BL-15A2/2015G547

Orientation Control of Inverse Bicontinuous Cubic Phase by using Shear Alone: Effects of Strain Amplitude of "MAOS" and Replacement of LAOS by Steady Flow.

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1 Introduction

The inverse bicontinuous cubic phase (V_2 phase) is one of the lyotropic phases consisting of bilayer networks with a long-range order. Recently, we have performed simultaneous measurements of rheology/small-angle X-ray scattering (rheo-SAXS) for V_2 phase with a space group Ia3d formed in a nonionic surfactant ($C_{12}E_2$)/water system [1]. It has been found that the application of the large amplitude oscillatory shear (LAOS) with the strain amplitude (γ_0) of about 20 induces grain refinement, whereas sample orientation occurs by applying the "medium" amplitude oscillatory shear ("MAOS") whose strain amplitude is much smaller than the LAOS but still in the nonlinear regime just after the LAOS. In these studies, the frequency of the MAOS has been chosen to be much higher than of the LAOS. In the present study, the frequency of the MAOS has been set to the same value as the LAOS and studied relations between the strain amplitude of the MAOS and the sample orientation. We have also examined effects of replacing the LAOS by steady flow with various shear rates.

2 Experiment

Rheo-SAXS measurements were performed on the beamline 15A2 by using a stress-controlled rheometer AR550 (TA Instruments). The details of the shear cell have been reported previously. The scattered beam was recorded with the camera length of 2.6 m using the PILATUS3 2M. The approximate q range is from 0.1 to 2.4 nm^{-1}. The exposure time was 10 s.

3 Results and Discussion

Figure 1a shows time evolution of 2-D SAXS patterns in the tangential configuration with variation of the angular frequency (ω) and the strain amplitude (γ_0) of the oscillatory shear (see the table above the figure). In the steps ①, ③, ⑤, and ⑦, we applied the LAOS with the same ω and γ_0. In the steps ②, ④, ⑥, and ⑧, on the other hand, we applied the MAOS with the same value of ω as the LAOS except for the step ② whose conditions are the same as in the previous study (the steps ① and ② were added for comparison). For quantitative discussion, we use the degree of the orientation α defined by

$$\alpha \equiv \frac{\dfrac{1}{8\delta\phi}\displaystyle\sum_{n=0}^{3}\int_{(n\pi/3)-\delta\phi}^{(n\pi/3)+\delta\phi} I(\phi)\,\mathrm{d}\phi}{\dfrac{1}{\pi+2\delta\phi}\displaystyle\int_{-\delta\phi}^{\pi+\delta\phi} I(\phi)\,\mathrm{d}\phi} \quad (1)$$

where $I(\phi)$ is the peak intensity of the (211) reflection at the azimuthal angle ϕ (ϕ = 0 corresponds to the velocity gradient direction). The peaks for n = 4 and 5 were excluded because they are superimposed to the gap between the detectors. For the "ideal" powder sample, α becomes 1. When all the peaks are included within $(n\pi/3) \pm \delta\phi$, on the other hand, α should be 2.5 because we set $\delta\phi = \pi/18$. Time evolution of α is shown in Figure 1a. For the steps ①, ③, ⑤, and ⑦, α is close to 1 as expected. The results for the steps ②, ④, ⑥, and ⑧ indicate that the sample orientation can occur even if the frequencies of the LAOS and MAOS are the same and that the large difference in the strain amplitude between them should be important for the sample orientation.

Figure 1b shows effects of replacing the LAOS by the steady flow (the steps ①, ③, ⑤, and ⑦). The conditions of the MAOS are the same as in the previous study. It can be seen from the figure that the higher the shear rate of the steady flow just before the MAOS, the higher the orientation degree during the MAOS. These results suggest that the grain refinement processes in the steady flow strongly affects the sample orientation during the MAOS applied after the steady flow.

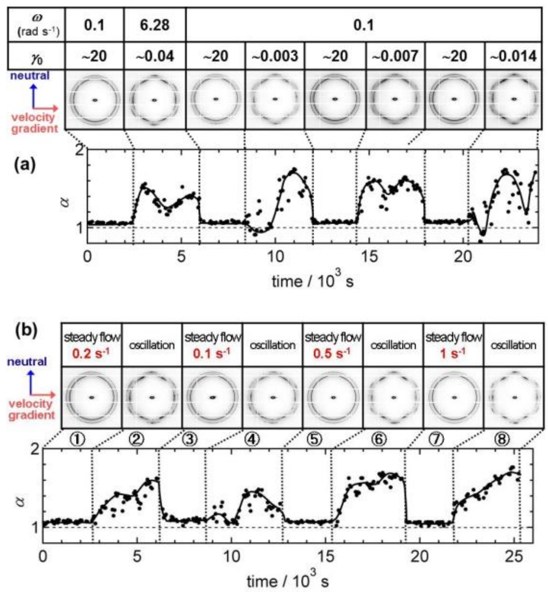

Fig. 1 Time evolution of 2-D SAXS patters in the tangential configuration and the degree of orientation obtained from the peak intensity of the (211) reflection using Eq. 1 with the change in the shear conditions shown in the tables where ω and γ_0 in the panel (a) are the angular frequency and the strain amplitude of the oscillatory shear, respectively. In the table of the panel (b), the shear rates of the steady flow are indicated.

References

[1] M. Yamanoi, Y. Kawabata, and T. Kato, *Langmuir* , **32**, 2863 (2016).

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