| ESRF | **Experiment title:** ***In-situ*** **investigation of the anisotropic scaling properties of nanorippled Si surfaces during low energy ion sputtering** | **Experiment number**: SI-1591 |
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**Report:**

The purpose of this experiment was the *in-situ* investigation of the evolution and the anisotropic dynamic scaling behavior of ion-induced ripple morphologies during oblique high-fluence ion sputtering of silicon surfaces. For this, a high-flux Kaufman type ion source has been mounted onto the vacuum chamber of the beam line. After some initial experiments, e.g. to determine the erosion rate of the silicon surface under the present experimental conditions, Si(100) samples have been bombarded with 500 eV Ar ions at different angles of incidence $\theta_{ion}$ = 65°, 67°, and 69° with respect to the surface normal. The highest fluence applied was $\Phi = 1.6 \times 10^{19}$ cm$^{-2}$. The setup of the vacuum chamber and the high heat load introduced to the sample by the ion flux made it necessary to stop the sputtering and realign the sample prior to each measurement. The surface morphology has been characterized by grazing incidence small angel X-ray scattering at fixed incident angle ($\theta$ = 0.3° with respect to the surface) and energy (E = 17.5 keV). The diffuse scattering has been monitored using a cryogenically cooled charge coupled device camera with 1024 x 256 pixels and a pixel size of 19 µm.

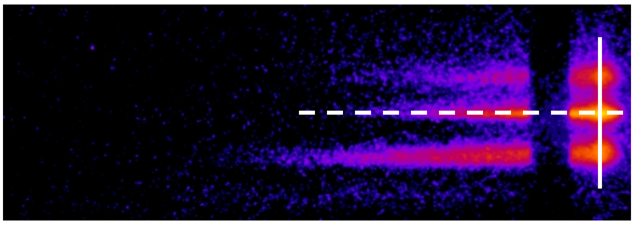

**Fig. 1**: Scattering diagram of the Si(100) surface recorded after 240 s of sputtering at $\theta_{ion}$ = 67°.

Fig. 1 shows a scattering diagram recorded after 240 s of sputtering at $\theta_{ion} = 67°$. The one-dimensional power spectral density (PSD) functions $S_{n,p}(k_{n,p})$ in the direction normal and parallel to the ion beam have been determined from cuts of the scattering diagrams along the dashed and the solid white lines in Fig. 1, respectively. The $S_{n,p}(k_{n,p})$ functions at different fluences for 67° incidence are shown in Fig. 2(a,b). In both directions, a power law scaling regime develops with increasing fluence. Power law fits to the $S_{n,p}$ curves yield roughness exponents $\alpha_n = 0.72$ and $\alpha_p = 0.42$ in the direction normal and parallel to the ion beam.

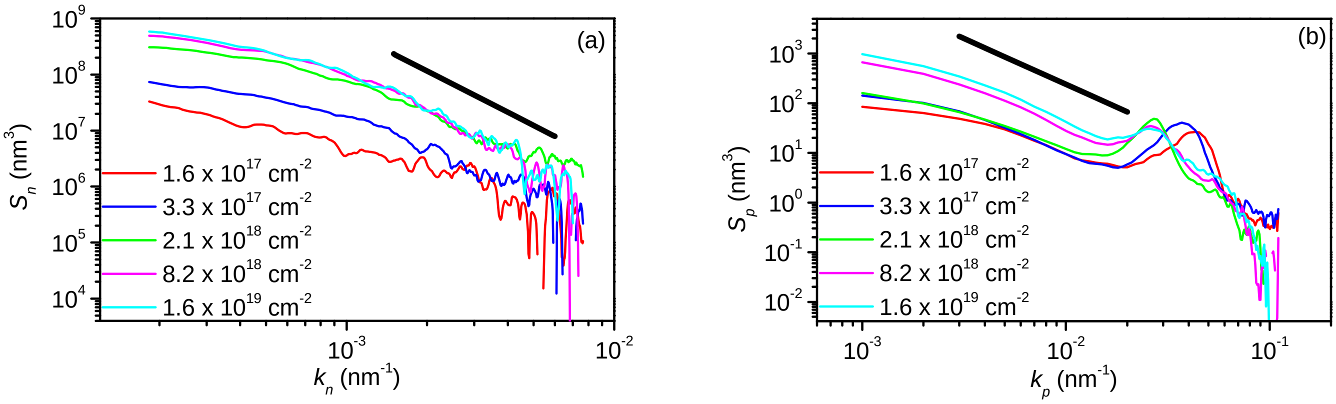


**Fig. 2**: PSD functions obtained from the scattering diagrams for 67° ion incidence in the direction normal (a) and parallel (b) to the ion beam. The thick lines correspond to roughness exponents $\alpha_n = 0.72$ (a) and $\alpha_p = 0.42$ (b), respectively.

In Fig. 3(a), the roughness exponents for the three incident angles under investigation are depicted. At $\theta_{ion} = 65°$, a strong scaling anisotropy is observed with $\alpha_n = 0.81 \pm 0.05$ and $\alpha_p = 0.04 \pm 0.02$. At $\theta_{ion} = 67°$, however, this anisotropy is substantially reduced, and at $\theta_{ion} = 69°$, the scaling of the surface becomes isotropic with $\alpha_p \approx \alpha_n = 0.46 \pm 0.07$.

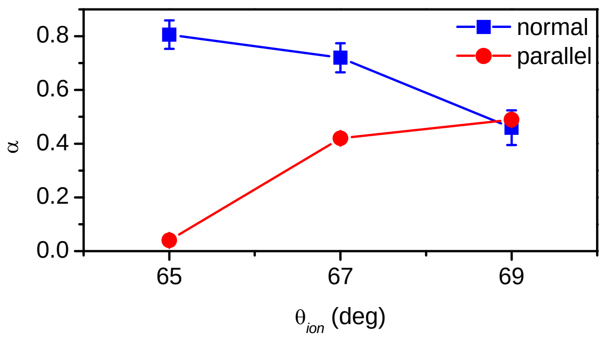


**Fig. 3**: Roughness exponents in the direction normal and parallel to the ion beam, respectively, for the three different incident angles.

This observed transition from strong anisotropic to isotropic scaling can be interpreted in terms of a phase transition with a critical point at $\theta_{ion} = 69°$. This critical point then can be considered as an isotropic metastable fixed point, and small deviations of the incident angle will cause the system to leave this fixed point and get attracted to another anisotropic fixed point. The fact that the observed transition from isotropic to anisotropic scaling is rather continuous indicates that the system (for $\theta_{ion} \neq 69°$) has not yet reached its final state and the determined scaling exponents are not the asymptotic but rather some intermediate ones.