ESRF	**Experiment title:** The Surface Versus Bulk Electronic Structure by Magnetic X-Ray Scattering From NiO(111) Single Crystal Surfaces with Respect to the Néel Transition.	**Experiment number**: HE-1496
Beamline: ID-20	**Date of experiment**: from: 09.04.2003 to: 15.04.2003	**Date of report**: 06.09.2004
Shifts: 18	**Local contact(s)**: Flora Yakhou	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

A.Barbier* CEA/Grenoble DRFMC/SP2M/IRS 17, Rue des Martyrs 3 8054 Grenoble
F.Yakhou* ESRF (ID20)
M.Mulazzi* ELLETRA (Italy)
C.Mocuta* MPI-Stutgart – Germany
S.Stanescu* ESRF
R. Belkhou* LURE (Orsay – France)
C.Vettier* ESRF / ILL
F. de Bergevin* ESRF

Report :

The present report summarized the results obtained during experiments HE-1496 and HE-1691 and will thus appear as report for both experiments.
Magnetically coupled ferromagnetic (F) metals / antiferromagnetic (AF) oxides interfaces are major constituents of magnetic sensors based on the so-called spin-valve geometry which are studied at CEA laboratories. Because of the high stability of NiO, sensors operating in harsh environments can be expected. The magnetic exchange coupling is used in the sensors but is still not well understood especially because of the lack of characterization of antiferromagnet (NiO) in the surface and interface regions. In order to tackle this issue, X-ray magnetic scattering is a method of choice. Since NiO(111) has no magnetic peak in the surface plane it was necessary to work in an asymmetric diffraction geometry keeping the incidence angle constant (and small) in order to obtain the necessary surface sensitivity. This corresponds to standard surface diffraction geometry. However the diffraction plane is then no longer only vertical or only horizontal and the polarization analysis become complex. To overcome this difficulty we have introduced the rotation angle of the detector in the geometry in order to discriminate between charge and magnetic signals (HE-813). A furnace with accuracy +/-0.1° was implemented (HE-929).We could establish that the domain structure of NiO(111) evolves in bulk and surface with respect to the temperature well before the Néel temperature is reached, which was found 5° higher for the surface than for the bulk.

Experiments HE-813 and HE-929 were performed in non resonant conditions with photon wavelength λ=1.58Å allowing to have the PG(006) crystal analyzer at its Brewster angle and thus a measured rejection efficiency of 99.98%. For all experiments the surface was held vertical, the incidence polarization was thus always more or less close to π.

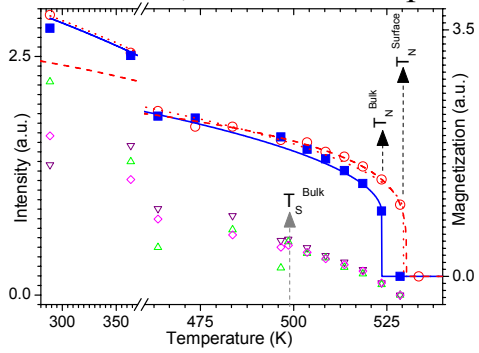

Figure 1: Quantitative measurements obtained during previous experiments. From complete peak families the magnetization in the surface (circles) and bulk (square) were derived with 0.3° and 3° incidence angles respectively. The surface Néel transition temperature is about 5 K larger then the bulk one.

The lower graphs reveal the large intensity dispersion that exists within a given peak family ((1,1,3/2), (2,-1,3/2) and (1,-2,3/2) in the triangular surface lattice mesh) due to unbalanced S-domain population. The effect vanishes at about 500 K.

The aim of experiments HE-1496 and HE-1691 was to investigate the bulk versus surface electronic structure at least at 3 temperatures corresponding to coherent domain rotation (room temperature), statistic fluctuation of the domains (210°C) and to surface magnetic order without bulk magnetic order (261.5°C). A quantitative measurement, for a given energy, requires the measurement of a complete family of peaks (equivalent by symmetry but non equivalent because of the magnetic domain structure) and necessitates thus 3 rocking scans and 3 longitudinal scans for a complete integration of the intensity. Since the magnetic signal decreases strongly around the Néel transition the measurement times became large (several hours for the last points) to get a reasonable integrated intensity.

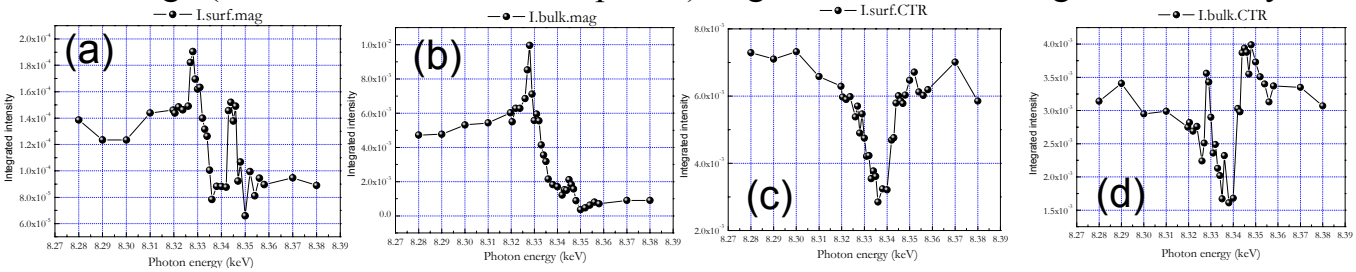

Figure 2: Quantitative measurements obtained for the sample held at T=210°. (a,b) in the magnetic and (c,d) in the charge sensitive channels. (a,c) (resp. (b,d)) in surface (resp. bulk) sensitive conditions (incidence angle = 0.3°, (resp. 3°)).

Complete data sets of quantitative measurements across the Ni K-edge (8.28 to 8.38 keV photon energy) were obtained in the direct (charge sensitive) and indirect (magnetization sensitive - for which the polarization experiences a π/2 phase shift) channels for sample temperatures of 40, 210, 261 and 264°C (see figure 2 for T=210°C). As expected from previous qualitative measurements the electronic structure of NiO(111) in surface and in bulk with respect to the intensities of the quadrupolar (E1=8328 eV) and dipolar transitions (E2=8346 eV) are different from each other and also from the anomal scattering (compare figure 2a and 2c or 2b and 2d). Importantly, surface scattering does almost not suffer the limitations due to self-absorption (compare fig 2a and 2b). The crosstalk between channels (that was measured independently), due to less efficient polarization rejection, remains at an acceptable level for which correction will be possible. With respect to the temperature the E1 and E2 transition exhibit different behaviors that deserve further investigation.

In summary, experiments HE-1496 and HE-1691 run smoothly and provided the expected data in very good experimental conditions. However, further work is necessary to fully determine the electronic structure of NiO(111) in surface and in bulk across the Néel transition.