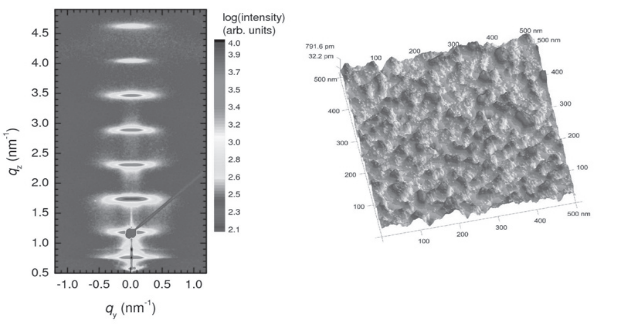
Lanthanum based multilayer mirrors have been employed for soft X-ray fluorescence analysis and recently for processing femtosecond light pulses from free-electron lasers. The contribution is devoted to a detailed interface study in La/B_4C mirror in terms of roughness, correlation properties and thermal 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.



(a) (b)

Fig. 1

Keywords: multilayer mirror, GISAXS, thermal stability

MS21.P05

Acta Cryst. (2011) A**67**, C336

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

Isao Takahashi, Tsuyoshi Kumagai, *Department of Physics, Faculty of Science and Technology, Kwansei Gakuin University, Gakuen 2-1, Sanda 6691337 (Japan).* E-mail: z96019@kwansei.ac.jp

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

Acta Cryst. (2011) A**67**, C336-C337

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

Laure Bourgeois,[a,b,c] Christian Dwyer,[a,b,c] Matthew Weyland,[a,c] Jian-Feng Nie,[b,c] Barrington C. Muddle,[b,c] [a]*Monash Centre for Electron Microscopy, Monash University, Victoria 3800, (Australia).* [b]*ARC Centre of Excellence for Design in Light Metals, Monash University, Victoria 3800, (Australia).* [c]*Department of Materials Engineering, Monash University, Victoria 3800, (Australia).* E-mail: laure.bourgeois@monash.edu

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 phases associated with intriguing solid-solid phase transformations), the atomistics of interfacial structure and growth mechanisms of many precipitate phases remains poorly understood.

Here we present a structural study by aberration-corrected scanning transmission electron microscopy (STEM) of Al-Cu precipitate phases in several model aluminium alloys. These phases, Guinier-Preston (GP) I zones, theta" (Al_3Cu) and theta' (Al_2Cu) phases, are classic crystalline precipitates of the simplest age-hardened alloys [1], [2]. Using a combination of experimental and simulated high-angle annular-dark-field STEM imaging, we demonstrate that these well-known phases in fact exhibit structural features hitherto unreported. Atomic-scale models of interfacial structures are provided. Furthermore, trace additions of Sn are shown to result in precipitate nanoscale thicknesses exhibiting "magic" values. These findings resolve a long-standing mystery [3] and provide new insights into the atomistic mechanisms of precipitate growth in these systems.

[1] A. Guinier, *Nature* **1938**, *142*, 569-570. [2] G.D. Preston, *Nature* **1938**, *142*, 570. [3] J.M. Silcock, T.S. Heal, H.K. Hardy, *J. Inst. Metals* **1955-56**, *84*, 23-31.

Keywords: aluminium, precipitate interface, TEM.

MS21.P07

Acta Cryst. (2011) A**67**, C337

A simultaneous multiple angle-wavelength dispersive X-Ray reflectometer

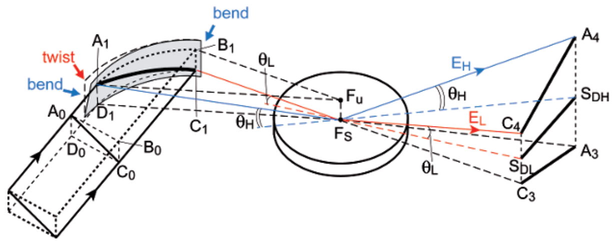
Etsuo Arakawa,[a] Tadashi Matsushita,[b] Wolfgang Voegeli,[b] Yasuo Higashi,[c] Yohko F. Yano,[d] [a]*Department of Physics, Tokyo Gakugei University, Koganei, Tokyo, (Japan),* [b]*Photon Factory, Institute of Materials Structure Science, KEK, Tsukuba, Ibaraki, (Japan),* [c]*Engineering Center, KEK, Tsukuba, Ibaraki, (Japan),* [d]*Department of Physics, Kinki University, Higashiosaka, Osaka, (Japan).* E-mail: arakawae@u-gakugei.ac.jp

In previous studies[1], [2], [3], we reported a method of measuring specular X-ray reflectivity curves with no need of angle scanning of the sample, detector and monochromator crystal during the measurement. In this method, the reflectivity curve is measured with a position sensitive detector as a function of X-ray energy using a convergent X-ray beam which has a one-to-one correspondence between direction and energy. Because the practically covered energy range was limited, the measured range of the momentum transfer (q=4πsinθ/λ: θ and λ being the glancing angle and wavelength of the X-ray beam) was not wide enough for quantitative analysis.

In the present study, we report improvements of the method for widening the simultaneously covered rang of the momentum transfer by realizing a convergent X-ray beam with which the wavelength (energy) and the glancing angle to the specimen surface of the X-ray beam change continuously at the same time as a function of direction. For realizing such a convergent beam, we used a bent and twisted crystal. An inclined slit is placed upstream of the crystal, so that the foot print of an X-ray beam is along a line from the upper left to lower right corners of the crystal. The crystal is elliptically bent in the horizontal plane and furthermore twisted around the cross line of the crystal surface and the horizontal plane. The beam reflected at the upper left corner is slightly deflected downward, while that at the lower right keep the same vertical direction. Another big improvement is the use of a pixel array detector (PILATUS 100K) instead of an X-ray CCD.

Reflectivity curve profiles from a silicon wafer were simultaneously recorded covering a q range from 0.05 to 0.5 $Å^{-1}$. Measured minimum reflectivities were 1×10^{-8} and 1×10^{-6} with data collection times of 100 s and 1 s, respectively.

We will report results of a performance test experiment and discuss means to further improve the performance of the reflectometer including the time resolution.



Geometry of simultaneous multiple angle-wavelength dispersive X-ray reflectometer

[1] T. Matsushita, Y. Niwa, Y. Inada, M. Nomura, M. Ishii, K. Sakurai, E. Arakawa, *Appl. Phys. Lett.* **2008,** *92***,** 024103. [2] T. Matsushita, E. Arakawa, Y. Niwa, Y. Inada, T. Hatano, T. Harada, Y. Higashi, K. Hirano, K. Sakurai, M. Ishii, M. Nomura, *Euro. Phys. J. Special Topics* **2009,** *167***,** 113. [3] T. Matsushita, E. Arakawa, T. Harada, T. Hatano, Y. Higashi, Y. F. Yano, Y. Niwa, Y. Inada, S. Nagano, T. Seki, *AIP Conf. Proc.* **2010,** *1234***,** 927-930.

Key words: X-ray_reflectivity_curve, simultanous_measurement, multiple_angle-wavelength_dispersive

MS21.P08

Acta Cryst. (2011) A**67**, C337-C338

Long-range order and interface stability in Co_2FeSi/GaAs hybrid structures

B. Jenichen, J. Herfort, T. Hentschel, A. Trampert, *Paul-Drude-Institut, Berlin, (Germany).* E-mail: bernd.jenichen@pdi-berlin.de

Ferromagnet/semiconductor hybrid structures are well suited for spin injection as a first step towards the design of spintronic devices. The half-metallic and ferromagnetic Heusler alloy Co_2FeSi exhibits a high degree of spin polarization as well as a large Curie temperature of about 1,100 K and is perfectly lattice matched to the semiconductors GaAs and Ge. Spin injection has already been demonstrated. The properties of the ferromagnet/semiconductor interface and long-range order in the Heusler alloy are important for an improvement of the spin injection efficiency. The Co_2FeSi structures on GaAs are grown by solid source molecular beam epitaxy. The long-range order and the interface stability of the structures are investigated by transmission electron microscopy (TEM), x-ray diffraction, atomic force microscopy (AFM) and secondary ion mass spectrometry. At high substrate temperatures T_S during growth, a precipitation process near the Co_2FeSi/GaAs interface is observed, which is connected to an enhanced diffusion of Co, Fe, and Si into GaAs. We use the value of T_S at which precipitation begins as the limit of the interface stability. The critical value of T_S depends on the crystallographic orientation of the interface. For (001) and (110) interfaces, these temperatures are near T_S= 250°C and T_S= 200°C, respectively, whereas for the (111) interface this temperature limit is considerably higher: T_S= 325°C. As a result, the (111) interface is the most stable interface, and an overgrowth of the ferromagnetic film with Ge could be possible for this orientation [1]. The figure demonstrates the surface roughness of the Co_2FeSi film measured by AFM for two orientations of the interface (110 and 111) and several growth temperatures T_G.

For a quantitative characterization of long-range order we determine the average order parameters in Co_2FeSi using x-ray diffracttion and image the lateral inhomogeneities of the compositonal order in the films on the nanometer scale using the dark-field mode of TEM with superlattice reflections. A fundamental reflection is insensitive to long-range order, and the dark-field image of that reflection is usually almost homogeneous. A superlattice reflection images the distribution of long-