

# Effects of Reheating on Strength of Glass Fibers

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The glass fibers of the composition,  $\text{SiO}_2$  67,  $\text{B}_2\text{O}_3$  12,  $\text{BaO}$  2,  $\text{K}_2\text{O}$  9,  $\text{Na}_2\text{O}$  9.5,  $\text{Al}_2\text{O}_3$  0.5 % (in weight) were reheated at the different temperatures between the room temperature and the softening temperature of the glass, and then were subjected to the etching with hydrofluoric acid. The tensile strengths were determined with the reheated fibers before and after the etching.

It was found that, although the tensile strength of fibers decreases on reheating, it regains its original value if its thin surface layer is removed by etching with hydrofluoric acid.

## INTRODUCTION

Extensive studies on many physical properties of glass fibers by W. H. Otto and F. W. Preston,<sup>1)</sup> and R. T. Brannan,<sup>2)</sup> gave many indications that glass fibers, being imposed the severe chilling during forming, must be in a special form which is so far from equilibrium with the room temperature configuration. Recently, in support of this assumption, W. H. Otto<sup>3)</sup> gave a firmer experimental evidence that the strength of fibers does not depend on the fiber diameter but is significantly affected by the temperature of the glass from which the fiber is formed. Otto found that higher glass forming temperatures result in increased strengths of fibers. The same experimental results were found concurrently by G. M. Bartenev and A. V. Bovkunenko.<sup>4)</sup> They also found that fibers formed from the glass of higher temperature have higher strength even after the surface layer of fibers have been removed by treating with hydrofluoric acid.

The present investigation was started with an aim to get a more clear picture on the internal structure of the chilled glass in such a form of fiber. The present paper deals with the effect of reheating on the strength of fibers. As has already been reported by many authors,<sup>5)</sup> the measured strength of fibers decreases on reheating. In the present investigation it was found that the fibers once reheated can regain their original value of strength if their thin surface is removed by etching with hydrofluoric acid. The unusual nature of the internal structure of fibers on annealing have been discussed.

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## EXPERIMENTAL

The glass fiber of the following composition was used in the present investigation :  $\text{SiO}_2$  67.7,  $\text{B}_2\text{O}_3$  12.0,  $\text{BaO}$  2.0,  $\text{K}_2\text{O}$  9.2,  $\text{Al}_2\text{O}_3$  0.5%. The glass was melted from materials of the highest chemical purity that were available. The melting was carried out in a platinum crucible at  $1500^\circ\text{C}$ . The glass thus prepared were transferred into a platinum container-melter of bushing having a single orifice of 4 mm in diameter. The size of the bushing was 3 cm in diameter and 3 cm in height. The bushing was heated electrically using silicon carbide elements (Fig. 1). The glass temperature was kept at  $1170^\circ\text{C}$  during the fiber forming process. The glass issuing from the orifice was attenuated into fibers of 12 and 22  $\mu$  (0.00047 and 0.00086 in.) in diameter by pulling, respectively, at speeds of 500 and 150 m. per min. The glass filaments were measured off approximately 30 mm. from portion of the fiber between the bushing and the winder for the elimination of surface damage.

The glass filaments thus prepared were treated in either one of the following two methods : (Method 1) reheating at different temperatures between the room temperature and the softening temperature of the glass, and (Method 2) reheating and etching with hydrofluoric acid. For reheating in Method 1, the filaments were placed on a platinum sheet and were put in an electric furnace with nichrome heating elements which had previously been

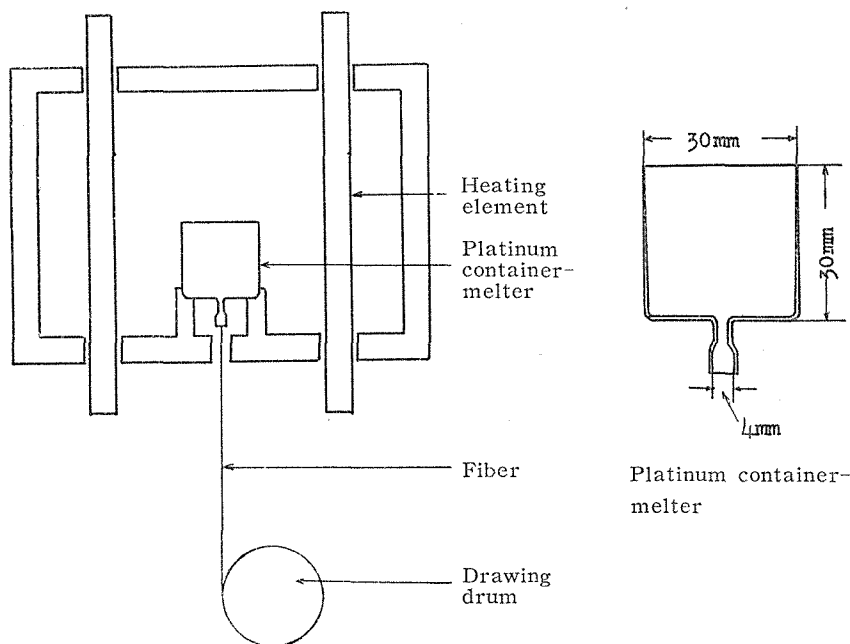


Fig. 1. Schematic representation of fiber drawing apparatus.

heated to the desired temperature. After kept for 1 hour, the filaments were taken out from the furnace and allowed to cool to the room temperature. The method of reheating in Method 2 was the same as that in Method 1. Surface etching after reheating was carried out using 10 % hydrofluoric acid. The time of immersion was 3 min., since the preliminary experiments indicated that further immersion over 3 min. caused no change for the filaments in the value of strength to be measured. After etching, the filaments were cleaned with distilled water and were dried at 70°C. for 10 min. The filaments thus treated were subjected to the tensile strength measurement. A tensile strength apparatus of the Mackenzie type, which is frequently used for the measurement of strengths of organic fibers, was used for this purpose. Both ends of the glass filament were held between two brass chucks having a span of 20 mm and were stucked on their surfaces with picein. The load was applied with a speed of 2 gr. per sec.

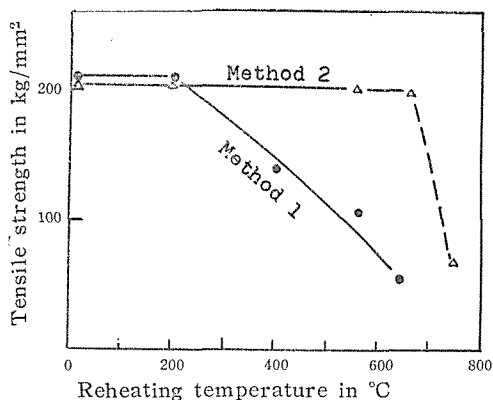


Fig. 2. Relation between tensile strength and reheating temperature of the fiber 12  $\mu$  in diameter.

●, before etching ;  $\Delta$ , after etching.

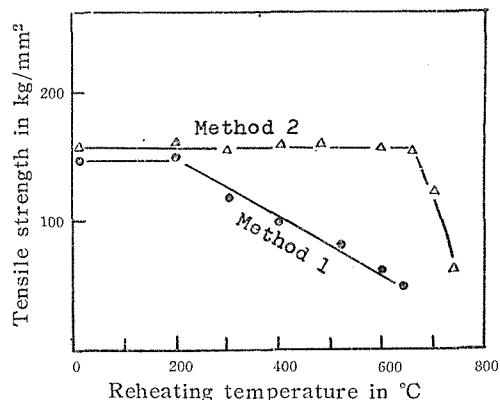


Fig. 3. Relation between tensile strength and reheating temperature of the fiber 22  $\mu$  in diameter.

●, before etching ;  $\Delta$ , after etching.

## RESULTS AND DISCUSSION

The results are shown in Figs. 2 and 3. Their ordinates are the tensile strengths of the filaments and their abscissa, the temperature at which the filaments were reheated. The curves denoted by Method 2, show data obtained on the filaments which were treated, respectively, with the methods 1 and 2 described above. A point corresponding to the room temperature denoted by solid circle is the datum for the filament which had no treatment and a point corresponding to the room temperature denoted by triangle is that for the filament which was subjected only to the etching by hydrofluoric acid. As these points may be regarded as the data for the filaments reheated at the

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room temperature in the methods 1 and 2, they were put, respectively, together with other data on the curves, Method 1 and Method 2. Each point is the average of five tests. It is seen that the strengths of filaments decrease on reheating (Method 1) while they stay constant on reheating and etching (Method 2) except for the data on the filaments reheated over 660°C. Microscopic examinations of the appearance of the filaments showed that the surface of the filaments reheated over 660°C. was corroded irregularly by hydrofluoric acid while that of the filaments reheated below 660°C was kept smooth even after the etching. The irregular corrosion would be probably due to the inhomogeneous structure at the glass surface caused by the heat treatment. With the exception of the data obtained on the filaments reheated over 660°C, a combination of the two curves of Method 1 and Method 2, would lead to the conclusion that, although the strength of the fibers decreases on reheating, it can regain its original value if the thin surface layer of the fibers are removed by etching with hydrofluoric acid. If the strength of a fiber is assumed to consist of the strengths of the surface and the inner part of the fiber, i. e., of the surface strength and the inner strength, the above conclusion can also be expressed in such a way that the reheating does affect the surface strength but not the inner strength. Generally, properties of chilled glass change on reheating at least near its softening temperature. Actually, the measurements on the density of a lump of the glass having the same composition as that of the fiber used in the present investigation revealed that its density changes from 0.002 to 0.02 g/cm on reheating at transition temperatures. Consequently, it would be reasonable to assume that in the inner part of the fiber, its properties such as density should also change on reheating. It should be emphasized, however, that this rule can not apply to the strength of the inner part of the fiber: the inner strength of the fiber does not change on reheating even near the softening temperature. It might be interesting to speculate that the part of the structure which governs the nature of strengths is somewhat different from that which governs ordinary properties such as density, and that this part of structure, in the inner part of the fiber, can preserve its characteristics even after reheating near the softening temperature of the glass.

### SUMMARY

The effects of the reheating and the subsequent etching with hydrofluoric acid on the strength of glass fibers were investigated. It was found that, although the tensile strength of fibers decreases on reheating, it regains its original value if its thin surface layer is removed by etching.

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