ESRF	**Experiment title:** In-situ study of nanopatterning on GaSb surfaces by low-energy ion beam sputtering	**Experiment number**: SI-1056
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Report:

Ion beam erosion has been shown to be a promising technique for the formation of nanostructures on a surface. In particular, ripple morphologies and self-organized regular arrays of dots can be produced on the irradiated surface. The ripple morphology is attained for off-normal ion irradiation whereas nanodots are obtained under normal incidence. In order to understand and control the production of nanodots on GaSb by ion erosion, we have studied the temporal evolution of the surface morphology during the irradiation process. From this measurement we obtain, for the first time, detailed information about short and long sputtering time behaviour of the pattern and their dependence on ion energy in the range between 100 eV and 1000 eV. The time evolution of the pattern has been seen to strongly depend on the ion beam divergence. Also, we evidenced the thermal stability of the pattern for annealing temperatures up to 650 C, close to GaSb melting point at 711 C.

For this study, we used a compact portable UHV chamber with a 360 degree Be window that has been recently implemented on ID01 Beamline. This chamber was mounted on the "Huber tower" installed at the centre of the goniometer, allowing for x, y and z translations as well as two perpendicular tilt angles for sample alignment. The chamber is equipped with a turbo-molecular pump (base pressure 10^{-7} mbar) and with a 3 cm diameter Kaufman ion source (supplied by Veeco) that delivers a high current density (0.1-1 mA/cm^2) in a wide energy range (100-1300 eV). We have studied the formation and temporal evolution of nanodots on a clean GaSb surface during ion sputtering at 100, 450 and 1000 eV with *in-situ* Reflectivity, Grazing Incidence Small Angle X-ray Scattering (GISAXS) and Grazing Incidence Diffraction (GID) measurements. Figures 1a and 1b show, respectively, the reflectivity and the GISAXS measurements performed during the ion erosion process at 450 eV. One can follow the surface evolution in the first minutes of sputtering, and its stabilization for longer erosion times.

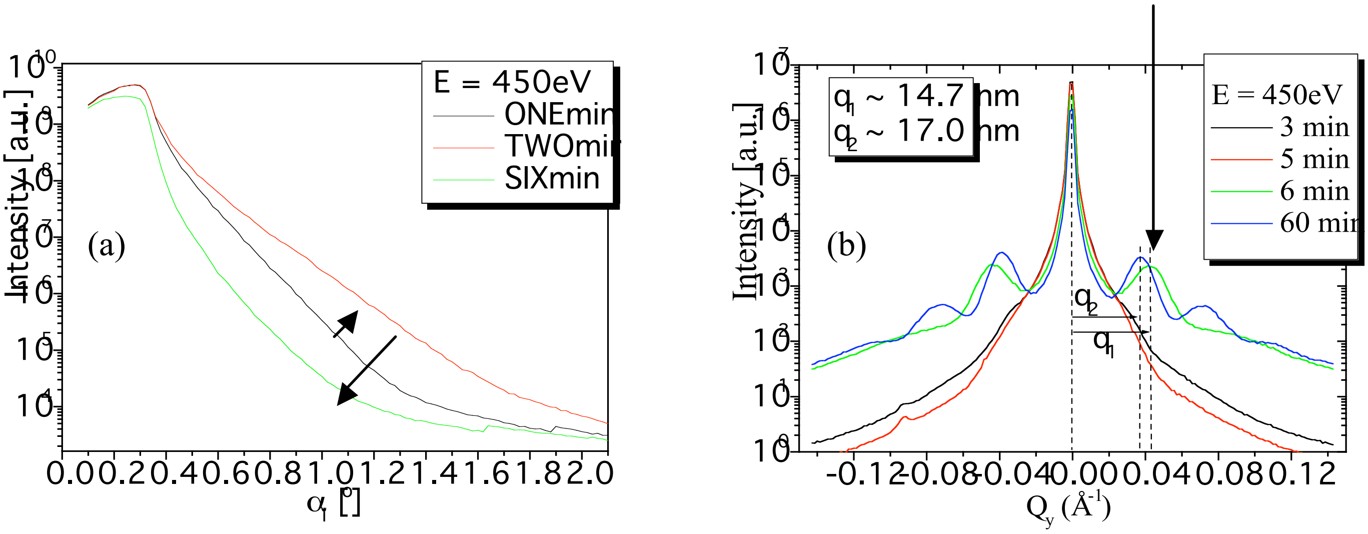

Fig. 1 : ...

After two minutes of sputtering at 450 eV, the reflected intensity shows an increase, indicating a smoothening of the surface. This can also be seen from fig.1b where the diffuse scattered intensity level measured after 5 minutes of sputtering is lower if compared with the measurement after 3 minutes of sputtering. This behaviour is totally new and unexpected on the basis of the last theoretical developments that describe the nanodots formation (S. Facsko et al., PRB 69 (2004) 153412 ; M. Castro et al., PRB 94 (2005) 16102). Previous works show that one needs ion beam energy in the MeV range to smoothen a surface (D.K. Goswami et al., PRB 68 (2003) 33401). After 6 minutes sputtering, the reflected intensity has fallen below the starting value, indicating the beginning of a roughening process. This roughening corresponds to the appearance of a periodic pattern, characterised by a specific wavelength, on the surface. This can be seen from the GISAXS signal, where two correlation peaks appear at a distance +/-q_1 from the specular position, (indicated by the black arrow in fig.1b), which corresponds to a wavelength of 15 nm on the surface. This value does not agree with previous AFM measurements quoting 40 nm for 500 eV ion beam energy (T. Bobek et al., PRB 68 (2003) 85324). This discrepancy may be due to the different ion sources used in the two studies. For long sputtering time (60 min), the correlation peaks shift to lower q values, indicating a slight increase of the wavelength (~17 nm). Also a second correlation peak appears at higher q, indicating an improved short-range order of the nanodots, in agreement with simulations (S.Facsko et al., PRB 69 (2004) 153412). The intensity in the correlation peaks starts to decrease for an annealing temperatures of 650 C, indicating a very good stability of the pattern.

Similar results have been found using different ion energy. Hovewer, the temporal evolution of the morphology, i.e. the onset of the nanopatterning and the stabilisation of the nanostructures, strongly depend on the ion energy. In particular longer sputtering times are needed to stabilise structures obtained with lower ion beam energy. This wealth of information will certainly be taken into account for a better modeling of the formation process. Finally, the short-range ordering of the nanodots has been found to be strongly dependent on the ion beam condition, which we have optimized when using the filament neutraliser at the end of the ion gun (more parallel beam).