| ESRF | **Experiment title:** Unravelling the Half Metallic Ferromagnetism of Metal/Oxide epitaxial Interfaces | **Experiment number**: 1237 |
|---|---|---|
| **Beamline**: ID08 | **Date of experiment**: from: 05/05/2002 to: 14/05/2002 | **Date of report**: 18/02/2002 |
| **Shifts:** 18 | **Local contact(s)**: Celine De Nadaï | *Received at ESRF:* |


**Names and affiliations of applicants** (* indicates experimentalists):

Dr. TAGLIAFERRI Alberto, Dr. DE NADAÏ Celine, Dr. BROOKES Nick / ESRF

Dr. ANDREIU Stéphane, Dr. SICOT Muriel, Dr. TURBAN Pascal,
Laboratoire de Physique des Materiaux, UMR 7556, Universite Henri Poincare/Nancy I, Boulevard des Aiguillettes - BP 239, F-54506 Vandoeuvre-Les-Nancy, France

Dr GOTA Suzana, Dr. MOUSSY Jean-Baptiste, Dr. GAUTIER-SOYER Martine
DSM - DRECAM - SPCSI, Service de Physique et Chimie des Surfaces, CEA Saclay, F-91191 Gif-sur-Yvette Cedex, France

Dr. CROS Vincent, Dr. PETROFF Frédéric,
Unité mixte de Physique CNRS/THALES, Domaine de Corbeville, 91404 Orsay Cedex, France

Dr. SACCHI Maurizio / L.U.R.E. bâtiment 109D, Campus Universitaire d'Orsay, F-91405 Orsay Cedex, France


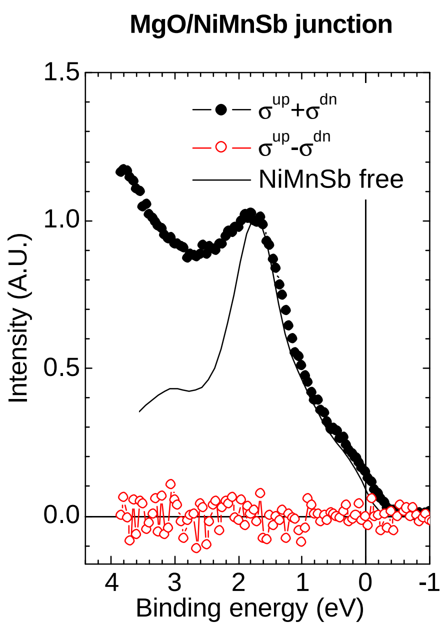


Fig. 1

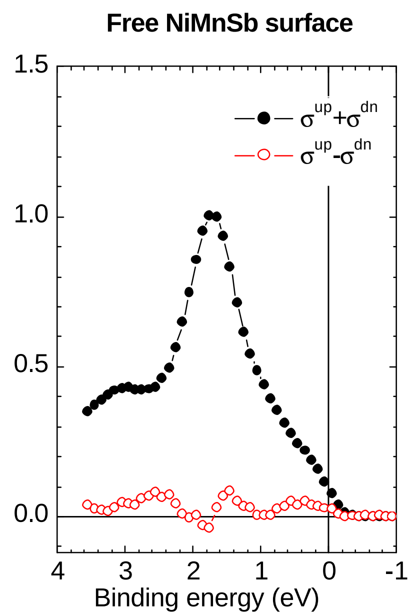


Fig. 2

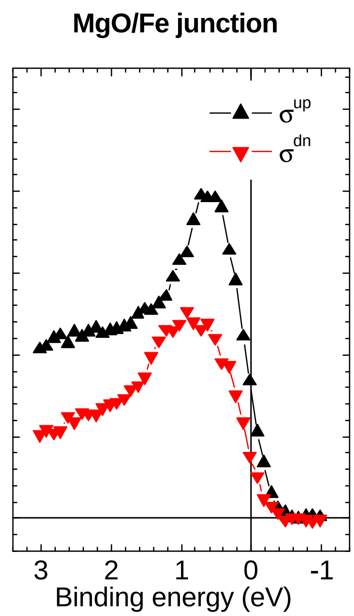


Fig.3

In a metal/insulator/metal Magnetic Tunnel Junction (MTJ), the magnetoresistive effect is related with the spin polarisation of the density of states at the Fermi level [1]. Thus the search for materials having higher spin polarisation is one of the possibilities in order to increase the magnetoresistive effect in commercial devices. In the limit of a 100% spin polarisation the material is called a Half metallic Ferromagnet (HFM) and the MTJ device becomes an electron polariser and a spin switch. Some materials like $LaSrMnO_3$, NiMnSb, $Fe_3O_4$, $CrO_2$, seem to be good candidates for this goal since the density of states at the Fermi level is in theory close to zero for minority spins. This behaviour was only evidenced in $LaSrMnO_3$ and $CrO_2$ at low temperatures [2], but not in other materials up to now.

The crystalline quality of the MTJ plays a fundamental role on the performances of the device, as it is supported by the wide range performances obtained in the literature on junctions made of the same materials, and a good matching between the electronic structures of metal and insulator seems to be also crucial [1].
The aim of the experiment was to measure the spin polarisation of the occupied states at the metal/insulator interface. We investigated the following systems:
The epitaxial MgO/NiMnSb [2] and MgO/Fe are prepared by MBE in Nancy. They are protected towards contamination by an Sb overlayer, which is removed *in situ* by heating the sample. The $Fe_3O_4$(111) and $Al_2O_3$/$Fe_3O_4$(111) layers are epitaxially grown by MBE on $\alpha$-$Al_2O_3$(0001) in Saclay [3]. The samples are fully characterized by a combination of RHEED, LEED and XPS analysis. Before the experiment, the $Fe_3O_4$(111) samples was annealed *in situ* at 800 K ($PO_2 = 10^{-6}$ Torr) to restore the surface crystallinity after the passage through air.
After the preparation in the experimental apparatus at ESRF, each sample was characterised for crystallinity and contamination by LEED and XPS, respectively.

**MgO/NiMnSb:**

We performed non resonant SRXPS on MgO/NiMnSb junction at room temperature. Fig. 1 shows the sum and difference spectra of MgO/NiMnSb, together with the one from the free NiMnSb surface as a reference (thin line). In the first 1.8 eV below the Fermi level the spectral shape is very similar to the one of the free surface. This confirms that the electrons emitted by the metal in this energy range are nearly not affected by the presence of the MgO insulating overlayer. At higher binding energies the contribution from the occupied states below the insulating gap of the oxides becomes substantial and overrides the decreasing contribution of the buried NiMnSb. Despite a good initial crystalline quality of the sample, as supported also by STM investigations on samples prepared with the same procedure and apparatus [2], the magnetic properties at the MgO/NiMnSb interface was extremely reduced with respect to the free NiMnSb surface. SRXPS difference spectra don't show a sizable polarisation, while absorption XMCD sum rules measurements give a magnetic moment of roughly 1/3 with respect to the value measure on the free surface. This seems to be due some intermixing of the first layers at the junction of the barrier during the heating procedure needed for the desorption of the protecting Sb overlayer. This model could justify also the non zero moment obtained in XMCD, which is more bulk sensitive with respect to XPS and averages the properties of the surface and of deeper layers. To check the quality of the metallic electrode in the particular sample measured, we removed the insulating barrier by sputtering and restored the NiMnSb surface by annealing. The polarisation in SRXPS and the magnetic moment measured in XMCD on the free surface were recovered (Fig. 2), so that a bad crystalline quality of the NiMnSb electrode away from the interface seems to be ruled out.

**Fe/MgO:**

The sample was prepared with the same procedure as the NiMnSb, i.e. desorbing the Sb protecting overlayer by heating to 500 C. Fig. 3 shows the up and down non-resonant SRXPS spectra, to allow a better comparison with data published in ref. 4. The spectral shape within the energy gap of the insulating covering barrier is consistent with the published data taken on the free Fe surface [4]. This suggest that the properties of the free Fe surface are preserved or slightly modified. The absorption XMCD sum rules give a moment close to the 2.1 $\mu_B$/at for bulk Fe at room temperature. Thus the junctions preserves a good crystalline quality even after the removal of the protecting capping layer, opposite to the MgO/NiMnSb case.
We enphasise that this is the first experimental demonstration of the possibility of measuring the spin polarisation through an insulating barrier by SRXPS.

**$Fe_3O_4$(111) and $Fe_3O_4$(111)/ $Al_2O_3$:**

The $Fe_3O_4$ has been prepared and measured for few hours, due to the ESRF brekdown on the 11$^{th}$ and 12$^{th}$ of May 2002 and the consequent loss of more than 2 days of beamtime. The SRXPS data show a negative polarisation at the Fermi energy. The low statistics doesn't allow to draw up more precise conclusions. The $Fe_3O_4$/ $Al_2O_3$ could not be measured for the same reason.

In conclusion, we have demonstrated for the first time the possibility to measure the spin polarisation of the occupied states near the Fermi energy through an insulating barrier, an information which is of crucial importance for the understanding and improvement of MTJ devices. The MgO/Fe junction shows magnetic properties similar to Fe bulk and confirms its good crystalline quality and stability under thermal treatement below 800 K. The MgO/NiMnSb junction degrades under the thermal treatement needed for the preparation of the sample after transportation under $N_2$ atmomosphere and exposure to air during few tens of minutes.

---

**References**


[1] J. De Teresa *et al.*, *Phys. Rev. Lett.* **82**, 4288 (1999).
[2] P. Turban *et al.*, *J. of Mag. And Mag. Mat.* **240**, 427 (2002).
[3] S. Gota J. B. Moussy, M. Henriot, M. J. Guittet, M. Gautier-Soyer, *Surf. Sci.***482-485**, (2001) 809.
[4] A. K. See and L. E. Klebanoff, *J. Appl. Phys.* **79** (8), 4796 (1996).