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Molten glass viscosity measurement with open-type EMS system

オープン型 EMS システムによる溶融ガラス粘度測定

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1. Introduction

The investigation of the glass transition phenomena using the ultrasonic spectroscopy has been widely promoted from the academic and industrial interests. As for the observation of the mechanical relaxation accompanying the glass transition, the light scattering method has been an effective tool, since it can easily applied for the high temperature samples because of its feature of non-contact measurement. In the Brillouin scattering measurement, the wavelength of phonons to be observed is in the order of the optical wavelength, which gives the higher frequency limit of the complex elastic modulus. In order to obtain whole information of the relaxation, we should also determine the low frequency limit of these mechanical properties.

The corresponding technique is the rheology measurement, which provides us with the knowledge of slow dynamics of glasses; the complex elastic modulus is determined as a function of the frequency. However, much experience and skills are required to design and fabricate a sample container for the rheology measurement of high temperatures. In the conventional method of viscosity measurement of molten organic glasses, a high temperature environment above 1000 °C is required and the all the measurement apparatus should be contained in the electric furnace; the time required for the temperature increase and the cooling exceeds several hours, which restrict the rapidity in obtaining the rheological data of the glasses. In addition, the probe in contact with the sample should be made of chemically and thermally tough materials and the candidate is restricted only to platinum and its alloys at present. Further, the size of the sample cell should be commonly be in cm order, and the cleaning after the experiments requires the chemical process with hydrofluoric acid and the expense and the cost for the environment are serious problem.

To settle these experimental difficulties, we improved the electromagnetically spinning (EMS) viscometer, which can measure the viscosity of the fluid sample set in a confined space in a non-contact manner.[1-3)] In our presentation, we would introduce an advanced form of the EMS viscometer especially designed for the viscosity measurement at high temperatures. The new apparatus allows half an infinite space above the viscometer to be free for the sample area, and the concept is represented as the trade name of 'open-type' EMS.

2. EMS measurement of inorganic molten glasses

Figure 1 shows the schematic view of the open-type EMS viscometer. A pair of magnets is set under the table top of the apparatus, which generates the magnetic field in the horizontal direction at the position of the metal probe in the figure. Rotation of magnets supported by a motor generates the rotating magnetic field, which induces an eddy current in a metal probe set in a sample. The Lorentz interaction between the magnetic field and the eddy current drives the probe to rotate following the motion of the magnetic field.

We can roughly estimate the magnitude of the torque applied to the probe with spatial size L and the electric conductivity σ to $T_B \sim \sigma B_0^2 L^5 \Omega$, where B_0 and Ω are the magnitude and the angular velocity of the magnetic field, respectively. On the other hand, the torque required for a probe to rotate with an angular frequency ω in the sample fluid with the viscosity η is roughly given by, $T_\eta \sim \omega\eta L^3$. By substituting typical experimental values of B_0=0.1 T, Ω=100 rad/s, σ=4x10^7 Ω^{-1}m^{-1}, and assuming the resolution of angular velocity of the probe to be 10^{-4} rad/s, the higher limit of the viscosity of the measurement is estimated to about 10^2 Pa·s.

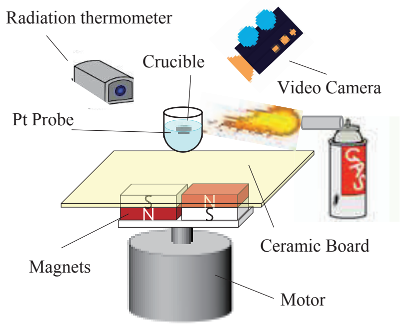

Fig.1 Schematic view of the Open EMS system for high temperature measurement.

Most remarkable feature of the system is that the specimen is apart from the measurement apparatus and can be isolated to each other. For the application to the high temperature measurement, an important specification of the instrument is the distance between the top plate and the expected position of the metal probe. In the above experiment, the probe is designed to set at the height of h=20 mm above the surface of the magnets to keep an appropriate distance for the heat insulation and the optical measurement of the probe rotation. In the actual experimental set up, two magnets of 30 x 60 mm^2 in face and 20 mm in thickness is employed.

We actually examined the thermal isolation between the space including magnets and that above the ceramic board and the temperature higher than 1300 °C can be safely achieved without the damage of the measurement apparatus. For the simple method of sample heating, we employed the direct radiation of the frame from a gas burner. The temperature is measured by a remote radiation thermometer.

Another important point is the selection of the metal probe; highly chemical toughness as well as high melting temperature is required. Therefore, a probe is especially designed for the experiment; a circular platinum plate with 0.5 mm thickness is molded into the shape of half sphere shell, which floats on the molten glasses. We chose B_2O_3 glass as the sample since pure material is commercially available and the established values of the viscosity is known.[4] The sample was purchased from Nacalai Tesque, Inc. and used without further purification.

3. Measurement of inorganic glass viscosity

Using the system described above, we carried out the measurement of the viscosity of the melt of an inorganic glass. The sample is set in a melting pot made of aluminum oxide and is directly heated by exposing it to a flame from a blowtorch. The temperature was measured by a radiation thermometer. The heat insulator is a ceramic board with the thickness of 2 mm, whose thermal conductivity is 31.4 W/(m･K).

Figure 2 shows the temperature dependence of the obtained viscosity of B_2O_3 glass (a) and its Arrhenius plot (b). According to the previous studies, the viscosity is known to rapidly increase towards the temperature of 318 °C, while the temperature dependence is well described by an Arrhenius law in the range of 1000-1500 °C. The dashed lines in the figures show the result of these previous results and the present experiment gives agreeable values of the viscosity. In Fig.2 (b), we can examine the obtained results are well fitted by a straight line showing the Arrhenius law.

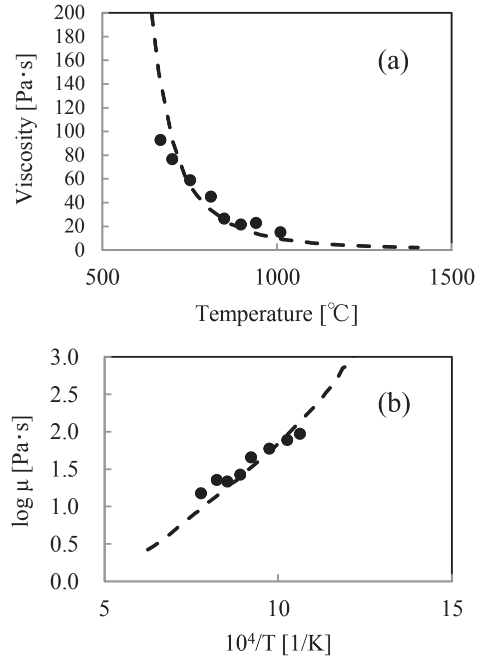

Fig.2 Temperature dependence of viscosity of the molten B_2O_3 glass (a) and its Arrhenius plot (b).

Note here, that the time required for the present experiment is only several tens of minutes and is much shorter than that of the conventional method. In this experiment, the angular resolution of the rotation of the probe is limited by the direct image analysis of the picture showing the motion of the probe. Instead by employing the observation of the laser speckle pattern, the resolution can be improved to better than 10^{-6} rad/s. The corresponding higher limit of the viscosity is 10^4 Pa·s and we can expect more rapid measurement within a few minutes.

In conclusion, we succeeded in measuring the rotation of the probe due to the application of the remote torque by EMS system even in very high temperature. The temperature dependence of the viscosity obtained with much rapidity is in good agreement with the literature value. We are now applying the laser light scattering measurement of the probe rotation, which would lead to further high time resolution of glass viscosity measurements.

Acknowledgment

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