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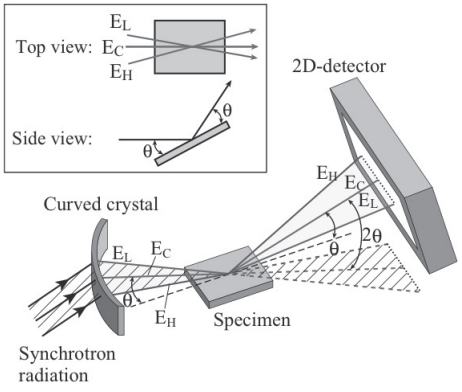
To observe structural dynamic behaviors at surfaces and interfaces, we developed a new method of simultaneously measuring X-ray crystal truncation rod (CTR) scattering profile without mechanical motion of the specimen, detector and X-ray optics during the measurement, using a geometry shown in the figure. A curved crystal polychromator produces a horizontally convergent X-ray beam having a one-to-one correlation between energy and direction. The convergent X-ray beam components of different energies are diffracted within corresponding vertical scattering planes by a specimen placed at the focus. In the specular geometry, although the glancing and exit angles, θ, are the same for all the directions, the momentum transfer continuously varies because the X-ray energy (wavelength) changes as a function of direction. The normalized horizontal intensity distribution behind the specimen represents the CTR scattering profile.

A convergent X-ray beam covering an energy range of 16 to 22 keV was produced by a curved crystal (Si 111 reflection). Scattering intensity was detected by a two-dimensional pixel array detector, PILATUS-100K. For 00L reflection (specular geometry) of a GaAs/AlAs superlattice on GaAs(001) substrate, reflected intensity was simultaneously measured in the range 1.6<L<2.4 around the GaAs 002 Bragg reflection. The CTR profile down to reflectivity of 1×10^{-10} was measured with a sufficient data collection time 1000-7000 s. The CTR profile was well reproduced by that measured by the conventional step-by-step angle scan method with a monochromatic X-ray beam.

Its potential for time-resolved measurement was demonstrated by measuring CTR profile in short data collection time. With an exposure time of 10 ms, CTR profiles down to reflectivity of 1×10^{-7} could be measured. Changes in CTR profile during rotation of the specimen were successfully measured with time resolution of 1.0 and 0.1 s. The present method can be a powerful tool to study irreversible structural changes at surface and interface such as material growth and reactions.



The simultaneous multi-wavelength dispersive diffractometry.

Keywords: surface_x-ray_diffraction, synchrotron_radiation, time_resolved_measurement

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Ordering of pores in InP (001) subsurface multilayers: formation and structural characterization

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Porous single- and multilayed semiconductors are of significant interest both as an object with advanced physical properties and a material for different applications. Typically these layers with meso- and nanopores are formed by anodization technique. Etching under passing of electrical charge produces the confused morphology of pores and architecture of porous multilayers. The structural characterization of porous layers is a main task.

In the present study we show an application of high-resolution X-ray diffraction and scanning electron microscopy methods for determination of structural parameters and space configuration of pores. The porous structures (≈6mkm) with single- or four bilayers were formed by anodic oxidation of n-type InP(001) substrates in aqueous HCl solution. The structural parameters of the sublayers were varied by changing the electrochemical etching mode (potentiostatic/galvanostatic). The X-ray diffraction experiment was performed on the E2 Station of Hasylab with radiation energy of 10 keV. To collect data one dimensional MYTHEN detector was used. The reciprocal space intensity maps (RSM) near the InP 004 reflection were obtained at different azimuth angle (φ=0,90) (Fig). The map's features demonstrate two completely different types of pores in InP oriented particularly along <111>B and <001> directions. To extraction features of pore a model for scattering from such systems is proposed based on the statistical dynamical diffraction theory. Theoretical scattering maps have been fitted to the experimental ones. It is shown that a mathematical analysis of the scattering intensity maps makes it possible to determine the structural parameters of sublayers. The reconstructed parameters (thickness, strain, porosity of sublayers and the shape and space arrangement of pores) are in satisfactory agreement with the scanning electron microscopy data.

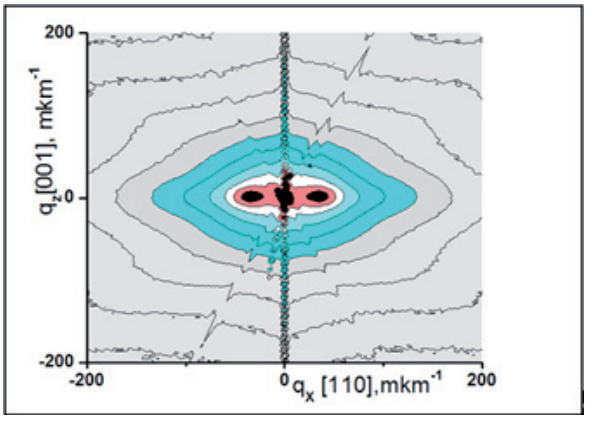

Figure. X-ray RSM from InP porous multilayers obtained in the vicinity of 004 reflection at φ=0. Two black sports demonstrate presence in the structure a middle ordering with correlation length 170nm.

Keywords: characterization, porous, multilayer

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GISAXS study of interfaces in high-performance La/B_4C multilayer mirrors

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Lanthanum based multilayer mirrors have been employed for soft X-ray fluorescence analysis and recently for processing femtosecond light pulses from free-electron lasers. The contribution is devoted to a detailed interface study in La/B_4C mirror in terms of roughness, correlation properties and thermal behaviour which is completed by probing the internal layer structure. These are decisive factors for mirror performance.

The mirror with 20 periods of 11 nm was prepared by magnetron sputtering. Grazing incidence SAXS (GISAXS) measurements were performed at HASYLAB BW4 beamline with Pilatus 300K detector at a wavelength of 0.138 nm and an angle of incidence of 0.7 degree. Laboratory X-ray reflectivity (XRR), XRD and AFM measurements complete the study.

The GISAXS pattern of the as-prepared mirror exhibits sheets of enhanced intensity around positions of the multilayer Bragg points (Fig. 1a) which indicates vertical correlation of the interface roughness. There is no spreading of the sheets along q_z for higher multilayer orders which implies equidistant interfaces without cumulative position error. The lateral cuts of the sheets show a maximum at q_y=0.06 nm^{-1} which is typical for mounded interfaces with characteristic lateral feature (mound) of ~100 nm. The mounds on the surface are visible by AFM (Fig. 1b). The origin of mounds is in the deposition process rather than in the layer structure which was found to be amorphous. A strong intensity decrease from the center to extremities of the lateral cuts of the sheets (~1 order of magnitude on the 1st order) suggests decay of vertical replication for higher roughness frequencies.

A series of 120 s rapid thermal vacuum annealings was performed on separate sample pieces from 150°C to 950°C with a step of 50°C. After an initial increase by 0.8% at 250°C, the multilayer period decreases by up to 5% at 950°C. However, the multilayer stack with vertically correlated mounded interfaces is still preserved. The neighboring XRR peak intensity ratios exhibit oscillatory thermal behavior from the lowest temperatures suggesting interface shifts while interface widths increase steadily but do not exceed 1 nm. The layer structure undergoes substantial changes above 750°C which result in LaB_6 compound formation. Obviously, decomposition of B_4C layers limits thermal stability.

The results have direct implications for application of La/B_4C mirrors, typically in the 100-190 eV energy range.

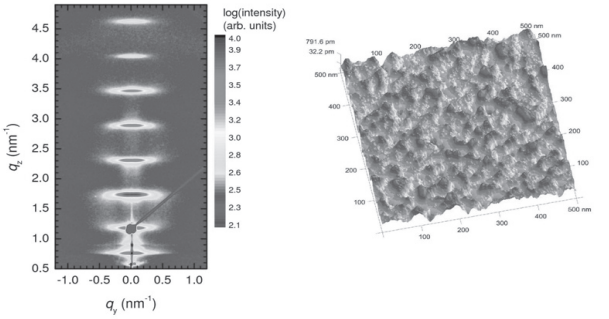

Fig. 1

Keywords: multilayer mirror, GISAXS, thermal stability

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Temperature evolution of dauphine twins terminated at quartz (001) surface revealed by X-ray reflectivity

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SiO_2 is a ubiquitous material in the lithosphere and known to exhibit several isomorphisms under high temperatures and pressures. Especially, structural phase transition between alpha quartz and beta quartz has attracted great attention for the decades owing to the triangular domain structure (Dauphine twins) peculiar to the alpha phase on cooling from the hexagonal beta phase. Group theoretical considerations had predicted an incommensurate (IC) phase as an additional intermediate phase, which was subsequently confirmed experimentally. It is not so surprising not to be reported by the middle of 1980s, if relatively high transition temperature (T_c = 846 K) and extremly narrow temperature range of the IC phase (ca. 1.3 K) are recalled. Although there are a lot of dielectric materials showing the Normal - IC – Commensurate (N-IC-C) phase successive phase transitions, we must regard quartz as a unique substance in which instability of acoustic phonon slightly away from the Gamma point in reciprocal space constructed by hexagonal symmetry plays an crucial role in developing the IC structure and responsible for the Dauphine twins formation. In spite of our understandings on bulk structure on N-IC-C phase transitions, we might admit that less is known for surface structure and morphology when it undergoes the successive phase transitions. In the present study, we investigated the surface structure of quartz with surface-sensitive-X-ray diffractions so as to give a novel insight into this phase transitions.

Sample was a (001) plane of synthesized quartz polished at room temperature with dimension of 20 mm x 20 mm x 0.15 mm. It was placed into a vacuum chamber installed at BL13XU of SPring-8 mounted on a multi-axis X-ray diffractometer. Sample temperature was controlled between room temperature and 980 K with a stability of 0.5 K. Surface-sensitive X-ray diffractions we exploited were crystal truncation rod (CTR) scattering emanating from the 003 Bragg point and X-ray reflectivity (XR). Rocking curves (q_x scan) and longitudinal curves (q_z scan) of XR in 2theta range between 0 and 5-8 degrees were collected at each temperature.

In the alpha phase, a noticeable increase in width of specular XR (q_x scan) beyond the total reflection regime is reproducibly observed as the sample temperature T when it approaches T_c. Furthermore, specular XR in total reflection regime shows an anomalous decrease in intensity. Both anomalies can be fitted by $C/(T_c - T)$, indicating that the variation of surface morphology in alpha phase would accompany some critical feature.

Keywords: quartz, surface, reflectivity

MS21.P06

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Atomic-scale structure of plate-shaped precipitates in Al-Cu-based alloys

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Many engineering aluminium alloys owe their high strength to low-dimensional precipitate phases generated by the age hardening process. Such precipitates, usually plate or rod shaped with very high aspect ratios (> 50:1) and one or two dimensions at the nanoscale (≤ 10 nm), are dominated by interfaces. In fact the precipitates' interfacial structure and composition to a large extent determine the properties and microstructural stability of the material. Despite the importance of such aluminium alloys both practically (as structural materials) and fundamentally (the age hardening process often results in metastable