ROBL-CRG	**Experiment title:** *In-situ ion beam irradiation: X-ray scattering & diffraction experiments*	**Experiment number:** **20-02-716**
Beamline: BM 20	**Date of experiment:** from: 10.11.2012 to: 16.11.2012	**Date of report:** 3.11.2013
Shifts: 12	**Local contact(s):** Dr. Carsten Bähtz and Dr. Jörg Grenzer	*Received at ROBL:*

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Report:

The aim of this experiment was to setup an in-situ ion beam implantation experiment at ROBL/MRH. An ion gas source was used with a maximal acceleration voltage of 5kV. To realize sufficient volume damage the ion energy was raised further by increasing the electrostatic potential of the irradiated sample to 15kV using an additional power supply. The idea of this setup was to mimic an ion beam implanter in the low keV range. X-ray diffraction experiments were carried to probe the sensitivity and stability of such an in-situ experiment.

Two tasks should be solved during this experiment:

1st modification of the existing in-situ setup, test of the HV interlock system and remote control;
2nd test of different measurement strategies.

We were able to carry out a first in-situ real time experiment but the time resolution was rather limited (~5min/receprocal space map see Fig. 1). Ion implantation is a very fast process and it is almost impossible to monitor it in-situ with the present x-ray sources. However, the accumulation of damage is a much slower process and was accessible during this experiment. The experiment was carried out using He^+ at an ion flux of about 10^{13}ions/cm^2/s implanting it into a Si(001) substrate. The experimental setup is shown in Figure 2.

Figure 1 shows two reciprocal space maps around the (004) Bragg reflection. An almost symmetrical crystal truncation rod can be observed. Implantation leads to a strained layer which expands in the direction normal to the substrate surface. Figure 3 shows the time evolution of this strained layer during continuous irradiation. In the first minutes of irradiation (time between starting irradiation, searching the hutch and first measurement) a weakly strained layer due to the generation of point defects was build up (red arrow in Fig.2).

Since the bulk material prevents any lateral macroscopic expansion, the (thin) irradiated layer is subjected to an in-plane biaxial compressive stress due to the continuous accumulation of point defects. This leads to an lattice expansion in the direction of the surface normal. This process leads to the appearance of a peak in the diffraction curve (black arrow fig. 3). The width of this peak is almost constant which can be related to a layer thickness of about 100nm. The peak position shifts constantly with the irradiation time reflecting the increasing strain in the irradiated layer. After an irradiation time of ~1h a lattice expansion of about $(\Delta a/a)_\perp \approx 0.3\%$ was found.

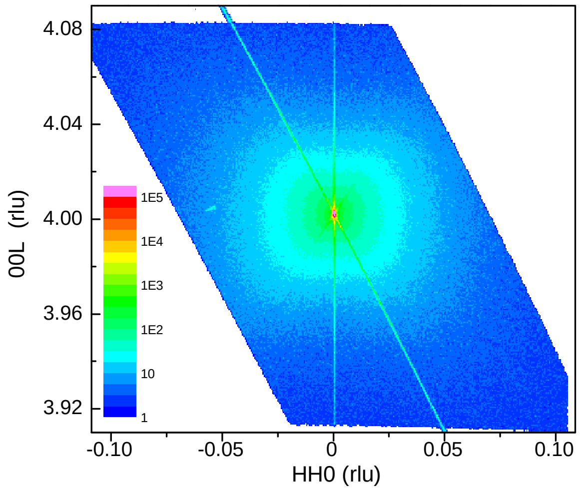

Fig 1a: Reciprocal space map at the (004) reflection before irradiation. The tilted line running trough the centre of the Bragg reflection is a position sensitive detector artefact due to very high intensity.

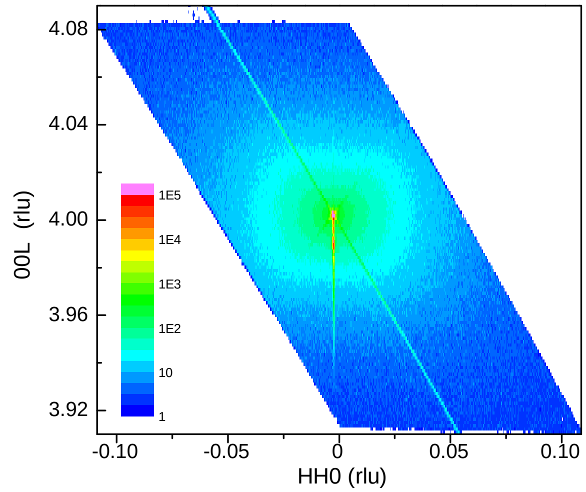

Fig 1b: Reciprocal space map at the (004) reflection after 60min irradiation at ~15keV. A second peak appeared characterizing a layer with a vertical lattice expansion due to irradiation.

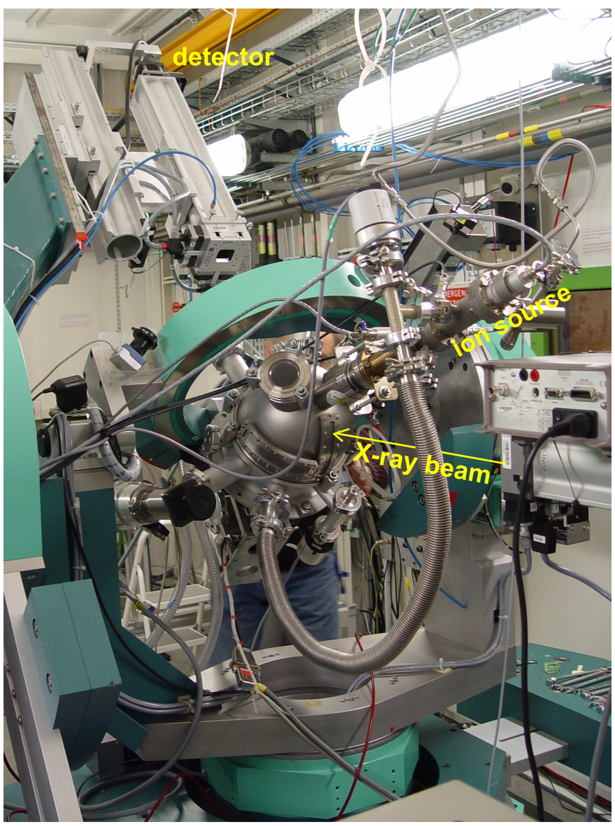

Fig. 2 Experimental setup with an alignment of the diffractometer for an asymmetrical (115) reflection in shallow incidence. The ion source is on the right pointing towards the X-ray source; the position sensitive detector is on the left.

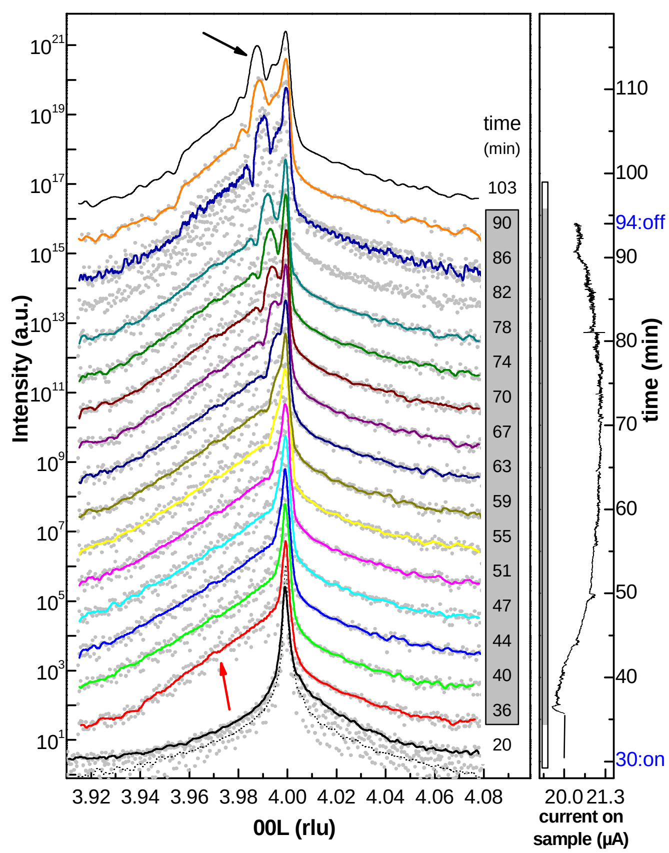

Fig 3. Line scans at the (004) reflection before, during and after irradiation. The inset shows the variation of the irradiation current during the experiment. The grey bars highlight measurements during irradiation; arrows are explained in the text.