7th World Tribology Congress, WTC 2022
July 10-15, 2022, Lyon, France

Observation of Shear-Induced Orientation Change of Grease Thickener Structure Using Ultra-Small-Angle X-ray Scattering

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The insight of microscale shear-thinning mechanism of lubricating grease has a great importance for the development of high-reliability greases. In this study, shear-induced mechanical structural changes of thickeners can be observed by means of an ultra-small-angle X-ray scattering measurement. Examining the relation between microstructural behavior and apparent viscosity of greases based on the subtracted scattering patterns, microscale grease behavior such as alignment and collapse of the thickener structure came out.

Keywords: grease, thickener structure, ultra-small-angle X-ray scattering, Rheo-USAXS

1. Introduction

Grease is a semisolid substance mainly comprising a base oil and thickener, and functions as a lubricant owing to be subjected to shear forces. Although the lubricity obtaining process of greases based on the shear-thinning effect is supposed to be deeply involved in a thickener structure behavior: molecular micelles orientation and collapse of its structure [1], there hardly exists definitive observation method to explain microstructural dynamics of greases. The insight of microscale shear-thinning mechanism has a great importance for the development of high-reliability greases. In this study, shear-induced mechanical structural changes of thickeners can be observed by means of an ultra-small-angle X-ray scattering (USAXS) measurement.

2. Methods

In order to observe microscale grease behavior such as the alignment and collapse of the thickener structure under the shear flow, in situ rheological USAXS (Rheo-USAXS) in BL08B2, SPring-8 (Super Photon ring-8 GeV) was performed.

2.1. Measurement conditions
The X-ray energy and the sample-to-detector distance were set to 12.4 keV and 16 m respectively. The shearing stage was filled with an appropriate amount of a barium complex grease without air bubbles, and then the gap length between parallel shearing plates was controlled to be 0.15 or 1 mm. After filling with grease, the tester was left for at least 1 minute to remove the initial thickener structure orientation. From static condition, shear rate was varied to 0.05, 0.1, 0.3, 1, 3, 10, 30, 100, 300 s^{-1}. Exposure time was set to 1 minute at each shear rate.

2.2. Results
To observe shear-induced orientation change, the initial USAXS pattern was subtracted from the one under shear flow. Figure 1 shows the subtracted USAXS patterns of a barium complex grease at each shear rate. With increasing shear rate, the strong scattering was detected in vertical/horizontal components of the USAXS patterns, which implies the thickener would move and align perpendicular/along to the shear direction.

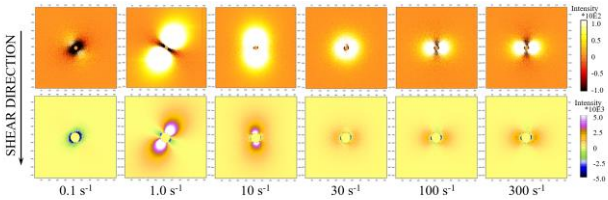

Figure 1: Subtracted USAXS patterns of barium complex grease in 0.15 mm gap: X-ray scattering variation induced by the shear flow (top) and the one partially zoomed in around an ultra-small-angle regime (bottom).

3. Discussion

The relationship between apparent viscosity of grease and microstructural behavior is shown in Fig. 2. The subtracted USAXS patterns were divided into two components because greases show remarkable scattering characteristics in vertical/horizontal directions. As shown in Fig. 2, thickener began to move in two steps, one is around 1 s^{-1} in a vertical component and the other one is around 10 s^{-1} in a horizontal component. They correspond to the edge of the intermediate plateau of the flow curve, it might be seen that the former shows the orientation start and the latter shows active alignment including collapse of the thickener structure. The mechanisms of microstructural behavior of thickener were confirmed by simulation models.

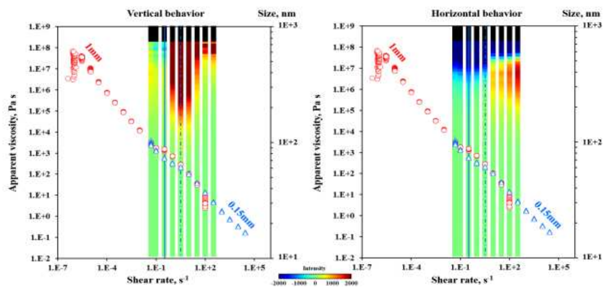

Figure 2: The relationship between thickener structure behavior and apparent viscosity of barium complex grease (left: vertical component, right: horizontal component of the subtracted USAXS patterns).

4. References

[1] Lugt, P.M., “Grease Lubrication in Rolling Bearings”, John Wiley & Sons, Ltd., 2013.