



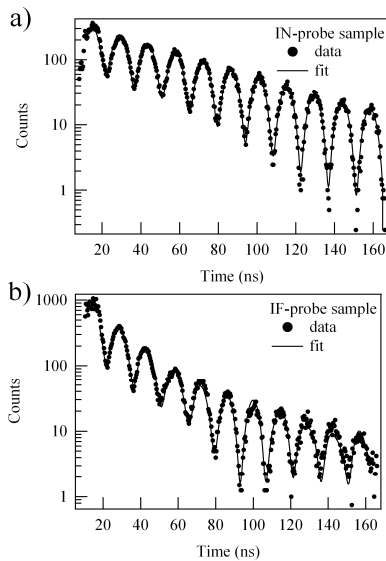
	<b>Experiment title:</b> Depth-resolved Fe spin structure at the Fe/NiO(001) interface	<b>Experiment number:</b> SI 1705
<b>Beamline:</b> ID18	<b>Date of experiment:</b> from: 12/03/2008 to: 18/03/2008	<b>Date of report:</b> 30/08/2009
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## Report:

The aim of this experiment was to perform a depth-resolved magnetic characterization in the proximity of the epitaxial Fe/NiO(001) interface. The starting point for this study was our previous structural and chemical characterization of the system, which indicated that a buckled FeO-like layer is formed at the interface and that the metallic Ni resulting from NiO reduction interdiffuses in the overlying Fe layer forming an alloy with a distorted bcc structure involving  $\sim 3$  ML [1,2]. Information on the magnetic coupling between these phases is expected to be very important to understand the exchange bias mechanism for this system.

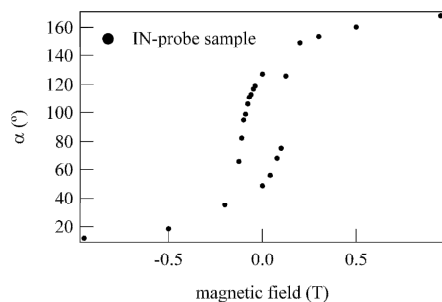
The three samples used for the present experiment were grown in ultra-high vacuum by molecular beam epitaxy in our laboratory at the S3 center in Modena and carried to the ESRF in non-reactive atmosphere. The substrates used were three Ag(001) single crystals, prepared by repeated cycles of sputtering and annealing. A 50 ML thick epitaxial NiO film was grown on the three substrates by reactive deposition of Ni in O<sub>2</sub> atmosphere. Three different Fe films were subsequently grown at RT on the NiO/Ag(001) layers: the first one, hereafter referred to as the interface- (IF-) probe sample for simplicity, consists of a 2-ML thick <sup>57</sup>Fe probe layer (95.5% isotopical enrichment) located at the interface with NiO and a 10-ML thick layer of natural Fe on top of it; the second sample has the same overall Fe thickness (12 ML), but the 2 ML <sup>57</sup>Fe probe layer is located 6 ML above the interface and will be referred to as the inside- (IN-) probe sample in the following; the third sample used for reference was a 12 ML <sup>57</sup>Fe film. All samples were capped by a 5 nm-thick Ag layer, which proved to be effective in protecting the samples from oxidation when exposed to the atmosphere. The nuclear resonant scattering (NRS) experiment was performed at the ID18 beamline with the storage ring operating in the 16-bunch mode, with subsequent bunches separated by 176 ns. The radiation was monochromatized to a bandwidth of 6 meV around the nuclear resonance of Fe<sup>57</sup> (14.413 keV). The beam cross section at the sample position was focussed to  $15 \times 15 \mu\text{m}^2$  size. The measurements were performed at grazing incidence at the critical angle for total reflection in order to maximize the nuclear reflectivity signal. The three samples were measured at RT both before the application of the magnetic field H and in applied field in cycles from 1 T to -1 T and back. The field was applied in the sample plane along the [100] direction of Fe and the NRS spectra were measured with the k vector of the photon beam parallel to H. In some cases also spectra with k perpendicular to H were measured. We also performed field cooling (FC) of the samples, following the same procedure used in our laboratory to induce exchange bias (heating to 270° C and cooling

in applied magnetic field of 1 T), but unfortunately the samples were damaged by this treatment due to the poor vacuum conditions at which FC was performed.



**Fig.1** NRS spectra of 12 ML Fe/NiO(001) samples with a 2 ML  $^{57}\text{Fe}$  probe layer inside (a) and at the interface (b). The solid line is the fit of the data.

The data analysis, still in progress, has been performed by fitting the time spectra with the MOTIF program [3]. For the fitting of some of the spectra, we also used the NRS package based on the CONUSS program [4], which takes into account the reflectivity and standing wave phenomena in the grazing incidence geometry, obtaining very similar results in the two cases. Fig.1 shows the NRS spectra for the IN- and IF-probe samples measured at saturation ( $H=1\text{T}$ ) with  $k$  parallel to  $H$  and the relative fits. A good-quality fit of the IN-probe sample spectrum (Fig.1 a) was obtained using a majority component with a hyperfine field  $B_{\text{hf}} = 32.94(3)$  T, a value compatible with bulk Fe. The fit quality improves if a second component with similar  $B_{\text{hf}}$  (31.8(2) T) and a slightly larger value of isomer shift IS (0.02(6)) is included with a weight of approximately 10%. This component can be possibly ascribed to Fe partially coordinated with Ni (or Ag). The situation of the IF-probe sample is, as expected, much more complicated. The NRS data for this sample and the relative fit are shown in Fig.1 b. It has to be noticed that the IF-probe sample spectrum shows a quantum beat structure very similar to the IN-probe sample spectrum, but the damping of the scattered intensity is much faster. Since the overall thickness of the two samples is the same, this effect can be due to a broadening of the hyperfine field distribution and/or to the presence of different components reflecting the structural and chemical complexity of the interface. A good quality fit of this spectrum could be only obtained if we introduced magnetic components with non zero IS, ascribed to oxygen coordinated Fe atoms at the interface. In particular the best fit was obtained by using four components. While for the first component the IS was fixed to zero, the fitting procedure resulted in a similar value for the second one and in a non zero positive IS=0.35(4) and IS=0.36(9) for the third and fourth component. The first two components, with an overall weight over 70%, are ascribed to metallic Fe and their  $B_{\text{hf}}$  values are 31.9(2) and 33.9(1) T. The third and fourth components, ascribed to Fe partially coordinated with oxygen atoms, have  $B_{\text{hf}}$  values of 32.6(1) and 34.7(6). The values of  $B_{\text{hf}}$  of the different components in this sample are related to the structural complexity of the interfacial phase. The models here described gave a very good fitting also for the spectra measured with  $k$  perpendicular to  $H$  for the two samples. The results of this analysis have been presented as an oral communication at Symposium R of E-MRS 2009 Spring Meeting and have been accepted for publication in the conference proceedings to appear on NIM B.



**Fig.2** NRS hysteresis cycle for a 12 ML Fe/NiO(001) sample with a 2 ML  $^{57}\text{Fe}$  probe layer inside.

The analysis of the IN-probe sample spectra measured as a function of  $H$  along a magnetization cycle has been performed using one metallic bulk-like component and leaving as a fitting parameter the angle  $\alpha$  such that  $B_{\text{hf}} \cos\alpha$  represents the average  $B_{\text{hf}}$  projected along  $k$ . The resulting NRS hysteresis loop is shown in Fig.2. The analysis of the IF-sample spectra along the same magnetization cycle is in progress. We expect to be able to extract the evolution of the  $B_{\text{hf}}$  component along  $k$  for both the metallic and oxidized components at the interface. A comparison between the cycles obtained is expected to give information on the coupling between the different magnetic phases in this sample. In particular the oxidized phase, theoretically predicted to have an increased magnetic moment compared to the case of the ideal interface [2], is expected to play an important role in exchange coupling and in exchange bias in this system.

## References

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