[Reprinted from the Journal of the American Ceramic Society, Vol. 18, No. 7. July, 1935.]

THE TIME FACTOR IN THE TESTING OF GLASSWARE*

By F. W. Preston

Abstract

A preliminary report on the effect of the time element on the apparent strength of glass is given. Tests have been run on bottle glass from three seconds to 30 minutes duration and on polished plate and drawn window glass (in the form of laths) from five seconds to several hours.

I. Introduction

When a piece of glass is twisted or stretched or distorted for any but the briefest time, it gets "cramps" in its distorted position. This was shown recently by Spencer, who wrapped the fibers of glass round a small glass cylinder, held them there for months, and then found they had a "permanent set." They did not become straight again on being cut loose from the cylinder. But the "set" was not so permanent after all; if left floating on mercury, they gradually straightened.[1] Thus it was not a plastic or viscous flow but an elastic phenomenon.

II. Elastic After-Working

This "elastic after-working" has been known for some time. Much work was done on it in Germany a generation or more ago in connection with the change of zero point in thermometers, where a "thermal after-working" is encountered.[2] The strictly elastic (as distinct from thermal) effect was investigated also by Phillips some thirty years ago.[3] Phillips does not give the composition of his glass or any references to the literature. He experimented with fibers of glass in direct tension, and measured the stretch (x) under various loads and after various lengths of time, as given in the following example.

Stress (779 kg./cm.²) Stretch (x) in arbitrary scale divisions	In Corning's units (7.79 kg./mm.²) Log (time)	In English units (11,000 lb./sq. in.) $(x - a)/\log t$
289.7	0.38	2.63
290.2	.50	3.0
290.6	.66	2.88
291.0	.86	2.68
291.3	.94	2.77
291.75	1.15	2.66
292.3	1.32	2.73
292.5	1.40	2.72
292.9	1.54	2.73
293.2	1.65	2.73
293.8	1.90	2.69
296.7	2.92	2.74

The last column is given to show the agreement of the experimental results with the empirical formula.

$$\text{Stretch } (x) = a + b \log t.$$

Where a and b = constants for the particular experiment.

For above load of 11,000 lb./sq. in. or 7.79 kg./mm.², $b = 1.55 \times 10^{-5}$

For load of 5.99 kg./mm.², $b = 1.16 \times 10^{-5}$

For load of 4.22 kg./mm.², $b = 0.78 \times 10^{-5}$

By plotting these values of b against the loads producing them, Phillips concludes that b would become zero at a load of 2.84 kg./mm.² or 4000 lb./sq. in. In other words, for stresses as low as this there would be no "creep" or gradual stretching. This point does not appear to have been investigated experimentally.

III. Nature of Creep

The nature of the observed creep is an interesting matter for speculation. When a sudden tension is applied to a glass fiber, the process is adiabatic. The temperature of the specimen falls slightly, so that a slight thermal contraction takes place and opposes the stretch due to the load. As the temperature equalizes, the fiber extends a slight additional amount, giving the isothermal stretch.

This, however, does not explain the observations, since temperature equilibrium is attained quickly, and the slow stretch persists for a long period.

For the same reason, any supposition (in its ordinary form) that the stretch produces an electric polarization or piezo-electric effect, which tends to oppose the stretch, must be rejected. Spencer[1] floated fibers on mercury, which would give surface electricity an excellent chance to attain prompt equilibrium.

For the present it seems convenient to regard it as a slow mechanical polarization of the glass; certain atoms or ions in the structure are forced into different positions or are rotated slightly into attitudes more convenient in the strained state. On releasing the stress, they slowly return to the

* Presented at the Annual Meeting, American Ceramic Society, Buffalo, N. Y., February, 1935 (Glass Division).

[1] C. D. Spencer, "Discussion of Flow of Glass Subjected to Strain." Presented at the Annual Meeting, American Ceramic Society, Cleveland, Ohio, February, 1931; not published.

[2] H. Hovestadt, Jena Glass, p. 304. English ed., 1902.

[3] Percy Phillips, *Phil. Mag.*, 225 [Ser. 6, Vol. 9] 520–22 (1905).

attitudes or positions convenient in the unstrained state. This mechanical polarizing and depolarizing is probably effected with the help of the thermal vibrations of the ions, which is present at all temperatures above absolute zero, and thus the rate of creep observed will probably be found to vary considerably with any large variation of temperature. It seems likely also that it could be accelerated in an alternating electric field.

Be that as it may, it is known that all glasses are not alike in exhibiting creep.

IV. Deformation Preceding Rupture

A little earlier than Phillips' experiments in England, Grenet's experiments were made in France.[4]

NOTE: Inasmuch as the English version in a recent issue of *Glass Industry* was curiously curtailed, the significant parts of the missing paper are given here.

"The writer has tried to determine whether the delay in fracturing in the cross-bending tests was accompanied by a permanent deformation or 'set,' increasing slowly from the time the load was applied up to the moment of rupture.

"For this purpose, a plate of No. 4 glass, 25 by 3 millimeters in section (1 by $^1/_8$ inch) with smoothed edges, was loaded in the same manner as in the breaking tests, applying the load at a rate of 1 kilogram in 2 minutes until a stress of 4 kg./mm.^{2} was developed.

"A 'test plate' of glass was placed on the specimen whose strength was under investigation, and the combination was illuminated with the yellow sodium flame.[5]

"With the aid of a magnifying glass, one of the interference fringes thus produced was watched attentively.

"While the specimen was being loaded, the deflection of the glass produced a displacement of the fringes, and as soon as the flow of water into the bucket was stopped and the load became constant, the fringes ceased to move.

"When a load of 6 kg. was reached, corresponding to a stress of 4 kg./mm.^{2}, no further load was added; thereupon the fringes ceased to move and at the end of 2 or 3 minutes fracture took place.

"While the last kilogram was being added, 70 fringes were observed to pass (black band to black band, extinction to extinction); no displacement of the fringes was observed from the moment when the writer ceased to add weight until the moment of fracture.

"As a movement of $^1/_4$ of a fringe would certainly have been detected, it is seen that if there had been a permanent deformation (or creep) it must have been of the order of magnitude which corresponds to a very small increment of elastic deformation; in fact, the permanent set must have been less than the deflection caused by adding $3^1/_2$ grams weight.[6]

"The increase of stress or strength which would result from this increased load under a more rapid rate of loading would be insignificant, *viz.*,

$$R = 4.002 \text{ kg./mm.}^2, \text{ in place of } 4.000,$$

and does not suffice to explain the differences of strength found with different rates of loading.

"In another similar test, the glass plate was loaded up to 3.8 kg./mm.^{2}, and 80 fringes were observed to pass during the loading of the last kilogram; no sensible displacement of the fringes from the time the loading stopped up to the time the glass broke, several minutes later, could be detected.

"This delay in fracturing is known to the champagne makers, who never refill their bottles a second time, in spite of their relatively high price; it is likely that the explosions of bottles of Seltzer water, taking place long after the bottles are placed in service, may be connected with this phenomenon.*"

Thus, while Phillips found creep, Grenet did not. Incidentally, Grenet gives as the composition of the glasses:

St. Gobain's No. 4 glass (%)		St. Gobain's "Cathedral" glass (%)	
SiO_2	71.75	SiO_2	70.98
$Al_2O_3 + Fe_2O_3$	1.50	$Al_2O_3 + Fe_2O_3$	1.56
CaO	14	CaO	14.94
Alkali and SO_3	12.75	Alkali and SO_3	12.52

Grenet's tests were made in cross-bending ("transverse tests"), and the load was carried to the breaking point, while Phillips' were not.

Spencer's glass, which showed creep or mechanical polarization, was presumably a lamp glass.

[4] *Bull. soc. encour. ind. nat.* [Ser. 5] **4**, 838 (1899); partial English version in *Glass Ind.*, **15** [11] 277–80 (1934).

[5] *Translator's note:* This "interference" test is unfamiliar to most glass manufacturers, but is well understood in physical laboratories, and is shop practice in optical glassworking plants.

[6] *Translator's note:* In the French version, this figure is derived by a formula, but as usual, the formula has a misprint, though the result is right.

* End of Grenet translation.

The composition was SiO_2 63%, K_2O 6%, Na_2O 9%, and PbO 22%.

Littleton,[7] experimenting at about the same time as Spencer, on strong rods of borosilicate, found no creep. Very high stresses and delicate optical tests were used with negative results.

G. A. McKee, experimenting for the present writer on the cross-bending of plate-glass laths, found virtually no measurable creep in tests lasting 4 to 15 hours. (See page 223 following.)

Borchard, in contemporaneous tests in Germany, seems to have found considerable creep.[8]

Fused silica is the most elastic (*i.e.*, creep-free) material available for delicate scientific work. Thus it is used in the suspension fibers of galvanometers. High silica glass comes next and is used for thermometers. Plate glass is also quite good and is used in all the big telescope mirrors in effective use to date, though apparently destined to be superseded by the low-expansion borosilicates which are equally free from creep. Some of the softer glasses are apparently not free from creep, and it is supposed that the mechanical polarization consists in squeezing the sodium ions, or similar loosely held atoms, out of their normal attitudes in the (more or less) octahedral spaces of the glass "lattice."

But most experimenters seem agreed that the total amount of creep observed is very small in comparison with the elastic part of the stretch. Thus, Phillips found an elastic stretch of 290 scale divisions and a creep of 7 divisions after a long period. Spencer wrapped the fibers (of about 0.1 mm. diameter) round a rod or tube 2 centimeters in diameter and held them there for about 1000 hours. On cutting them loose, they immediately opened up to coils some 60 centimeters in diameter, and this radius of curvature continued to increase with time, *i.e.*, the fiber continued to straighten. In the case of a few fibers still in existence after being cut loose five years ago, there is still evidence of curvature.[9]

V. Breaking Load under Continued Stress

Turning to the breaking load under continued stress, a totally different state of affairs is found. Glass will not stand a long-continued load of anything like the proportions it can stand for a few moments. It is not a difference of $1/2$ of 1%, as in Phillips' stretch, but a reduction of strength of 25 or 50% in a comparatively short time.

This fact too has been known for many years, but the available data are most unsatisfactory. Grenet gives information, but the variability of his specimens in size and manufacture do not permit very definite conclusions. The measurement of breaking strength is a vastly more difficult thing than the measure of stretch, however small the latter may be. The trouble arises largely from the fact that what is being measured is not really known. The mechanical strength is always a small fraction of what it ought to be; it is extremely variable from specimen to specimen, even when the specimens are prepared at one time from one particular batch and under the most controlled conditions possible; it varies also with the state of the surface of the specimen and enormously with the size of the specimen. As ordinarily tested, it varies greatly too with the rate of loading. On the other hand, it seems to have a nearly zero temperature coefficient, *i.e.*, for practical purposes, strength is independent of temperature to a greater extent than with most other materials.

The writer's staff has been engaged for a year or two past in experimenting on these subjects, more particularly in the decrease of strength with continual loading. The experiments are far from complete, and it seems desirable here to give only a few outlines.

The following strength values were obtained on a plate glass of high quality, polished on the faces, diamond-cut on the edges, the diamond mark being in the compression face in a transverse test of the specimen, which was roughly $1/4$ inch thick, 2 inches wide, 16 inches between supports, loaded centrally, and of the following chemical composition:

	(%)
Silicon dioxide	71.32
Sodium oxide	13.00
Calcium oxide	13.34
Magnesium oxide	0.23
Sodium sulfate	0.95
Sodium chloride	0.73
Arsenic pentoxide	0.17
Iron and aluminum oxides	0.20
	99.94

Duration of test	Average breaking (lb./in.²) strength of 20 specimens	
5 sec.	11,900	These specimens from a single sheet of polished plate
1 min.	8,600	
30 min.	6,000	
4 hr.	5,420	This from another plate

[7] J. T. Littleton, private communication.
[8] K. H. Borchard, "Elastic After-Effect in Glass"; *Sprechsaal*, 67 [20] 297 (1934); *Ceram. Abs.*, 13 [11] 290 (1934).
[9] Private communication, January 7, 1935.

Tests of a fine-grade Fourcault drawn window glass showed a rather similar course.

5 sec.	13,150 (av. of 25 specimens)	Glass 2 by 0.0125 in. on 4-in. centers, centrally loaded
1 min.	9,700 " " " "	
30 min.	7,950 " " " "	

The composition of this glass is more complex and will not be given until more extensive mechanical tests have been made. The surfaces are fire-polished and the edges diamond-cut with diamond mark on compression face.

The rate of decline of strength is similar for the two glasses above.

On the other hand, when tests were made of the endurance strength of bottles, the strength did not decline so fast with time. It fell off markedly, and the fact that it did not fall faster is something of a mystery. The bottle glass is higher in soda and lower in lime than the lath specimens, and it may be necessary to prepare laths of the bottle glass to see if the cause of the discrepancy is in the composition or in the method of testing.

The bottle tests were made on the precision testing machine designed by the author.[10] This machine imitates the conditions of service perhaps better than any other device, and the fact that the strength does fall off appreciably with time under these conditions warrants the attention of glass manufacturers to the inadequacy of "instantaneous tests."

The satisfactory determination of strength on the longer tests is a slow process, and the writer hopes soon to have a larger number of testing machines available so that a more complete investigation can be made.

The tests of long duration are particularly important, because it is far from clear whether the strength declines to some finite limit or whether it actually goes to zero on a very long test. What percentage of the strength (as ordinarily measured) is left at the end of a couple of years of testing should be determined.

Several experimenters have suggested that the strength declines to about one-half; Littleton, in conversation, has suggested one-third; the present writer is inclined to think the ultimate value may be zero. Borchard,[11] in the case of bottles, believes that if the specimen survives a 2-hour test it will last indefinitely.

VI. Real Instantaneous Strength

A point of less practical importance, but of considerable scientific interest, is, what is the real instantaneous strength? No actual test is instantaneous, for the inertia and elastic properties of the materials prevent that. Under impact tests, which is the nearest approach to instantaneity, a sharp increase of maximum stress is noticed as compared with tests lasting a few seconds.[12] The writer is, however, fairly well convinced that the true instantaneous strength is infinite, or that at least it equals the theoretical strength of the glass (perhaps a hundred times any of the ordinarily observed strengths). This conclusion is reached partly from the shape of the curve as shorter and shorter durations are used and partly from theoretical requirements. An ordinary crack must be propagated with a finite velocity, so that under an instantaneous test (lasting an infinitesimal time) no crack, such as we know it, could be propagated at all.

When the duration of the test becomes so short that there is no time for a crack to be propagated from one molecule to the next (about 10^{-8} of an inch), it is necessary for every thread or chain of molecules to break simultaneously if the specimen is to rupture as a whole. Not only that, but for the specimen to break as a whole, the points of rupture of the individual chains must be adjacent. This can only be attained in practice if every link of every chain breaks, a process which requires as much energy as one-third of the heat of vaporization and carries us to the theoretical strength of the glass.

With still shorter tests, it is doubtful if any meaning can be attached to the concept of strength.

The "set" acquired by the plate-glass laths on the 4-hour and 15-hour test was measured in each instance, except when the specimen broke. In all cases, it was very small and appeared to be about 1% of the elastic deformation. It was not sensibly different at 15 hours from its 4-hour value, and was measured immediately after taking off the load. Some "set," from $\frac{1}{2}$ to $1\frac{1}{2}$%,

[10] F. W. Preston, "Pressure Testing Machines," *Glass Ind.*, **15** [11] 252 (1934).

[11] K. H. Borchard, "Testing Bottles," *Sprechsaal*, **67** [38] 576–77 (1934).

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was always observed on loads near the breaking point, but the accuracy of the observations is not much greater than the apparent "sets." After careful examination, Mr. McKee came to the conclusion that the whole of the apparent set, amounting to a couple of ten-thousandths of an inch, was in the steel work of the apparatus and that no part of it that could be measured at all was in the glass.

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