ESRF	**Experiment title:** Microstructure and composition versus Tunnel Magneto-Resistance in fully epitaxial magnetite-based magnetic tunnel junctions	**Experiment number**: HS 2971
Beamline: ID-01	**Date of experiment**: from: 21 june to: 26 june 2006	**Date of report**: 4/02/2008
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Report:

The results of this experiment were published in [1]: C. Mocuta, A. Barbier, A. V. Ramos, M.-J. Guittet, J.-B. Moussy, S. Stanescu, R. Mattana, C. Deranlot, F. Petroff, *Effect of optical lithography patterning on the crystalline structure of tunnel junctions*, Appl. Phys. Lett. **91**, 241917 (2007).

***Abstract**:* The crystalline structure of metal-oxide-based magnetic tunnel junctions patterned by optical lithography was resolved locally using a microfocused x-ray spot. We evidence several micron-sized lithography-induced distortion effects on the crystalline structure of the layers near the edges of the junction. The distortions translate into tilts (up to 1^o) of the crystalline planes in the vicinity of the edges and propagate toward the center of the junction. They are attributed to the release of the elastic strain in the layers during the lithographical process. For the smallest junctions, size effects limiting the amplitude of the tilt are also evidenced.

We studied [Pt (10nm) /$CoFe_2O_4$ (5 nm) /γ-Al_2O_3 (1.5 nm) /Co (15 nm) /Au (15 nm)] structures grown by atomic oxygen assisted molecular beam epitaxy on an α-Al_2O_3(0001) single crystalline substrate, then patterned *ex situ* by optical lithography. The resulting junctions had square shapes and dimensions ranging from 10×10 to 150×150 μm^2, and 0.5 mm average spacing between their centers.

An x-ray beam of energy E=7 keV was focused using 18 individual beryllium-compound refractive lenses down to a clean spot size of about 6×9 μm^2 (horizontal × vertical, FWHM). The photon flux in the spot was about 5×10^{10} photon/s (focusing gain of ~1200). The sample was placed on the microdiffractometer in diffraction condition. The lateral scanning translation stages are used to bring into the x-ray spot the different areas of interest (junctions). A point detector with a 0.03° angular acceptance in 2θ vertical angle was used. The layers in the junction have different crystalline structures and thus different characteristic d spacing. Scanning the lateral position of the sample, while recording the signal in the detector for a given 2θ position, provides an image (two-dimensional map) sensitive to the chosen layer (Fig. 1). We thus have a point-probe scanning technique which allows to image a chosen d spacing (which can then be related to lattice spacing, crystalline structure, etc.). In this way, all of the constituent layers in the same junction are probed individually. Using the Bragg peak positions in the reciprocal space as a probe for the imaging of the junction as described above, Co, $CoFe_2O_4$, and Pt can be traced individually (Fig. 1). All layers were characterized using line sections, as shown in (Fig. 1 left), but we will focus on the results corresponding to the Co layer.

By using this method, when approaching the edges of the junction, a significant shift (~1^o) in the position of the Bragg peak is detected. This shift was of opposite sign on either side of the junction and is

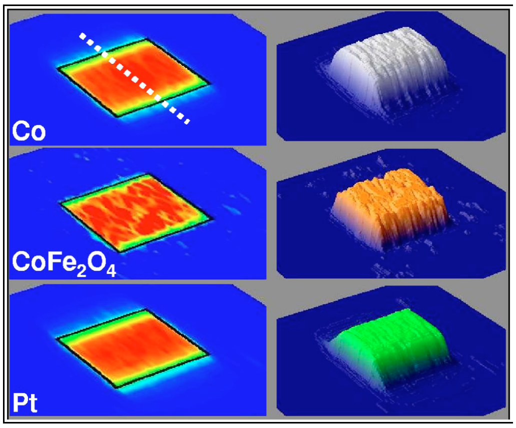

Fig 1. (from [1]) Color intensity maps (left) and the three-dimensional corresponding representation (right) of a 150×150 μm^2 junction. All the intensities were normalized. From top to bottom, positions corresponding to Co(0002), $CoFe_2O_4$(333), and Pt(222) interplane spacing were imaged. The black line square contour (left panels) represents the exact size of the junction. The smearing along one direction in the maps comes from a smeared footprint of the beam (the incident angle) in this direction only. The dotted line shows the scanning direction (Fig. 2).

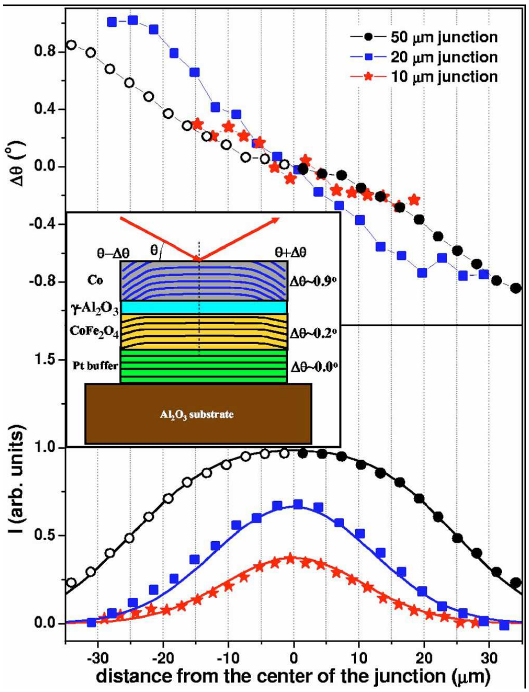

Fig. 2: (from [1]) Measurements for square junctions of sizes of 50 (●), 20 (■), and 10 μm (*) are reported. (Bottom) Measured integrated intensity (I) of the Co peak, function of the lateral position of the x-ray spot on the junction is shown by the symbols. The continuous line is the calculated convolution of a square junction with the beam footprint. In the case of the 50 μm junction, only half of it was measured (●); the symmetric data are shown by the open symbols (o). (Top) Angular shift (Δθ) of peak's center of mass, across the junction. (Inset) Cartoon of the tilt of the crystalline planes in the 50 μm junction. The values of the shift (tilt) angle Δθ for Pt, $CoFe_2O_4$, and Co layers are reported.

explained by a tilt of the crystalline planes (image sketched in the inset of Fig. 2). A similar sign variation is reported for the $CoFe_2O_4$ layer. For the Pt layer, the observed shift lies within the error bar of the measurement (0.05° - 0.1°). This smaller effect may be explained by the fact that the Pt layer is relaxed and in direct contact with the substrate. The amplitude of the effect (especially for the Co case) is by far above the error bar, so beam divergence and less probable rotation effects can be excluded.

The curvature of the crystalline planes in the vicinity of the edges occurs in all of the investigated junctions (Fig. 2), with an extension of about 5 μm, but with different amplitudes. In the case of the 10 μm junction, the reduced amplitude of the tilt is a consequence of its reduced size: if the junction is small enough, the effect on one edge counterbalances the effect induced by the opposite one. The extension of the perturbation is quite large and is comparable to the typical junction sizes. This effect should thus be considered when measuring magneto-transport properties of tunnel junctions, and, in particular, those which are fully epitaxial, since the crystalline structure and the transport properties are correlated.

We have proposed a method enabling the investigation of patterned devices which requires almost *no sample preparation*, is *nondestructive*, and has *high sensitivity to individual (buried) layers*. The use of the microfocused x-ray beams brings all these advantages together into a local (spatially resolved) powerful technique to complete information obtained from well established microscopy techniques. We have shown that after optical lithography, for all investigated junctions, the atomic planes in the vicinity of the edges have the tendency to subside, especially for the Co layers. The extension and strength of the deformation may be related to the release of epitaxial strain promoted by the lithography process.

References:

[1] C. Mocuta *et al.*, *Effect of optical lithography patterning on the crystalline structure of tunnel junctions*, Appl. Phys. Lett. **91**, 241917 (2007) (and references therein).