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Effect of Thickness on Phase Transitions of Organic Thin Film by DSC

Nobuaki Okubo, SII NanoTechnology Inc. - nobuaki.ookubo@siint.co.jp

With decreasing the material size, the structure and the properties are influenced by the interaction between the materials and the material-gas surface or the material-solid interface due to the increase of the surface area to volume ratio. The structure of monolayer and multi-layer of amphiphilic di-block copolymer is in the metastable state within 40 nm from the copolymer-silicon interface (1, 2). The melting temperature of poly(ethylene oxide), PEO, in the confined space decreases discontinuously at 100 nm with decreasing sample thickness due to the PEO-silicon interface. In this study, the phase transition temperature and structure of PEO ultrathin film with the thickness from ?m to nm were investigated using the high sensitive DSC and AFM.

The ultrathin PEO film was prepared by solvent casting from 10 ?L toluene solution with various concentrations. Phase transition behaviors of ultrathin PEO film were investigated at 5 K/min under nitrogen atmosphere by X-DSC7000 Differential Scanning Calorimeter (SII NanoTechnology Inc.) and the morphology was observed by the dynamic forth mode by E-sweep Scanning Probe Microscope (SII NanoTechnology Inc.). The thickness of PEO thin film was determined by X-ray reflectivity measurement.

DSC heating curves, normalized by sample mass, of PEO ultrathin film of the different sample weight from 0.0004 to 3.0811 mg, different film thickness from 40 nm to 1 ?m, on 1st and 2nd heating were observed. On the 1st heating, the solvent casting sample showed the double melting peaks around 60 ºC, and the bulk sample showed the single melting peak at 65.5 ºC. Although the accuracy of sample mass less than 10 ?g, the DSC heating curves normalized by sample mass were almost the same, which indicated that the melting enthalpy was scarcely influenced by sample mass. On the other hand, the melting behavior of samples prepared by cooling from the molten state at 5 K/min was influence by sample mass on the 2nd heating. All sample showed the single melting peak, and the melting temperature decreased with decreasing the sample mass. The morphology of the solvent casting sample was fringe micelles consisted of small thin lamellar observed by AFM height image. However, the morphology of sample prepared by cooling from the molten state was lamellar with 1 ?m of the diameter. The melting temperature difference between ultrathin films prepared by solvent casting and cooling from the molten state was due to the morphology difference.