ESRF	**Experiment title:** Quantitative analysis of nanoripple patterns by 3D GISAXS mapping	**Experiment number**: 02-01-831
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Report:

The goal of this proposal was to perform 3D GISAXS mapping experiments to study the influence of various parameters on the formation of nanoripples produced by Xe^+-ion beam sputtering (IBS) of amorphous alumina surfaces. These experiments were motivated to provide new quantitative information helping to understand the evolution of dielectric surfaces nanopatterned by IBS. Samples were prepared at Institut P' (CNRS Poitiers) by deposition of amorphous alumina layers onto Si substrates followed by IBS at oblique incidence by a defocused Xe^+ ion-beam with the fluence and flux being fixed at $\Phi = 6.8\times10^{16}$ ions/cm^2 and $\phi = 2\times10^{14}$ ions/cm^2/s, respectively. Samples sputtered at different energies (E = 500 eV, 750 eV, 1000 eV, 1250 eV, 1500 eV), different temperatures (T = 300 K, 400 K, 500 K, 600 K), and different angles of incidence (θ = 50°, 55°, 60°, 65°) were investigated. GISAXS measurements were carried out at 9.8 keV with the GISAXS chamber of the D2AM beamline using an angle of incidence α_i = 0.25° close to the critical angle of total reflection. In order to obtain full 3D maps of the GISAXS intensity, series of 2D GISAXS patterns were collected with a CCD detector for different azimuthal angles ω (from ω = -10° to ω = 30°) by increments of 1° (ω = 0° corresponds to the direction perpendicular to the projection of the Xe^+ beam).

As typical examples, Figs. 1(a)-1(c) show 2D GISAXS patterns (ω = 0°) collected from samples sputtered at E = 500 eV, 1000 eV, and 1500 eV. Vertical streaks, whose position and shape depend on the sputtering parameters, were systematically observed as a result of lateral correlation between unidirectional ripples oriented perpendicular to the incident ion beam. The experimental data were analyzed with the FITGISAXS package using the method described in Ref. 1 to determine statistical and quantitative information about the morphology (period, length, height, and asymmetry) and organization (short-range order) of the ripples. The main results are summarized below:

- a minimum for the ripple period associated with an optimum for the ripple organization is observed when ion sputtering is performed at θ = 60°;
- the ripple period increases linearly with temperature and ion energy;

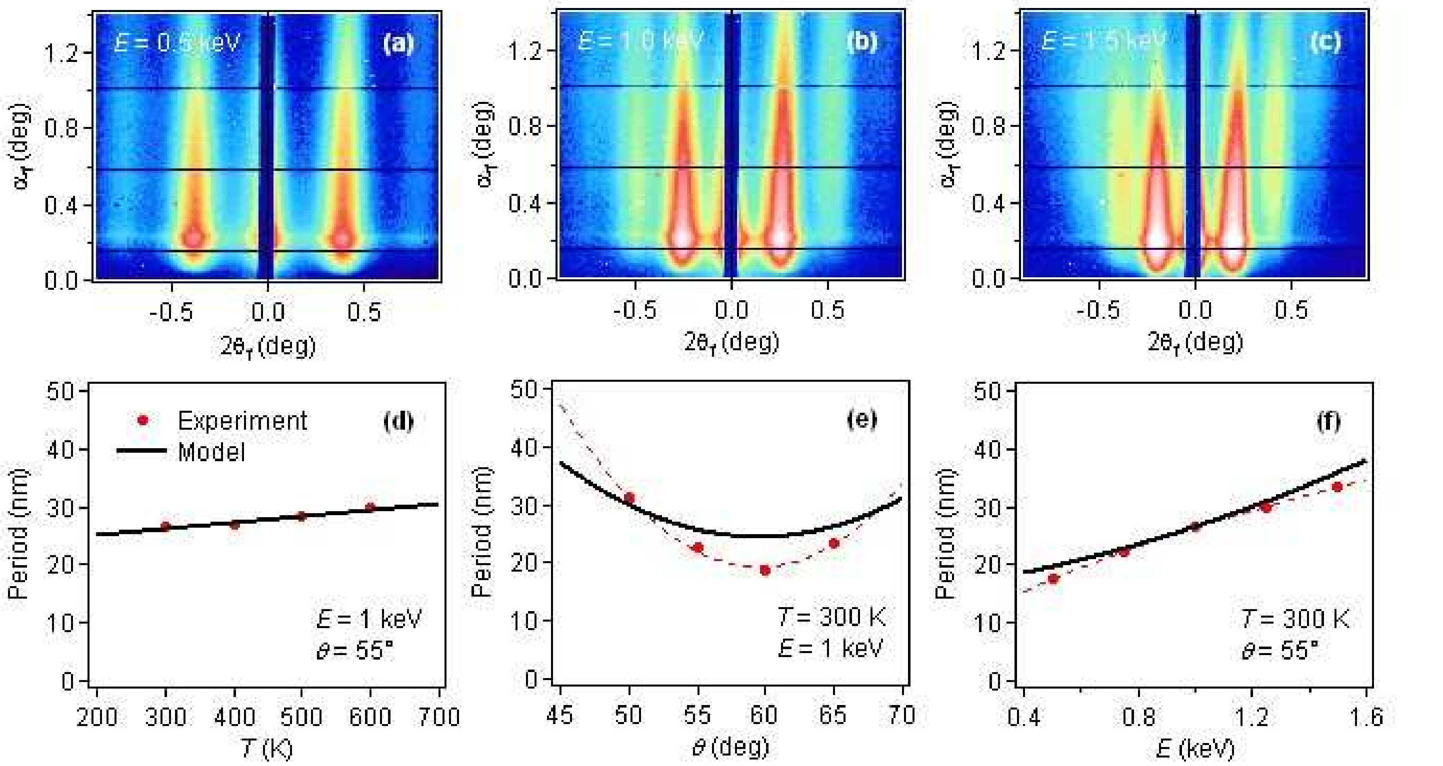

Fig. 1: (a)-(c) GISAXS patterns of amorphous alumina layers sputtered with Xe^+ ions at different energies *E* (*T* = 300 K, θ = 55°). (d)-(e) Dependence of the ripple period on the substrate temperature *T*, incidence angle θ, and ion energy *E*. The solid lines represent the dependence of the period predicted by a linear continuum model assuming an interplay between a surface curvature dependence of the sputtering yield and an ion-induced surface relaxation mechanism.

- the ripple organization is improved at low temperature and is independent of the ion energy;
- the ripple height increases with the incidence angle and energy, while it decreases with the temperature;
- the asymmetry of the ripple profile increases with the incidence angle, while it decreases with the temperature, and is independent of the ion energy.

The dependence of the morphological parameters was compared to simulations using linear continuum models [see Figs. 1(d)-1(f)]. Our results show that the period of the ripples can be fairly well reproduced by assuming an interplay between a surface curvature dependence of the sputtering yield (that gives rise to surface roughening) and an ion-induced surface relaxation mechanism (that causes smoothing). However, evolution of the ripple shape (height and asymmetry) and organization show that nonlinear effects also contribute to the dynamic of ripple formation during low energy IBS of amorphous alumina surfaces.

Additional GISAXS experiments were performed in order to study the organization of Ag nanoparticles grown by glancing-angle deposition on such nanorippled alumina surfaces. The results state unambiguously that a replication of the lateral order between ripples is achieved [2], leading to the formation of nanoparticle chains with a strong plasmonic coupling.

[1] D. Babonneau et al., Phys. Rev. B 85 (2012) 235415.
[2] S. Camelio et al., Nanotechnology 25 (2014) 035706.