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## **Experimental Report:**

The magnetic coupling across the interface between a ferromagnet (FM) and an antiferromagnet (AFM) gives rise to several interesting phenomena, usually summarized under the term "exchange bias". During the past decades, extensive efforts have been devoted to experimental and theoretical investigations of these phenomena, in part due to their technological relevance in magnetoresistive applications. Although the exchange bias is now globally quite well understood, many questions still remain open. For example, it is not clear whether or not the amplitude of the exchange bias depends on the crystalline quality, structural properties and/or on the roughness of the FM/AFM interface.

This work is part of a long-term research project dedicated to the study of exchange bias and related phenomena in Fe/NiO/Fe(001) trilayer structures. Using Spin-Polarized Low-Energy Electron Microscopy (SPLEEM), we showed that incorporation of a NiO layer within a simple Fe multilayer structure reduces the minimal size of stable magnetic domains in the top Fe layer by one order of magnitude compared to Fe films grown without AFM spacer layer. In addition, X-ray PhotoEmission Electron Microscopy (X-PEEM) carried out at ELETTRA (Nanospectroscopy beamline) showed that the NiO film has large AFM domains with twofold symmetry, which mimic the magnetic domain structure of the bare Fe substrate. To explain the unusual magnetic domain structure of the top Fe layer, we proposed a model based on the presence of a random exchange field at the Fe/NiO interface resulting from magnetic/structural roughness. FM-NiO based systems have been extensively studied these last years using spectroscopy experiments (XAS, XMCD, XPS..). All these studies point towards the important role of the interface structure and morphology, and especially the presence of uncompensated spins at the interface due to the Ni reduction. The formation of NiO-FM mixed interface is also believed to be decisive for the exchange bias phenomenon. However, only few atomically resolved structure analyses have been carried so far, and the subject suffers from the lack of structural characterization. In these experiments, we were interested in completing our magnetic data set with structural information.

Three samples were prepared with the following sequence:

(1) Fe(001), intentionaly oxidized Fe(001), NiO deposits of 25 min and 46 min, followed by 20 min and 18 min Fe deposition.

(2) Fe(001), intentionaly oxidized Fe(001), NiO deposits of 1, 2, 4, 8, 16, 32 and 52 min.

(3) Fe(001), intentionaly oxidized Fe(001), evaporation of a thin Au layer and NiO deposits of 1 min 20 sec and 20 min.

For experiments (1) and (2) complete measurements were performed including specular reflectivity (see figure), in-surface-plane linear scans and rocking scans on all peaks of interest. Linear scans along the [10L] and [20L] directions, as well as integrated rocking scans were also performed. The NiO film grows epitaxially with a c(2x2) epitaxial relationship with respect to the Fe substrate, but strongly increases the surface roughness, even for ultra-thin layers (a fraction of a NiO monolayer is enough to induce dramatic changes in the surface roughness). The subsequent Fe layer grows epitaxially but experiences a clear residual stress. The mosaic spread and domain size could be determined in each case.

Experiment (3) was a test situation to show that a significant reduction of the surface roughness occurs when a monolayer-thick Au layer is deposited on the Fe(001) substrate prior to the NiO growth (see figure). The remaining beamtime only enabled specular reflectivity measurements, but this last situation clearly deserves further measurements.



Figure: (left) Reflectivity measurment during *in situ* preparation of a NiO / Fe(001) bilayer structure. (rigth) Reflectivity measurement corresponding to the growth of a NiO / Fe(001) bilayer with incorporation of a Au spacer layer.

In summary, all measurements in our proposal have been fully carried out and the expected structural data could be recorded in very good experimental conditions. The quantitative interpretation of the data is in progress.