ESRF	**Experiment title:** *Nano-crystal formation by high-temperature annealing of ion-beam induced lateral nano-structures in silicon*	**Experiment number**: SI-1307
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Report⁺:

We report on in-situ investigations of a recrystallization process of amorphous and damaged crystalline parts generated during ion-beam induced rippling on a Si (100) surface. The ripple structure was created by 60 keV $^{40}Ar^{+}$ irradiation with a dose of ~$5x10^{17}$ ions/cm^2 at ion incident angle of 60° with respect to the surface normal. At this dose the ripples have average spatial periodicity of about 715 nm and surface undulations with an amplitude of about 40 nm. Structure and morphology of ripples were studied by two types of x-ray scattering (grazing incidence diffraction and amorphous scattering) methods as well as by transmission electron microscopy and atomic force microscopy. X-ray grazing-incidence amorphous scattering pattern were recorded in-situ for a temperature range from 250°C to 750°C. Up to about 500°C mainly we found a single broad scattering maximum corresponding to the Si(111) inter-planar distances. At higher temperature these peaks become sharp and intense indicating the onset of a re-crystallization process in the amorphous top layer.

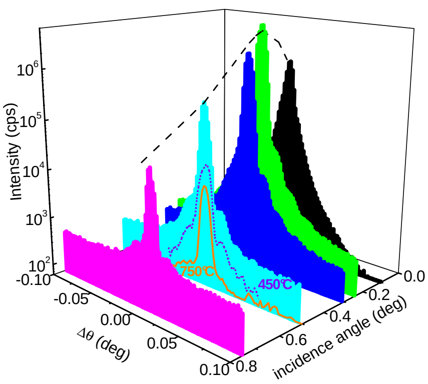

Figure 1a: GID curves around (220) Bragg peak of Si (001) substrate for different incidence angles (α_i), i.e. penetration depth. The filled curves are measured before annealing; the dotted line at 450°C and the solid line at room temperature after annealing up to 750°C. The dashed curve shows the α_i dependence at Bragg peak position.

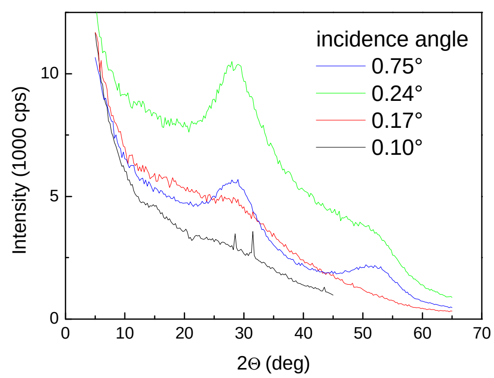

Figure 1b: Amorphous scattering as a function of the incidence angle α_i. The broad diffraction maxima refer to the next-neighbour distances of crystalline silicon.

Figure 1a shows GID patterns taken at different incidence angles α_i, i.e. different penetration depths. It shows the diffraction at the Si(220) Bragg reflection. For large penetration depths >100nm (α_i >0.35°) a number of satellites measuring the average lateral spacing of the buried ripple structure appeared in addition. These satellites indicate that the crystalline-amorphous interface shows a periodical modulation. The buried ripple wavelength (680nm) is very close to surface one (715nm), as it was determined by AFM. The satellites disappear at low penetration depths ($\alpha_i \leq 0.35°$) accompanied by an increase of the full width half maximum of the main Si peak. This is caused by an almost complete loss of the crystalline modulated structure within the damaged near surface region. The GIAS scans (Fig.1b) show the defect structure near the rippled surface of the as-irradiated sample. The curve at α_i=0.1° is flat but shows two small peaks at 2θ = 28.5° and 2θ = 31.5° originating from tiny crystallites at the sample surface. These angular 2θ positions correspond exactly to (111) and to the forbidden (200) reflections from strained Si crystallites. Such crystallites, sticking at the whole sample surface, are mainly caused by the cleavage process of the wafer prior to ion bombardment. With increasing α_i the scattering pattern of the implanted area is dominated by broad intensity humps with two clearly visible maximum positions on top of a nearly uniform background. Both features are increasing with increase of α_i. The maxima (2θ = 28.5° and 2θ = 47.3° for 8 keV) correspond to lattice distances of the (111) and (220) net planes but their width indicates that they come from very small clusters diluted in the amorphous matrix.

After annealing at about 750°C, we found a reduction of the amorphous scattering and an increase of the Si (111) Bragg diffraction intensity indicating a growth of crystalline material. In-situ measurements reveal different processes. At the beginning dominates the GIAS scattering. In the next phase the crystalline scattering prevails mirroring the process of recrystallization and correspondingly the amorphous scattering decreases. The formation of a buried defect-rich region close to the former amorphous-crystalline interface was confirmed by TEM investigations. The formation of strained crystalline islands on top of the former surface ripples was supported by AFM observations. Whereas these crystallites are randomly in azimuthal orientation (as random as these small crystallites before annealing) there are some indications that the ripple morphology, especially the formed (111) side facets at the amorphous-crystalline interface, trigger the growth of twined crystallites into the amorphous layer towards the surface.
This regrowth process was clearly observed by GID and GIAS x-ray techniques. The combination of complementary *in-situ* and *ex-situ* methods afford a detailed evolution of the re-crystallization process itself.

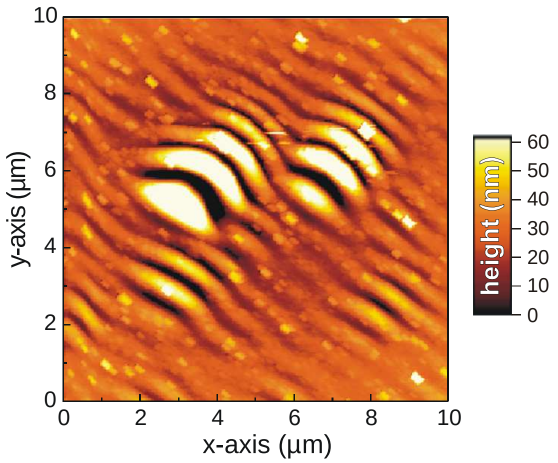

Figure 2: AFM picture of the rippled sample surface after annealing at 750°C. We find islands with height up to 200nm spread over several ripples.

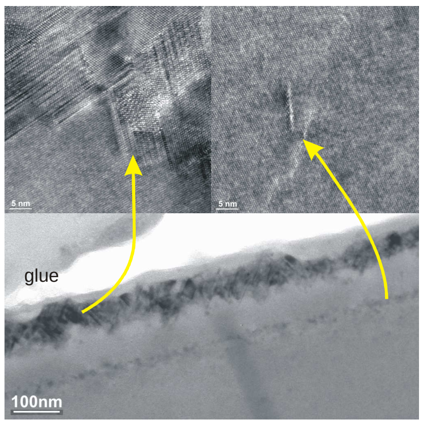

Figure 3: Cross-section TEM of an annealed (750°C) sample. In the region of the amorphous ripples twinned Si crystals are grown up (left inset). At a depth of about 200nm a region of defects is visible (right inset)

[+] J. Grenzer, A. Mücklich, S. Grigorian , U. Pietsch, D.P. Datta, T.K. Chini, S. Hazra, and M.K. Sanyal, *High-temperature induced nano-crystal formation in ion beam-induced amorphous silicon ripples*, accepted by physica status solidi (a) (2007).