ESRF	**Experiment title: The $Ni_{81}Fe_{19}$/NiO(111) interface during its formation – structure and morphology by grazing incidence x-ray diffraction (GIXD)**	**Experiment number:** SI-470
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Report:

The first GIXD experiments on this interface (SI-337) showed that the system is very interesting and worth future investigations. In particular the improvement of the crystalline quality of the film by a 500°C annealing led to a reactive interface. A spinel interfacial compound has been identified by HRTEM after the experiment. Since this one is ferrimagnetic it prevents the exchange coupling between the permalloy (Py) film and the NiO(111) substrate. The present experiment aimed at describing more precisely the growth of Py on NiO(111) with respect to the growth temperature and to the chemical nature of the interface.

The $Ni_{81}Fe_{19}$ (Permalloy)/NiO(111) interface is involved in spin valves sensors. NiO(111) is the antiferromagnetic (AF) layer used to magnetically pin the ferromagnetic (F) layer. Although industrial devices are already in production, the exchange coupling phenomenon appearing at the interface, which makes the devices functioning, is still not yet well understood. With additional structural and magnetic measurements, we hope to be able to correlate the crystallographic structure of the interface and its magnetic properties. From this correlation one could expect improvements of the properties of spin-valves. A fine description of the interface is only possible by use of single crystals that have a GIXD compatible quality.

Our single crystal preparation method has been improved. NiO(111) single crystals with a surface mosaic spread better than 0.03° and terraces sizes of about 2000 Å are routinely obtained. Such a quality is ideal for performing accurate quantitative measurements. All substrates were found to be p(2×2) reconstructed. The large scattered signal along the crystal truncation rods (CTRs) of the substrate shows the very good quality of the physical surface.

Two new growth temperatures were investigated (260°C and 300°C). The surface temperature of the substrates was measured and controlled using an IR pyrometer.

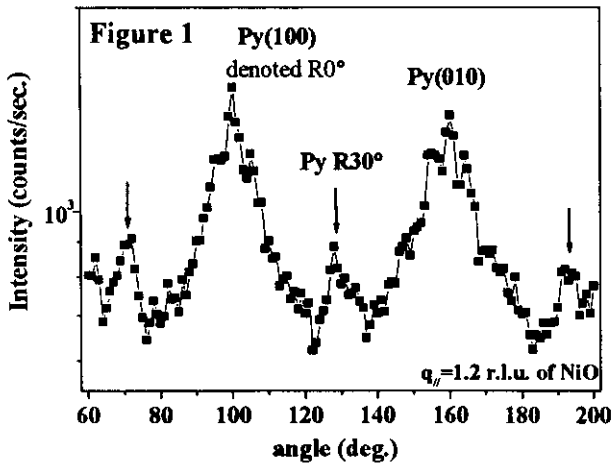
The cleanliness of the surface was checked by Auger Electron Spectroscopy (AES). The main contaminants found were C and Ca. While C can be completely removed by an oxygen annealing (10 min. at 800°C under $p(O_2)=10^{-5}$ Torr), Ca segregates at high temperature. However, the residual Ca corresponds to very small sub-monolayer (ML) quantities without any effect on the structure of the reconstruction. The Py film was obtained by co-evaporation from two calibrated sources (using a quartz balance). Cumulative deposits were performed.

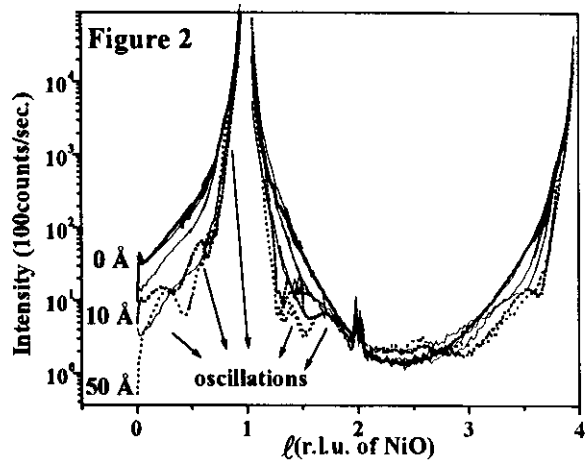
The growth of the Py film was investigated in each situation. As expected from the previous experiment (**SI-337**), 3D growths with a rapid coalescence and a rather good crystallographic quality are obtained. At some particular thickness (θ_{Py}) the Py film has been fully quantitatively characterized. The initial NiO(111) surface reconstruction was fully characterized by quantitative measurements of the in-plane reconstruction peaks. The same was done at each θ_{Py} allowing a direct monitoring of the surface coverage since metallisation un-reconstruct the surface. The out-of-plane scattering from the Py film was also carefully investigated showing that for $\theta_{Py}<1$ML islands of at least 3 ML heigth exist. It also allows to determine the quantitative proportion of each stacking present in the Py film.

The Py film is always mainly FCC (the stacking continuing the NiO(111) one, ~80%) with twins (i.e. a FCC structure rotated by 60°: R60°). Structures rotated by 30° and 90° were found in small quantities (fig.1). The relative proportion of the different variants will be deduced from corrected integrated intensities of characteristic Bragg peaks of each structure. For each structure accurate values of the mosaic spread and diffracting domain size will be deduced from the quantitative measurements. Qualitatively it appears already that, by increasing temperature, the film crystallographic quality improves but the interface become reactive above 260°C. At 300°C the interface presents already a small amount of diffuse spinel layer (peaks at ~ half integer values in the reciprocal space) with a poor crystallographic quality (the peaks are 5° large).

A quantitative characterization of the oscillations observed along NiO Crystal Truncation Rods (CTRs) was performed at each θ_{Py} (figure 2 :scan along the [10ℓ] out-of-plane direction). Rocking scans were performed all along two rods. These data are expected to lead to an improved understanding of the formation of the interface.

Transmission electron microscopy (high resolution and determination of diffusion profiles by chemical analysis in electron energy loss spectroscopy) will complete the understanding of this rather complex interface.





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