

Simultaneous 2D SAXS/WAXS study of crystallization of *n*-Hexadecane in emulsion droplets

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Introduction

Normal alkanes are among the most basic components of soft materials such as surfactants, lipids, liquid crystals and polymers. It is, therefore, important to study the nature of *n*-alkanes for the further understanding of soft materials. Precise kinetics of crystallization of *n*-alkane, especially *n*-Hexadecane (C16), has not clarified yet in spite of large number of studies.

In the present study, we have performed simultaneous time-resolved 2D SAXS/WAXS measurements of C16 in emulsion droplets during crystallization. With this technique which provides the information about nano- and subnano-structural change, the transient phase of C16 has been observed at the initial stage of the crystallization.

Experimental

The sample used was of droplets of C16 of an oil-in-water (O/W) emulsion. By using the novel method called Microchannel method [1], monodisperse and large droplets were prepared. The average size of droplets was $32.6 \pm 3.2 \mu\text{m}$.

SAXS/WAXS measurements together with Differential Scanning Calorimetry (DSC) were performed at BL-15A. The X-ray wavelength was 1.50 Å and the beam size at sample position was 0.5 mm × 0.5 mm. We performed the experiments at two different settings (setting A), we used two X-ray CCD detectors coupled with 230 mm diameter X-ray Image Intensifier (XRII) for SAXS and 150 mm XRII for WAXS simultaneously (Fig.1). The camera length was 870 mm for SAXS and 200 mm for WAXS, respectively. At the other setup (setting B), the images of SAXS and WAXS were recorded with an X-ray CCD detector coupled with 230 mm diameter XRII. The distance between the sample and the detector was 239 mm. The sample was sealed in a 1.0 mm thick cell made of aluminum and cooled from 25 °C to -5 °C with a cooling rate of 2K/min. SAXS/WAXS images were recorded at the intervals of 10 s.

Results and Discussion

Figure 2 shows the time evolution of SAXS intensity profile from C16 during crystallization which was obtained at the setting A. It is clearly seen that transient peaks appear at the initial stage of crystallization.

Transient peaks have been also observed in WAXS images which are not shown here. The positions of these peaks agree with the rotator phase of C16 which was observed by Sirota et al., in bulk sample with one-dimensional detector [2], which was detected 9 times out of 85 measurements. In our experiments, the peaks from the rotator phase were observed in every measurement. This result shows that the time-resolved 2D SAXS/WAXS measurement is a powerful tool for the observation of peaks which was difficult to be detected.

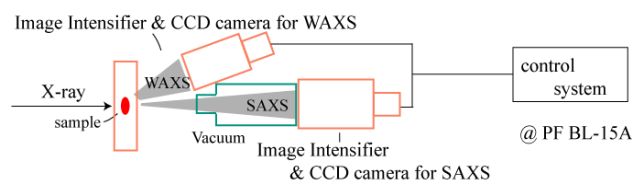


Figure 1: Schematics of experimental setup

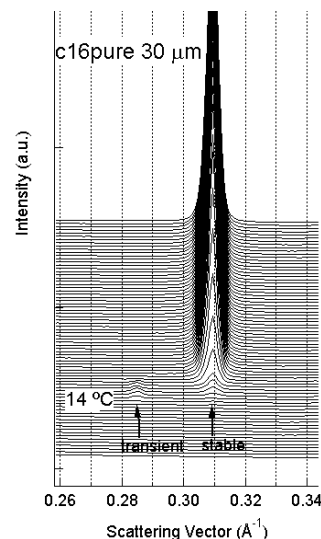


Figure 2: SAXS profiles during crystallization. Transient peak first appeared first at 14 °C and stable peaks appeared later.

References

- [1] T. Kawakatsu et al., J. Am. Oil Chem. Soc. 74, 317 (1997).
- [2] E. Sirota and A Herhold, Science 239, 529 (1999)

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