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μGISAXS and μXRR experimental set-up for thin film characterization

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Introduction

In recent years, a grazing incidence small-angle x-ray scattering (GISAXS) technique has become widely used for the characterization of surface or buried structures of thin films. From the GISAXS pattern, we are able to determine the size, shape, size distribution of the particles and/or the information of the nm ordered structures. In addition, X-ray reflectivity (XRR) measurement gives us information on thin films such as a film thickness, film density, surface roughness and interface roughness. A combination of these techniques is preferable for the understanding of thin film structures. Both GISAXS and XRR measurements are carried out under the grazing incidence configuration, which is around the total external reflection angle. So the footprint of the incident beam becomes large, up to a few cm under the conventional experimental condition. That means we can only get average information on the film parameters and that need a uniform and large sample. If we use the X-ray micro-beam as an incident beam, the footprint along the x-ray beam becomes small. And it is desirable that two techniques are used for same area with the same experimental set-up. But the combination of μGISAXS[1] and XRR measurements of the same area has been only recently tried [2].

So, we developed μGISAXS [3] and μXRR system for measuring the same small area using X-ray micro-beam.

Experiment

μGISAXS and μXRR measurements were carried out on the BL4A at the Photon Factory. The X-ray micro-beam was formed by the Kirkpatrick-Baez focusing system. The size of incident beam was around 5μm*5μm and the divergence of the incident beam was around 0.2mrad. X-ray energy was 11keV (λ=0.11nm). The scattered/reflected x-ray beam was detected using an image-intensified CCD detector (II-CCD) for μGIASXS and μXRR. An X-ray path from the sample to the detector about 150cm long was evacuated to reduce air scattering. The present μGISAXS set-up adopted a specular beam attenuator which was not so thick that it could stop the speculary reflected beam. At the set-up of the μXRR, we used another set of the specular beam attenuator that had a suitable thickness which enabled transmitted X-rays to be measured within the dynamic range of the II-CCD. Since the exchange of the specular beam attenuator of μGISAXS for μXRR, and vice versa, was easy, we could measure the μGISAXS and μXRR at the same area under the same condition.

Results and Discussion

By using this set-up, we measured indium contained oxide film of about 20nm thick evaporated on a Si substrate. The μXRR measurement was performed by scanning of the incident angles. The X-ray reflectivity curve was calculated by extracting the specular spot region. Fig.1(a) is the obtained μXRR pattern from this film. The obtained reflectivity curve exhibits a typical interference pattern for the thin film. From this XRR curve, we performed the calculation in order to obtain parameters of the film based on the one layer model. After the μXRR measurement, by changing the specular beam attenuator and setting the sample to the particular incident angle, we can easily move on the μGISAXS measurement. Fig1(b) shows a μGISAXS image taken at 0.30° incident angle.

The combination of μXRR and μGISAXS is simply realized by changing the specular beam attenuator optimized for each measurement. Using our system, we could measure the μGISAXS and μXRR from the same small area of the thin film.

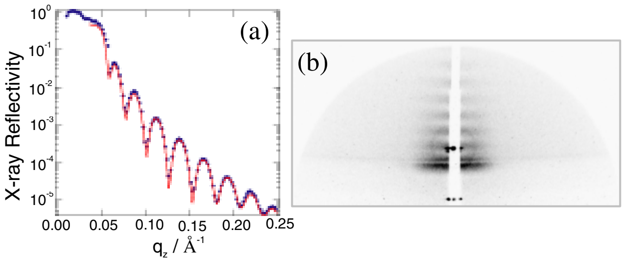

Fig.1 (a) shows μXRR profile. The blue circles are experimental data and the red line is the calculated one. (b) μGISAXS image taken at 0.30° incident angle.

References

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