ESRF	**Experiment title:** Surface Versus Bulk Magnetic X-Ray Scattering From NiO(111) Single Crystal Surfaces And Co/NiO(111) Magnetically Exchange Coupled Interfaces.	**Experiment number**: HE-929
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Report (preliminary) :

The present experiment belongs to a larger research program started several years ago, aiming at the understanding of magnetically coupled ferromagnetic (F) metals / antiferromagnetic (AF) oxides interfaces. They are a major constituent of magnetic sensors based on the so-called spin-valve geometry which are studies in the SP2M (CEA/Grenoble) laboratory. 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. From our previous experiments and recent literature it appears that the origin of the effect probably lies in the antiferromagnet (NiO) . It is thus mandatory to investigate the antiferromagnetic structure of NiO(111) near the surface. A method of choice is X-ray magnetic scattering. 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 a 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 the charge and the magnetic signal. We used the geometry successfully set-up in experiment HE-813 and worked in similar conditions.

In the present experiment we wished to mesure the surface versus bulk Néel transition. For that purpose a motorized sample holder and a small furnace were developped and tested in the months before the experiment. The furnace was build to fit in an available small Be chamber and a vacuum of about 10^{-5} mbar was obtained in the small chamber. The regulation thermocouple was placed below the sample in the Ta plate supporting the sample. A temperature regulation in the range +/- 0.1° could be obtained by fine tuning the heating parameters. Moreover the device was cooled by water and/or compressed air below the furnace and also by and additionnal external fan avoiding any heat on the Be cap.
After some setting up the furnace and the sample holder worked accordingly to our expectations. The experiment was delayed because of the lack of availability of the heater. This successfull developpement was largely made with the efficient help of the ID20 staff and now belongs to the ID20 beamline. It is thus available for other users.

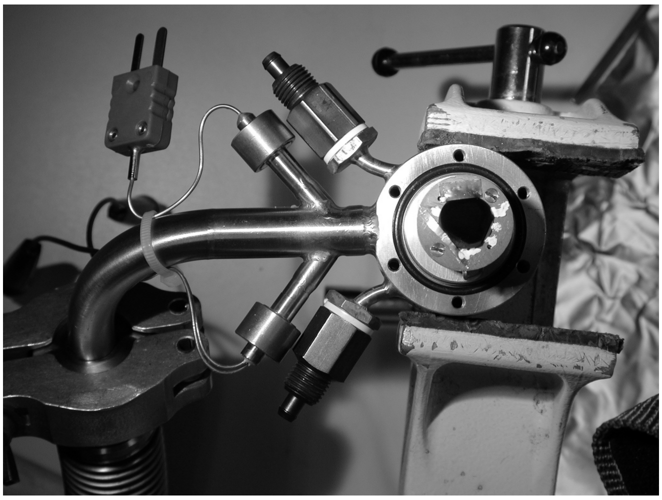

Figure : Top view of the furnace without the Be cap. The NiO sample si visible on top. The water, thermocouple and vaccum feedthroughs in the lower part of the device are also visible.

Several hours were necessary to stabilise the temperature and to realign the diffractometer because of the thermal drift at each new temperature point. Several incidence angles were tested in order to find the best signal/noise position.
Between room temperature and 270°C 12 complete quantitative data sets were taken at 0.3° (surface sensitive) and 3° (bulk sensitive) incidence angle. A quantitative measurement correspond for a complete familly 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 measurment times became large (about 4 hours for the last points) to get a reasonable integrated intensity.

The result is a major success since we could measure the domain structure evolution in bulk and surface with respect to the temperature. The Néel transition for NiO has been found of second order in bulk and surface and the transition temperatures showed to be different of only several degrees.

Since less time was available than asked the Co/NiO(111) part could not be done.