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Orientation of *n*-Hexadecane molecules in a single emulsion droplet revealed by microbeam two-dimensional SAXS-WAXS measurement

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Introduction

Alkanes are among the most basic components of soft materials; nevertheless the precise mechanism of crystallization has not been clarified. Metastable phase of n-alkanes, rotator phase, is a key for the understanding of the crystallization. Recently, the existence of the rotator phase in C16 [1] in emulsion droplets was experimentally certified [2], while the precise mechanism of crystallization through the rotator phase remains unsolved. For the study of the structural inhomogeneity in a single droplet, scanning microbeam X-ray scattering is a promising tool. In this study, we used scanning microbeam 2D-SAXS-WAXS in order to investigate crystal orientations in a single droplet.

Experimental

The sample used was C16 droplets in O/W emulsions. Samples of C16 with > 99 % purity were purchased from Sigma Chemicals and no further purification steps were taken. The O/W emulsion droplets were made by microchannel emulsification technique [3], by which we obtained large droplets with nearly monodisperse size distribution. We added 1 wt.% concentration of emulsifier before emulsification.

Experiments were performed at BL-4A. The X-ray wavelength was 1.54 Å and the beam size was around 5 μm × 5 μm. A large aperture X-ray Image Intensifier (270 mmϕ) coupled X-ray CCD detector [4] was used as a detector. The sample-to-detector distance was around 160 mm. Various positions in a droplet were irradiated by the microbeam X-ray and 2D-SAXS-WAXS image was simultaneously recorded.

Results & Discussion

Figure 1 shows the result of scanning microbeam SAXS-WAXS. A droplet with a diameter of around 50 μm was scanned by the microbeam X-ray. The SAXS-WAXS image from the center part of the droplet showed isotropic scattering pattern, while the SAXS-WAXS from the upper side oil-water interface and from the lateral side oil-water interface showed anisotropic scattering pattern. There is a decisive difference between the scatterings from the upper and the lateral sides of interface. In the SAXS-WAXS image from the upper interface, SAXS is observed in the vertical direction, and WAXS is observed in the horizontal direction. On the other hand, the SAXS-WAXS image from the lateral side interface was rotated by 90°. These results show that the C16 molecules align their axes parallel to the hydrophobic base of the surfactant. This finding strongly suggests that the surfactant at oil-water interface plays a precursor role for the crystallization.

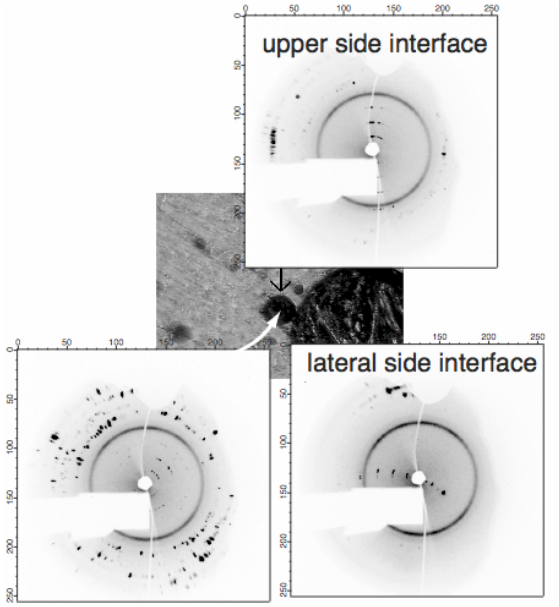

Fig. 1: Microbeam SAXS-WAXS images from three positions in a droplet. The central image is obtained by optical microscope. Black circle in scattering patterns corresponds to the scattering from Myla films used as the windows of the sample cell. Scattering inside the circle corresponds to the SAXS, which is from lamellar spacing, and scattering outside the circle corresponds to the WAXS, which is from the packing of the molecules.

References

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