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Development of laser-trap technique for microbeam SAXS study of crystallization in droplet floating in solution

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Introduction

Microbeam X-ray scattering is a useful method to get information about inhomogeneity of nano- and subnano-scale structure in materials. Conventionally, a sample is scanned by a microbeam X-ray by moving the sample itself. But, when a sample of interest is floating or sinking in solution, it is impossible to hold and/or move the sample at an X-ray beam position, because it is moving involuntarily in the sample cell. So, it is required to develop a technique to hold a sample in solution, such as an emulsion, so that the microbeam X-ray scattering method can be applied. If this technique is realized, microbeam X-ray scattering method will be used more widely.

Experimental

We have adopted a "laser trap" technique[1]; a focused laser beam produces optical forces enough to hold a small particle. This is widely used as noninvasive micro-manipulators for biological objects.

We have built a laser optical system, and combined it with microbeam X-ray optical system at BL-4A. (Fig.1) The laser beam was incident diagonally to a sample holder so as not to obstruct an X-ray beam. The scanning of the laser beam was performed by moving the entire laser system. The beam from a fiber laser (wavelength: 1064nm, power: 1.0w) was focused by objective lens (×10, NA=0.28).

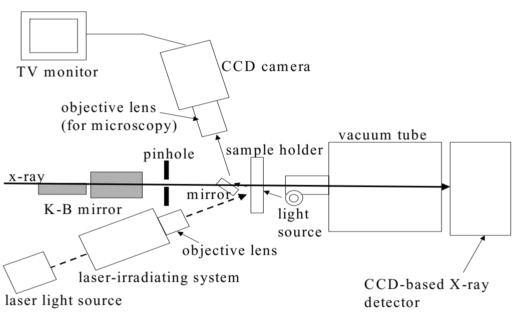

Fig.1: Optical system including a laser beam and an microbeam X-ray.

As a sample, we used oil-in-water (O/W) emulsion, which consisted of distilled water as continuous phase, n-hexadecane (oil) as disperse phase and Tween20 as surface-active agent. The size of oil droplets was 30μm in diameter. We tried to examine the structure of oil crystal in a single oil droplet.

X-ray beam size at the sample position was 5μm and X-ray wavelength was 1.5A.

Results and discussion

We have successfully picked up a single oil droplet in the sample cell, and moved it at will to the X-ray beam position by moving the laser beam. Then, the droplet was crystallized upon cooling. Small-angle X-ray scattering (SAXS) pattern was obtained from the single droplet as shown in Fig.2.

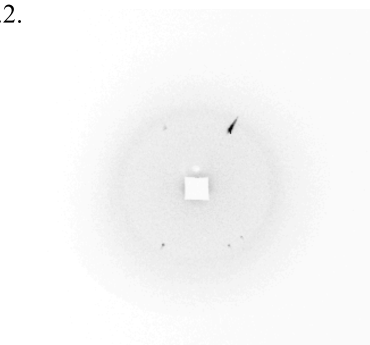

Fig.2: SAXS pattern from n-hexadecane lamellae in a single oil droplet.

In Fig.2, X-ray scattering from n-hexadecane lamellae appears as a spot-like pattern rather than a ring pattern. In addition, the spot-like pattern seems to be rather oriented but not at random. This means that the size of n-hexadecane crystal in a single droplet is comparable to that of the X-ray beam, and that the crystal grains have a certain preferred orientation in a single droplet.

This SAXS pattern was obtained from a droplet in which the oil was crystallized. However, we couldn't examine the crystallization process, because the droplet moved when it crystallized, in spite of being trapped by laser. The reason may be that the refractive index of oil changed when it crystallized and that optical force given to the droplet was changed. We need to build an improved system, in which a droplet doesn't move even during the crystallization by using two laser beams.

References

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