ESRF	Experiment title: Manipulation of the growth of the magnetically coupled Co/NiO(111)-p(2x2) interface
Beamline:	Date of experiment:
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Report:

NiO(111) is a good potential antiferromagnetic (AF) candidate for the magnetic pinning of ferromagnetic (F) layers in spin-valve sensors. It is highly resistant to corrosion, insulating and has a high Néel temperature (523 K) compatible with the work conditions of hard drive read-back heads. The study of interfaces grown on NiO(111) is difficult because most electron based techniques are hampered by the insulating character of this oxide. This also explains the lack of detailed structural characterization of these interfaces. However, it is generally accepted that the exchange coupling phenomenon making these devices working takes place at the interface between the AF and F materials.

The present study is part of C.Mocuta's Ph.D. work (foreseen in 2000) aiming at the understanding of the correlation existing between the crystallographic structure and the magnetic properties of such F/AF interfaces. In turn it should allow to better understand the magnetic exchange coupling phenomenon itself.

For the present experiment the preparation conditions of the NiO(111) single crystals have been greatly improved. The substrates were provided by Crystal GmBH, Berlin, Germany, The NiO single crystal boule was first air annealed for 24 hours at 1550°C, then cut and polished and at last again air annealed for 3 hours at 1000°C. The last annealing re-establishes the stoïchiometry of the surface. This yields NiO(111) single crystals with a surface mosaic spread typically of 0.03°. The improved preparation was necessary to follow the evolution of the surface reconstruction with the Co deposit but leads to segregation of some additional contaminants (about 1/10 of monolayer).

After introduction in the UHV chamber (base pressure 10-10 mbar), Auger Electron Spectroscopy (AES) showed that the main contaminants were carbon and calcium. Several cleaning procedures were tested. An oxygen annealing (10 minutes at 750°C with p(O₂)=10⁻⁵ Torr) easily removes the C (see Figure 1). Unfortunately, the Ca is not removed with this treatment. Longer annealing periods (with or without O₂) at high temperature leads to the segregation of additional Ca and K, as seen by AES. The lower limit temperature where no segregation is observed is around 700°C.

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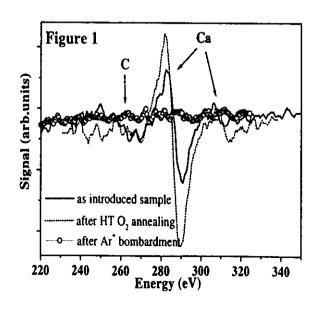
After air annealing, the surface is $p(2\times2)$ reconstructed. Removing the contaminants by ionic bombardment (either Ar^+ or O^{2-}) leaves the surface rough: the crystal truncation rods (CTRs) signal vanishes. There is also a modification of the surface stoïchiometry: additional peaks appear, at this stage the surface is similar to the not annealed as-polished sample. Attempts to re-anneal at 1000° C in air the sample re-establish the stoïchiometry and the physical surface (the CTRs signal is restored - Figure 2), but the quantity of the surface Ca is also restored. Air annealing at 700° C prevents the segregation but the surface remains of poor crystallographic quality limiting the intensity of the peaks of the $p(2\times2)$ reconstruction. A set of data was recorded showing that the residual Ca contamination has no effect on the atomic structure of the surface reconstruction: the relative intensities of the peaks are the same than for the initial surface.

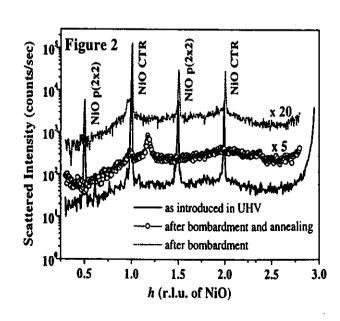
The Co was deposited on the surface from an evaporation cell. The flux ($\sim 1\text{Å/min}$) was calibrated using a quartz micro-balance. Two growth situations, at 300°C deposition temperature, were investigated: UHV deposition and the effect of a 10^{-8} Torr partial pressure of O_2 in order to check the surfactant effect of O. Different cumulative Co thicknesses, θ_{Co} , were investigated in the range 0 to 10nm. During all X-ray measurements, at each θ_{Co} , AES spectra were recorded. This provides another way to follow the quantity of deposed metal on the surface and may also allow evaluating the growth mode independently. For a several times cleaned and annealed surface, the wetting of the surface by the Co layer at 300°C, deposited in UHV, was found to be fairly better than in previously investigated situations (experimental report 32.3.015 and in-house research). At each θ_{Co} , the evolution of the reconstruction was carefully followed by quantitative measurements of the in-plane reconstruction peaks. These data will show if the Co layer changes the structure of the reconstruction during the metallization of the surface that is an important open question in this system. Thicknesses up to 30 Å were investigated quantitatively.

The evolution of a particular in-plane Co Bragg peak (the (110) peak where FCC, *Twinned-FCC and HCP stacking scatter) was also followed in detail. The decrease of the intensity of the surface signal is larger than expected for the attenuation due to the metallic layer. Large signal is still measured at large values of θ_{Co} evidencing a 3D growth mode, also supported by the existence of out-of-plane Bragg peaks of the Co for sub-monolayer thickness. The substrate CTRs were quantitatively measured at different coverage. Oscillations appeared rapidly on these rods with increasing Co coverage indicating either a precise epitaxial site of the Co or a new structure (maybe a spinel like NiCoO?) with a poor crystallographic quality. Further detailed analysis of these data is necessary to conclude. This sample will be used for magnetic measurements (dichroïsm).

The same study was performed in the case of Co on NiO(111) with a small partial pressure of O_2 . The O_2 was not found to have a clear surfactant effect but changes the overall growth behavior. On the other hand neither CoO nor NiCoO were found, indicating that O does not react with Co at these levels of partial pressure. Similar measurements with respect to the previous growth were performed.

Note: * The FCC structure is the one continuing the stacking of the NiO(111) substrate (ABC stacking), while the twinned-FCC is the one rotated by 60° (ACB stacking).





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