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Keywords: total reflection X-ray fluorescence, dynamical theory of X-ray diffraction, Darwin profile

P12.04.15

Acta Cryst. (2008). A64, C551

Micro and quick reflectometry with high-energy white synchrotron X-rays

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X-ray reflectivity is sensitive to slight structural changes along the depth of layered materials in the order of sub-nanometers or even smaller. This property is extremely promising for the observation of buried layers and interfaces of multilayered thin films. So far, the method looks at quite wide area (mm²~cm²) and requires typically 20min~1h for one measurement. Here we report the instrumentation for upgrading the technique to give it a much higher spatial resolution with very quick measurement. The experiment has been done with high-energy white synchrotron beam ranging up to 100 keV, at BL28B2, SPring-8. Parallel small beam of 17micron(H) x 5.5micron(V) has been formed by several pairs of thick blade of tungsten. The data have been taken by a silicon drift detector as X-ray reflection spectra. One can analyze inhomogeneous layered thin films with around 20 micron resolution, and even scanning has become possible. Figure shows the results of the line scan of a patterned sample which has Cu, Cr and Au layer on the same substrate. Further experimental details and results will be presented.

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Keywords: grazing incidence, microbeam analysis, energydispersive analysis

P12.04.16

Acta Cryst. (2008). A64, C551

Melting behavior of substrate-free polystyrene surfaces studied by X-ray reflectivity

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Glass transition, melting and solidification in the surface region of polystyrene (PS) were observed by surface-sensitive X-ray reflectivity (XR). Unlike the studies reported, in which ultrathin layers with several nm thick had been prepared by the spin coating method on flat substrates, we used the surface of bulk PS with several mm thick. The reason why we adopt such a PS surface is to reveal the kinetics and dynamics of glass-forming polymers on the surface without any effect from the substrates. Not a few results on physical properties on thermal behaviors of glass-forming polymer surfaces would severely be affected by the physical and chemical properties of substrates, and by residual solvents. A lump of solventfree PS (glass transition temperature: ca. 373 K) melted on a Si (100) wafer at 450 K in low vacuum for 6 hours was cooled down to the room temperature with a cooling rate of 1 K/min. Then, the PS was removed from the Si to obtain the flat surface of PS with 2 cm X 2 cm in area and 2 mm thick. The surface of PS was found to be extraordinary flat; the root-mean-square roughness of the surface was fitted to be 1 nm by XR measured at room temperature, which was also confirmed by AFM. XR at various temperatures up to 400 K is now undertaken to obtain the temperature dependence of surface roughness, height-height correlation function, and electron density of the novel PS surface.

Keywords: melting, surfaces, X-ray reflectivity

P12.04.17

Acta Cryst. (2008). A64, C551

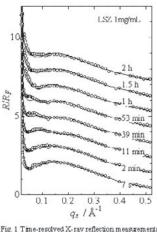
Time-resolved X-ray reflectivity investigation of lysozyme adsorption at the air-water interface

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X-ray reflection measurements of a globular protein adsorped at the air-water interface was demonstrated using recently developed liquid interface reflectometer at SPring-8. The reflectometer equipped with two-dimensional hybrid pixel array detector, PILATUS,

achieved only 1 sec, having enormous potential for quick measurements. A globular protein, lysozyme (LSZ) was injected into a phosphate buffer solution. The reflectivities shown in Fig.1 were measured under non-equilibrium condition, 4 sec after the protein injection. The time required each measurement was 150 sec. A broad peak observed in R/RF moves towards small qz, suggesting that the thickness of protein layer becomes thicker during adsorption. More details will be discussed.



Time-resolved X-ray reflection measurements regrame(LSZ) adsorption at the arrivater interface

Keywords: X-ray reflectivity, protein unfolding, SPring-8

P12.06.18

Acta Cryst. (2008). A64, C551-552

Resonance shear measurement on liquid crystal confined between solid surfaces under electric field

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It is known that the properties of liquids confined in the nanometer scale spaces are quite different from those of bulk liquids due to the structuring. We developed a resonance shear measurement¹⁾