Although temperature-controlled sample holders for electron microscopes have been available since the 1930s for the study of organic materials, they have not been frequently employed for a quantitative cryotransfer specimen holder with temperature range -170°C to 150°C allows one to follow their thermotropic phase behavior as illustrated by three examples:

1. Electron diffraction data (Okv) from solution-grown crystals of normal even chain paraffins clearly reveal the transition from the orthorhombic to the hexagonal pre-melt state. Data (Okv) from crystals epitaxially-grown on boric acid allow one to distinguish among several suggested chain melting models; reduced analysis of lamellar 00l reflections but relative intensity of such lattice sites. In the case of ammonium alums the anomaly temperature dependency of the occupancies of such lattice sites is observed on the Fourier transform of the chain zig-zag in accordance with a kinked chain model which includes chain end voids.

2. Microcrystals of di(n-C14 rady) phosphatidyl-ethanolamines epitaxially crystallized on naphthalene give basically the same smectic liquid crystal form. Some difference in the melting temperatures of chiral and racemic dipalmitoyl compounds was also found.

3. Solution-grown microcrystals of cholesteryl myristate reveal the transition of the crystal form to the smectic mesophase (diffuse ring at 5.3A) as shown by Price and Wendorff (J. Phys. Chem., 75, (1971) 2839-2840). Larger area crystals (250m diam.) are obtained by cooling the smectic phase but cooling from the cholesteric phase (+10°C) through the smectic produce spherulitic crystals. A previously uncharacterized crystal form exists (at least) below -145°C with the shortest unit cell spacings double the room temperature c axis as long as b, although the symmetry is the same as that of room temperature. The present work is to make clear a mechanism of the appearance of the modulated structures in the lower temperature phases. Experiments were performed by taking X-ray precession and Laue photographs at temperatures down to 85K. In consideration of an aspect of the molecular configuration, the modulated structures are explained by phase modulation arising from nonsinoidal distortion. The displacements of CTFP molecules have a component along the unique axis of the crystal. On the other hand, one dimensional diffuse scatterings originating in the modulated structure are observed on the c-planes at 85K. These scatterings leave the traces even if the temperature rises above 175K. This fact indicates a presence of another mode of displacement by phase transitions.

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