Experimental Report
"Self-assembling of metal nano-particles on nanostructured MgO film"
(Proposal Code 32-03-660) BM32, ESRF, 21-27 November 2007

The growth of MgO on Mo(001) was studied by Grazing Incidence X-ray Diffraction (GIXD) at BM32. We have observed a network of regularly spaced misfit dislocation at the MgO/Mo interface due to the lattice mismatch between the substrate and the overlayer. The influence of this coincidence lattice on the successive growth of Au thin layers has been investigated by GISAXS.

Experimental

The substrate used was a Mo(001) single crystal prepared by repeated cycles of sputtering (Ar^+, 600 eV) keeping the substrate at 900°C. On the clean Mo surface we have evaporated MgO films by reactive deposition of Mg in O_2 partial pressure of $1x10^{-7}$ mbar at room temperature (MgO deposition rate = 1.22 ML/min).

The GIXD measurement was performed at 18 KeV of photon energy while the incidence angle was set at 0.166°. During the experiment the evolution of the MgO lattice parameter and the formation of an interfacial dislocation network have been studied as a function of the film thickness and of the annealing temperature.

Moreover we have evaporated thin Au deposit on 4 ML of MgO at a rate of 0.03 Å/min and the influence of the interfacial dislocation network on the Au growth has been investigated by GISAXS.

In the following the relative lattice units (r.l.u.) are always referred to the Mo reciprocal space.

MgO film structure

The evolution of the Bragg peaks along the (h,0,0.03) and (h,h,0.03) directions as a function of the MgO thickness of the as-grown films were recorded in order to study the relaxation of the in-plane lattice parameter of the MgO film during deposition (Fig. 1).

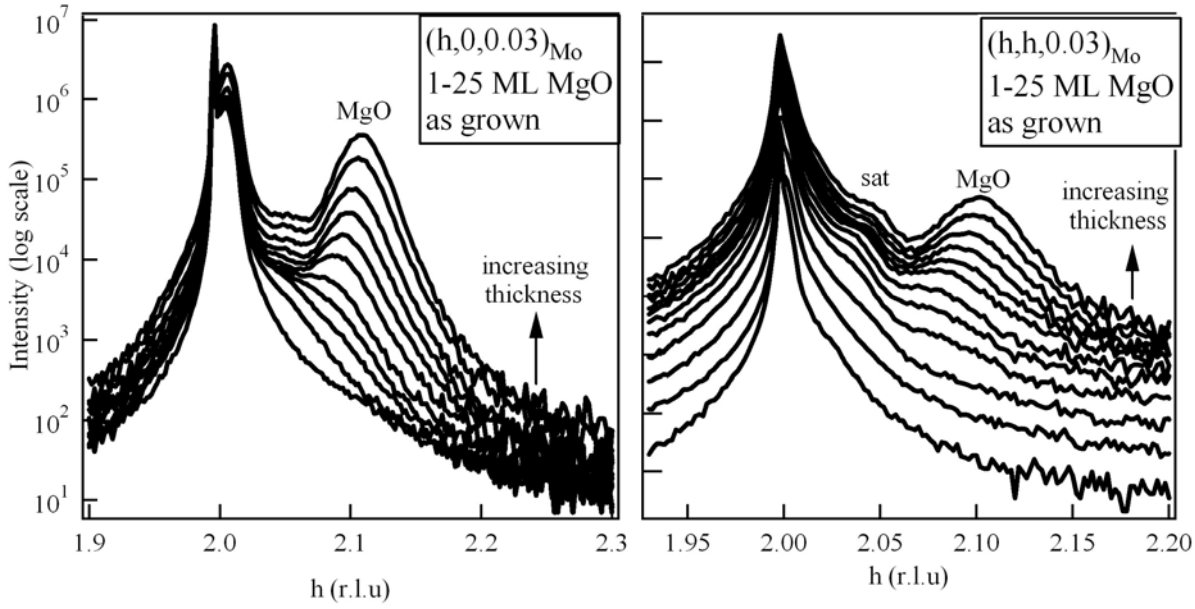

Fig. 1:Radial scans along the (h,0,0.03) direction around the (2 0 0) Mo Bragg peak (left panel) and along the (h,h,0.03) direction around the (2 2 0) Mo Bragg peak (right panel) as a function of MgO thickness on the as deposited films.

From these scans we observe the appearance of the MgO shoulder placed at 2.05 r.l.u. at about 2 ML and the successive dispersion to higher h values as a function of the MgO thickness. Furthermore it can be clearly observed the presence of the dislocation pattern peak in between the

Mo and the MgO Bragg peaks on the (h,h,0.03) scan. The values of the MgO in-plane lattice parameter as a function of thickness are shown in Figure 2. It is important to notice that the evolution of the in-plane parameter is not the same for the two azimuths: this result is ascribed to a non-isotropic plastic deformation of the film lattice due to the presence of the interfacial misfit dislocations. The same measurements have been repeated for MgO films that have been annealed at 800°C for 10 min after growth. The thermal treatment accelerates the relaxation of the MgO in-plane parameter toward the bulk value (see figure 2) and drastically improves the order of the dislocation network. The difference between the two analysed azimuths is reduced with respect to the as-grown sample but not cancelled.

To study the evolution of the MgO structure as a function of the annealing temperature, a film of 7 ML of thickness has been chosen and annealed at subsequent steps of 100°C for 10 min starting from 400°C (Fig. 3). The MgO peak moves toward higher r.l.u., indicating a further relaxation with increasing temperature, while the dislocation peak appears more evident and after the annealing at 800°C several orders are clearly visible.

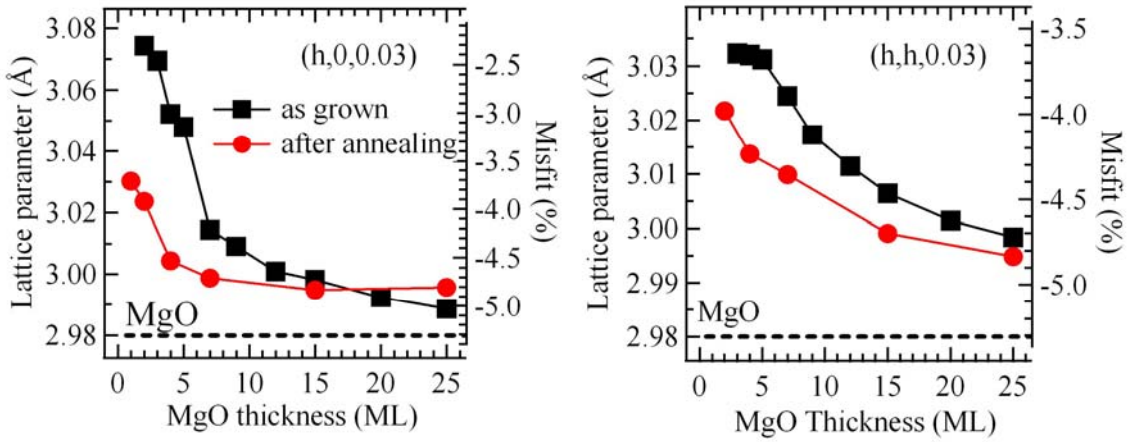

Fig. 2: Evolution of the in-plane lattice parameter along the two main azimuths of MgO film on Mo(001) as a function of thickness before (black squares) and after annealing at 800°C (red circles).

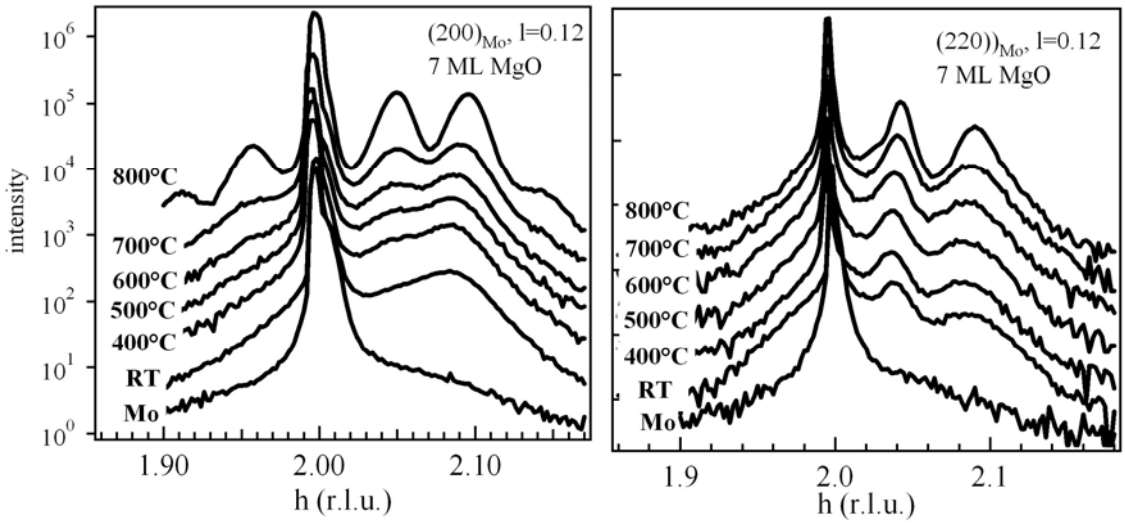

Fig. 3: Radial scans along the (h,0,0.03) around the (200) (left panel) and (h,h,0.03) around the (220) (right panel) Mo Bragg peaks as a function annealing temperature on a 7 ML MgO film.

Analysis of the dislocation network

In all measured radial scans satellite peaks between the Bragg peaks of Mo and MgO are present. These peaks arise from the presence of an ordered distribution of misfit dislocations formed at the interface between the Mo substrate and the MgO overlayer, induced to relax the strain. In Fig. 4 scans around the (200), (220), (400), (440) and (600) Mo Bragg peaks are shown for an L value of

0.12 r.l.u. (that maximize the contribution from the satellite peaks) measured on a 15 ML thick MgO film on Mo(001) after annealing at 800° C. Comparing the positions of these satellites with respect to the Mo peak with a model of the reciprocal space expected for the coincidence lattice between MgO and Mo, we can have information concerning the orientation of the dislocation pattern. This is a square network aligned along the Mo[100] (parallel to MgO[110]) direction. Quantitative information can be obtained about the periodicity, that decreases with increasing MgO thickness and with annealing temperature. It corresponds to 66 Å at 2 ML, to 63 Å at 25 ML.
On a MgO film of 4 ML thickness, displaying a well defined dislocation network, we have evaporated 0.5 ML of Au. The GISAXS patterns recorded before and after the Au evaporation have not revealed a correlation between the spatial distribution of the deposited gold and the underlying dislocation network.

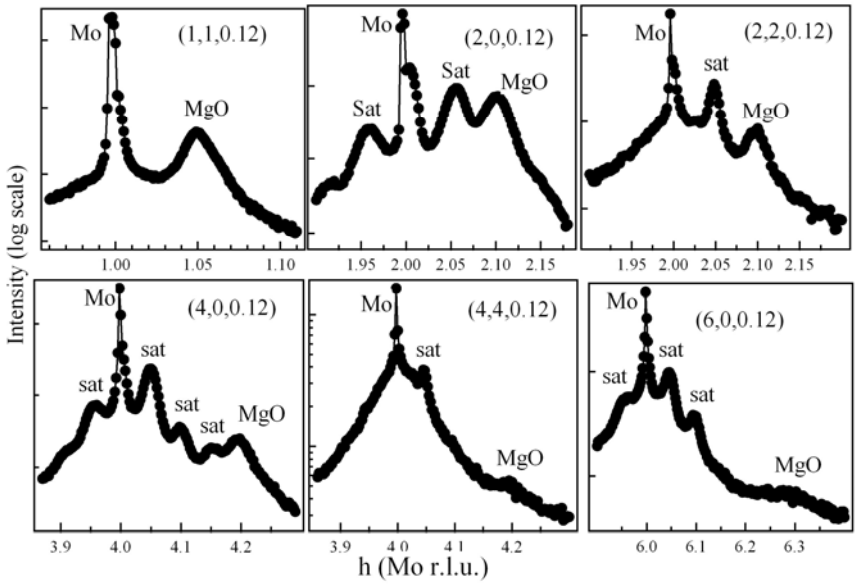

Fig. 4: Radial scans on a 15 ML MgO film on Mo(001) annealed at 800°C. Diffraction satellites due to the interfacial dislocation network are evidenced.