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Determination of Exact Gelation Point and Measurement of Tiny Elastic Modulus Using Disk-type EMS

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1. Introduction

In the spectoscopic study, it is commonly noted whether and how physical properties depend on frequency, in other words, time scale of observation. If a physical property of a sample shows a frequency dependence due to a relaxation behavior, useful information about degrees of freedom of motions in the sample can be obtained. The relaxation time and strength are especially on our interest, and give knowledge on the charactericatic time scale and magnitude of the degrees of freedom, respectively.

Ultrasonic measurement is a powerful tool for the spectroscopy, in which the measureable frequency region is from dozens of kHz to MHz, and the elastic modulus and shear viscosity are the typical physical properties to be obtained. For the accurate determination of these values, a reference value at the low-frequency limit is required, however, the value is rarely measured by itself.

Recently, a measurement system of the rheological properties has been developed using the electromagnetically spinning (EMS) technique[1]. In the newly developed system, a floating disk-shape plobe is used as a probe, called Disk-type EMS[2], and then, the mechanical friction that will be a large error factor in the conventional methods becomes absolutely zero. This extraordinary feature provides us with the accurate spectroscopic measurement in ultra-slow time scale of observation.

In this work, we obtained responce curves for creep tests under the different stress conditions with the Disk-type EMS, and measured the elastic modulus and shear viscosity of an aqueous solution containing worm-like micelles. In addition, we also challenged the simple determination of the exact gelation and solation point.

2. Disk-type EMS

In the EMS system, a temporally modulated magnetic field is applied to an aluminum probe, and the eddy currents are induced in the probe. The Lorentz interaction between the currents and magnetic field generates the driving torque to rotate the probe. For the sample in liquid state, the angular speed of the rotating probe is determined by the viscous resistance, and then the relation between the angular speed and the electromagnetically induced torque gives the viscosity of the sample. On the other hand for the sample in solid state, the angular displacement similarly gives the elastic property of the sample.

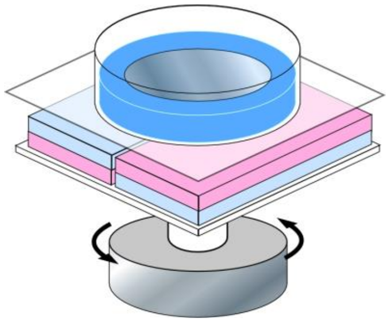

Fig. 1 Schematic image of Disk-type EMS.

A schematic image of the Disk-type EMS is shown in Fig. 1. A penetrating magnetic field in the perpendicular direction to the plane including the disk probe is the source of the driving torque. The magnitude of the driving torque depends on the rotational speed and the intensity of the magnetic field. The rotational speed is controlled by a brushless DC motor, to which a pair of magnets is attached, and the intensity is roughly estimated from the distance from the magnets to the probe.

The Disk-type EMS system has a great advantage of applying an arbitrary low torque in a noncontact manner. Therefore, this system would be a useful tool for the direct measurement of slow dynamics and weak interaction, which is peculiar to complex fluids.

3. Creep test for worm-like micellar solution

A creep test is an experimental method for observing the strain response to a constant stress applied to the sample. To analyze the response curve, we can measure the viscoelastic properties mainly for slow dynamics. In the creep test with the Disk-type EMS, the variation of rotational angle of the probe was observed under the torque induction of a step function.

The sample used in our experiments was an aqueous solution of cetyl trimethyl ammonium bromide (CTAB) and sodium chloride (NaSal). The CTAB is a detergent and forms warm-like micelles

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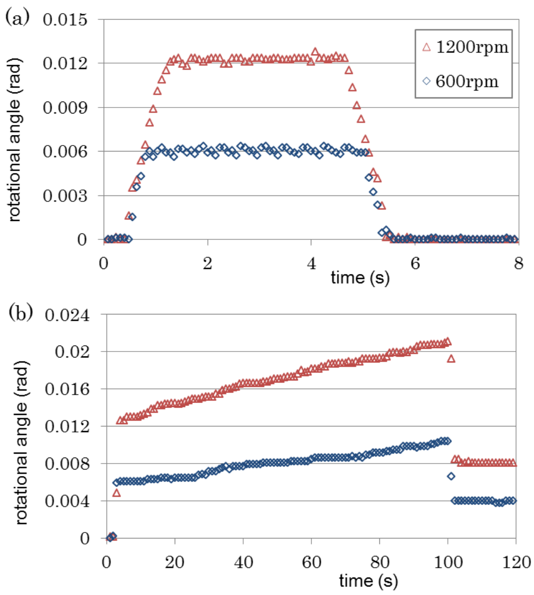

Fig. 2 Response curves to the creep tests in short(a) and long(b) duration for aqueous solution of CTAB/NaSal.

affected by the sufficient amount of counter ions[3], and the warm-like micelles show a viscoelastic behavior described by the Maxwell model[4].

Figure 2 show the time-series data of the rotational angle during short (a) and long (b) testing time for the CTAB/NaSal solution of 20mM/20mM at 25 degrees Celcius. In Fig. 2 (a), the rotational displacement returned almost zero, being the initial position, after the reduction of torque induction. This instantaneous response indicates that the sample is in the solid state in the short time scale. On the other hand, in Fig. 2 (b), a certain amount of displacement remains due to the long-term torque induction, which means the static state of the sample is liquid.

The instantaneous displacement and the remaining displacement correspond to the elastic modulus G and the shear viscosity η, respectively. As a result, G and η were determined to be 2.7 Pa and 400 Pa・s. The time scale of observation for the η value is the shear rate of the order of 10^{-4} s^{-1}, which is an unprecedented time scale and may be the low-frequency limit.

In addition, we conducted a similar creep test using the probe with larger radius. The time-series data only in short time scale for the same concentration and temperature is shown in Fig. 3. In this case, the instantaneous response to the step function of the torque shows an overshoot and an oscillating behavior. Now, we are ready to analyze the oscillating behavior, which can give another G and η in the time scale of its frequency.

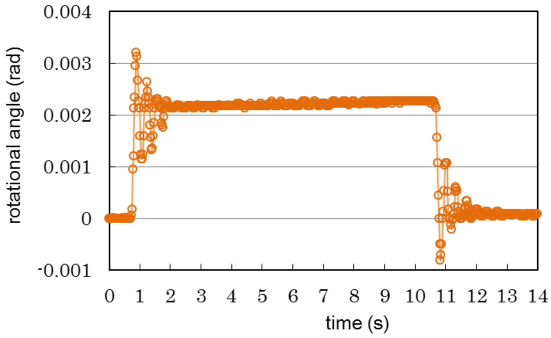

Fig. 3 Response curve to the similar creep test using the larger probe.

4. Determination of sol/gel transition point

As mentioned above, we can clearly distinguish whether the sample state is solid or liquid using the Disk-type EMS, and then accurately determine the exact sol/gel transition point, which were defined formally as the crossover point of complex moduli (G' and G''). We investigated the variation of the rotational speed of the probe during a gelation and solation process of an aqueous solution of methylcellulose.

The temperature dependence of the observed rotational speed is shown in Fig. 4. The moment of the rotation stopping and restarting were found in the heating and cooling process, and then the gelation and solation temperature were determined, respectively. Additionally, it was clarified this phase transition has a hysteresis.

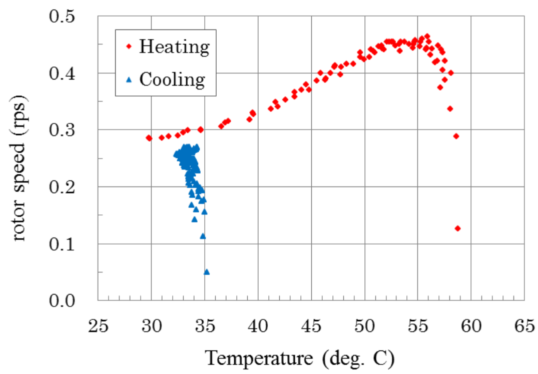

Fig. 4 Temperature dependence of the rotational speed of the probe for gelatin and solation process.

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