

Development of viscosity measurement method in ultra-low shear rate

Mika Iga^{1†}, Satoshi Ishida¹, and Keiji Sakai² (¹Nippon paint corporate solutions Co., Ltd.; ²IIS, The Univ. of Tokyo;)

1. Introduction

In coating industry, shear viscosity has been contributed to determine proper fluid properties for each process from painting to film building. Thoroughly understanding of viscosity behavior during film building process, however, remains unclear because of the lack of the measurement method for viscosity in extremely lower shear rate region under $10^{-2} \text{ sec.}^{-1}$. In addition, solvent evaporation makes paints highly solid or viscous. Many previous experiments show that a various qualities of coatings depend on this process.

We focus on EMS (Electro Magnetically Spinning) system. It has been developed for accurate measurements of low viscosity fluids like water as its subjects¹⁾. From the viewpoint of the principal, EMS is available to our samples.

The application of a time-varying magnetic field by the electromagnet produces an induced current in the metal part of the rotor. The Lorentz interaction between the current and the magnetic field generates a non-contacting force on the rotor in the direction that follows the rotation of the magnetic field. As the magnetic field rotates, a torque acts on the rotor, but the viscous resistance of the sample causes a delay in the rotational speed of the rotor. The delay is proportional to the viscosity, and therefore, the viscosity of the sample can be determined from the known magnetic field rotation speed and the measured rotational speed of the rotor.

In the development of the disk-type EMS, floating the rotor on the fluid surface eliminates friction between rotor and sample petri dish and gives higher detection ability²⁾. Furthermore, highly accurate viscoelasticity measurement in the low-strain region with extremely small torque was yielded practical applications.

2. Experiments and Results

Figure 1 shows a schematic view of the first prototype of viscosity measurement system using disk-type EMS system.

A sample fluid is filled into a glass petri dish with an inner diameter of 26.57mm and a disk-shaped aluminum rotor with a diameter of 10mm is floated on the fluid surface. A pair of permanent magnets is placed under the sample stand.

A reference line is marked on the surface of the

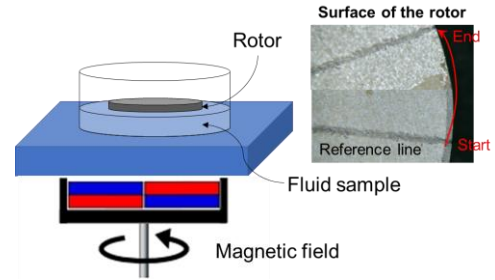


Fig. 1 Schematic view of the first prototype of viscosity measurement system using disk-type EMS system.

rotor and its motion is monitored with microscope.

Shear rate of the measurement is calculated from the time dependent motion of the reference line, together with the sample thickness determined by the distance between the rotor and the bottom of the petri dish.

The viscosity η of the sample can be determined from the known magnetic field rotation speed and the measured rotational speed of the rotor, which is given by

$$\eta \propto \frac{\Omega_M - \Omega_D}{\Omega_D} \quad (1)$$

where Ω_M is angular speed of magnetic field, and Ω_D is angular speed of rotor.

Experiments were carried out for newton fluids. Here, silicone oils with viscosities of 10^2 Pa s (Shinetsu silicone KF-96-1000cs), 10^3 Pa s (KF-96H-10,000cs), and 10^4 Pa s (KF-96H-100,000cs) are used as standard samples. Note that the typical viscosity of common water-borne paints for interior

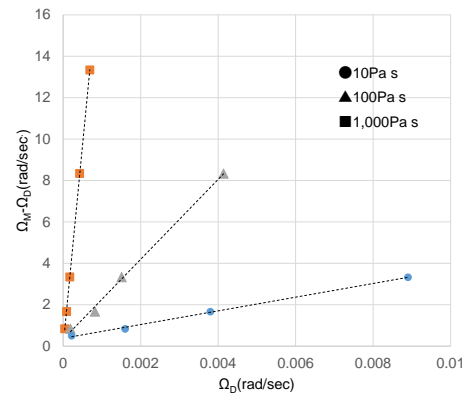


Fig. 2 The linear relation between angular frequency of rotator and differential angular rate (25°C).

is from 10^2 to 10^3 Pa s at shear rate of 10^{-2} sec. $^{-1}$.

Figure 2 shows the linear relation between differential angular rate and angular frequency of rotor at 25°C. It indicates that the viscosity η is constant in the selected angular frequency range. The lowest shear rate for silicone oil with 10^3 Pa s is calculated to be 4.8×10^{-4} sec. $^{-1}$

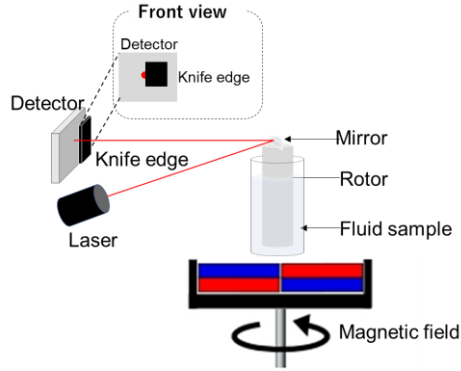


Fig. 3 Schematic view of viscosity measurement system improved from previous system.

In the next experiment, some improvements were made for the measurement system. **Figure 3** shows a schematic view of EMS system improved from previous system.

Cylindrical rotor floats by buoyancy in non-contact way in a sample tube along its center axis. The rotor has a larger contact area with sample than the disc type rotor so that it effectively gives torque to sample.

Glass sample tube with an external diameter of 14mm is filled with a fluid sample and an aluminum rotor with a diameter of 10mm and height of 70mm is set into the sample tube. The weight of the cylindrical rotor is adjusted to avoid immersing the rotor head in the sample.

The optical lever method is used to detect the rotation of the rotor. A mirror is placed on the rotor head to reflect the laser beam and the slight rotation of the rotor is captured as the shift of the reflected beam. A detector is placed 1.2m away from the mirror to amplify the motion of the mirror.

Furthermore, the knife-edge technique gives the higher resolution of detection³⁾. A knife edge is placed in front of the detector and intercepts laser beam. The rotation angle of the viscosity probe is detected as the change of the transmitted power of the laser beam. The intensity distribution of the laser has Gaussian profile and total intensity $P(a)$ is given by

$$P(a) = \int_{-\infty}^{\infty} dy \int_a^{\infty} dx I(x,y) = (Pt/2) \operatorname{erfc}(a\sqrt{2}/\omega_s) \quad (2)$$

where $I(x,y)$ is the intensity distribution, ω_s is the

beam width and a denotes the position of the laser beam at the knife edge. The integration in eq.(2) can be expressed by the error function of,

$$\operatorname{erfc}(a) = \frac{2}{\sqrt{\pi}} \int_a^{\infty} \exp(-t^2) dt \quad (3)$$

The angular speed of the rotor is calculated from the motion of the laser beam.

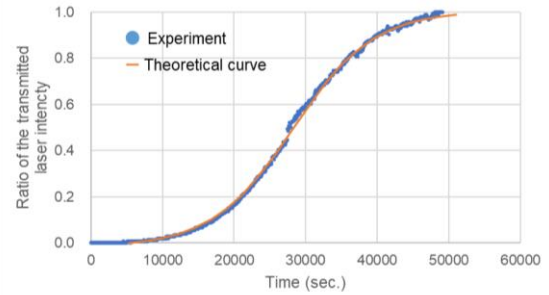


Fig. 4 Comparison between ratio of the transmitted light volume measured by detector and the error function with time (25°C).

Figure 4 shows the experimental result obtained for silicone oil with viscosities of 10^3 Pa s. It shows the comparison between the transmission ratio of the light measured by the detector and the error function (theoretical curve). In this figure, we see the laser intensity increased with moving rotor and the measured values well fit the theoretical curve. It indicates that the rotor moves at a constant speed. Shear rate $\dot{\gamma}$ is calculated using double cylindrical measurement method by,

$$\dot{\gamma} = \frac{2\omega R^2}{(R^2 - r^2)} \quad (4)$$

where ω is angular speed of rotor, R is external diameter, and r is inner diameter. The lowest shear rate was 5.6×10^{-6} sec. $^{-1}$ This value would be the lowest in the history of the rheology.

3. Conclusion

In this study, we improved viscosity measurement system using EMS and measured some samples' viscosity at much low shear rate region.

References

1. K. Sakai, T. Hirano, and M. Hosoda, *Appl. Phys. Express*, **3**, 016602 (2010).
2. Mitani S, Hirano M, Hirano T, Sakai K, *Annu Trans Nord Rheol Soc*, **25**, 329 (2017).
3. Shigeharu Kimura, Chusuke Nunakata, [Methods for Measuring the Spot Size of Focused Laser], *Japanese Journal of optics*.1990; **19(2)**, 74-79.