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NOTES

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Improvement of a beam-bending viscosimeter for fast measurement

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The beam-bending method is very useful for the viscosity determination of glasses, but the time spent to run the measurement is usually very long. The present note describes the performance of an improved beam-bending viscosimeter at the support stand and loading rod to get the faster measurements of high viscosity glasses with better reliability and precision. The testing time was reduced from 8 to less than 4 hours to run one measurement at three different temperatures (1100, 1200, and 1300 °C), with the precision of almost one order of magnitude better (± 0.01 for 1g η in poise).

The viscosity measurement of glasses by means of a beam-bending method is very useful for viscosity values between 10^8 to 10^{15} P.[1]

The statement of this method lies on the relation between the bending velocity (rate of midpoint viscous bending of a simply loaded glass beam) and the viscosity. A ceramic support stand and a ceramic loading rod are provided for supporting the specimen and applying the load to the specimen. The materials of the stand and the loading rod must have similar thermal expansion rates in order to minimize the relative motion between them.[2]

Usually, the beam-bending viscosimeter has a problem of excessive time required for recording one measurement. The main limitations are imposed by the low heating rate (about 15 °C/min) required by the ceramic support stand to prevent failure since one part of it is placed inside the furnace chamber and another part is placed outside, thus causing a large gradient of temperature along the stand. It also requires a long time to stabilize the thermal expansion equilibrium between the support stand and loading rod. For example, to measure viscosity at three different temperatures, 1100, 1200, and 1300 °C, the time required with this type of viscosimeter is about 10 h.

The beam-bending viscosimeter used is a Rheotronic model[3] made by Tokyo Industries, Inc. as represented in Fig. 1. The displacement of the sample midpoint viscous bending and the temperature are recorded by an x-t plotter. The bending velocity of the order of 10^{-6} cm/min can be determined with the temperature variation of the furnace smaller than 1 °C at 1200 °C.

Samples for viscosity measurements were cut with a diamond saw and polished with abrasive white alumina to have a rectangular cross section with final dimensions of approximately $3\times5\times50$ mm^3. Special care was taken to have the uniformity and parallelism of the faces, with dimension differences smaller than 0.01 mm along the sample.

The viscosity deviation due to the specimen dimensions was estimated as $\Delta \lg \eta = \pm 0.005$ (η in poise) in these experiments. Another error for viscosity determination originated by the imprecision of the effective load weight Δw, that is affected by the friction of the displacement transducer ($\Delta w = \pm 1$g). This effect contributes to the viscosity deviation by Δ 1g $\eta = \pm 0.02$. Using the original ceramic support stand and loading rod, the difference of the thermal expansion was responsible for a false midpoint deflection rate of the order of 10^{-4} cm/min, even after 30 min at a constant temperature of 1100 °C. The influence of this false deflection on the viscosity values can reach a deviation of as much as ± 0.2 in logarithmic scale. As can be observed here, the major error to determine the viscosity is caused by the uncertainties in the

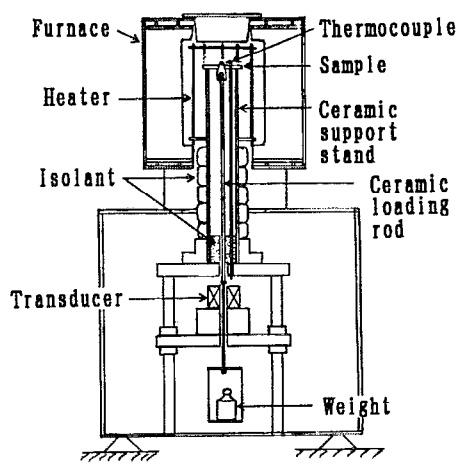

FIG. 1. Schematic representation of the beam-bending viscosimeter.

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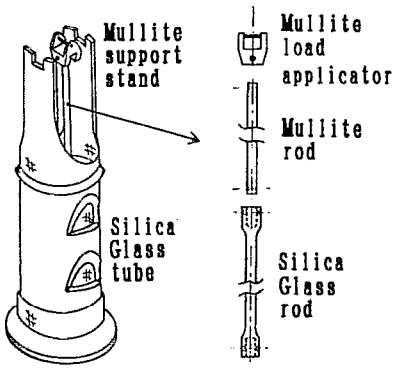

FIG. 2. The new support stand and loading rod composed by mullite and translucent silica glass (tube and rod).

determination of the midpoint deflection rate of the specimen.

To reduce the test time and improve the precision of the viscosity measurements, a new support stand and loading rod was designed, dividing them in two parts: (1) the upper part that is placed inside the furnace, composed of a tube and rod of mullite; and (2) the lower part, placed outside the furnace, composed of a tube and a rod of translucent silica glass. The purpose is to reduce the temperature gradient along the ceramic stand in order to increase the heating rate and consequently to decrease the thermal expansion difference between the stand and loading rod. The schematic representation of this new support stand and loading rod is shown in Fig. 2, where the load applicator is made by using mullite.

With these improvements, the thermal expansion difference between the support stand and loading rod was smaller than 1×10^{-2} cm from room temperature to 1100 °C. The expansion matching of the support stand and loading rod was tested by using an alumina beam (5 mm in diameter) as a sample at a real test condition. Using a heating rate of 30 °C/min, a false bending rate as high as 8×10^{-6} cm/min was observed 10 min after reaching 1100 °C, just after loading by 224 g of weight. This false bending rate is caused mainly by the elastic elongation of the loading rod which was estimated to be 7×10^{-6} cm/min for the above condition. At 1200 and 1300 °C, with a lighter load, the false bending rates observed were smaller than about 5×10^{-6} cm/min.

TABLE I. Viscosity values of the standard HLX silica glass using the new support stand and the original one. (Obs: Samples HLX-1,2 were measured with the improved viscosimeter, and samples HLX-3,4 were measured with the original one.)

Sample	$\lg \eta$ (P) (1100 °C)	$\lg \eta$ (1200 °C)	$\lg \eta$ (1300 °C)
HLX-1	14.03	12.81	11.62
HLX-2	14.07	12.83	11.61
Average	14.05±0.02	12.82±0.01	11.61±0.01
HLX-3	14.05	12.88	11.72
HLX-4	14.28	12.99	11.81
Average	14.16±0.12	12.94±0.06	11.76±0.05

The performance of the improved viscosimeter was tested by using various samples of flame-fused silica glasses from high purity sol-gel,[4] and a commercial HLX silica glass (by Shin-Etsu Co.). The viscosities were measured at the temperatures of 1100, 1200, and 1300 °C, loaded by 215.0, 65.0, and 15.0 g, respectively. Table I shows a comparison measurement for the HLX glass taken before and after the viscosimeter improvement. We can observe that the precision is one order of magnitude better by using the new support stand and loading rod. One advantage of the reduced measurement time is the possibility to minimize the influence of structure changes due to the fictive temperature of glasses.

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[1] H. E. Hagy, J. Am. Ceramic Soc. **46**, 93 (1963).
[2] Standard Test Method for Annealing Point and Strain Point of Glass by Beam Bending, ASTM Designation: C 598-72 (Reapproved 1978).
[3] Tokyo Industries Inc., Instruction Manual of the Rheotronic Viscosimeter.
[4] D. Torikai, C. K. Suzuki, H. Shimizu, T. Ishizuka, J. Yagi, K. Orii, and T. Miyakawa, J. Non-Cryst Solids (to be published).