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FOREWORD

This report was prepared by the National Bureau of Standards, Washington, D. C. under Air Force Order No. AF(33-657)62-362. The contract was initiated under Project No. 7381, "Materials Application", Task No. 738106 "Design Information Development". The work was administered under the direction of the AF Materials Laboratory, Research and Technology Division, with Mr. R. E. Wittman as project officer.

This report covers work conducted from January 1965 to December 1965.

The mechanical testing was performed in the Glass Section under Mr. C. H. Hahner, the Section Chief. The statistical analysis was made by J. M. Cameron of the Statistical Engineering Section.

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This report has been reviewed and is approved.

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ABSTRACT

This report is concerned with one phase of an overall program established to provide realistic design criteria on several oxide glasses. These glasses were chosen on the basis of their present and possible future use as windows and viewing ports in aircraft and space vehicles.

A relationship between the stress at the point of fracture and the size of the mirror on the fracture face of glass rods of varying diameters has been established, and shown not to be affected by a change in the diameter of the rod. Although similar work was not done with plate glass, it is believed that here again, specimen size does not affect the relationship.

The elastic properties of two chemically strengthened glasses were determined at elevated temperatures. Young's modulus and shear modulus show a sharp drop in the temperature range from 23.9°C (75°F) to 249°C (480°F). Other glasses in the program did not show this inflection. The strength of these glasses as well as the other glasses in the program were determined at -17.8°C (0°F) and -45.6°C (-50°F), with the results showing that the strength increased at these temperatures as compared to the room temperature strength.

Creep curves for the chemically strengthened glasses were obtained at 75°F, and appeared similar to those of earlier glasses in the program. Stress-rupture tests on abraded specimens revealed failures at 75% stress level at most test temperatures, but no failures at 67% at 75°F. Unabraded specimens tested only at 75°F, had failures at 90% but not at 75%. Curves and stress-rupture information are in Appendix II.

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INTRODUCTION

The development of military aircraft to meet advanced requirements in terms of speed, operating heights, etc., together with the needs for space vehicle viewports has created problems in finding enclosure materials that will withstand high temperatures as well as stresses introduced by thermal gradients and loading. At present glass appears to be the most suitable material for glazings that operate under conditions of high temperatures; however, for the proper utilization of glass in these applications accurate information on its properties at elevated temperatures is required.

In order to determine some of these properties this program was initiated by the Air Force Materials Laboratory with the objectives of: 1) developing test methods for measuring the effect of temperature on the physical properties of glass, and 2) determining the properties of some presently available commercial glasses that appear to be suitable for aircraft glazing.

This report contains the results on the investigation of the relationship of the stress at fracture to the mirror portion of the fracture face for annealed glass rods of various diameters; the elastic properties of two chemically strengthened glasses at elevated temperatures, and the modulus of rupture values of the glasses in the program determined at two temperatures below room temperature.

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SUMMARY

Annealed Pyrex brand glass rods ranging in diameter from 0.16 inch to 1.5 inch were broken in flexure at room temperature. The results indicate that for the range of diameters studied, the strength decreases as the rod diameter increases; but that the size of the rod does not have a discernible affect on the relationship between the size of the mirror portion of the fracture face and the strength.

The elastic properties of two chemically strengthened glasses, CGW 0311 and PPG Herculite II, were determined at temperatures ranging from 23.9°C (75°F) to slightly less than 593°C (1100°F). The values of Young's modulus and shear modulus for both glasses showed a sharp drop when the temperature was raised from 23.9°C (75°F) to 249°C (480°F) followed by a continuing, but gradual, decrease as the temperature was increased above 249°C (480°F). Poisson's ratio was not affected by an increase in temperature.

The modulus of rupture of all of the glasses in the program was determined at -17.8°C (0°F) and -45.6°C (-50°F) after about a fifteen minute exposure to these temperatures. The strength at these temperatures was greater than at room temperature. For the two chemically strengthened glasses the strength was found to be highest at -17.8°C (0°F), while for all of the other glasses the highest strength was attained at -45.6°C (-50°F).

Stress rupture tests conducted with chemically strengthened glasses involved mainly abraded specimens at test temperatures of 75°, 200°, 300°, 400° and 500°F, and stress levels of 90%, 75% and 67%. At the 75% stress level there were failures at most test temperatures, but no specimens failed at 67% at 75°F. Unabraded specimens were tested, but only at 75°F. There were failures at 90%, but none at the 75% stress level. Creep curves for these glasses were obtained only at 75°F, and appeared similar to those of earlier glasses in the program.

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RELATION OF THE FRACTURE STRESS TO THE FRACTURE PATTERN FOR GLASS RODS OF VARIOUS DIAMETERS

Introduction

Previous work on annealed glass laths (1) showed that such factors as surface conditions, rate of loading, and temperature, which affect the strength of glass do not affect the relationship between the fracture pattern and the modulus of rupture. The fracture face consists of a smooth area which includes the fracture origin bounded by an area of increasing roughness, graduating from a stippled area to a rougher hackled area. The smooth area is referred to as the "mirror" in this paper. Another factor that affects the strength of glass is specimen size: This had not been previously investigated, so this study was made to determine if specimen size affected the strength-mirror size relationship for glass rods.

Experimental

Pyrex brand glass rods were used for the specimens in diameters of: 0.16 inch (4 mm), 0.24 inch (6 mm), 0.39 inch (10 mm), 0.75 inch, 1.0 inch, and 1.5 inch. The three smaller diameter rods were cut into 4.5 inch lengths and the three larger diameter rods were cut into 10 inch lengths. After cutting, the rods were rinsed with tap water and placed in a furnace and well annealed. Once annealed, the specimens were stored at room temperature and 50 per cent relative humidity, until tested under the same conditions. Mid-point loading was used, with the three smaller diameter rods supported over a four inch span and the three larger diameter rods supported over an eight inch span. All were loaded at a rate of 10,000 psi/min. Test groups ranged in size from 22 to 73 specimens. After fracture the diameter of the specimens was measured, taking care to measure the diameter that was in line with the applied load, for the rods were slightly elliptical in cross-section. The distance the

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fracture occurred from the point of maximum load application was measured, as was the angle between the point of maximum stress and the fracture origin. These values were used to calculate the stress at the fracture origin, which is used in this work instead of the modulus of rupture.

The size of the mirror was measured with a travelling microscope. It was measured along a diameter that led from the fracture origin on the periphery of the rod to the inner edge of the stippled area surrounding the mirror.

Results and Discussion

The average mirror size and the average fracture stress are plotted against rod diameter in Figure 1. Here it is shown that the fracture stress varies inversely and the mirror size directly with rod diameter.

The individual specimen data for the various sizes of rods are plotted in Figure 2, where on a log-log basis, the typical linear relationship between fracture stress and mirror size is obtained. Also revealed here is the inverse relationship between the strength of the rods and the size of their mirrors. It can be seen that the data from each diameter rod group lies well distributed about the line with the data of the smaller, stronger rods tending to occupy the upper end and the larger, weaker rods the lower end of this line.

The equation $MR^b = A$ was used to describe the relation between the mirror size and the strength of glass. In this equation M is the stress at the fracture origin in psi, R is the size of the mirror in inches, and b and A are constants.

The most common presentation of this equation has b equal to $\frac{1}{2}$. With this value of b the equation has been found to describe the data with fair approximation (2,3). However, previous work (1,4) has shown that when a "best fit" determination is made on the data a value of b lower than $\frac{1}{2}$ is

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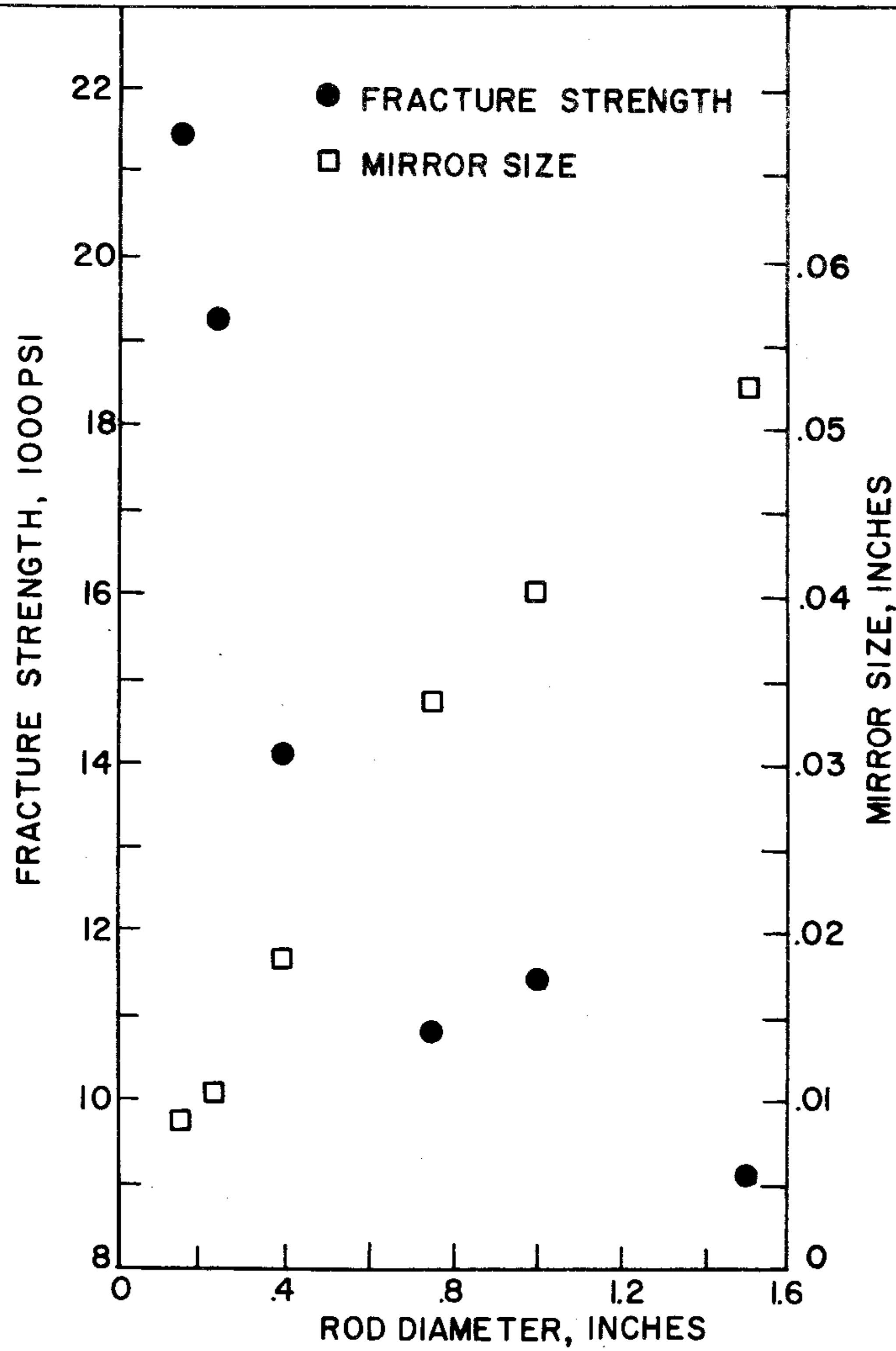

FIGURE I. STRESS AT FRACTURE AND MIRROR SIZE FOR VARIOUS ROD DIAMETERS

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generally derived. With this in mind the equation was solved with b as $\frac{1}{2}$ and also for the best fit with no restrictions placed on b. This was done by applying the method of least squares to the logarithmic form,

$$\log M = \log A - b(\log R).$$

The values obtained for A and b are presented in Table 1, and indicates that for each rod diameter the value for b of less than $\frac{1}{2}$ has a smaller standard deviation, and thereby fits the data better than the corresponding value of $\frac{1}{2}$; a finding similar to that for annealed glass laths (1,4).

The average values of the best fit b's are plotted in Figure 3 along with two standard deviations on either side of the average. The figure shows that the values of b are not significantly different one from another and that they fail to establish any kind of pattern with the size of the rod. This information together with the small differences among the A values shows that within the range of the rod diameters tested the size of the rod does not have a discernible effect on the relation between mirror size and strength.

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TABLE 1

CONSTANTS FOR THE EQUATION $\log M = \log A - b(\log R)$.

Rod Diameter (inches)	Number of Specimens	A	b	$\sigma(b)$[1]	$\sigma(Eq)$[2]	A	b	$\sigma(Eq)$[2]
0.16	26	2224	0.467	0.0271	1451	1900	0.5	1574
0.24	22	2306	0.461	0.0397	1002	1930	0.5	1043
0.39	29	2337	0.434	0.0227	899	1777	0.5	990
0.75	73	2219	0.452	0.0297	1105	1878	0.5	1130
1.00	51	2373	0.446	0.0139	764	1971	0.5	806
1.50	58	2271	0.441	0.0218	744	1890	0.5	819

1/ Standard deviation of slope b as determined from least squares fit.

2/ Standard deviation (individual points about the line).

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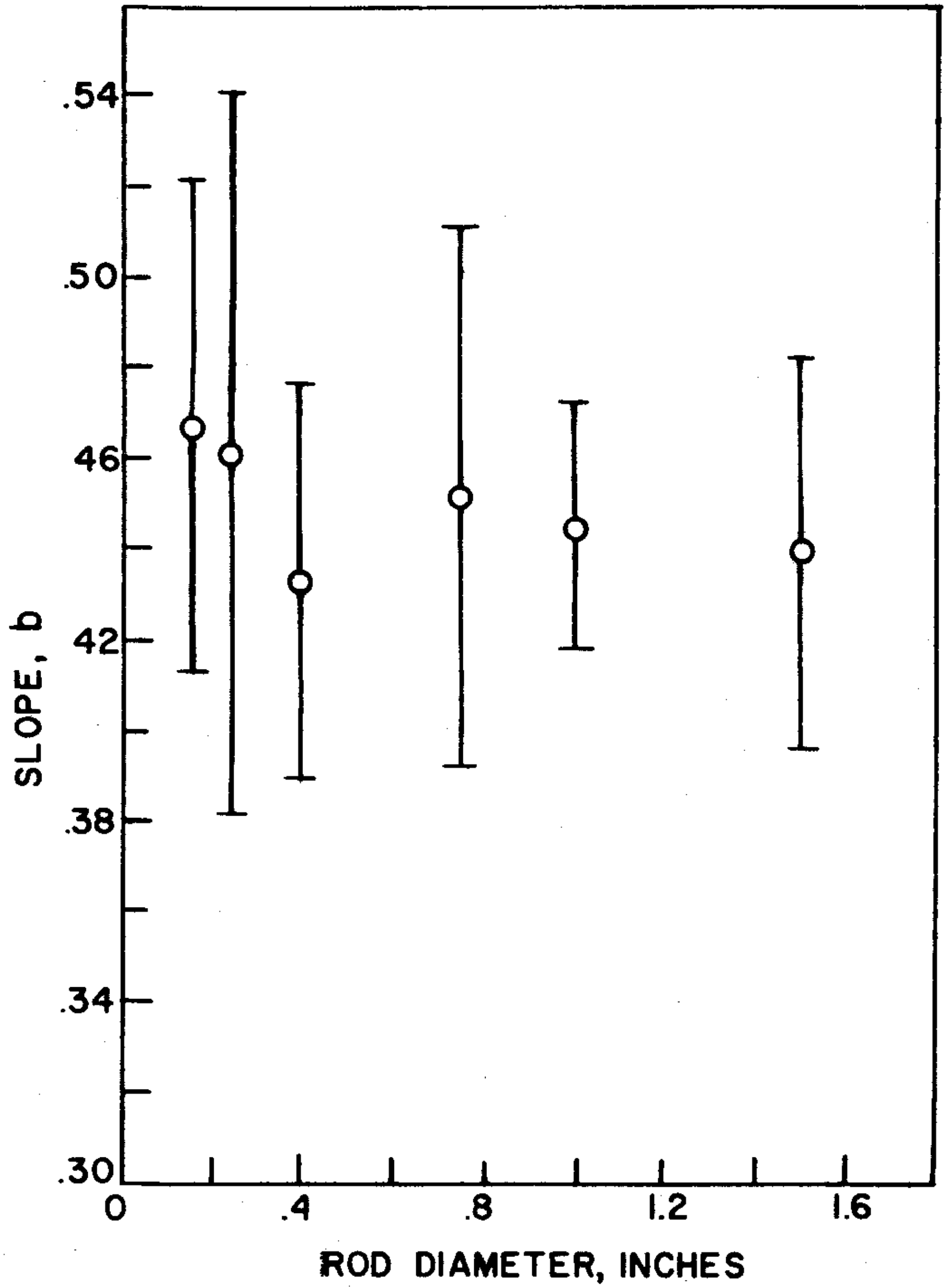

FIGURE 3 SLOPES OF THE BEST FIT LINEAR EQUATIONS VS. ROD DIAMETER

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ELASTIC PROPERTIES OF CHEMICALLY STRENGTHENED GLASSES AT ELEVATED TEMPERATURES

Introduction

At the Conference on Transparent Materials for Aerospace Enclosures held at Dayton, Ohio in December 1964, a number of people expressed an interest in the shape the Young's modulus curve for glass would assume upon cooling from an elevated temperature. We had reported the effect of temperature on Young's modulus (5) as the specimen was heated but did not show the curve developed on cooling.

The work reported here is the effect of change in temperature, both heating and cooling, on the elastic properties of several strengthened glasses.

Experimental

The specimens tested were: thermally tempered soda-lime-silica, thermally semi-tempered borosilicate (Corning Glass Works Code No. 7740), chemically strengthened Pittsburgh Plate Glass Co.'s Herculite II (No. 7265), chemically strengthened Corning Glass Works' Chemcor (Code No. 0311), and a specimen of Corning Glass Works' Chemcor that showed no indication of internal stress when inspected with crossed polaroids and had only 45 psi center tension when measured with the Babinet compensator. This was the only specimen found in which no internal stress was indicated. The thermally tempered specimens were 10 inches by 1½ inches by ¼ inch while the chemically strengthened specimens were 6 inches by 1 inch by 0.1 inch. One specimen of each type was tested, and the amount of center stress, before and after the heating and cooling, is given in Table 2.

The elastic properties were determined by a dynamic method in the same manner as previously used and reported (6). The flexural and torsional modes were measured for the determination of Young's modulus and shear modulus respectively.

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Both the fundamental and first overtone frequencies were measured. However, the fundamental frequencies were used exclusively for the computation of the moduli, since in each case their response was strong and their variation with temperature was not significantly different from that of the overtone.

TABLE 2

AMOUNT OF CENTER TENSION IN SPECIMENS
USED FOR ELASTIC PROPERTY DETERMINATIONS
AT ELEVATED TEMPERATURES

Specimen	Center Tension Before Heating	After Second Cooling
	psi	psi
Soda-lime-silica	7540	0
CGW 7740 (Borosilicate)	2656	0
PPG No. 7265 (Herculite II)	6994	647 1/
CGW Code No. 0311 (Chemcor)	8165	1737
CGW Code No. 0311 (Chemcor) showing no strain pattern	45	0

1/ This is center compression and not center tension.

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The temperature of the furnace in which the specimens were tested was raised at a rate of approximately 80°C (176°F) per hour until a top temperature in the range of 565-600°C (1049°F-1112°F) was reached. At several temperatures between room temperature and the top testing temperature the temperature was held for about ten minutes, until the specimen came to temperature equilibrium, and a measurement of the various frequencies was made. Once the top temperature measurements were made the same measurement procedure was followed during the cooling cycle. The specimens were cooled at a rate of approximately 50°C (90°F) per hour until a temperature of about 150°C (302°F) was reached. At this point the furnace was allowed to cool naturally overnight.

All of the specimens were run through two heating and cooling cycles with the exception of Corning Glass Works Code No. 7740; no measurements were made during the second cooling cycle for this specimen.

Results and Discussion

Figures 4 and 5 show the effect of heating and cooling on thermally tempered soda-lime-silica glass and thermally tempered borosilicate glass (CGW 7740), respectively. These curves are typical of Young's modulus at elevated temperature for these glasses, with the borosilicate showing an increase in Young's modulus with temperature and the soda-lime-silica showing a decrease with temperature. The top temperature reached in each case was well above the strain point of the glass, so that the induced stress was removed at the higher temperatures and the glass approached the annealed condition; then upon cooling the Young's modulus curve assumed the curve for annealed glass. The second heating and cooling effected only very small changes in the moduli of both glasses, showing that the induced stress had largely been removed during the first heating. Upon removal from the furnace after the second cycle both glasses were measured to see if any center tension remained. No measurable stress was present.

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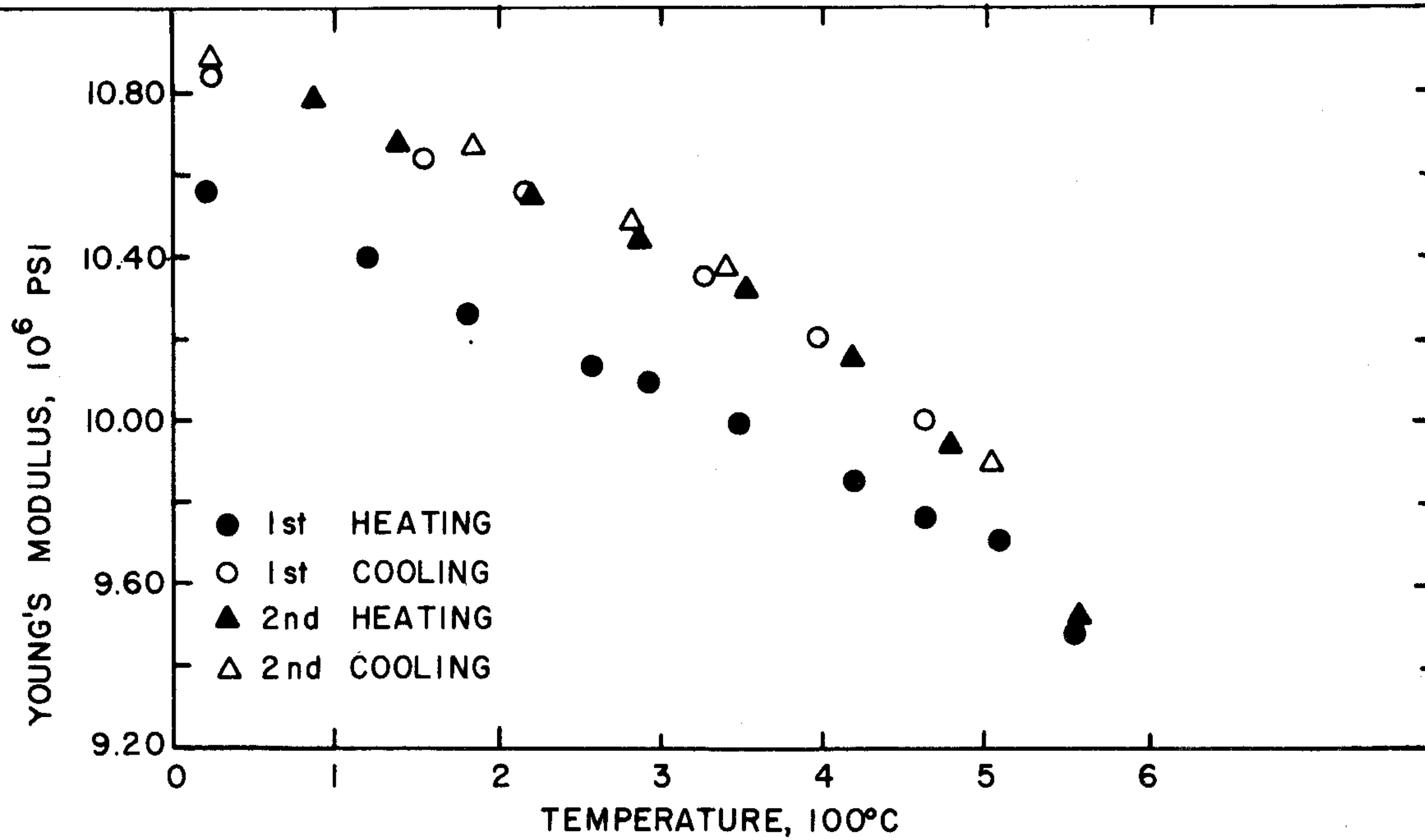

FIG. 4. YOUNG'S MODULUS AT ELEVATED TEMPERATURES FOR THERMALLY TEMPERED SODA-LIME-SILICA GLASS

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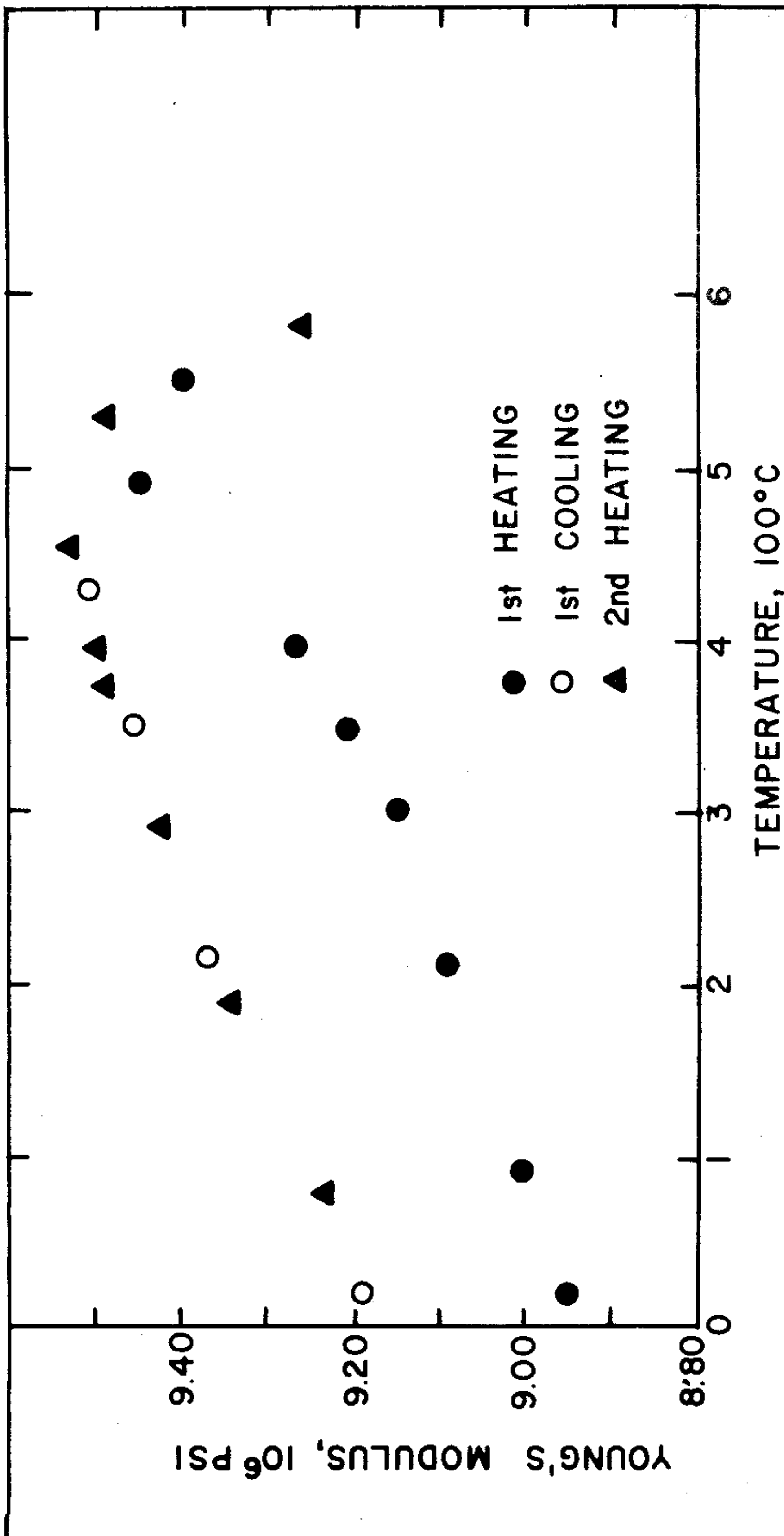

FIG. 5. YOUNG'S MODULUS AT ELEVATED TEMPERATURES FOR THERMALLY TEMPERED BOROSILICATE GLASS

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The Young's modulus versus temperature curves for the two chemically strengthened glasses that are shown in Figures 6 and 7 are typical for these glasses. There is a slight increase in Young's modulus after heating and cooling but very small when compared to the thermally tempered glasses. The degree of temper (center tension stress) in the specimens is given in Table 2, and it can be seen that the stress in the specimens was considerably altered by the heating and cooling, for the Chemcor specimens had about 20 per cent of its original stress remaining while the Herculite II specimen showed a center compressive stress. The large change in the amount of stress in the specimens and the small change in the elastic properties after the heating cycles indicate there was little change in the structure of the glass caused by the heating.

The chemically strengthened specimens were heated in an annealing furnace for 5 hours at 600°C (1112°F) and then slowly cooled to room temperature. Measurement of the degree of temper showed the Chemcor specimen to have a center compressive stress of 1125 psi and the Herculite II specimen to have center compressive stress of about 30 psi. The heating cycle was then repeated and the measurements showed the Chemcor specimen to have center tension of about 45 psi and the Herculite II specimen to have no measurable stress.

The Young's modulus values of the Herculite II specimen were determined again after the heat treatment in the annealing furnace and are shown in Figure 6. It can be seen that this heating caused an increase in the elastic modulus.

Figure 8 shows the effect of temperature on Young's modulus for the unstrained Chemcor specimen. It can be seen that the same type of curve was obtained for this specimen was obtained for the strained Chemcor specimen, except that the curve is slightly lower.

The values obtained for the Young's modulus, shear modulus, and Poisson's ratio are given in Appendix I.

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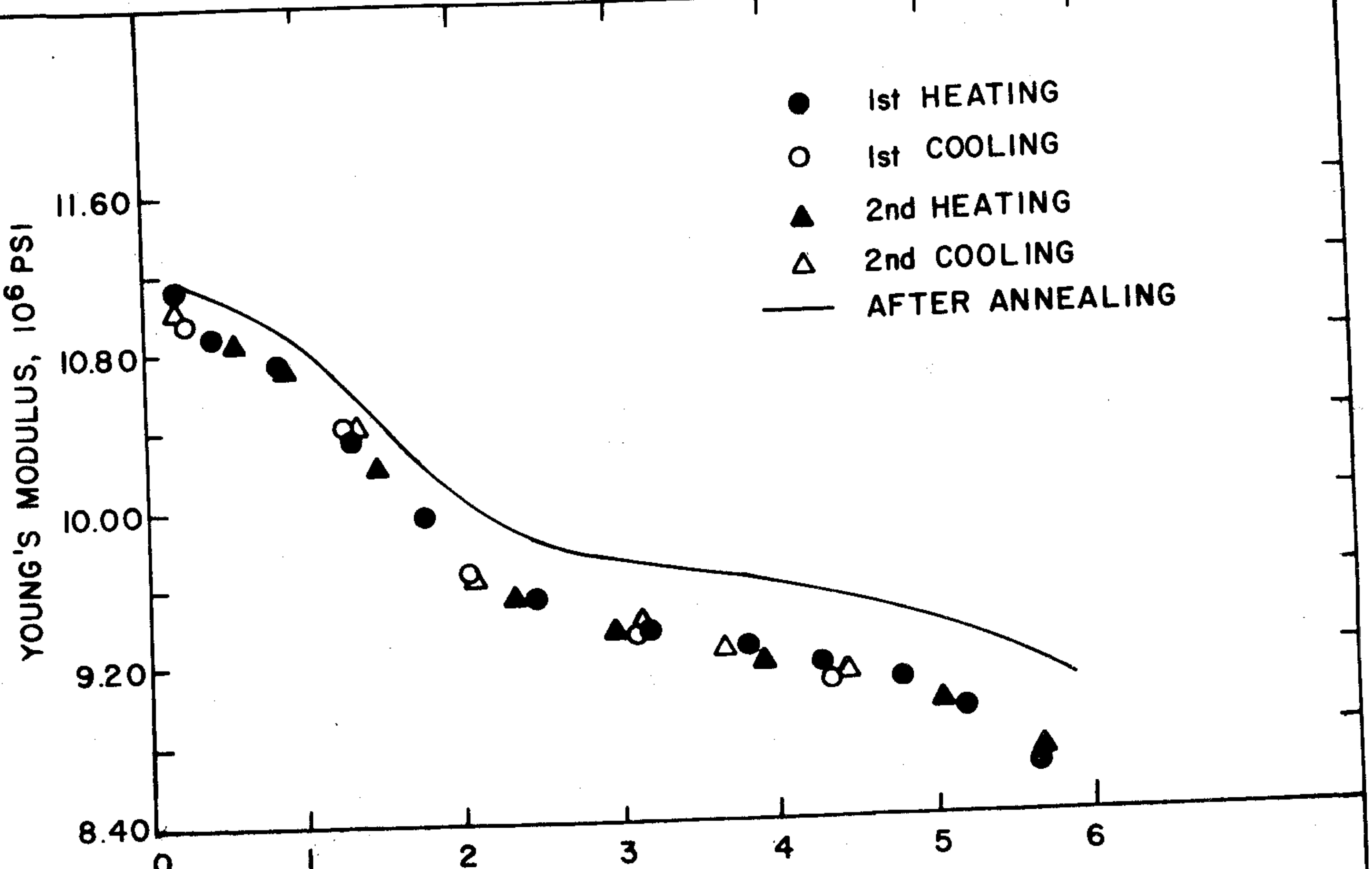

FIG. 6. YOUNG'S MODULUS AT ELEVATED TEMPERATURE FOR PPG HERCULITE II

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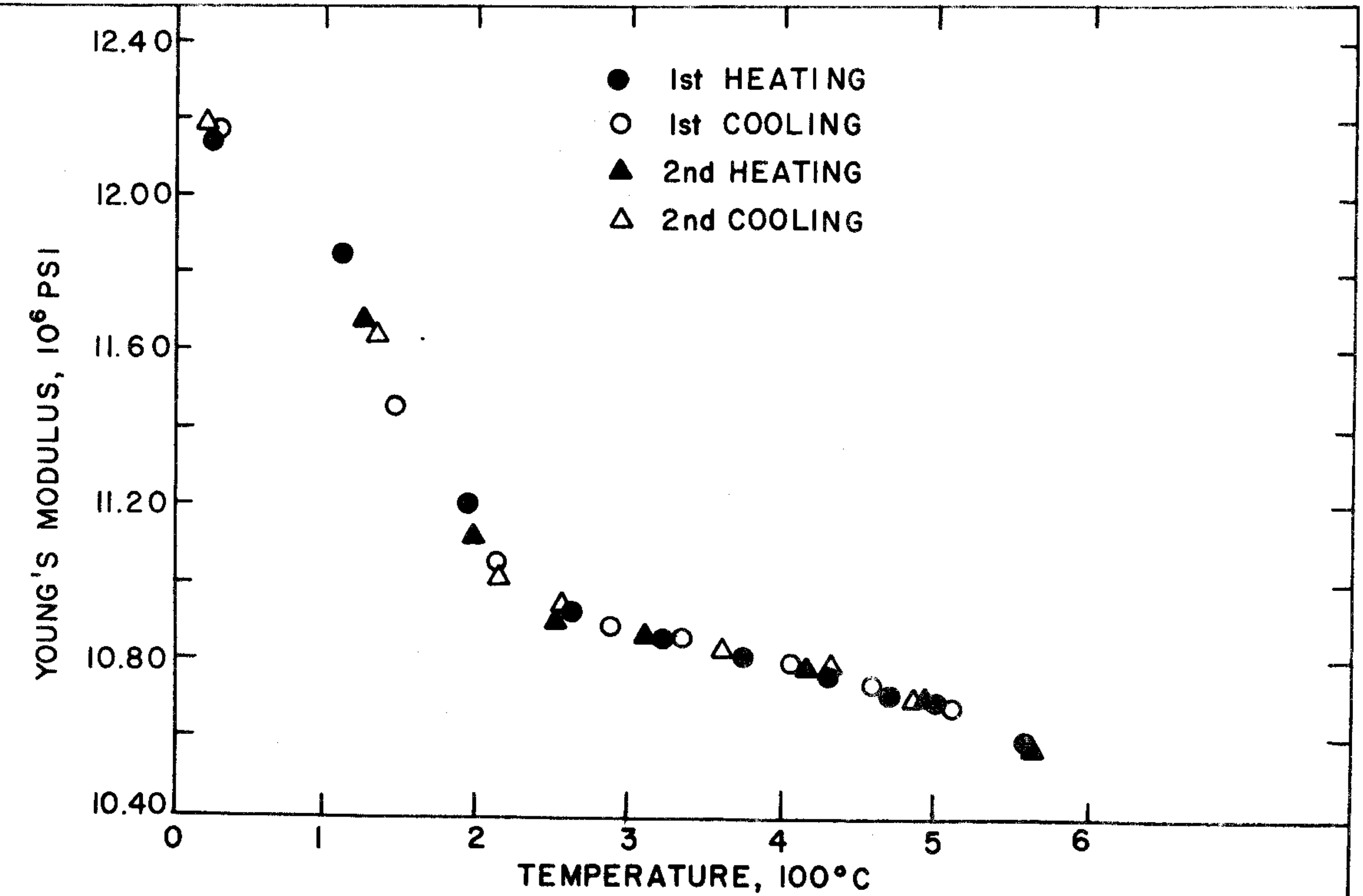

FIG. 7. YOUNG'S MODULUS AT ELEVATED TEMPERATURES FOR CGW CODE 0311

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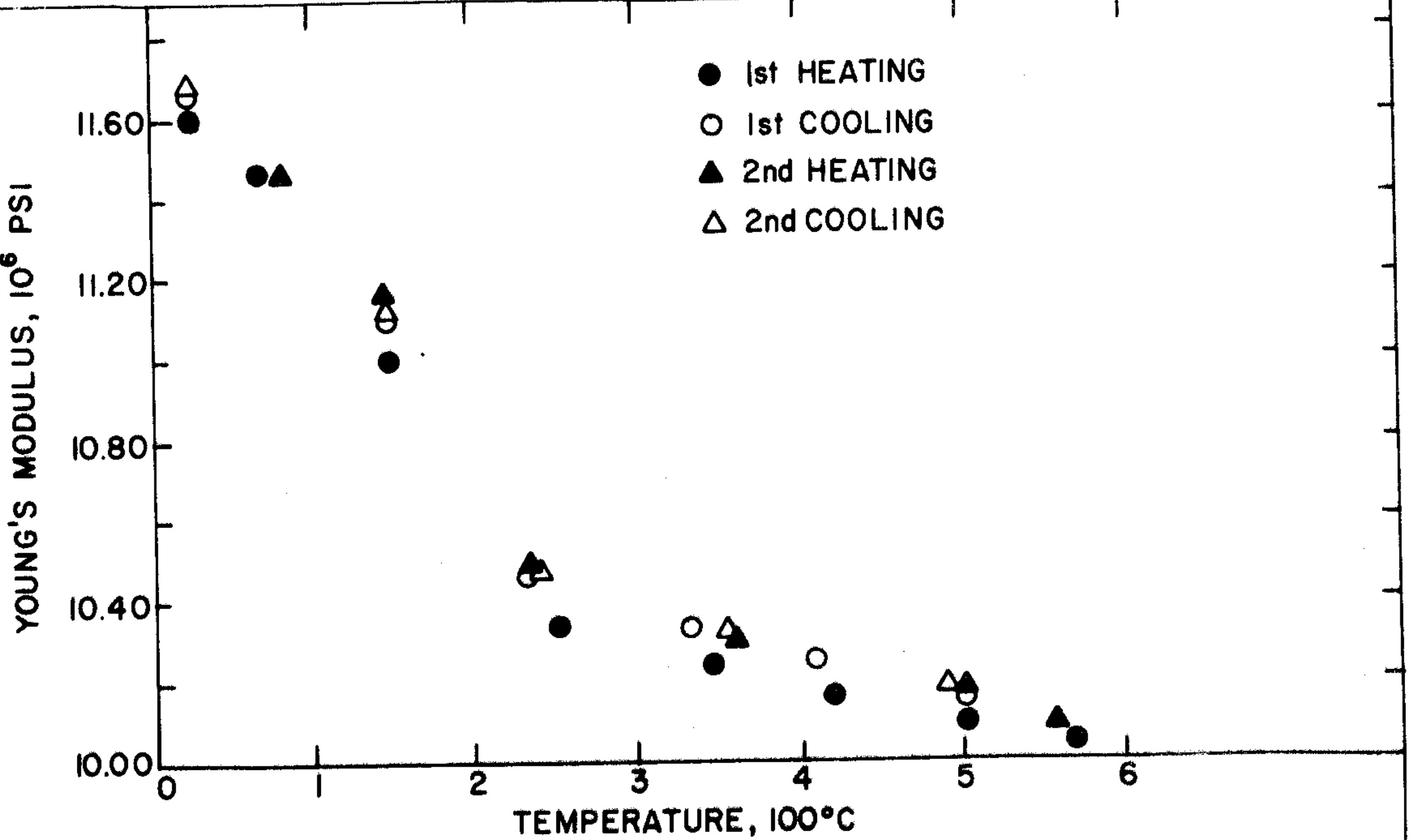

FIGURE 8. YOUNG'S MODULUS AT ELEVATED TEMPERATURES FOR A CGW 0311 SPECIMEN SHOWING NO STRAIN PATTERN

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It should be noted that the values of the elastic properties presented here may vary slightly from the true values, for the equations used to calculate the properties assume the specimen is a rectangular parallelpiped. The specimens used in these tests all had rounded edges and some had rounded corners or ends rounded to various diameters, and were not rectangular parallelpipeds.

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MODULUS OF RUPTURE DETERMINED BELOW ROOM TEMPERATURE

Experimental

Some specimens remained in five of the original samples of seven different glass compositions that were tested previously at elevated temperatures. These remaining specimens were used for a low temperature study. The specimens were 10 inches by $1\frac{1}{2}$ inches by $\frac{1}{4}$ inch with "as cut" edges. The surface opposite the surface with the scored edges had been abraded by the manufacturer. They had been wrapped in brown wrapping paper and stored in a laboratory since they were received from the manufacturer over five years ago. Measurement of the center tension of the semi-tempered and tempered specimens showed these specimens to have the same amount of center tension as the specimens tested previously.

Two of the seven glass compositions tested previously, soda-lime-silica and CGW 1723 (aluminosilicate), had no specimens remaining; however, these two glasses were studied in another work at low temperatures and are included in this report. These specimens were the same size as above but did not have abraded surfaces, and were recently obtained from the manufacturer.

Specimens of the two chemically strengthened glasses, CGW 0311 and PPG Herculite II were also tested. These specimens were 6 inches by 1 inch by 0.1 inch and were abraded at this laboratory. These specimens were from the same lots tested previously at elevated temperatures.

The loading apparatus and the procedure for testing the specimens at low temperatures were the same as those used for the previous work. The ten inch specimens were tested with an 8 inch support span and a two inch loading span with a loading rate of 10,000 psi/min. The six inch specimens were tested with a 5 inch support span and a loading span of $1\frac{1}{2}$ inches and a loading rate of 60,000 psi/min. Specimens were

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stored at least 48 hours at 23.9°C (75°F) and 50 per cent relative humidity before testing, and those tested at room temperature were loaded to failure under these conditions. Specimens tested at low temperature were placed in the cold box and held at the testing temperature for ten minutes. After it had been reached they were loaded to failure. The total time in the cold box was about 15 minutes.

Results and Discussion

Abraded Glasses

The modulus of rupture values obtained for the abraded glasses are presented in Table 3. In addition, the modulus of rupture values obtained by the original testing at 23.9°C (75°F) over five years ago are presented. Comparing the two room temperature values shows there is no appreciable difference between the glasses tested previously and those recently tested. This is probably explained by the fact that the glasses were stored under rather dry conditions, and as Mould (7) has pointed out, the aging effect appears to be eliminated when glass is stored in very dry air.

The modulus of rupture values determined at low temperatures show an increase over those determined at room temperature, regardless of composition or amount of temper. The glasses were stronger at -45.6°C (-50°F) than at -17.8°C (0°F).

Unabraded Glasses

The modulus of rupture values of the two glasses tested with unabraded surface, soda-lime-silica, and CGW 1723, are presented in Table 4. These values show that for both glasses, and all three conditions of temper, the strength increases as the temperature decreases.

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TABLE 3

MODULUS OF RUPTURE VALUES FOR ABRADED GLASS SPECIMENS TESTED BELOW ROOM TEMPERATURE

Glass	Degree of Temper [1]	Fracture Origin [2]	Original Testing (75°F)				75°F				0°F				% [7] Diff.	-50°F				% Diff.
			n [3]	$\bar{x}$ [4] psi	S.D. [5] psi	v [6] %	n	$\bar{x}$ psi	S.D. psi	v %	n	$\bar{x}$ psi	S.D. psi	v %		n	$\bar{x}$ psi	S.D. psi	v %	
PPG 6695	A	All	15	6974	1251	17.9	12	7172	826	11.5	11	8374	631	7.5	16.8	13	9050	540	6.0	26.2
		S	12	7059	1379	19.5	11	7297	739	10.1	11	8374	631	7.5		10	9164	505	5.5	
		E	3	6633	-	-	1	5803	-	-	0	-	-	-		3	8670			
PPG 6695	S	All	15	14660	993	6.8	15	14769	1038	7.0	15	17673	961	5.4	19.7	15	19154	854	4.5	29.7
		S	15	14660	993	6.8	15	14769	1038	7.0	15	17673	961	5.4		15	19154	854	4.5	
		E	0	-	-	-	0	-	-	-	0	-	-	-	-	0	-	-	-	
PPG 6695	T	All	15	22105	1654	7.5	15	22498	1319	5.9	15	26231	2229	8.5	16.6	15	27943	1968	7.0	24.2
		S	15	22105	1654	7.5	15	22498	1319	5.9	15	26231	2229	8.5		15	27943	1968	7.0	
		E	0	-	-	-	0	-	-	-	0	-	-	-		0	-	-	-	
PPG 3235	S	All	15	13380	682	5.1	15	14292	971	6.8	15	17396	1241	7.1	21.7	15	18637	1228	6.6	30.4
		S	15	13380	682	5.1	14	14227	973	6.8	15	17396	1241	7.1		15	18637	1228	6.6	
		E	0	-	-	-	0	-	-	-	0	-	-	-		0	-	-	-	
PPG 3235	T	All	15	21770	823	3.4	15	21551	1291	6.0	15	25522	1180	4.6	18.4	15	26556	2499	9.4	23.2
		S	15	21770	823	3.4	15	21551	1291	6.0	13	25660	1195	4.7		12	26691	2785	10.4	
		E	0	-	-	-	0	-	-	-	2	24625	-	-		0				
CGW 7740	A	All	15	5860	795	13.6	15	6682	770	11.5	15	7536	1114	14.7	12.8	15	8021	903	11.3	20.0
		S	13	6100	233	3.8	13	6738	793	11.8	10	7699	1112	14.4		13	8123	883	10.9	
		E	2	4300	-	-	2	6318	-	-	5	7210	1167	16.2		2	7356			
CGW 7900	A	All	15	5953	737	12.4	15	6204	555	8.9	15	6943	849	12.2	11.9	15	7478	687	9.2	20.5
		S	13	6150	627	10.2	13	6289	548	8.7	13	6957	855	12.3		10	7796	609	7.8	
		E	2	4700	-	-	2	5651	-	-	2	6850				5	6843	249	3.6	
CGW 7900	S	All	14	8990	2034	22.6	15	8639	1099	12.7	15	9974	1285	12.9	15.5	15	10583	1510	14.3	22.5
		S	14	8990	2034	22.6	15	8639	1099	12.7	15	9974	1285	12.9		15	10583	1510	14.3	
		E	0	-	-	-	0	-	-	-	0	-	-	-		0	-	-	-	
CGW 7940	A	All	15	6753	346	5.1	10	6784	658	9.7	12	7812	1053	13.5	15.2	13	8424	869	10.3	24.2
		S	14	6800	306	4.5	9	6846	666	9.7	10	8055	909	11.3		12	8353	868	10.4	
		E	1	6100	-	-	1	6225	-	-	2	6599	-	-		1	9278	-	-	

1/ A - annealed; S - semi-tempered; T - tempered.

2/ All indicates all of the specimens regardless of fracture origin, S indicates fracture originated on the surface, E indicates the fracture originated on the edge of the specimen. When S plus E does not equal the number of specimens in All, this indicates some specimens could not be identified as to fracture origin.

3/ Number of specimens.

4/ Average.

5/ Standard deviation.

6/ Coefficient of variation.

7/ Difference between testing temperature values and 75°F values divided by the 75°F value and multiplied by 100.

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TABLE 4

MODULUS OF RUPTURE VALUES FOR UNABRADED GLASS SPECIMENS TESTED BELOW ROOM TEMPERATURE

Glass	Degree of Temper[1]	Fracture Origin[2]	75°F n[3]	75°F $\bar{x}$[4] psi	75°F S.D.[5] psi	75°F ν[6] %	0°F n	0°F $\bar{x}$ psi	0°F S.D. psi	0°F ν %	%[7] Diff.	-50°F n	-50°F $\bar{x}$ psi	-50°F S.D. psi	-50°F ν %	% Diff.
CGW 1723	A	All	50	14455	3743	25.9	50	15171	2662	17.5	5.0	50	18629	3498	18.8	28.9
		S	21	14885	3125	21.0	3	18236	-	-		5	22223	3823	17.2	
		E	29	14143	4160	29.4	46	14891	2584	17.4		39	17968	3312	18.4	
CGW 1723	S	All	49	27234	3777	13.9	50	30655	4492	14.7	12.6	49	33734	4175	12.4	23.9
		S	42	26898	3857	14.3	21	30245	4244	14.0						
		E	7	29248	2638	9.0	26	30906	4642	14.9						
CGW 1723	T	All	50	29697	3956	13.3	50	34186	4142	12.1	15.1	50	37503	3839	10.2	26.3
		S	40	29468	3770	12.8	26	33961	4119	12.1						
		E	10	30613	4734	15.5	22	34515	4427	12.8						
PPG Soda-Lime-Silica	A	All	50	16917	5215	30.8	50	17453	3369	19.3	3.2	50	19925	4274	21.5	17.8
		S	24	19208	4338	22.6	3	19716	-	-		3	21898			
		E	26	14801	5127	34.6	47	17309	3383	19.5		37	19223	4167	21.7	
PPG Soda-Lime-Silica	S	All	50	24871	4923	19.8	48	28435	4551	16.0	14.3	50	32767	5042	15.4	31.7
		S	36	23426	3995	17.1	24	27373	4445	16.2						
		E	14	28587	5265	18.4	21	29858	4695	15.7						
PPG Soda-Lime-Silica	T	All	50	36345	5888	16.2	35	42157	3882	9.2	16.0	50	43707	4854	11.1	20.3
		S	40	35590	5073	14.3	7	40185	3539	8.8						
		E	10	39363	8037	20.4	13	44016	4101	9.3						

1/ A - annealed; S - semi-tempered; T - tempered.

2/ All indicates all of the specimens regardless of fracture origin, S indicates fracture originated on the surface, E indicates fracture originated on the edge of the specimen. When S plus E does not equal the number of specimens in All, this indicates some specimens could not be identified as to fracture origin.

3/ Number of specimens.

4/ Average.

5/ Standard deviation.

6/ Coefficient of variation.

7/ Difference between testing temperature values and 75°F values divided by the 75°F value and multiplied by 100.

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With the exception of the two unabraded glasses tested at -17.8°C (0°F), both the abraded and unabraded glasses showed similar increase in strength with decrease in temperature. It appears that the degree of temper of the glass does not have a discernible effect as far as the amount of increase in strength at a particular temperature is concerned.

The overall average of a 25 per cent increase in strength at -45.6°C (-50°F) obtained here agrees rather well with the 25 per cent increase found by Holland (8) on unabraded sheet glass at -40°C (-40°F) and the 27 per cent increase in strength found by Kropschot and Mikesell (9) on abraded borosilicate crown glass at -79°C (-110°F).

<u>Chemically Strengthened Glasses</u>

The strength values for the two abraded chemically strengthened glasses are presented in Table 5. These values show that the strength for both glasses increase at temperatures below room temperature; however, the strength at -17.8°C (0°F) is greater (about a 50 per cent increase over the room temperature strength) than at -45.6°C (-50°F) (about a 29 per cent increase over the room temperature strength.

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TABLE 5

MODULUS OF RUPTURE VALUES FOR ABRADED CHEMICALLY STRENGTHENED GLASSES DETERMINED BELOW ROOM TEMPERATURE

Glass	Temperature	Fracture Origin[1]	n[2]	Modulus of Rupture $\bar{x}$[3]	Modulus of Rupture S.D.[4]	Modulus of Rupture ν[5]	Birefringence of Center $\bar{x}$	Birefringence of Center S.D.	Birefringence of Center ν
	°F			psi	psi	%	mu/in	mu/in	%
CGW 0311	75	S	18	38194	3512	9.2	3514	93	2.6
		E	2	32491	-		3492	-	-
CGW 0311	0	All	20	57403	2675	4.66	3430	97.6	2.8
		S	16	57405	2779	4.84	3429	104.8	3.1
		E	2	56149	-	-	3409	-	-
CGW 0311	-50	All	20	49235	4400	8.9	3306	139.6	4.2
PPG Herculite II	75	S	20	36174	3695	10.2	3172	96	3.0
		E	0	-	-	-	-	-	-
PPG Herculite II	0	All	20	55658	3906	7.0	3084	103.6	3.4
		S	18	56441	3248	5.8	3087	105.9	3.4
		E	0	-	-	-	-	-	-
PPG Herculite II	-50	All	20	46844	3297	7.0	3078	81.9	2.7

1/ All indicates all of the specimens regardless of fracture origin, S indicates fracture originated on the surface, E indicates fracture originated on the edge of the specimen. When S plus E does not equal the number of specimens in All this indicates some specimens could not be identified as to fracture origin.

2/ Number of specimens tested.

3/ Average.

4/ Standard deviation.

5/ Coefficient of variation.

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APPENDIX I

Values obtained for Young's modulus, shear modulus, and Poisson's ratio at various temperatures for the glasses tested.

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TABLE 6

ELASTIC PROPERTIES FOR THERMALLY TEMPERED
SODA-LIME-SILICA GLASS AT ELEVATED TEMPERATURES

	Temp. °C	Young's Modulus 10^6 psi	Shear Modulus 10^6 psi	Poisson's Ratio
First Heating	21	10.56	4.24	0.24
	119	10.40	4.18	.25
	182	10.27	4.12	.25
	258	10.14	4.07	.25
	294	10.10	4.04	.25
	350	10.00	4.01	.25
	421	9.86	3.97	.24
	465	9.78	3.94	.24
	509	9.72	3.92	.24
	555	9.49	3.83	.24
First Cooling	462	10.01	4.03	.24
	398	10.21	4.11	.24
	328	10.36	4.18	.24
	216	10.56	4.26	.24
	156	10.65	4.31	.24
	22	10.85	4.39	.24
Second Heating	89	10.79	4.36	.24
	137	10.68	4.28	.25
	221	10.54	4.26	.24
	287	10.44	4.21	.24
	353	10.32	4.16	.24
	419	10.15	4.10	.24
	479	9.94	4.02	.24
	557	9.53	3.86	.24
Second Heating	505	9.90	3.99	.24
	340	10.38	4.19	.24
	282	10.49	4.23	.24
	186	10.67	4.30	.24
	23	10.89	4.40	.24

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TABLE 7

ELASTIC PROPERTIES FOR THERMALLY SEMI-TEMPERED CGW 7740 (BOROSILICATE) GLASS AT ELEVATED TEMPERATURES

	Temp. °C	Young's Modulus 10^6 psi	Shear Modulus 10^6 psi	Poisson's Ratio
First Heating	24	8.95	3.70	0.21
	96	9.01	3.72	.21
	114	9.04	3.72	.21
	215	9.10	3.74	.22
	304	9.15	3.75	.22
	352	9.22	3.76	.23
	400	9.28	3.79	.22
	494	9.45	3.85	.23
	554	9.40	3.84	.22
First Cooling	432	9.51	3.90	.22
	355	9.46	3.88	.22
	219	9.37	3.86	.21
	25	9.19	3.81	.21
Second Heating	83	9.23	3.83	.21
	195	9.34	3.85	.21
	294	9.42	3.88	.22
	378	9.49	3.89	.22
	400	9.50	3.89	.22
	458	9.54	3.90	.22
	534	9.49	3.88	.22
	586	9.26	-	-
After[1/] Second Cooling	23	9.20	3.82	.20

1/ No measurements were made during the cooling cycle.

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Table 8

ELASTIC PROPERTIES FOR CHEMICALLY
STRENGTHENED GLASS PPG NO. 7265 (HERCULITE II)
AT ELEVATED TEMPERATURES

	Temp. °C	Young's Modulus 10^6 psi	Shear Modulus 10^6 psi	Poisson's Ratio
First Heating	22	10.98	4.54	0.21
	46	10.91	4.51	.21
	87	10.76	4.44	.21
	133	10.36	4.34	.19
	180	9.98	4.21	.19
	248	9.54	3.92	.22
	321	9.39	3.88	.21
	383	9.29	3.83	.21
	429	9.19	3.79	.21
	479	9.10	3.74	.22
	520	8.96	3.70	.21
	566	8.65	3.57	.21
First Cooling	434	9.12	3.77	.21
	308	9.35	3.88	.20
	208	9.69	4.02	.20
	133	10.44	4.36	.20
	28	10.99	4.56	.20
Second Heating	59	10.87	4.52	.20
	92	10.73	4.46	.20
	150	10.23	4.31	.19
	235	9.55	3.99	.20
	298	9.37	3.86	.21
	394	9.20	3.81	.21
	506	8.97	3.71	.21
	567	8.72	3.61	.21
Second Cooling	448	9.13	3.78	.21
	368	9.28	3.85	.20
	314	9.38	3.90	.20
	211	9.66	4.01	.20
	137	10.44	4.38	.19
	23	11.02	4.58	.20

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TABLE 8 (CONT'D)

	Temp. °C	Young's Modulus 10^6 psi	Shear Modulus 10^6 psi	Poisson's Ratio
After[1] Annealing First Heating	22	11.15	4.62	0.21
	77	11.00	4.58	.20
	150	10.46	4.36	.20
	210	9.99	4.14	.21
	270	9.77	4.04	.21
	332	9.67	4.00	.21
	368	9.63	3.98	.21
	427	9.52	3.93	.21
	507	9.37	3.87	.21
	563	9.19	3.79	.21
	588	9.07	3.75	.21
Second Heating	26	11.12	4.61	.21
	92	10.89	4.52	.20
	192	10.00	4.18	.20
	254	9.69	4.03	.20
	315	9.59	3.97	.21
	378	9.51	3.93	.21
	440	9.41	3.89	.21
	481	9.35	3.86	.21
	525	9.28	3.82	.21
	563	9.16	3.77	.21
	599	8.89	3.67	.21
After Second Heating	23	11.11	4.60	.21

1/ Specimen was heated at 600°C (1112°F) for 5 hours and slowly cooled and showed a center compression of 30 psi. Specimen was again heated in the same manner and showed no measurable center strain. The after annealing elastic properties were then determined.

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TABLE 9

ELASTIC PROPERTIES FOR CHEMICALLY
STRENGTHENED GLASS CGW CODE NO. 0311 (CHEMCOR)
AT ELEVATED TEMPERATURES

	Temp. °C	Young's Modulus 10^6 psi	Shear Modulus 10^6 psi	Poisson's Ratio
First Heating	25	12.15	4.82	0.26
	111	11.86	4.71	.26
	196	11.21	4.47	.25
	262	10.93	4.34	.26
	322	10.86	4.32	.26
	375	10.81	4.29	.26
	432	10.76	4.27	.26
	474	10.72	4.25	.26
	501	10.70	4.23	.26
	560	10.59	4.21	.26
First Cooling	513	10.68	4.24	.26
	460	10.74	4.27	.26
	406	10.79	4.29	.26
	336	10.86	4.32	.26
	288	10.89	4.34	.25
	214	11.06	4.43	.25
	147	11.47	4.61	.25
	25	12.19	4.84	.26
Second Heating	125	11.69	4.70	.24
	200	11.12	4.44	.25
	254	10.91	4.33	.26
	311	10.87	4.33	.26
	419	10.78	4.29	.26
	498	10.70	4.25	.26
	566	10.58	4.21	.26
Second Cooling	491	10.70	4.25	.26
	434	10.77	4.28	.26
	362	10.83	4.31	.26
	260	10.93	4.34	.26
	215	11.01	4.42	.25
	135	11.65	4.69	.24
	22	12.20	4.85	.26

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TABLE 10

ELASTIC PROPERTIES AT ELEVATED TEMPERATURES
FOR A CGW CODE NO. 0311 (CHEMCOR) GLASS SPECIMEN
SHOWING NO STRAIN PATTERN[1/]

	Temp. °C	Young's Modulus 10^6 psi	Shear Modulus 10^6 psi	Poisson's Ratio
First Heating	25	11.60	4.87	0.19
	68	11.47	4.82	.19
	148	11.00	4.62	.19
	253	10.34	4.38	.18
	349	10.24	4.30	.19
	424	10.17	4.27	.19
	505	10.10	4.25	.19
	571	10.05	4.22	.19
First Cooling	503	10.17	4.27	.19
	414	10.26	4.31	.19
	334	10.34	4.35	.19
	233	10.48	4.44	.19
	146	11.10	-	-
	23	11.66	4.89	.19
Second Heating	83	11.47	4.82	.19
	141	11.17	-	-
	234	10.52	4.42	.19
	362	10.31	4.34	.19
	505	10.18	4.27	.19
	560	10.10	4.24	.19
Second Cooling	493	10.19	4.28	.19
	358	10.32	4.34	.19
	237	10.48	4.43	.18
	144	11.12	4.60	.21
	22	11.67	4.90	.19

1/ Examination of this specimen through crossed polaroids showed it to be of uniform color, the same as annealed glass. Measuring the amount of center tension with the Babinet compensator showed it to have 45 psi center tension, well within the range of annealed glass.

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APPENDIX II

Creep curves and stress-rupture data for chemically strengthened glasses, CGW Chemcor and PPG Herculite II

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STRESS-RUPTURE AND CREEP RESULTS FOR CHEMICALLY STRENGTHENED GLASSES

Introduction

Stress-rupture is a term used to denote failure of a specimen that has been under constant stress for a period of time. The term stress-rupture is often used interchangeably with fatigue and static-fatigue. Creep is a term applied to the deformation of a solid material over a period of time. Creep most generally occurs under load and at elevated temperatures. However, some material will deform at room temperature.

The stress-rupture and creep work included in this report was designed to complement the modulus of rupture tests performed as part of this program, and reported in report WADC-TR-56-645, Part X, March 1965. The modulus of rupture tests were made with a continuously increasing load for a short period of time (generally under one minute). For the stress-rupture and creep tests reported here, the specimens were under constant load and temperature until they fractured or survived 500 hours.

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Specimens, Apparatus, and Procedures

The specimens used for the stress-rupture and creep tests were all of the same size, 5 inch by 1 inch by 0.1 inch, and were randomly selected from the specimens supplied by the manufacturers. Two glass companies supplied chemically strengthened specimens for test. Corning Glass Works provided Chemcor (Code No. 0311) and the Pittsburgh Plate Glass Company provided Herculite II. The specimens were provided in the ground and polished condition with ground edges. For most of the specimens an area of 3/4 inch diameter was abraded in the center of the surface to be tested in tension. The abrasion, performed at the National Bureau of Standards, was made in the same manner as had been made for the other specimens in the program.

Specimens were tested over a five inch support span and loaded with a one and one-half inch load span. Knife edges were 1/8 inch diameter rods that rolled when the specimen deflected. Weights were hung from a yoke that fitted over the loading knife edge assembly and a jack was used to lower the weights and apply load to the specimen. A motion transformer measured the amount of deflection and also indicated the time at which a specimen broke.

Specimens tested at room temperature (75°F) were tested in a controlled atmosphere chamber maintained at a relative humidity of fifty percent. Those tested at elevated temperatures were tested in individual furnaces. These latter specimens were first heated to the test temperature and then loaded. The test temperatures used were 75°, 200°, 300°, 400° and 500°F, and were maintained within 5°F. There were 10 specimens in each test group.

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RESULTS

Stress-Rupture

The individual stress-rupture results for the two chemically strengthened glasses are presented in Tables 11 and 12. The tables show the time the individual specimens sustained load before they failed. An asterisk indicated the specimen survived for the 500 hour duration of the test. The results for abraded specimens show that at 90 percent stress there were failures at all temperatures. At 75 percent stress there were failures at all temperatures with the exception of CGW Chemcor at 200°F and PPG Herculite II at 400°F. The time-to-failure results are interesting for they show that at 300°F and below the failures tended to occur as load was applied or shortly after the application of load. At 400 and 500°F, the failures occured after the specimens were under load for some time. Apparently the exposure to elevated temperatures reduces the strengthening effect of the induced compressive layer and weakens the specimen.

These findings agree well with the modulus of rupture results, for these showed no change in strength after long exposure to temperatures of 200 and 300°F, but prolonged exposure to temperatures of 400 and 500°F greatly reduced the strength of these glasses.

The abraded specimens showed no failures at a stress level of 67 percent at 75°F.

Unabraded specimens were tested at 75°F; there were failures at 90 percent stress level but none at the 75 percent stress level.

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The stress levels used were percentages of the average modulus of rupture values determined at the same temperature at which the glass was to be tested for creep and stress-rupture. When there was a difference between the modulus of rupture values (at the elevated temperatures the specimens exposed to the testing temperature for long periods of time had considerably less strength than those exposed for short periods of time), the lower of the two modulus of rupture values was used to determine the stress levels. Stress levels of 90 percent and 75 percent of the average modulus of rupture were used at all temperatures while a stress level of 67 percent was used at 75°F for abraded specimens.

The normal duration of the test was 500 hours. Fracture occuring before this time terminated the test for that particular specimen. A creep curve that does not extend for 500 hours indicated that not enough specimens remained after the last period indicated on the graph to give meaningful data.

Unfortunately useful creep curves were not obtained at elevated temperatures. The accuracy of the apparatus at these temperatures was found insufficient to render curves truly representative of the creep activity. Failure of the apparatus and the termination of the project halted the work at elevated temperatures, leaving the gaps in the tables at the 75 percent and 67 percent stress levels.

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Creep

At elevated temperatures difficulties with the apparatus caused the data to be inadequate to obtain meaningful creep curves. Indications were that there was some slight creep for both glasses at both 400 and 500°F. At 200 and 300°F the creep curves were similar to those presented for 75°F.

At room temperature the creep data was obtained and the curves for both glasses are presented in Figure 9. The curves are similar in appearance to those obtained for the other glasses in the program and reported in WADC-TR-56-645, Part VIII, March 1963.

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TABLE 11

Individual Stress-Rupture Results for Abraded CGW Chemcor Specimens

Time to Failure in Seconds [1]

	90% Stress	75% Stress	67% Stress
500°F	17393 PSI [2] 378000 423000 902700 992700 1028700 1042200 1127700 1616400 1683900 *	14494 PSI 1296600 1341900 1470600 1673100 * * * * * *	
400°F	27106 PSI 435600 544500 1052400 1602900 1618200 * * * * *	22589 PSI 946800 * * * * * * * * *	
300°F	34088 PSI 0 0 0 0 0 0 5 777600 * *		
200°F	34367 PSI 0 0 0 0 0 0 5 40 460 1590	28639 PSI * * * * * * * * * *	
75°F	34375 PSI 0 0 0 0 0 0 0 90 95 2860	28646 PSI 20700 * * * * * * * * *	25590 PSI * * * * * * * * * *
75°F Unabraded 3]	48534 PSI 240 900 5400 21000 28800 70800 90000 171900 * *	40445 PSI * * * * * * * * * *	

1] Numbers indicate the length of time specimen survived. Asterisks indicate specimens survived 500 hours.
2] Stress in outer fiber calculated from the modulus of rupture equation.
3] Specimens unabraded.

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TABLE 12

Individual Stress-Rupture Results for Abraded PPG Herculite II Specimens

Time to Failure in Seconds 1]

	90%	75%	67%
500°F	15409 PSI 2] 211500 282600 295200 336000 548400 691200 811800 952200 1217700 1345200	12841 PSI 913500 1053900 1157400 1170000 1373400 1656000 1677600 * * *	
400°F	25608 PSI 662400 713700 906300 1087200 1286100 1405800 1492200 * * *	21340 PSI * * * * * * * * * *	
300°F	33058 PSI 0 0 0 0 0 0 0 0 15 45		
200°F	33400 PSI 0 0 0 0 0 0 0 0 900 *	27833 PSI 600 * * * * * * * * *	
75°F	32557 PSI 0 0 0 0 0 45 50 85 828000 *	27131 PSI 0 35 600 54900 1330200 * * * * *	24237 PSI * * * * * * * * * *
75°F Unabraded 3]	46955 PSI 190 330 420 660 213000 * * * * *	39129 PSI * * * * * * * * * *	

1] Numbers indicate the length of time the specimen survived. Asterisks indicate specimens survived 500 hours.
2] Stress in outer fiber calculated from the modulus of rupture equation.
3] Specimens unabraded.

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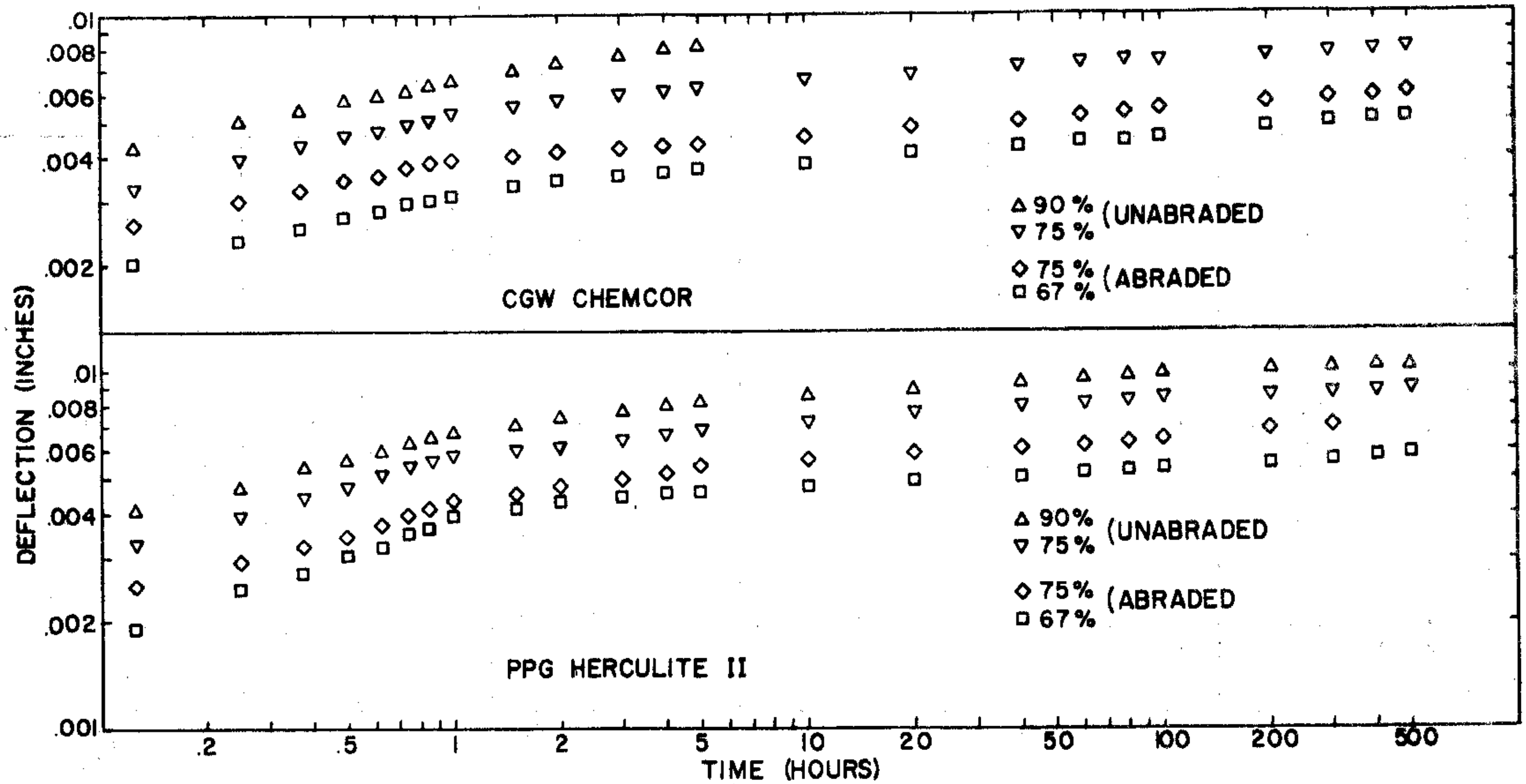

FIGURE 9 DEFLECTION-TIME CURVES FOR TWO CHEMICALLY STRENGTHENED GLASSES AT 75° F.

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