<b>ESRF</b>	Experiment title: Investigation of ion beam induced nano-wire formation by x-ray grazing-incidence diffraction	Experiment number: SI-1062
Beamline:	<b>Date of experiment</b> : from: 16.09.04 to: 20.09.04	<b>Date of report</b> : 23,02,05
Shifts:	Local contact(s): H. Metzger	Received at ESRF:
15   U. Pietsch*, J. Grenzer*, S. Hazra <sup>a,*</sup>		

Institute of Physics, University of Potsdam, Am Neuen Palais 10, 14415 Potsdam, Germany

<sup>a</sup>Saha Institute of Nuclear Physics, Binhagar, Calcutta

## **Report:**

The formation of a periodic ripple structure from the nanometer to the micrometer length scale at semiconductor surfaces has become a topic of intense research in the context of fabrication of nanoscale textured materials.<sup>1</sup> Ion-induced ripples are produced as result of the interplay between a roughening process originated by ion beam erosion (sputtering) and smoothening processes caused by thermal or ion-induced surface diffusion.

The ESRF-Highlights 2004<sup>2</sup> have kindly considered the results of our previous investigations where we have investigated the self organized periodic ripple formation at Si(001) surfaces. The sample surface was bombarded with <sup>40</sup>Ar<sup>+</sup> ions under 60° incidence with respect to surface normal using different doses and ion beam energies. The lateral ripple wavelength  $\lambda$  of about 1000 nm is found to be dependent on the implantation energy; the ripple depth can exceed 100 nm.<sup>3</sup>

In contrast to previous TEM investigations, the crystalline nature of the ripples in the top region of sample implanted with low doses is evident from the appearance of diffraction peaks even at a very low penetration depth (5 nm) of the probing x-ray beam. Modelling of the measured Yoneda wings reveals a 25% lower material density in the top region compared with the undisturbed crystalline material.<sup>3</sup> The presence of a crystalline structure in the top region of  $Ar^+$  implanted silicon at dose of about  $10^{17}$  ions/cm<sup>2</sup> is surprising. Amorphization of Si by heavy ion (such as  $Ar^+$ ) bombardment at keV energies typically occurs at a dose of about  $10^{14}$  ions/cm<sup>2</sup>. Further experiments are required to understand the process of ripple formation and to distinguish between amorphous and crystalline scattering.

In the present experiment we have tried to resolve the amorphous scattering in grazing-incidence geometry. The measurements have been performed at the ID1 beamline at photon energy of 8 keV. We have performed in-plane scans through reciprocal space in a direction far away from any reciprocal lattice point but under shallow angle of incidence. In particular, we scanned the in-plane detector angle  $2\Theta$  for fixed azimuth angles  $\phi$  and fixed out-of-plane incidence angles  $\alpha_i < \alpha_c$ , where  $\alpha_c$  is the critical angle of total external reflection of crystalline silicon. Figure 1 shows the amorphous scattering for a sample after Ar<sup>+</sup> implantation of  $8 \cdot 10^{16}$  ions/cm<sup>2</sup>. It clearly shows two maximum above the background which refer to the amorphous scattering of damaged silicon. The peak position of the first maximum at  $2\Theta = 24^{\circ}$  fairly well corresponds to the next-neighbour distance of crystalline silicon - Si(111). The angle position of the second peak might be influenced by the neighbourhood to the (220) reflection of the underlying crystal; it shifts towards the respective Bragg angle for increasing  $\alpha_i$ . Figure 1b shows similar scans for a sample implanted with  $7 \cdot 10^{17}$  ions/cm<sup>2</sup>. In contrast to the low dose material the angle position of the first peak is shifted to larger scattering angles, i.e., to smaller next neighbour distances. The structural origin of this distance is not clear yet, but it is close to the oxygen-oxygen distance in SiO<sub>2</sub>. Additional sharp peaks appear for decreasing  $\alpha_i$ 

which can be interpreted by micro-crystals onto the silicon surfaces, probably created from eroded material while the implantation process.



**Figure 1:** Amorphous scattering of low (a) and high dose (b) implanted silicon as a function of the incidence angle  $\alpha_i$ . The first diffraction maximum of (a) refers to the next-neighbour distance of crystalline silicon, whereas the respective maximum in (b) gives a smaller distance. At the high-implanted materials we also find additional sharp peaks which refer to the existence of micro-crystallites at the sample surface.

Varying the azimuth angle  $\phi$  of the sample at fixed 2 $\Theta$  position the intensity of the first maximum of amorphous scattering is not constant. It appears a pronounced texture for samples with low implantation doses (see Fig.2) but a more uniform distribution for higher implantation doses. This documents the strong directional dependence of the amorphization process as the amorphous scattering is strongly correlated with certain crystallographic directions of the crystal. Due to channelling effects the ions can penetrate deeper into the crystalline material and initiate self-diffusion of displaced host atoms and subsequent amorphization in certain directions. The interplay between this directional diffusion and the appearance of ripples at the sample surface is still a matter of question and needs further experiments.



(a)



<sup>1</sup> U. Valbusa, C. Boragno and F. B. de Mongeot, J. Phys.: Condens. Matter **14**, 8153 (2002). <sup>2</sup> ESRF Highlights 2004, page 79-80.

<sup>3</sup> S. Hazra, T. K. Chini, and M. K. Sanyal, J Grenzer and U. Pietsch, "*Ripple structure of crystalline layers in ion beam induced Si wafers*", Phys. Rev. **B70** 121307 (2004).