ESRF	**Experiment title:** ***Determining the structure of exchange bias stabilized Co-CoO core shell nanoparticles embedded in Ag***	**Experiment number**: 25-02-622
Beamline: BM25-B	**Date of experiment**: from:7/02/2008 to:12/02/2008	**Date of report**:
Shifts: 15	**Local contact(s)**: Manuela E. Medina Muñoz	*Received at ESRF:*

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Report:

The main study conducted during the 25-02-622 experiment has dealt with the multilayered system Co-CoO / Ag.

For exchange-bias (EB) systems involving CoO as the antiferromagnetic (AF) component, preparation of *overoxidized* (i.e. $CoO_{1+\delta}$) AF layers has previously been shown to have an important effect on EB properties, which arises due to point defects associated with such non-stoichiometric material. We have conducted a study of the effect of post-deposition annealing to progressively higher temperatures (*Tann)* on magnetic and structural parameters of a Co-CoO(bilayer) / Ag multilayered system [1]. The results indicate non-stoichiometry of a different type: $CoO_{1-\delta}$ (i.e. *underoxidized*) layers, with $\delta = 0.1$ for the as-deposited sample which in turn explains the magnetic behaviour observed. In fig 1 the evolution of the hysteresis loop shift (H_E) is shown as a function of the annealing temperature, from a rather high initial value of around 2.5kOe to almost zero at 500ºC . The crystalline parameters have been followed, their change being explained via a mechanism of diffusion, initiated at *Tann* ≈ 300ºC, of Co ions from the oxide to the Co layers, leading to the approach to stoichiometry (i.e. Co_1O_1) in the former layers. In an article **recently submitted** to "New Journal of Physics" [2] we show all these results together with a detailed study of the crystalline structure of Co / CoO / Ag multilayers. Preliminary XRD patterns acquired using laboratory x-rays in the standard θ-2θ geometry (i.e. scattering vectors perpendicular to the multilayer plane) were obscured by the strong peak of (002) Ag, very close to that of CoO -textured in the (002) direction as well- and appears only as a small shoulder

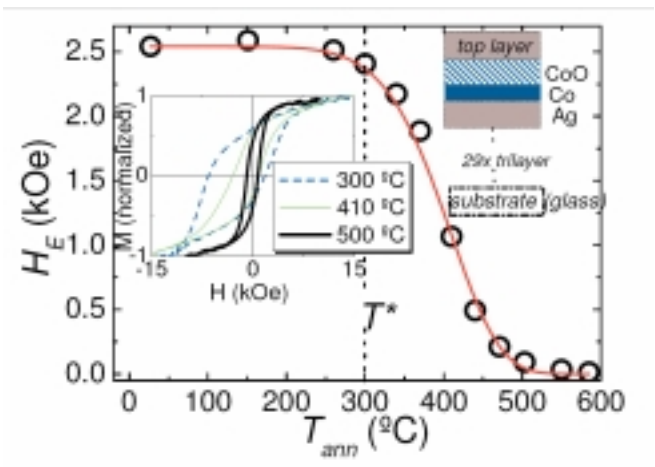

Figure 1. Post-deposition annealing (at temperatures T_{ann}) dependence of the EB field, measured at 80 K. Insets: (left) corresponding hysteresis curves for selected values of T_{ann} and (right) the multilayer structure of the *as-deposited* sample, comprising essentially Co-CoO bilayers (30 in total) separated by Ag layers, and with an Ag capping (top) layer.

in the low 2θ side of the peak. For this reason we have carried out a detailed XRD study at SpLine, with the aim of exploring the AF crystallinity **parallel** to the multilayer plane. To do so we employed a geometry typical for surface diffraction, with fixed incidence and exit angles grazing to the sample surface, and scanning the detector horizontally. In this way we acquired figure 2(a), where both position an width of CoO (002) peak is followed as a function of subsequent annealings to progressively higher temperatures (process performed ex-situ). The main conclusion drawn from here is to confirm that the effect of annealing is a improvement in CoO crystallinity by diffusing out the excess of Co, as CoO peak becomes increasingly sharper and moves towards to position of bulk Co_1O_1 [figure 2(b)]. This change is linked to the drastic reduction of the EB shift pointing out the importance of disorder in the exchange bias effect.

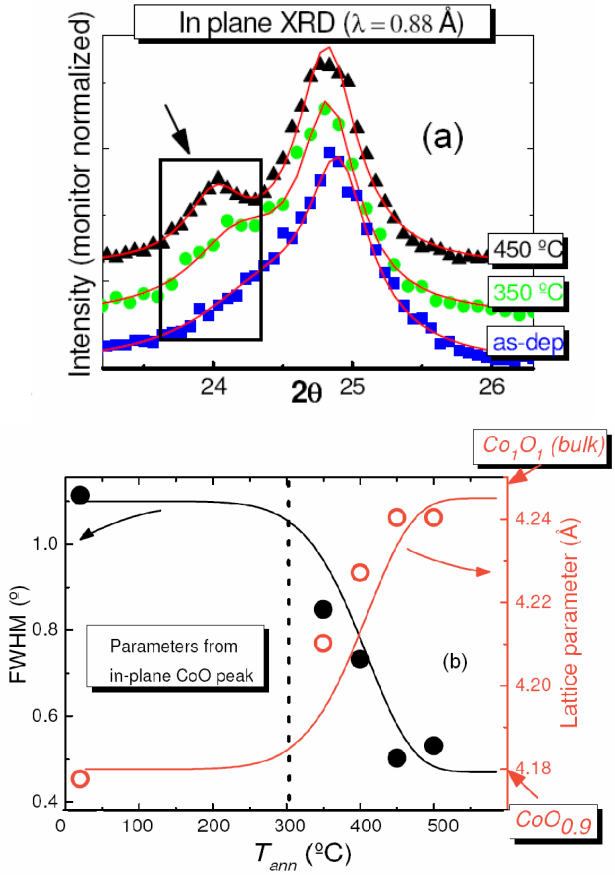

Figure 2. (a) in-plane x-ray diffraction for selected values of T_{ann}. taken using synchrotron radiation. The arrow indicates the CoO (002) signal. (b) shows the evolution of FWHM and lattice parameter with T_{ann} as extracted from the fits in (a)

A different system that has been also studied during this experiment: films made from Co-CoO nanoclusters obtained by deposition of preformed gas phase condensed Co clusters on silicon and glass substrates. A small amount of oxygen was allowed to enter either in the final deposition chamber or the aggregation chamber. The structure of these systems is very hard to study by using conventional (lab) x-ray techniques due to limiting sample volume. XRD specular scans measured at SpLine have demonstrated a difference between the two series. When O_2 is introduced in the aggregation chamber, the CoO formed is more disordered, which may explain differences in their magnetic behaviour. Further measurements and analysis are required to confirm this preliminary experiment, which could not be completed due to problems with beam quality and stability during the first 4 or 5 shifts of the experiment (injection problems at the main storage ring). In addition, our beamtime was allocated during a "single bunch" mode thus leading to limited diffraction intensities.

[1] For details on the multilayer structure and preparation method see P.S. Normile et al. Phys. Rev. B **76**, 104430 (2007)

[2] J.M. Riveiro, P.S. Normile, J. A. de Toro, T. Muñoz, P. Muñiz, J.A. González, J.P. Andrés. "$CoO_{1-\delta}$ layers in a reactively sputtered exchange bias system" Submitted to "New Journal of Physics" on the 8th June 2008. Article ID: NJP/282929/PAP