ESRF	**Experiment title:** **Investigation of amorphous-to-crystalline interface in Xe irradiated Si (001)**	**Experiment number**: Si-1550
Beamline: ID 01	**Date of experiment**: from: 14-02-2008 to: 19-02-2008	**Date of report**: 06-03-2008
Shifts: 15	**Local contact(s)**: Dr. Till Hartmut Metzger	*Received at ESRF:*

Andreas Biermanns*, Ullrich Pietsch*, Anton Davydok*, Souren Grigorian

FB7 - Physik , Universität Siegen, 57068 Siegen, Germany

Jörg Grenzer*, Antje Hanisch*

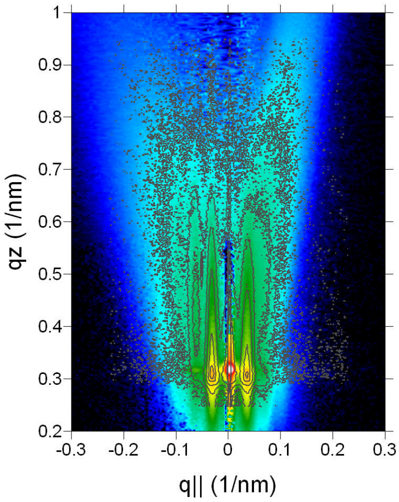
Forschungszentrum Dresden-Rossendorf, Institute of Ion Beam Physics and Materials Research
01314 Dresden, Germany

Report:

We report on the investigation of nanostructured silicon using grazing incidence small-angle scattering and – diffraction. For this study, Si samples were irradiated with energetic Xe^+-ions in the Forschungszentrum Dresden-Rossendorf with ion-energies between 5keV and 70keV under oblique incidence, leading to the process of ripple formation at the surface. The ripple patterns have a characteristic wavelength between 45nm and 200nm, depending on ion energy and incidence angle. The projection of the ion-beam on the surface was aligned along [110]. The ripples align perpendicular to the beam-direction and therefore also perpendicular to the [110] planes. Due to the damage created by the incident ions, the topmost part of the surface gets amorphous, followed by a transition region towards crystalline material. From TEM measurements it is known that this interface between the amorphous and crystalline part shows the same rippled morphology as the surface.

In this experiment we investigated the surface morphology using GISAXS and the interface structure and morphology using GID. In addition, the structure of the amorphous layer was studied using amorphous scattering [1]. The experiments were carried out at the beamline ID01 at a X-ray energy of 8.8keV using a position sensitive detector (PSD) placed perpendicular to the sample surface. The setup allows fast measurements of GISAXS and GID data with intermediate resolution that was sufficient for our purpose. Exemplarily, figure 1 shows GISAXS and GID (transverse scan) data recorded for a sample irradiated with 50keV Xe^+ ions. The presence of satellite peaks in the maps indicates the mean wavelength of the probed system, in this case ~190nm. Furthermore, an asymmetric distribution of diffuse scattered intensity indicates a clear asymmetric average shape of individual ripples that can only hardly be determined from AFM measurements due to tip-convolution effects. Furthermore, we found good agreement between GISAXS and GID data, allowing to deduce that both surface and amorphous-crystalline interface show similar morphology also on mm scale, a fact which can not easily be determined from TEM which probes the structures only very local.

Figure 2 shows examples of longitudinal GID scans taken either along (a) or across (b) the ripple direction [110] at an incidence angle of 0.1°, limiting the penetration depth to a few nm only.
As visible, the measurements show an asymmetric intensity distribution in the scan measured along the ripples, indicating an expansion of the lattice in the transition region toward the bulk material. This expansion may be a consequence of the creation of defects inside the material during irradiation. Surprisingly, there is almost no lattice expansion across the ripples.



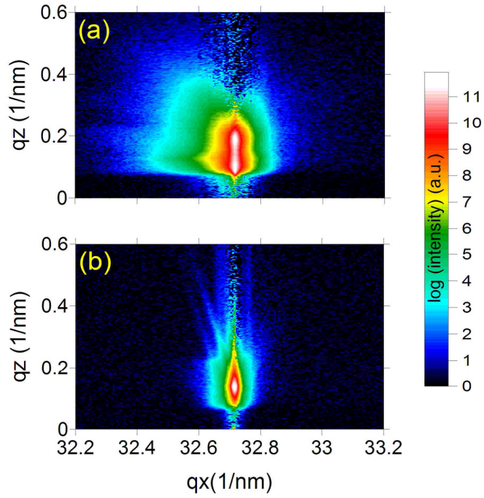
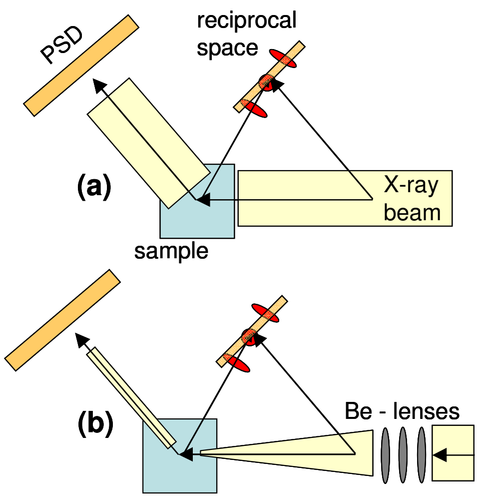


Figure 1 Reciprocal Space map comparing GISAXS (colour) and GID data (contours). The data shows satellite peaks as well as asymmetric diffuse scattered intensity, corresponding to an asymmetric average shape of the ripples

Figure 2 Longitudinal scans at shallow incidence angles comparing the lattice expansion at the amorphous-crystalline interface along (a) and across (b) the ripples. The asymmetry is caused by a net expansion of the transition region.

In addition to samples where the projection of the ion beam was aligned along the [110] direction, we exploratory investigated first samples with azimuthal orientations different from [110]. In this case, also the (GID-) satellite peaks rotate in reciprocal space and it is no more possible to obtain information from transverse- or longitudinal line scans only. To avoid time-consuming reciprocal space maps, we evaluated a setup using the PSD aligned parallel to the sample surface, i.e. covering a wide range in 2θ. However, for a conventional setup with a relatively broad beam and therefore large footprint on the sample surface, a large area on the sample creates a broad diffracted beam leading to a loss of resolution in reciprocal space (figure 3(a)). To overcome this problem, either an analyser in front of the detector or a small beam size has to be used which lead to significant losses of intensity if a conventional system of slits is used. To increase resolution without loosing too much intensity, we made use of the focussing option at ID01 using a set of compound refactive Be-lenses to focus the beam down to a size of 20×50µm² (figure 3(b)). Figure 4 shows the effect of this setup in reciprocal space, measured at a sample irradiated by 5keV Xe^{+}-ions along [110] (therefore the satellite peaks are not rotated with respect to the vertical direction). It's visible that the setup with the Be-lenses increases the resolution up to a sufficient level, allowing fast measurements of in-plane reciprocal space maps in further experiments (timescale: 10min for RSM with PSD and Be-lenses; 5h for conventional GID setup using a point-detector).



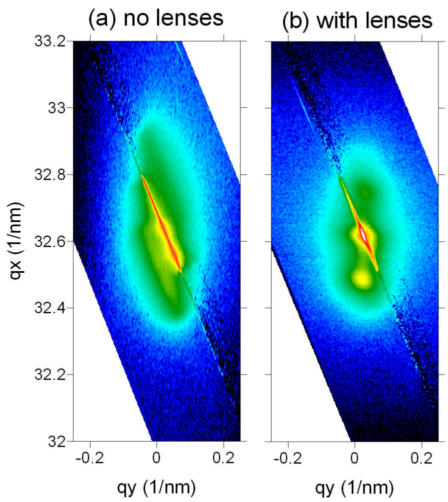


Figure 1 Sketch of the experimental setup for fast measurement of in-plane reciprocal space maps. With a large beam, the footprint counteracts the PSD-resolution (a). Be-lenses decrease the footprint while maintaining intensity and increasing resolution (b).

Figure 2 Comparison of a measurement without (a) and with (b) a focussed beam, showing strongly increased resolution when a focussed beam is used. The sample was irradiated with 5keV Xe-ions, leading to a wavelength of 45nm.

[1] S. Grigorian, U. Pietsch, J. Grenzer, D. P. Datta, T. K. Chini, S. Hazra, M. K. Sanyal *"Micro-structural anisotropy at the ion-induced rippled amorphous-crystalline interface of silicon"*, Appl. Phys. Lett. **89**, 231915 (2006)