ESRF	**Experiment title:** Nature of the p(2×2) reconstruction of NiO(111) by grazing incidence X-ray diffraction (GIXD)	**Experiment number**: SI-536
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Shifts: 12	**Local contact(s)**: Kevin PETERS	*Received at ESRF:*

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Report:

Polar (111) surfaces are theoretically unstable due to the divergence of the Coulomb potential, unless they are relaxed or reconstructed. On the other hand the magnetic and catalytic properties claim for a stable surface. In our previous experiments [1] we have shown by GIXD, directly on a single crystal, that the NiO(111) surface is stable and adopts the octopolar reconstruction after air annealing. A UHV annealing cleans the surface but leads to strong modifications [2] of the atomic structure of the p(2×2) reconstruction and to metallisation at rather low temperatures (<400°C). A two domain octopolar reconstruction was found on a NiO(111) film grown on Au(111). The proposed experiment aimed at measuring the structure of a clean NiO(111) surface prepared under high O_2 pressure to avoid the structural modifications observed in UHV annealing.

The recently commissioned UHV/High-Pressure chamber on ID03 was expected to allow the annealing of the NiO single crystal in similar oxygen environments to air (i.e. 0.2 atm.). Like in the air annealing case, the surface reconstruction is then not expected to change during an anneal at 700°C (during 30 minutes) but the surface should be clean after this treatment. The preparation method suggested here was expected to give a unique opportunity to measure the octopolar reconstruction on a clean surface.

Extensive data sets were collected for the as-installed surface, for comparison to the cleaned surface. The NiO(111) single crystal had the expected quality (mosaicity less than 0.03 degrees) and a large set of reference measurements (in plane measurement of the reconstruction peaks within the accessible reciprocal space, 5 crystal truncation rods and 6 reconstruction rods) were performed. As expected the sample was not modified through the bake out of the chamber.

Unfortunately, the in situ sample heater proved unable to provide the necessary heat treatment in the oxidizing environment of 0.2 bar O_2. Specifically, failure of the graphite electrical contacts occurred during heating above T~300-400°C and again, after an attempted repair, in a few seconds at 700C. After the unsuccessful attempts we have concluded that, although that heater worked with CO, we have not the necessary technical solutions to heat at 700°C under O_2 yet.

Once an oxygen-compatible heater has been designed and tested, we would like to re-propose the present NiO experiment.

With the remaining time we have started to investigate the MgO(111) surface. The corresponding proposal was on the reserve list of ID03 (SI-537). The special surface preparation has largely been inspired by our experience with NiO(111). The crystal appeared to have an extremely high structural quality (mosaicity of 0.01°, intense and sharp crystal truncation rods) and a very well defined p(2x2) reconstruction with sharp and intense peaks as it can be seen in figures 1 and 2. The overall quality of the MgO(111) surface is close to the best MgO(001) surfaces we have studied in past years.

Since the horizontal geometry tends to limit the accessible in-plane region of the reciprocal space because of the polarisation we could not measure the reconstruction peaks far away from the origin of the reciprocal space but we could evidence reconstruction rods and intense crystal truncation rods. Additional beam time on a better suited setup (vertical sample) will be necessary to complete these very promising first measurements.

References :

[1] A. Barbier, C. Mocuta, H. Kuhlenbeck, K. F. Peters, B. Richter, and G. Renaud, Phys. Rev. Lett. *In press*

[2] A.Barbier and G.Renaud, Surf. Sci. Lett. 392 (1997) L15

Figure 1 : In plane scan along the (h,0,0.05) direction. The lattice is indexed in the triangular surface lattice of the reconstruction.

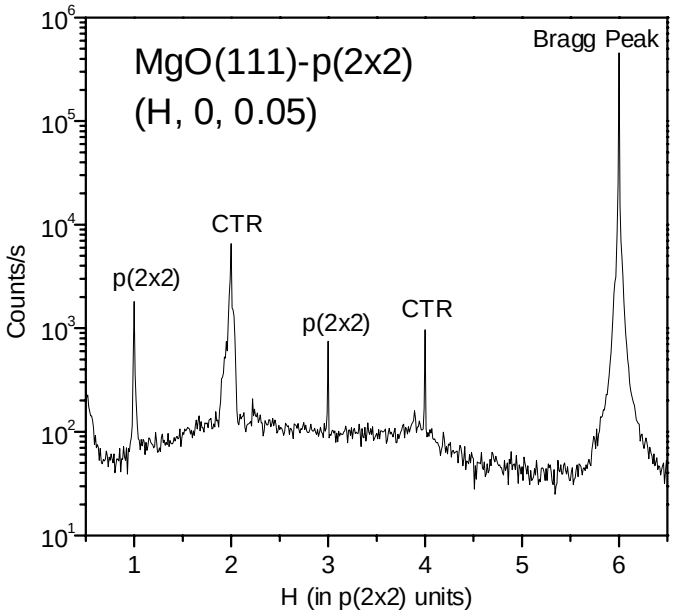

Figure 2 : Rocking scan around the (1,0,0.05) reconstruction peak. The lattice is indexed in the triangular surface lattice of the reconstruction.