ESRF	**Experiment title:** In situ GIXD and GISAXS study of the self-organized growth of Co nanostructures on Ag(001) patterned by an emerging 2D misfit dislocation network	**Experiment number**: Si-802
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Manufacturing ordered magnetic nanostructures on a substrate is of great interest both for applied and basic research. A promising method is based on self-organized growth (SOG) on a patterned substrate. Growth on substrates with a regular network of preferential nucleation-growth sites, such as 2D arrays of buried dislocations emerging at the surface, can result in a well ordered bidimensional lattice of nanometric size dots[1, 2]. A substrate of Ag(001)/MgO(001) is a good candidate, since the 3% lattice parameter mismatch produces a well-ordered square network of misfit dislocations at the interface [3]. In addition, Co is an important ferromagnetic material, which is immiscible with non-magnetic silver.

Our measurements during the growth of Co on Ag/MgO thin film can be divided into two parts: the manufacture of the Ag/MgO film and the Co clusters growth. They were investigated by the combination of three *in situ* techniques: Grazing Incidence X-ray Diffraction (GIXD), X-ray Reflectivity (XR) and Grazing Incidence Small Angle X-ray Scattering (GISAXS), which *in situ* application has been recently developed in our group [4,5], and by *ex situ* AFM.

Ag/MgO Film.

Nucleation-growth atop dislocation lines is predicted to be efficient only if the film thickness is of the order or smaller than the period Λ of the dislocation network (Λ ~100 Å) [6]. The first step was thus to obtain high quality ~100 Å-thick Ag(001) films on MgO(001) with a well ordered misfit dislocation network at the interface, but also with a high crystalline quality, large terraces (much wider than Λ), low roughness, and good thickness homogeneity. This is not trivial since thermodynamically and kinetically, the growth of Ag on MgO(001) is three-dimensional, and thus fairly thick (~1500 Å) buffers are needed to get a continuous film and next re-order the dislocation network by annealings.

High quality MgO(001) substrates were prepared according to our standard procedure [7] of Ar+ bombardment at 1500°C. Ag buffers (1500 Å-thick) were next deposited at room temperature and annealed by steps up to 670°C to get a bidimensional film as flat as possible with a well ordered dislocation network at the interface [3]. Thinning by Ar+ bombardement (IB) was first tried *ex situ* on a dedicated setup at LETI, but resulted in very inhomogeneous buffers. We then performed the Ar+ bombardment thinning *in situ*, while monitoring the conditions by anti-Bragg GIXD measurements, and the thickness by XR. Measurements of the intensity and FWHM of the Ag(110) anti-Bragg peak at different temperatures during IB have shown that the initial quality of the surface of the thick Ag film can be maintained during IB, and even improved. Indeed, at T~ 300 °C the diffusion of Ag smoothes the surface (Fig. 1) whereas at higher or lower temperature the surface roughens. The oscillations observed on the intensity (inset of Fig. 1) accurately yield the abrasion rate (~5 ML/min), which allows a precise determination of the thickness planed after bombardment.

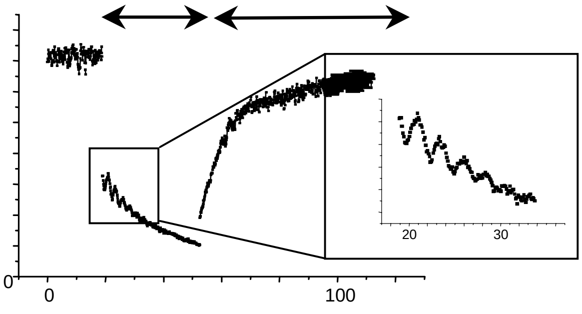

Fig. 1 Evolution of the intensity of the Ag(110) anti-Bragg before (left) and during Ar+ ion bombardment and at different substrate temperature (200 and 300 °C) Inset : Oscillations of the intensity corresponding to layer/layer ablation.

Several Ag(001)/MgO(001) very thin films (50-100 Å average thickness) were thus prepared with a very well ordered interfacial dislocation network, and also large (001) terraces (~1000 Å average size, i.e. 10 times the network period). The presence of a well ordered dislocation network at the interface was checked by diffraction measurements (GIXD) along (*hh0*) and (*h00*) or equivalent directions and the good crystallinity of the film by azimuthal scans on the (220) Ag Bragg peak. The Ag film was also characterized by GISAXS (Fig. 2) along the (*h00*) and the (*hh0*) directions. Scattering rods from the array of buried dislocations have been observed for the first time in GISAXS as far as we know. Comparison between the data obtained by the two techniques should give a better insight into the whole morphology of the film.

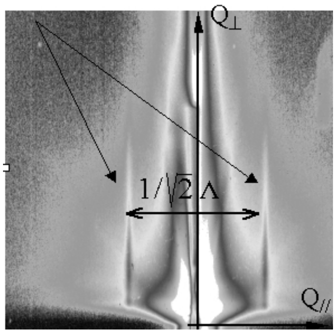

Fig 2 GISAXS image (along h00) of the dislocation network

However, XR measurements revealed that the films are extremely inhomogeneous in thickness.

Co/Ag/MgO.

Co deposition was performed at different temperature between 300 and 600 K with the hope to find the optimal conditions of diffusion and nucleation to make possible the self-organized growth of Co on this substrate, *i.e.* in order that Co atoms get enough mobility to reach nucleation-growth sites but not too much to ‘jump over’. Actually, we were not able to find the optimal temperature conditions yielding a better organization of Co at a macroscopic scale on thin films compared to growth on a few µm thick Ag/MgO buffer.

We believe this is due in part to the inhomogeneity of the Ag films, which has to be improved, and could also be due to the fact that the right conditions for SOG might be slightly below room temperature. As reflectivity suggested, and as later confirmed by *ex situ* AFM, after the ion bombardment process, only a portion (~30 %) of the deposit remains flat with its interfacial dislocation network; the remaining reaching its equilibrium shape of large clusters much thicker than the average, and onto which no organized growth is expected. The GISAXS measurements then result in the superposition of patterns from very different levels of organization, and thus do not reveal organized growth. A simple way to solve the problem could just be to cool down the sample rapidly after the IB, which was not done, and also to stop the IB slightly earlier, for thickness ~150 to 200 Å.

Nevertheless some aspects of the growth of Co clusters have been studied. GISAXS measurements (Fig. 3) show extremely flat clusters (~3 atomic planes), which, although not ordered, are separated by a preferential distance of ~100 Å, which is the distance between dislocations. A more detailed analysis should yield a precise determination of the morphology of the aggregates.

From GIXD measurements (Fig. 4), we have deduced the cube/cube epitaxial relationship between Co and Ag : $[100]_{Co}//[100]_{Ag}$ and $[001]_{Co}//[001]_{Ag}$. We have also found that Co aggregates are nearly completely relaxed from the very beginning of the growth because of the large (13%) lattice parameter mismatch.

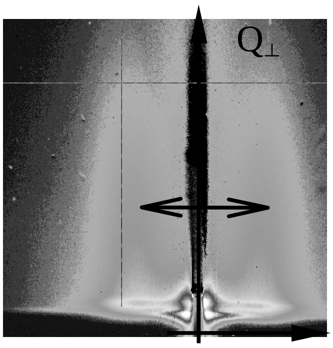

Fig. 3 Gisaxs Image of 4 Å of Co deposited. D~10nm.

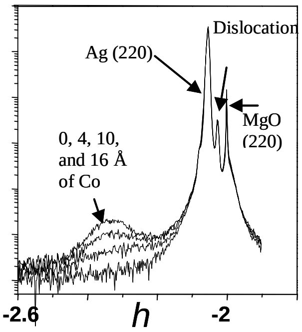

Fig.4 : (*-h-h0)* scan for 0, 4, 10 and 16 Å of Co deposited on Ag thin film on MgO(001).

To pursue this long term study, much efforts must be done on the macroscopic scale homogeneity of the Ag/MgO film. The deposit of Ag and the Ar+ ion bombardment will be performed while continuously rotating the sample. For the Co deposit, new experiments at lower temperature should be tried in order that Co atoms get less thermal energy to jump over the energy barrier of the surface strain. In addition, the samples will be cleaved after IB thinning and before Co deposit, in order to keep only the central part of the samples, believed to be much more homogeneous, for further measurements. A new proposal will be submitted in order to go beyond these first results.

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