Quick Measurement of Crystal Truncation Rod Profiles in Simultaneous Multi-Wavelength Dispersive Mode

A new method was developed which can simultaneously measure the whole profile of X-ray diffraction and crystal truncation rod (CTR) scattering of interest in a short time (seconds to sub-seconds) without needing to rotate the specimen, detector or monochromator crystal during the measurement. The method uses a bent-crystal polychromator to realize a convergent X-ray beam which has a one-to-one correspondence between energy and direction and a two-dimensional pixel array detector to simultaneously measure intensity distribution of diffracted/scattered X-rays as a function of the momentum transfer.

Crystal truncation rod (CTR) scattering is caused by the sharp truncation of the crystal at the surface and extends to angles far from the Bragg point along the direction of the surface normal of the sample crystal. The amplitude of this CTR scattering is of the same order as that of the scattering from surface monolayers. Atomic coordinations at surfaces and interfaces can be determined by analyzing the interference between these two. In the conventional angle-scan method, the CTR scattering profile is recorded by repeating scattered intensity measurements a few hundred times, each time after changing the incident angle of monochromatized and collimated X-rays. Data collection in the angle-scan mode therefore typically takes several tens of minutes to several hours even when using third-generation synchrotron radiation sources.

Figure 1 shows the geometrical arrangement of the present method. A white X-ray beam is diffracted by a polychromator curved crystal. Since the angle between the incident X-ray beam and the diffracting plane changes continuously along the surface, the energy E (wavelength λ) of the diffracted X-rays also varies as a function of the beam direction toward the horizontal focus. The convergent X-ray beam components of different energies are incident on a sample placed at the focus in a geometry such that the glancing angle θ in the vertical direction is the same and they are diffracted within corresponding vertical scattering planes by a specimen. The momentum transfer defined by $q_z = 4\pi\sin\theta/\lambda$ continuously varies because the wavelength λ changes as a function of direction. The normalized horizontal intensity distribution downstream of the sample represents the reflectivity curve profile both near to and far from the Bragg point.

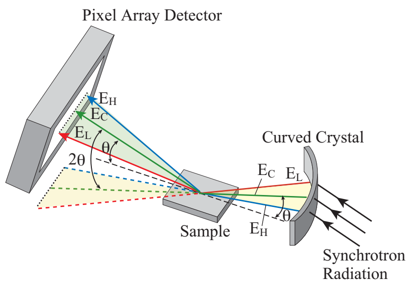

Figure 1
Geometrical arrangement for quickly measuring diffraction and crystal truncation rod profiles in simultaneous multi-wavelength dispersive mode.

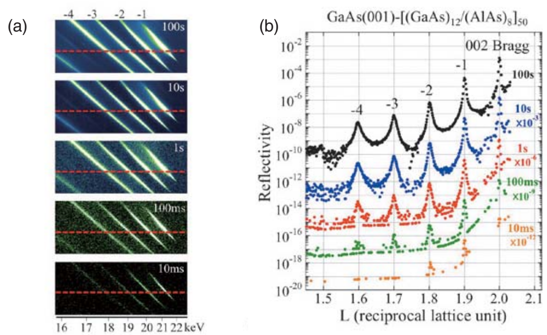

Figure 2
Diffraction and crystal truncation rod profiles from [GaAs(12ML)/AlAs(8ML)]$_{50}$ superlattice on GaAs(001) substrate measured in the simultaneous multi-wavelength dispersive mode. (a) Two-dimensional detector image recorded with data collection times of 100, 10, 1.0, 0.1 and 0.01 s. (b) Reflectivity along the vertical momentum transfer q_z in the reciprocal lattice unit obtained from intensity profiles along the red lines in Fig. 2(a).

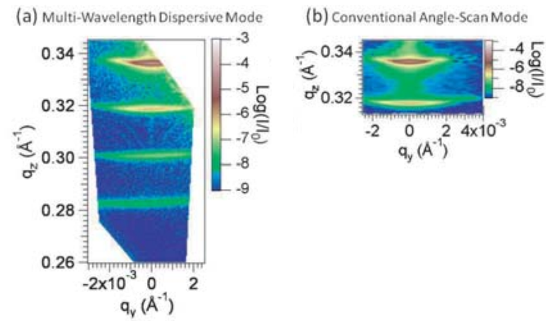

Figure 3
Reciprocal space maps around 002 reflection of [GaAs(12ML)/AlAs(8ML)]$_{50}$ super lattice on GaAs(001) substrate. (a) was obtained in 1 s with the present simultaneous multi-wavelength dispersive method. (b) was obtained in 30,000 s with the conventional angle scan method.

The experiment was carried out on the undulator beamline AR-NW2A. The 111 reflection of a 0.2 mm-thick silicon crystal bent to a radius of curvature of 425 mm was used in the transmission geometry covering an energy range from 15.8 to 22.7 keV. The beam was focused horizontally at a distance of approximately 210 mm from the center of the crystal. The sample was a GaAs/AlAs superlattice with 50 repeats of 12 GaAs (3.39 nm thick) and 8 AlAs (2.26 nm thick) monolayers on a GaAs(001) single-crystal substrate. A pixel array detector (PILATUS-100K) was mounted on a 2θ arm of a goniometer at a distance of 1000 mm from the specimen.

The panels in Fig. 2(a) show diffracted intensity patterns recorded on the detector for data collection times of 100, 10, 1.0, 0.1 and 0.01 s. The curves in Fig. 2(b) are normalized intensity profiles along the red lines in Fig. 2(a), representing diffraction and CTR scattering profiles around the 002 reflection from 1.46 to 2.04 in the reciprocal lattice unit of GaAs (from q_z = 0.26/Å to q_z = 0.36/Å). In the curve obtained with a data collection time of 100 s, reflectivity down to ~1×10^{-10} was simultaneously measured. Curves obtained with data collection times of 10, 1, 0.1 and 0.01 s are shown by shifting every three orders of magnitude along the vertical axis for clarity. With data collection times of 10, 1.0, 0.1, and 0.01 s, profiles down to a reflectivity of ~4×10^{-10}, ~1×10^{-9}, ~6×10^{-9} and ~1×10^{-7} were measured, respectively.

In a previous paper [1], it was shown that the intensity distribution along oblique intensity patterns recorded on the detector reflects the scattering profile along the q_y direction which is within the scattering plane and perpendicular to the surface normal. A reciprocal space map (RSM) in the q_y-q_z plane was obtained as shown in Fig. 3(a), for the 00 rod of the specimen. Figure 3(b) shows the RSM obtained with the conventional angle scan method on the bending magnet beamline BL-4C. The RSM by the multi-wavelength dispersive method was obtained with a data collection time of only one second, while that with the angle scan method required 30,000 seconds.

The present method opens up the possibility of obtaining time resolved CTR scattering profiles from samples undergoing irreversible structural change at surfaces and interfaces. Time-resolved CTR measurement is now under way for samples which undergo photo-induced irreversible surface structure changes.

REFERENCE

[1] T. Matsushita, T. Takahashi, T. Shirasawa, E. Arakawa, H. Toyokawa and H. Tajiri, *J. Appl. Phys.* **110** (2011) 102209.

BEAMLINE

AR-NW2A

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