tested, fewest data points were obtained because of a lack of specimens. Since no data was taken at 365 days, it is not known if the strength of this glass follows the drop off found for the Herculite II and Chemcor C-112. All that can be said is that Chemcor C-113 held under bending stress for 1100 days, Table 23, is highly significantly stronger than the initial glass and more normally distributed, Figure 21.

#### CONCLUSIONS.

The mean strengths ( $\bar{X}$ ) of all abraded glasses were increased by exposure to constant stress in salt water, and their coefficients of variation ( $100S/\bar{X}$ ) were decreased during three years of exposure. Their calculated reliable strengths ( $\bar{X}-3S$ ) were all increased significantly with time up to three years.

One glass (C-113) had an abnormal distribution of strengths after abrasion and before exposure, but three years of exposure to stress

in salt water had a remarkable effect increasing, normalizing, and equalizing the strength of this highly strengthened product.

Exposure in salt water had the effect of rendering abraded spots more visible.

A reduction in the mean ( $\bar{X}$ ) strengths and coefficients of variation of some glasses was observed between the one-year and three-year sets, while the calculated values of  $\bar{X}-3S$  increased.

The glasses with the highest strengths exhibited the smallest coefficients of variation, particularly after three years under stress in salt water.

No significant differences in strengths could be detected whether the abraded areas were more or less compressed during exposure or whether SCS glasses are ruptured in air or in salt water.

During the test the lath specimens were flexed like flat springs. Residual strains after unloading seemed to be asymptotic to zero. Weight losses after three years ranged from 0.112 to 0.315 milligrams/cm<sup>2</sup>. Two SCS glasses lost less weight than Pyrex. The highest average surface-regression rate was estimated to be  $4.5 \times 10^{-5}$  cm/year.

In general, the exposure of four types of abraded SCS glasses for three years to salt water and/or constant stress makes for a more uniform and predictable structural material.

Surface-compression strengthened glasses possess attractive properties of strength and endurance in salt water for use as naval structural materials.

#### RECOMMENDATIONS

A ten-year exposure test program on SCS glasses should be undertaken to determine whether these materials can attain Exploratory Development Goal 546 [1], i.e., "Survival for ten to twenty years between refurbishment opportunities and suffer deterioration no greater than 10% with 0.95 confidence." This program should include tests in the laboratory under controlled conditions and in the ocean under real conditions.

Other SCS glasses should be tested, including the "neutral" glasses which are extremely resistant to water. The strengthened glass-ceramics should be evaluated. The types of stress profiles should include the conventional tapered profiles tested in this program and the step profiles that can be generated by electrolytic ion displacements. SCS glasses with deep compression layers should be tested.

In the interest of making competitive comparison, other light-weight structural materials (aluminum, titanium, reinforced plastics) should be included, also without paint or protective barriers under constant stress.

Tests should include specimen geometries, some of which can be stressed uni-axially as in this program, and others that can be stressed multi-axially as can the NOL ring/disc test specimen.



Experiments at sea should be designed to differentiate between the effects of acidity or alkalinity, water temperature, salinity, pollution, water velocity, silt content, oxygen content, vegetation and animal growths, static and cyclic loads, freezing and thawing.

In view of the behavior of the flat specimens under load in sea water, it is recommended that SCS glass springs be considered for use underwater.

In general, it is recommended that SCS glass structures be considered for use underwater for periods of exposure up to three years.

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APPENDIX A  
DESIGN OF EXPERIMENTS

Grouping Specimens by Measurement of Their Internal Tensile Stresses

Statistical validity of the study of the effect on the modulus of rupture strength of the glass laths held under bending loads and stored in the circulating salt water was dependent upon the assumption that the initial lots of specimens could be divided into the several groups of specimens each having the same mean modulus of rupture. Rather than using large samples of specimens and relying on accidental randomization, it was decided to use a relatively small sample (12) and arrange them in groups having the same mean core tensile stress as determined using a polarizing microscope and Babinet compensator. The underlying principle, of course, is that the specimen strength is dependent on the degree of surface compression which in turn is reflected in the tensile stress in the center of the specimen. The annealed specimens having a zero core tensile stress could not be grouped in this way and accidental randomization was relied upon.

The general plan was to number the specimens of each type of glass consecutively, measure the internal stress of each, and through a computer program arrange them in groups of 12 having equal mean center stresses with minimum standard deviations from the mean for each group. Greater randomization of the specimens and uniqueness of the arrangement was assured by the latter stipulation.

Stress Measurement

Theory explaining the measurement of strain in glass is explained in detail by Morey and by Shand. In general, stress applied to a glass results in a strain of the glass which is reflected in change in the index refraction of the glass, which in turn is related in both magnitude and direction to the applied stress. Strained glass thus takes on the optical properties of a uni-axial mineral becoming "double refracting" and exhibiting, "birefringence." The degree of birefringence or fractional difference in retardation ( $r$ ) may be measured using a polarizing microscope equipped with a Babinet compensator which has been calibrated against mineral sections of known retardation. In determining the stress ( $\sigma$ ) in a section of glass, the retardation ( $r$ ) is measured and related to the stress by the equation:  $\sigma = \frac{r}{B^1}$ , where  $B^1$ , the relative stress-optical coefficient, for each type of glass is determined by applying a known stress to a section of the glass and measuring the resulting retardation with the compensator. By taking the reciprocal of the relative stress-optical coefficient and converting the stress units to psi, a more convenient factor (psi/nm) is obtained. The following factors were used for this series of glasses.

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<u>Glass</u>	<u>psi/nm</u>
Pyrex	1.47
Herculite II	2.15
Chemcor 14112	2.28
Chemcor 14113	2.28

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APPENDIX B

TABLE B-1

Strength<sup>1</sup> of Individual Glass Laths 0 Day controls <sup>2</sup>

	<u>Flexural Strength-Kpsi</u>				
	<u>Annealed Pyrex</u>	<u>Tempered Pyrex</u>	<u>Herclite II</u>	<u>Chemcor C-112</u>	<u>Chemcor C-113</u>
	6.4	12.4	42.6	38.8	62.9
	5.0	10.4	39.8	37.8	68.9
	5.5	12.8	41.4	38.7	62.2
	6.4	11.8	40.2	37.3	71.7
	5.6	12.6	43.2	37.5	68.2
	7.2	11.3	38.6	41.6	51.7
	7.9	11.3	42.3	38.5	74.2
	7.3	11.5	39.8	37.8	70.1
	4.0	12.3	39.5	37.2	57.6
	5.8	11.0	45.0	37.1	66.6
	8.6	11.6	41.8	37.4	73.8
	6.1	11.7	41.1	36.7	73.6
Mean Strength, $\bar{X}$	6.3	11.7	41.3	38.0	66.8
Std. Dev., S	1.28	0.702	1.82	1.30	7.01
Coefficient of Variation %	20.3	6.0	4.4	3.4	10.5
Design Allowable Strength ( $\bar{X}-3s$ )	2.5	9.6	35.8	34.1	45.8
$\sum (X-\bar{X})^2$	18.1	5.42	36.5	18.7	540.0

- (1) Modulus of Rupture (MOR) determined in a four point loading fixture placing abraded face in tension
- (2) Randomized groups of 12 specimens in each sample used to estimate the mean strength of the total population.

APPENDIX B

TABLE B-2

Strength<sup>1</sup> of Individual Glass Laths 176 day controls<sup>2</sup>

	<u>Flexural Strength-Kpsi</u>				
	<u>Annealed Pyrex</u>	<u>Tempered Pyrex</u>	<u>Herculite II</u>	<u>Chemcor C-112</u>	<u>Chemcor C-113</u>
	6.5	10.8	40.0	38.0	NA <sup>3</sup>
	4.9	12.2	42.6	39.8	
	6.9	11.3	43.5	38.1	
	7.1	12.5	42.4	38.3	
	6.9	12.5	41.9	40.9	
	7.0	12.9	47.1	39.7	
	6.6	12.5	24.1 <sup>4</sup>	40.3	
	7.2	11.9	42.1	38.0	
	7.5	12.7	39.9	36.6	
	6.4	12.4	43.1	48.2 <sup>5</sup>	
	7.2	12.2	41.0	40.2	
	5.9	12.7	42.0	40.7	
Mean Strength, $\bar{X}$	6.7	12.2	42.3	39.1	--
Std. Dev., S	0.706	0.615	1.96	1.40	--
Coefficient of Variation %	10.6	5.0	4.6	3.6	--
Design Allowable Strength ( $\bar{X}-3s$ )	4.6	10.4	36.5	34.9	--
$\sum (X-\bar{X})^2$	5.48	4.16	38.2	19.6	--

- (1) Modulus of Rupture (MOR) determined in a four point loading fixture placing abraded are in tension.
- (2) Randomized groups (see appendix A) of 12 specimens stored on the laboratory bench top.
- (3) Not applicable- no specimens available for this test.
- (4) Edge break, not used in averaging.
- (5) Unexplained datum- not used in average.

TABLE B-3

Strength<sup>1</sup> of Individual Glass Laths 3 year controls<sup>2</sup>

	Flexural Strength-Kpsi				
	Annealed Pyrex	Tempered Pyrex	Herculite II	Chemcor C-112	Chemcor C-113
	6.7	11.9	42.9	35.0	ND <sup>3</sup>
	5.9	11.2	42.0	36.9	
	7.9	11.7	42.9	27.2 <sup>4</sup>	
	5.5	10.2	40.8	34.4	
	7.0	11.2	40.9	35.5	
	6.0	11.4	42.9	*	
	5.8	11.9	37.8	36.7	
	6.2	11.8	42.0	36.6	
	6.7	11.9	42.8	36.9	
	6.6	10.9	42.1	36.1	
	6.6	11.4	40.0	37.5	
	5.4	11.8	41.4	38.7	
Mean Strength, $\bar{X}$	6.4	11.4	41.5	36.4	--
Std. Dev., S	0.705	0.514	1.51	1.25	--
Coefficient of Variation %	11.1	4.5	3.6	3.4	--
Design Allowable Strength ( $\bar{X}-3S$ )	4.3	9.9	37.0	32.7	--
$\Sigma(X-\bar{X})^2$	5.47	2.91	25.2	14.0	--

- (1) Modulus Rupture (MOR) determined in a four point Loading fixture placing abraded face in tension
- (2) Randomized groups of 12 specimens in each sample (see Appendix A) stored on laboratory bench top.
- (3) No data-no specimens available for this test.
- (4) Edge break not used in averaging, etc.
- \* specimen misplaced



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APPENDIX B

TABLE B-4

Strength<sup>1</sup> of Individual Glass Laths stored under Load<sup>2</sup>  
often 30 days in SSW

	<u>Flexural Strength-Kpsi</u>				
	<u>Annealed</u> <u>Pyrex</u>	<u>Tempered</u> <u>Pyrex</u>	<u>Herculite II</u>	<u>Chemcor</u> <u>C-112</u>	<u>Chemcor</u> <u>C-113</u>
	20	50	70	50.60	70.80
	5.36	13.1	41.4	37.4	78.3
	7.32	12.0	45.2	40.3	70.3
	6.55	13.3	43.2	43.0	73.5
	6.16	13.9	44.1	39.6	69.7
	7.27		44.4	39.8	70.7
	7.67		41.4	41.5	89.1
			44.9	41.3	70.5
			43.5	42.1	
				40.0	
				36.8	
				39.9	
				41.6	
Mean, $\bar{X}$	6.72	13.1	43.5	40.3	74.6
Std. Dev., $S$	0.867	0.793	1.46	1.82	7.07
$\Sigma (X-\bar{X})^2$	3.76	1.89	14.9	36.3	300.0
sample size, $N$	6	4	8	12	7

- (1) Modulus of Rupture (MOR) determined in a four point loading fixture, abraded face in tension.
- (2) Flexural four point load applied to laths so that the abraded face was placed in tension.

TABLE B-5

Strength<sup>1</sup> of Individual Glass Laths After 90 days  
Under Load<sup>2</sup> in salt water

	<u>Flexural Strength-Kpsi</u>				
	<u>Annealed</u>	<u>Tempered</u>	<u>Herculite II</u>	<u>Chemcor</u>	<u>Chemcor</u>
	<u>Pyrex</u>	<u>Pyrex</u>		<u>C-112</u>	<u>C-113</u>
	5.0	****	45.0	*	73.1
	+	11.5	*	34.9	67.3
	6.8	11.9	45.1	41.5	68.0
	5.8	11.4	45.9	39.7	86.6
	5.1	14.6	43.0	41.3	127.9 <sup>3</sup>
	6.7	11.9	45.6	43.0	74.7
	***	13.1	45.1	40.0	*
	6.3	12.4	45.5	*	*
	5.9	11.4	45.8	40.0	72.4
	5.5	11.9	44.5	**	*
	5.8	12.1	45.3	40.2	*
	4.6	12.7	46.4	39.1	70.4
Mean Strength, $\bar{X}$	5.75	12.3	45.2	40.0	73.2
Std. Dev., S	0.723	0.942	0.891	2.23	6.48
Coefficient of Variation %	12.6	7.7	2.0	5.6	8.9
Design Allowable Strength ( $\bar{X}-3S$ )	3.6	9.5	42.5	33.3	53.7
$\Sigma(X-\bar{X})^2$	4.71	8.87	7.94	39.9	252.0

(1) Modulus of Rupture (MOR) determined in a four point loading fixture, abraded face in tension.

(2) Flexural four point load applied to laths so that the abraded face was placed in tension.

3 Unexplained datum, not used in averaging, etc.

4 Accidentally broken just prior to strength test

\* loading failure

\*\* failed after 6 days immersion under load

\*\*\*failed after 27 days immersion under load

\*\*\*\* failed 48 days immersion under load

TABLE B-6

Strength<sup>1</sup> of Individual Glass Laths After 150 days Under Load<sup>2</sup> in salt water

	<u>Flexural Strength-Kpsi</u>				
	<u>Annealed Pyrex</u>	<u>Tempered Pyrex</u>	<u>Herculite II</u>	<u>Chemcor C-112</u>	<u>Chemcor C-113</u>
	6.8	11.0	47.6	41.2	NA <sup>3</sup>
	6.3	13.3	45.7	41.9	
	6.6	12.3	46.7	39.8	
	7.1	12.1	46.6	41.1	
	6.1	**	45.7	38.5	
	5.2	10.5	42.6	43.6	
	6.4	12.8	45.4	41.3	
	5.7	13.3	46.5	40.8	
	6.9	12.4	45.7	40.1	
	6.8	12.0	44.1	41.7	
	6.1	11.2	42.7	41.0	
	7.1	12.8	*	43.1	
Mean Strength, $\bar{X}$	6.43	12.2	45.4	41.2	--
Std. Dev., S	0.580	0.925	1.62	1.37	--
Coefficient of Variation %	9.0	7.6	3.6	3.3	--
Design Allowable Strength ( $\bar{X}-3s$ )	4.7	9.4	40.5	37.1	--
$\Sigma (X-\bar{X})^2$	3.70	8.55	26.3	20.8	--

- (1) Modulus of Rupture (MOR) determined in a four point loading fixture, abraded face in tension.
- (2) Flexural four point load applied to laths so that the abraded face was placed in tension.
- (3) Not applicable-no specimens available for this test.

\* loading failure

\*\* failed after 6 days immersion under load

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TABLE B-7

Strength<sup>1</sup> of Individual Glass Laths After 365 days  
Under Load<sup>2</sup> in salt water

	<u>Annealed</u> <u>Pyrex</u>	<u>Tempered</u> <u>Pyrex</u>	<u>Herculite II</u>	<u>Chemcor</u> <u>C-112</u>	<u>Chemcor</u> <u>C-113</u>
	5.0	14.5	45.8	40.8	ND <sup>3</sup>
	7.1	12.4	46.3	41.3	
	5.8	13.8	46.9	44.2	
	8.0	12.5	47.2	45.8	
	7.7	12.0	49.6	40.7	
	6.9	12.6	48.8	41.4	
	7.0	*	48.8	*	
	7.8	12.6	33.3 <sup>4</sup>	*	
	6.1	12.3	47.0	41.4	
	5.9	13.2	50.5	43.6	
	7.1	13.2	43.7	41.2	
	5.6	**	45.6	46.3	
Average Strength ( $\bar{X}$ )	6.7	12.9	47.3	42.7	
Std. Dev., S	0.97	0.77	1.98	2.13	
Coefficient of Variation %	15.0	5.9	4.2	5.0	
Design Allowable Strength ( $\bar{X}-3s$ )	3.8	10.6	41.3	36.3	
$\Sigma(X-\bar{X})^2$	10	5.31	39.4	40.8	

- (1) Modulus of Rupture (MOR) determined in a four point loading fixture, abraded face in tension.
  - (2) Flexural four point load applied to laths so that the abraded face was placed in tension.
  - (3) No data-no specimens available for this test.
  - (4) Unexplained datum, not used in averaging etc.
- \* loading failure
- \*\* failed after 3 days immersion under load
- 4 unexplained datum, not used in averaging, etc.

TABLE B-8

Strength<sup>1</sup> of Individual Glass Laths after various  
Time Periods Under Load<sup>2</sup> in air<sup>3</sup>

Time Under Load	Flexural Strengths-Kpsi				
	90 Days	150 Days		365 Days	
	Herculite II	Herculite II	Chemcor C-112	Herculite II	Chemcor C-112
	49.1	41.2	*	40.4	41.1
	48.4	40.3	44.9	44.0	40.0
	45.4	39.9	40.4	39.6	*
	41.4	42.6	37.2	41.6	49.3 <sup>4</sup>
	41.0	39.9	38.4	40.6	41.5
	40.3	39.2	36.5	43.5	40.1
	39.9	40.4	38.6	44.1	36.5
	43.2	43.7	38.1	44.5	39.0
	43.0	43.8	38.0	43.5	38.0
	41.4	40.6	39.5	37.8	40.4
	44.5	42.2	39.1	45.6	37.9
	41.9	46.6	37.8	41.9	40.0
Mean Strength, $\bar{X}$	43.3	41.7	39.0	42.3	39.6
Std. Dev., S	3.01	2.16	2.24	2.32	1.47
Coefficient of Variation %	6.9	5.2	5.8	5.5	3.7
Design Allowable Strength ( $\bar{X}-3s$ )	34.3	35.2	32.2	35.3	35.2
$\Sigma(X-\bar{X})^2$	99.6	51.3	50.3	59.4	21.6

- (1) Modulus of Rupture (MOR) determined in a four point loading fixture placing the abraded face in tension.
  - (2) Flexural Four point load applied to laths so that the abraded face was placed in tension.
  - (3) Instead of salt water, the specimens under load were in contact with the air in the laboratory at roughly 50% RH and 72 degrees Fahrenheit.
  - (4) Unexplained datum-not used in averaging.
- \* Loading failures

TABLE B-9

Strength<sup>1</sup> of Individual Glass Laths After 3 years  
Under Load<sup>2</sup> in Salt Water.

	Flexural Strength-Kpsi				
	Annealed Pyrex	Tempered Pyrex	Herculite II	Chemcor C-112	Chemcor C-113
	6.8	14.0	45.6	39.6	*
	7.4	11.7	45.6	*	*
	**	13.0	44.3	*	70.3
	5.5	12.8	45.6	39.8	75.4
	6.0	13.8	44.0	***	74.4
	4.6	13.0	46.8	39.7	77.2
	5.7	13.0	43.1	40.6	78.0
	7.1	13.0	45.8	39.1	74.5
	6.1	13.2	44.7	40.6	76.8
	6.7	11.8	45.5	34.8 <sup>3</sup>	*
	6.9	13.3	45.5	37.4	76.5
	5.5	13.6	45.4	41.3	*
Mean Strength, $\bar{X}$	6.2	13.0	45.2	39.8	75.4
Std. Dev., S	0.85	0.694	0.977	1.18	2.41
Coefficient of Variation %	14.0	5.3	2.2	3.0	3.2
Design Allowable Strength ( $\bar{X}-3s$ ) <sub>2</sub>	3.7	10.9	42.2	36.2	68.2
$\Sigma(X-X)$	7.2	5.30	10.5	9.82	41.0

- (1) Modulus of Rupture (MOR) determined in a four point loading fixture placing abraded face in tension.
  - (2) Flexural four point load applied to laths so that the abraded face was placed in tension.
  - (3) Edge break, not used in averaging etc.
- \* loading failure  
 \*\* failed after unknown number of days  
 \*\*\* failed after 4 days under load