Improvement of a beam-bending viscosimeter for fast measurement

Delson Torikai and Carlos Kenichi Suzuki
University of Campinas, Faculty of Mechanical Engineering, UNICAMP-FEM-DEMA, Campinas
13084-100, CP6122, SP, Brazil

Hiroshi Shimizu and Toshio Ishizuka
National Industrial Research Institute of Nagoya-NIRIN, 1-1 Hirate-cho, Kita-ku, Nagoya 462, Japan
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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 1 gη in
poise).

The viscosity measurement of glasses by means of a beam-bending method is very useful for viscosity values be-
tween 10⁸ to 10¹⁵ P.¹

The statement of this method lies on the relation be-
 tween 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 spec-
imen. 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 caus-
ing a large gradient of temperature along the stand. It also
requires a long time to stabilize the thermal expansion equi-
librium between the support stand and loading rod. For ex-
ample, 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
model² 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 bend-
ing velocity of the order of 10⁻⁸ 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 dia-
mond saw and polished with abrasive white alumina to have
a rectangular cross section with final dimensions of approxi-
mately 3×5×50 mm³. Special care was taken to have the

uniformity and parallelism of the faces, with dimension dif-
fferences smaller than 0.01 mm along the sample.

The viscosity deviation due to the specimen dimensions
was estimated as Δlgη = ±0.005 (η in poise) in these ex-
periments. Another error for viscosity determination origin-
gated by the imprecision of the effective load weight Δw,
that is affected by the friction of the displacement transducer
(Δw = ±1 g). This effect contributes to the viscosity deviation
by Δlgη = ±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.
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 $1 \times 10^{-2}$ 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 $8 \times 10^{-6}$ 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 $7 \times 10^{-6}$ cm/min for the above condition. At 1200 and 1300 °C, with a lighter load, the false bending rates observed were smaller than about $5 \times 10^{-6}$ cm/min.

The performance of the improved viscosimeter was tested by using various samples of flame-fused silica glasses from high purity sol-gel, 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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<table>
<thead>
<tr>
<th>Sample</th>
<th>$\log \eta$ (P)</th>
<th>$\log \eta$ (1100 °C)</th>
<th>$\log \eta$ (1200 °C)</th>
<th>$\log \eta$ (1300 °C)</th>
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<tr>
<td>HLX-1</td>
<td>14.03</td>
<td>12.81</td>
<td>11.62</td>
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<td>HLX-2</td>
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<tr>
<td>HLX-3</td>
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<td>Average</td>
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<td>12.94±0.06</td>
<td></td>
<td>11.76±0.05</td>
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