

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/B₄C 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 $q_y=0.06 \text{ nm}^{-1}$ which is typical for mounded interfaces with characteristic lateral feature (mound) of $\sim 100 \text{ nm}$. 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 LaB₆ compound formation. Obviously, decomposition of B₄C layers limits thermal stability.

The results have direct implications for application of La/B₄C mirrors, typically in the 100-190 eV energy range.

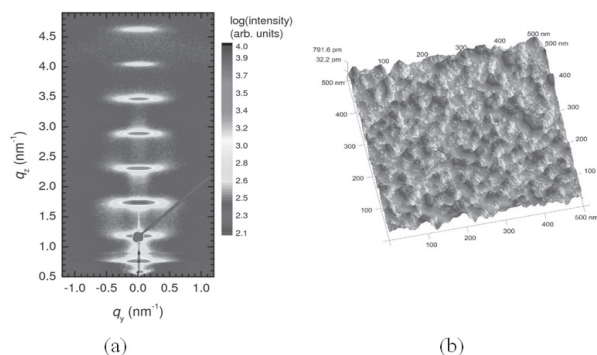


Fig. 1

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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SiO₂ 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 ($T_c = 846 \text{ K}$) and extremely 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

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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 ($\leq 10 \text{ nm}$), 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