BL08B2
Hyogo BM

1. Introduction

BL08B2 is one of the Hyogo prefectural beamlines. It is a hard X-ray contract beamline designed for industrial applications. X-ray absorption fine structure (XAFS), X-ray topography, imaging, computed tomography (CT), X-ray diffraction (XRD), and small-angle X-ray scattering (SAXS) measurements can be performed for industrial usage. Over the past few years, research to develop new materials to apply to informatics technologies (materials informatics) has been actively conducted. Since FY2018, the beamline has supported research and development by manufacturing through a coalition between synchrotron radiation measurements and informatics technologies. For materials informatics with synchrotron radiation, it is important to prepare large datasets for machine learning. Operando XAFS/XRD and high-throughput SAXS measurements have been conducted via automatic data acquisition. In situ measurements can also be performed using experimental utilities such as a heating/cooling stage, a heating tensile tester, and a rheological cell. Large amounts of data are obtained from in situ observations using these utilities, which are used for machine learning and simulation analysis. In this report, among the experiments using these utilities, we report on ultrasmall-angle X-ray scattering using the rheological cell (Rheo-USAXS).

2. Rheo-USAXS measurement system at BL08B2

Rheo-USAXS at BL08B2 uses a CSS-450 rheological cell [1]. The rheological cell has a plate–plate shear geometry and the gap between the two plates can be adjusted between 0.005 and 2.5 mm. The shear rate can be controlled in the range of 0.003 to 15,000 s^{-1}. In addition, the rheological cell of BL08B2 is specially made with synthetic quartz glass as the window material in order to obtain high-quality SAXS data. For Rheo-USAXS experiments of grease, which will be described later, an automated measurement system was constructed by combining the control of the rheological cell and USAXS measurements. Figure 1 shows the Rheo-USAXS setup at BL08B2.

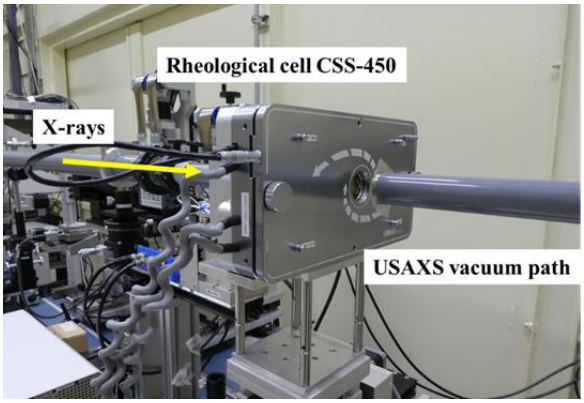

Fig. 1. Rheo-USAXS setup at BL08B2.

3. Observation of structural change of grease thickener using Rheo-USAXS

Grease is a semisolid substance mainly comprising a base oil and a thickener, and it functions as a lubricant that can be subjected to shear forces. The lubricity-obtaining process of grease based on the shear-thinning effect is considered to be deeply involved in a structure formed by thickeners [2]. Insights on the microscale shear-thinning mechanism are considerably important for the development of high-reliability greases. To

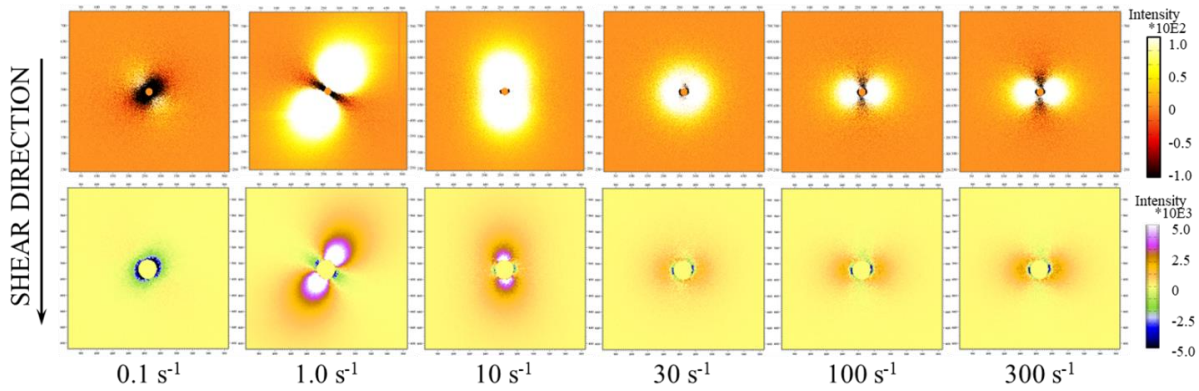

Fig. 2. Subtracted Rheo-USAXS patterns of barium complex grease: changes in overall scattering images (top) and zoomed images in the ultrasmall-angle region (bottom).

elucidate the shear-thinning mechanism of grease, the shear-induced structural changes of the thickener were observed by Rheo-USAXS.

In situ Rheo-USAXS measurements were performed at BL08B2. The X-ray energy and USAXS camera length were set to 12.4 keV and 16 m, respectively, and the X-ray detector was PILATSU-1M. Barium complex grease was used as the measurement sample. The gap between the two plates was controlled to 0.15 mm. The shear rate was controlled to 0.1, 1, 10, 30, 100, and 300 s^{-1}. The exposure time was set to 60 s at each shear rate.

Figure 2 shows the results of the in situ Rheo-USAXS measurements. To make it easier to observe the shear-induced orientation change, the initial USAXS pattern was subtracted from that under shear flow. With increasing shear rate, a change in the orientation direction of the USAXS pattern was observed. This behavior indicates a change in the orientation structure of the thickener and suggests that there is a relationship between the shear-thinning effect of the grease and the microstructural behavior of the thickener. The structure formed by thickeners in grease can be observed by in situ Rheo-USAXS. A simulation analysis of the microstructural behavior of the thickener will be performed in the future.

This study was conducted by Noda et al. of NSK Ltd. / University of Hyogo. The content of the publication was taken from the 7th World Tribology Congress (WTC2022) abstract [3].

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References:

[1] Linkam Scientific Instruments.

[2] Lugt, P. M., "Grease Lubrication in Rolling Bearings", John Wiley & Sons, Ltd. 2013.

[3] Noda T. et al., "Observation of Shear-Induced Orientation Change of Grease Thickener Structure Using Ultra-Small-Angle X-ray Scattering", 7th World Tribology Congress, Lyon, France, July 2022.