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ESRF	**Experiment title:** **Investigation of ion beam induced nano-wire formation by x-ray grazing-incidence diffraction**	**Experiment number**: SI-1135
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Report: Surface morphology evolution during ion sputtering has received an increasing attention recently [1]. In particular, the formation of periodic ripples in the nanometer length scale at metal and semiconductor surfaces has become a topic of intense research. Such ripples are produced due to of the interplay between a roughening process by ion beam erosion (sputtering) and smoothening processes caused by thermal or ion-induced surface diffusion. In previous investigations we have investigated the ripple formation and the amorphization the Si (100) subsurface region as a function of implantation dose and energy [2, 3]. A significant ripple formation was found only using an incidence angle of about 60° with respect to the surface normal keeping the implantation direction parallel to the [110] direction. Maximum amplitude of surface ripples was observed at an implantation energy of 60 keV ($^{40}Ar^+$ ion-beam).

Samples prepared under these optimal conditions at a high dose of implantation of 4 and $5\cdot10^{17}$ ions/cm^2 were studied by depth resolved x-ray grazing incidence diffraction (GID) and by atomic force microscopy (AFM) [2]. The GID intensity profiles show the presence of satellite peaks on both sides of the main (220) Bragg peak. The appearance of satellite peaks confirms the existence of lateral undulation of the buried crystalline part of the sample. The angular positions of these peaks allow extracting the wavelength of subsurface crystalline ripples, which agrees well with ripple wavelength on the top surface observed by AFM measurements.

In the current experiment, we studied the role of temperature on the surface and the subsurface morphology of modulated nanostructures as well as the behaviour of amorphous-crystalline interface. In particular, we have recorded the amorphous scattering in grazing-incidence geometry. The measurements have been performed at the ID1 beamline at a photon energy of 8 keV. We have measured 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 2Θ for fixed azimuth and fixed out-of-plane incidence angles $\alpha_i < \alpha_c$ and $\alpha_i > \alpha_c$, where α_c is the critical angle of total external reflection of crystalline silicon.

The samples were annealed in the range from 250°C to 750°C. Figure 1 shows the behaviour of the GID amorphous profiles versus temperature. For all these profiles the maximum intensity is found at $2\theta = 28.5°$ which corresponds to Si(111). Up to 500°C the shapes of amorphous scattering curve remains unaffected by temperature, but it changes dramatically at about 750°C. The change indicates that crystalline aggregates start to grow from the amorphous top layer. Figure 2 shows amorphous scans for one sample annealed at 750°C and cooled down to room temperature. At both temperatures amorphous profiles were measured at the surface and in bulk using incidence angles $\alpha_i = 0.20°$ and $\alpha_i = 0.24°$, respectively.

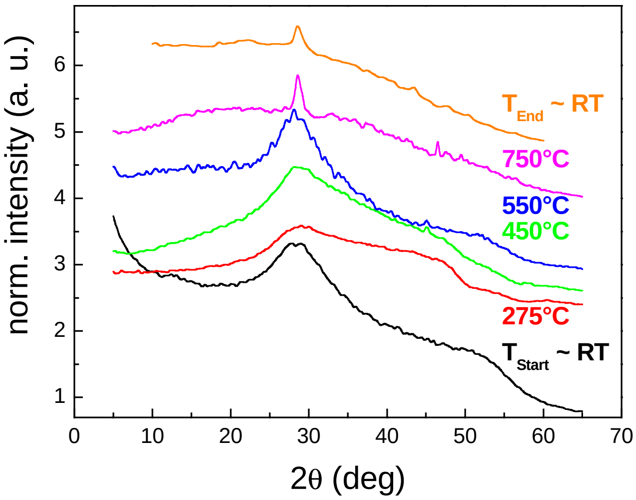

Figure 1: Amorphous scattering at $\alpha_i \approx 0.3°$ vs. temperature. The broad peak at $2\theta = 28.5°$ refers to the next-neighbour distance of amorphous silicon. At 750°C a regrowth process sets on.

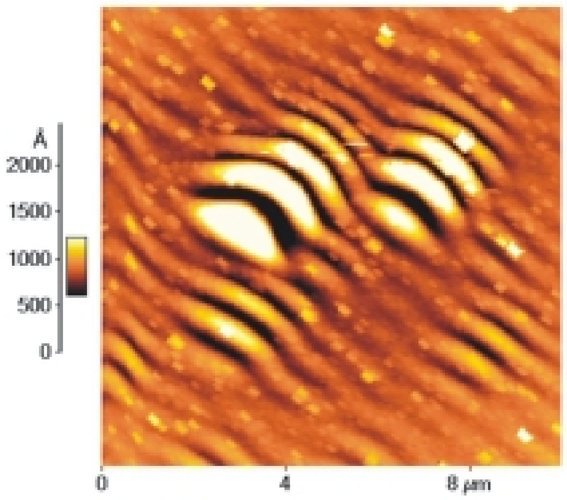

Figure 3: AFM picture of the rippled sample surface after annealing at 750°C. We finds islands with heigth up to 200nm spread over several ripples.

Red curves show the same profiles keeping the temperature constant for duration of about 3h at 750°C. The Si(111) peak becomes more pronounced after long time of annealing in both scans indicating the growth of crystalline material close to the surface. In addition to Si(111) one finds in bulk signal, Fig. 2b, a further peak close to $2\theta = 47.5°$ indicating a Si(220) reflection. Even more peaks were found at shallow angles corresponding to forbidden Si reflections such as (100), (200), (110), (112) and (113). Considering AFM measurements (Figure 3) the appearance of these peaks can be interpreted by the formation of strained crystalline islands on top of the surface ripples. The role of temperature on the formation of crystalline islands and kinetics of ripple reorganization need further investigations.

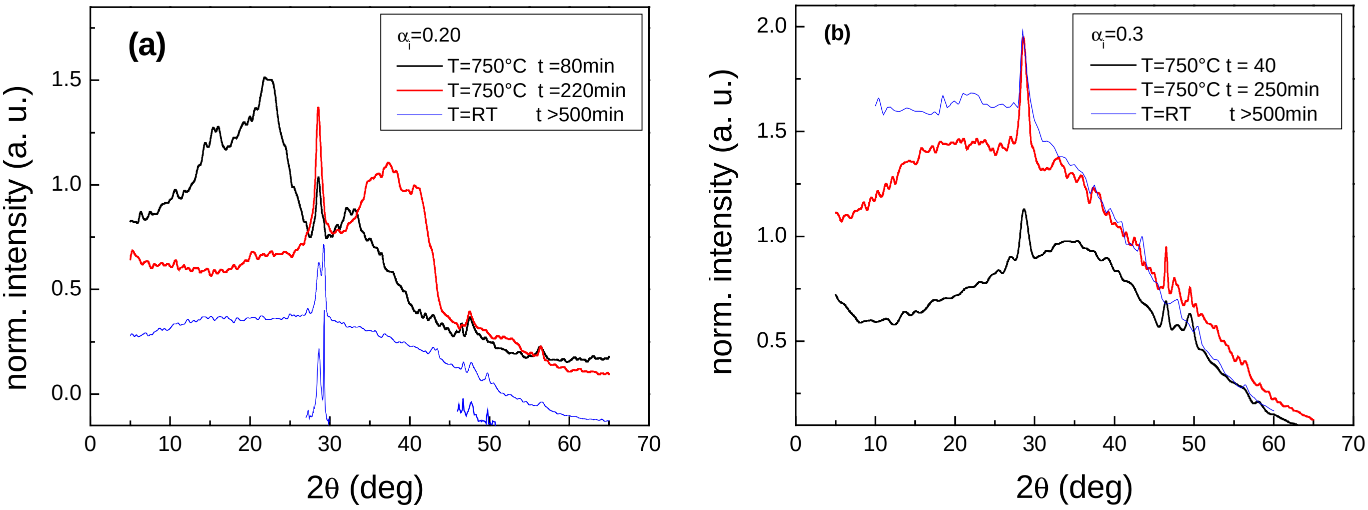

Figure 2: Surface (a) and bulk (b) amorphous scattering profiles at the high and low temperature measured over 8h. The sharp peaks refer to the Si(111) positon: note the double peak structure at the surface. this may be indicate different strain states.

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