

Micro-beam Scanning X-ray Analysis for Crystallization Mechanism of Two Dimensional Spherical Crystal of β' form and Polymorphic Transformation from β' to β Form of Trilaurin

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Crystallization Mechanism of two dimensional spherical crystal of β' form of trilaurin and its polymorphic transformation from β' to β form have been studied by micro-beam scanning X-ray method using synchrotron radiation source, BL-4A station at Photon Factory, KEK, Tsukuba, Japan. Micro-beam was prepared by reflecting synchrotron X-ray beam to K-B mirror and focusing the reflected beam to the sample position. The focused beam size was $5 \times 5 \mu\text{m}$. Micro-beam was scanned for each $20 \mu\text{m}$ for $200 \mu\text{m}$ length of x

and y direction, meaning 121 times (11×11) scanning was done for a spherical crystal. Two dimensional β' spherical crystal which was put between two glass plate was prepared

by cooling from $60 \text{ }^\circ\text{C}$ to $5 \text{ }^\circ\text{C}$ with cooling rate of $5 \text{ }^\circ\text{C}/\text{min}$. This β' spherical crystal consisted a lot of tiny β' single crystal. Scanning micro-beam to β' spherical crystal revealed that the orientation of lamellar structure of a spherical crystal radiated from the center of the crystal, and it meant that because the orientation of the trilaurin molecules was perpendicular to lamellar structure, therefore, molecules in the spherical crystal were placed along the concentric circle whose center was the center of crystal. During heating process with $5 \text{ }^\circ\text{C}/\text{min}$ to $40 \text{ }^\circ\text{C}$, β' form transformed to β form completely through not melt-mediation but the solid state transition. A lot of tiny β crystals appeared. The micro-beam scanning analysis at $40 \text{ }^\circ\text{C}$ also revealed that the orientation of the β crystals showed the same pattern, or trace, of that of the β' form crystals shown at $5 \text{ }^\circ\text{C}$. It means that β form crystallization would occur by tracing the β' spherical crystal, i.e., β' spherical crystal played the role of template for the β form crystallization.