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Rapid measurement of ultra-high viscosity with EMS system
EMS システムによる超高粘性試料の迅速測定

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1. Introduction

We developed a new type of a viscometer using Electro-Magnetic Spinning (EMS) method, [1)] which is at last commercially available from this year. **Figure 1** shows the photograph of the viscometer referred to as EMS-1000, manufactured by Kyoto Electronics Manufacturing Co., Ltd. It applies remote torque to the metal sphere probe in the sample in a non-contact manner, therefore, it is free from the problem of the contamination by using disposable sample cell. The viscometer has other unique features;[2)] such that measurements can be carried out under hermetically sealed condition and it requires very small amount of sample volume. In addition, the viscometer has a wide measurement range of 0.1～1,000,000mPa・s. In principle, it can measure much higher viscosity beyond the above commercially corroborated range. However, the measurement takes hours by the present detection system of the probe rotation. In this presentation, we introduce a rapid measurement method of ultra-high viscosity sample.

Fig. 1 Photograph of EMS system product.

2. Ultra-high viscosity measurement system

A typical example of high viscosity appears in the glass transition phenomenon, in which the characteristic viscosity varies in the range of 10^7~10^{14}Pa・s. Up to now, some conventional methods are developed, such as fiber elongation, beam bending and penetration methods. However for the observation of the glass transition at high temperatures, there have been some technical difficulties such that the apparatus with an electric furnace becomes huge. To overcome the problems we tried to apply the EMS methods for the measurement of high viscosities at high temperatures.

Figure 2 shows a schematic view of a developed detection system of the probe rotation. The light source is a semiconductor laser diode module driven at 635nm with the power of 10mW, (Global Laser Ltd.), and the detector is a smart line camera (LC100/M, Thorlabs Inc.). The motor generating the rotating magnetic field is a brushless DC motor (BMU230A-AC, Oriental Motor Co., Ltd.), and a probe sphere is made of aluminum and the diameter is 3/16 inches. Permanent magnets are a neodymium magnet.

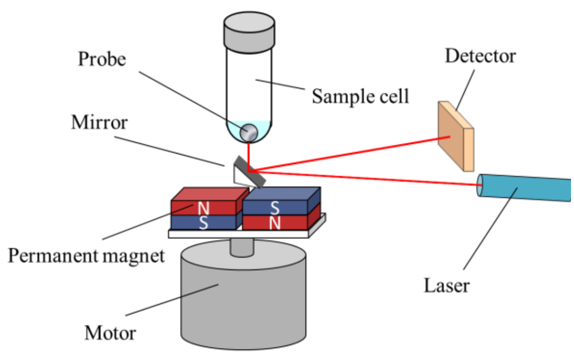

Fig. 2 Schematic view of a developed detection system

The principle of the detection of the probe rotation is as follows; the laser light is incident from the transparent bottom of the sample cell and illuminates the probe sphere. The surface of the sphere is smooth but has a slight roughness at the optical level, therefore, the reflected light shows the speckle pattern, which rotates with the sphere rotation. The speckle pattern is random and we detect the change in the intensity following its rotation at several observation points.

In the present system, the obtained signal shows the periodical change for a round and the auto-correlation function of the signal gives the period of the rotation. We have to wait for, at least a turn to detect the rotational speed. For the high viscosity measurement, the rotational speed of the probe sphere is expected to be extremely slow; it takes more than an hour if we measure the sample with the viscosity of 1,000Pa・s.

Instead in the present system, the several light detectors are placed close to the neighbor and aligned along the circular trace of the rotation of the speckle pattern. The distance between the center of the rotating speckle pattern and the detector is about 100 mm and the distance between the neighboring photo-detectors is 14 µm, therefore the resolution of the angle is better than 10^{-3} rad. It means that the time required for the measurement of the viscosity can be reduced by the factor of 1000.

To examine the rapidity of the measurement of high viscosity, we carried out the detection of the small rotating speed by the photo-detection system described above. The sample used is silicone oil often employed as the standard of the viscosity (KF-96, Shin-Etsu Chemical Co., Ltd.), whose viscosity is η = 1,000 Pa・s at 25 ℃. **Figure 3** shows the observed signal, obtained by five pixels arranged in distances of 70 µm. In addition, the sampling speed of the photo-detector is 5 ms, and the motor rotation speed is 100 rpm. As can be seen in Fig. 3, signals of the same pattern are observed with equal temporal delays. As a result of the analysis of the data, the time required for the speckle pattern to move between the neighboring photo-detector is calculated to 185 ms. For this sample, it takes about 17 min for a turn, while the time required by the developed method is in the order of 0.1 s; The increase in the time resolution is as high as 10^4.

Here, we estimate the magnitude of the error brought about by the thermal fluctuation. First, we consider the translational diffusion of a sphere by the Brownian motion. The translational diffusion constant is given by the following Einsten-Stokes equation as,

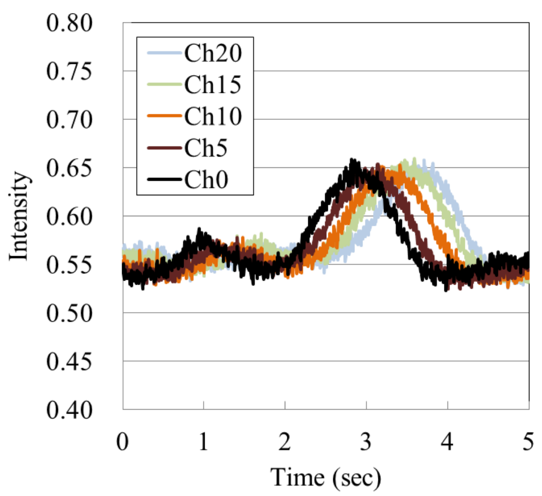

Fig. 3 Observed signal of the line sensor

$$D_T = \frac{kT}{6\pi\eta R}.$$

Here, k is the Boltzmann constant and T is the temperature. Actual physical quantities are substituted into the equation, and the translational diffusion constant in the system is calculated to D_T = 9×10^{-23} m^2/s. The fluctuation of the position of the sphere during 1 s is roughly estimated to $\ell = \sqrt{D_T\tau} \approx 10^{-11}$ m. On the other hand, the rotational diffusion constant is given by the Stokes-Einstein- Debye equation, which is

$$D_R = \frac{kT}{\eta V}.$$

Here, V is the volume of the probe sphere, and the value is calculated to $D_R = 7\times10^{-17}$ s^{-1}. The rotation due to the thermal fluctuation during 1 s is about $\delta\theta = \sqrt{D_R\tau} \approx 9 \times 10^{-9}$ rad, and the corresponding displacement at the equator is about 2×10^{-11} m. These values are quite small and the error due to the fluctuation can be safely ignored.

Next, we discuss the time constant required for the system to be stable after the application of the driving torque. The time constant τ_1, which represents the response time of the rotation of the probe sphere is about $\tau_1 = 10^{-6}$ s. On the other hand, the time τ_2 required to reach the steady state of shear flow in the medium is about $\tau_2 = 10^{-5}$ s. Therefore, the sampling interval of the system, which is 5 ms, is sufficiently large and we can safely observe the steady state of the flow.

3. Conclusion

The expected resolution of the angle detection is limited by the speckle structure, which is λ/R at the highest in the system, λ being the wavelength of the light. A simple extrapolation predicts that 1,000 Pa・s can be measured within in 10^{-2} s in principle. Another efforts, such as to increase the applied torque would also be effective for the high viscosity measurement. In the future, we aim to realize the rapidity to measure the viscosity of 10^{10} Pa· s within 10 minutes.

In conclusion, we can remarkably reduce the measurement time of ultra-high viscosity sample by improving the detection resolution of the rotation angle of the probe sphere. We are now trying to apply it for the real time observation of the glass transition.

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