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Rheology measurement under extraordinary environment with open-type EMS viscometer
オープン型 EMS による特殊環境レオロジー計測

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1. Introduction

In the study of ultrasonic spectroscopy, it is important to determine in advance the limited value of the mechanical relaxation at zero frequency. For example, the accurate measurement of the shear viscosity at the zero-shear limit gives information on the internal degrees of freedom, which can couple to the shear deformation of the material due to the ultrasonic propagation. The conventional methods of rheology measurements have been, however, not perfect for the measurement in the extraordinary conditions, such as the high pressure and high- and low-temperatures, since the mechanical probe connected to a precise torque motor should be in contact with the sample under the test.

To settle the problem, we developed the electromagnetically spinning sphere (EMS) viscometer, which measures 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 under the extraordinary conditions; half an infinite space above the viscometer is allowed for the preparation of the sample condition with the 'open-type' configuration.

2. EMS system for extraordinary sample conditions

As is known, much knowledge and the skill are required to design and fabricate a sample container for the special purpose of high temperatures and high- and low-viscosities, for example. In addition, it would be more troublesome to set it in the conventional type rheometers.

In our new system, we confined the mechanism to generate the magnetic field to apply torque to the probe below the top plate of the EMS viscometer. The system is referred to as the 'open type' EMS, representing the openness to half an infinite space above the apparatus to realize various sample conditions.

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 into 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 generates 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 estimate the magnitude of the torque applied to the probe with spatial size R and the electric conductivity σ to

$$T_B \sim \sigma B_0^2 R^5 \Omega, \qquad (1)$$

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

$$T_\eta \sim \omega \eta R^3. \qquad (2)$$

Here, we assume that a probe is set in an infinite surrounding media. In the actual experiment, the probe is a disk floating on the sample fluid, however, the magnitude of the viscous torque is correct in its order. We can determine the absolute value of the viscosity by equalizing these two torques. For more accurate measurement of the viscosity, we employ the viscosity standard liquids to determine the magnitude of the torque applied by the magnetic field.

In order to deliberately demonstrate a wide applicability of the developed system, we carried out the rheology measurement at high temperatures where inorganic glasses are expected to melt. In addition, a blow torch is employed to heat up the sample; we would demonstrate here that almost direct exposure of the top plate of the system to the high temperature flame brings any instrumental troubles.

For the application of the extraordinary environments, an important specification of the instrument is the distance between the top plate and

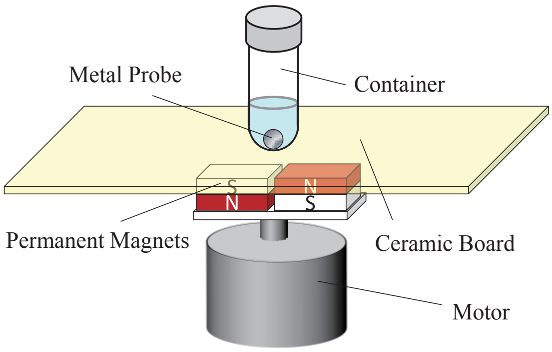

Fig.1 Schematic view of the Open EMS system

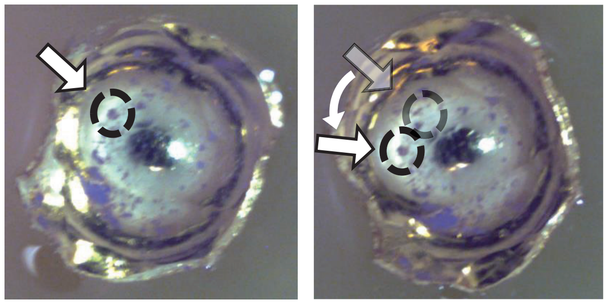

Fig.2 Characteristic mark moves as the disk rotates after an interval of 20 min.

the expected position of the metal probe. In the above experiment to keep an appropriate distance for the heat insulation and the optical measurement of the probe rotation, the probe is designed to set at the height h=20 mm above the surface of the magnets.

In the geometry shown in Fig.1, the magnetic potential ϕ generated by a pair of magnets with spatial size L at the height z is roughly given in the form of $\phi \simeq [z^2+L^2]^{-1/2}$ and, therefore, we can know that the spatial size of the magnet $L=h$ would be enough and appropriate to generate the magnetic field at the probe position. In the actual experimental set up, two magnets of 30 x 60 mm^2 in face and 20 mm in thickness is employed. The magnitude of the magnetic field at the probe position is B_0=100 mT. The torque applicable to the probe is in the order of 10^{-9} Nm.

Another important point is the selection of the metal probe; highly chemical toughness as well as high melting point are required. For the purpose, a gold and platinum disks with diameter of 1 mm and thickness of 0.5 mm are prepared.

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 a kind of lead glass with relatively low melting temperature. 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. We measured the temperature by a radiation thermometer, however the accuracy is not satisfactorily good and we can only know that the temperature exceeds 1000 °C at maximum. The heat insulator is a ceramic board with the thickness of 2 mm and the thermal conductivity of 31.4 W/(m・K).

Figure 2 shows the picture of a gold probe floating on the lead glass melt and we can see a characteristic mark on the disk, indicated by arrows, rotates during the induction of the torque. The mark traveled by approximately 36 ° in the anticlockwise direction for 20 minutes, suffering from high viscosity of the lead glass melt. The angular velocity is measured to be $\omega \simeq 10^{-4}$ rad/s, and the viscosity estimated from the rotational speed of the disk is in the order of 10^4 Pa·s.

We also carried out the viscosity measurement using a platinum probe, which has higher melting temperature than gold. It gave almost the same value of the viscosity, however, the electric conductivity is lower than that of gold and the accuracy of the rotation measurement is not satisfactory. Further improvement, such as the increase in the magnetic field would be required for more accurate determination of the viscosity. It is also effective to apply the sensitive detection of the probe rotation by the laser interferometry, which is reported in our previous report.[3]

As shown, 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, which would show rapid increase towards the glass transition temperature is a good evidence, if measured, and we are now planning to observe these critical behaviors.

In conclusion we can show the possibility of applying the EMS system to high temperatures. Our co-worker has also succeeded in measuring the gas viscosity as the demonstration of the open-type EMS at ultra-low pressures and the results would soon be reported.

Acknowledgment

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