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ESRF	**Experiment title:** **In-situ annealing of ion-beam induced lateral nanostructures in silicon**	**Experiment number**: SI-1226
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Report: The formation of periodic surface structures varying from several nm to a few µm caused by ion-beam sputter erosion processes on semiconductor surfaces has attracted significant interest for the fabrication of nanoscale laterally structured materials. Different morphologies of quadratic, hexagonal or wave like patterns under oblique or normal incident low energy (E < 5keV) ion-bombardment of solid surfaces are rather well described and explained by the interplay between a roughening process caused by ion beam erosion of the surface and smoothening processes caused by thermally or ion-induced surface diffusion and can be well described in terms of the Bradley-Harper model and respective extensions.

Recently we found that the surface ripple formation is accompanied by a ripple structure at the amorphous-crystalline interface with the same lateral spacing as found at the surface. A one-dimensional ripple formation on Si (100) surfaces has been observed after ion beam impact of about 10^{17} Ar^+ ions per cm^{-2} with energy of about 60 keV and incidence angles with respect to surface normal close to 60° [2,3]. Using X-ray diffraction they discovered that the ripples at the surface are followed by a nearly sinusoidal shaped interface between the strongly damaged, not completely amorphous near-surface region and the crystalline material. In contrast to low energy ion irradiation the ripple formation at the amorphous to crystalline interface is restricted to azimuth angles close to to the [110] direction, whereas the ripples at the surface appears independent from azimuth angle used. The crystalline ripple side planes are close to {111} side facets. These narrow fabrication conditions suggest that the erosion process is strongly influenced by the crystal lattice.

In the previous experiment [4], we studied the role of temperature on the subsurface morphology of modulated nanostructures and the behaviour of amorphous-crystalline interface. For a sample with high dose of Ar^+ ions ($7\ 10^{17}$ cm^{-2}) prepared under ion beam incidence close to [111] we recorded the temperature induced modifications of amorphous scattering in grazing-incidence geometry (GIAS). This amorphous scattering was found to be anisotropic in sample azimuth showing that sample damage appears in certain crystallographic directions only [5]. After annealing at about 750°C, we did not found major modifications in GIAS but an increase of the Si (111) and Si (220) Bragg diffraction intensities indicating the growth of crystalline material. Using AFM this finding could be associated with the formation of strained crystalline islands on top of the surface ripples.

In the present experiment, we repeated the annealing measurement at sample with low dose of Ar^+ irradiation (8×10^{16} cm^{-2}). This dose roughly corresponds to the onset dose of ripple formation at the amorphous to crystalline interface. The aim of this measurement was to study whether the temperature and subsequently the increased mobility of ion beam induced defect atoms can induce ripple formation after ion bombardment. The measurements are performed at the ID1 beamline at photon energy of 8 keV. We heated up the sample up to about 800°C and measured GIAS (2Θ- scans at fixed in-plane angle being 7 degrees off the 220 Bragg condition) as a function of time. After annealing a second peak appears and the first peak is shifted towards smaller angles compared with the virgin sample (see fig.1). The two peaks correspond to nearest next neighbour and next nearest neighbour distances in the damaged sample. The sharp peaks on top of the broad curves correspond to strained Si nano-crystals appearing on top of the sample, which are not caused by the ion beam irradiation but by mechanical treatment of the sample. At 800°C (see fig.2) the second peak is always stronger than the first one. Moreover, here we observed a strong variation of peak intensity with time, which might indicate reorganization processes within the sample.

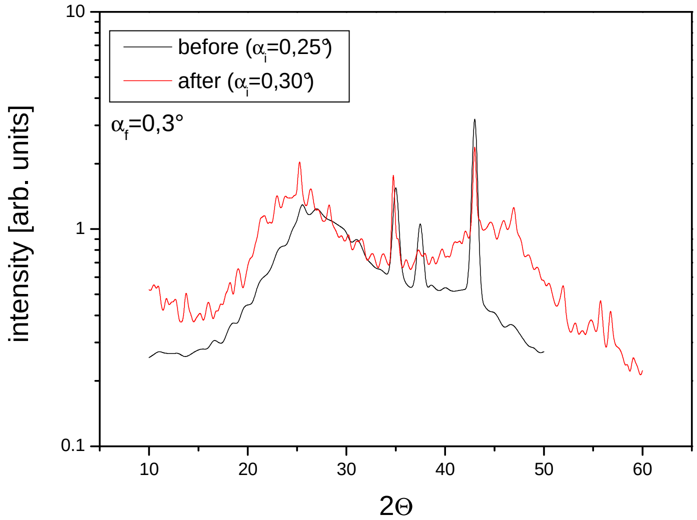

Figure 1: Amorphous scattering at shallow angle of incidence before and after annealing at 800°C.

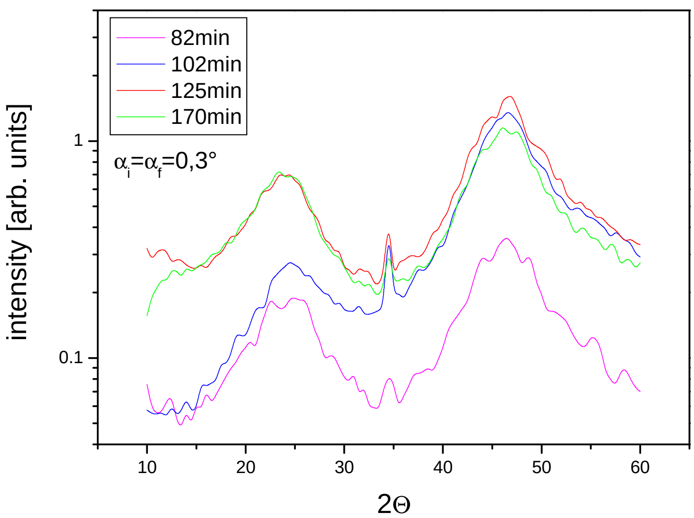

Figure 2: Amorphous scans during annealing at 800°C

As for high dose irradiated samples, the transverse scans through in-plane Bragg peaks did not show a change in the low performance of ripple formation at the damaged-to-crystalline interface. This finding corresponds with results of TEM investigations taken at this sample (fig.3). Therefore, we can conclude that the energy impact during ion beam irradiation is rather essential for ripple formation then the thermal mobility of defects. However, the details of this process will be investigated further by a scientific project submitted to German Science foundation recently.

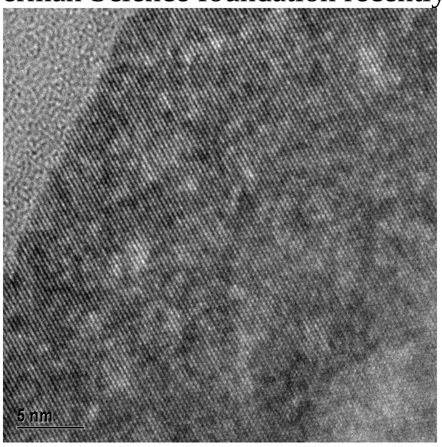

Figure 3: Cross-section TEM of an as-implanted low dose (8 $10^{16} Ar^{+}/cm^{2}$) sample. The amorphous region is clerly separated from crystalline part. The interface undulation is rather weak.

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