ESRF	**Experiment title:** Study of the formation and the termination of the NiO(11 l)-P(2x2) surfaces by grazing incidence x-ray diffraction.	**Experiment number:** SI-359
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Report:

In the present experiment we have continued experiment SI-265. Again, we have learned a lot about the very surprising NiO(111) surface. Although this surface is polar, we have shown that it is stable enough to be investigated by grazing incidence X ray scattering.

One of the challenges of the present experiment, to go beyond the results of SI-265, was to improve the single crystal quality, i.e. to get a smaller mosaic spread and less background due to bulk defects. A new sample preparation was thus used. As described in the proposal the sample was polished on both sides. Its surface mosaic spread was only 0.1°, as compared to 0.5-0.7° in the SI-265 experiment. The bulk mosaic spread was very small, (less than 0.01°, limited by the experimental resolution).

A drawback of this highly improved crystalline quality is that, in the well ordered state, the samples become very fragile and release constraints introduced by mounting the sample by a macroscopic deformation of the surface due to the magnetic domain wall motions. The sample mounting was thus particularly difficult. We have chosen to turn over the sample in order to investigate both sides rather than attempting a mounting which probably would have broken the sample. This choice lead to several sample transfers during the experiment.

Without any preparation, the well ordered sample was found to exhibit a p(2x2) surface lattice mesh with small structure factors on both sides. Figure 1 shows an in-plane scan in a direction where neither Bragg peaks, nor truncation rods of the unreconstructed crystal exists. A reconstruction peak is observed at each half integer value of the K index.

The in plane reconstruction peaks were measured on both sides : Typically 20-30 non equivalent reflections were accessible, equivalent reflections were recorded to assess the symmetry and to evaluate the systematic error. The Patterson maps analysis showed immediately that the structure is different on each face (figure 2 top and bottom). This seems to corroborate the predictions based on electrostatic arguments and answers one of the major questions of the proposal. Out-of-plane measurements were also performed along selected rods. The (0.5,0,L) (2,2,L), (0 ,1.5,L) and (0.5,0.5,L) rods were quantitatively measured up to high L values. These data should lead, to a structural model of both sides of this polar oxide crystal.

We have followed the transformation of the reconstruction on one face during anneals at increasing temperature. In- and out-of-plane measurements were performed with the sample held at 140°C, 172°C, 210°C, 320°C and 430°C. It appears that the in- and out-of-plane structure inside the p(2x2) mesh evolves at very low temperatures. At 430°C the reconstruction was almost completely transformed.

At 430°C metallisation occurred and the rods of the p(2x2) structure uniformly decreased. Moreover, the intensity along the crystal truncation rods decreased showing that the structure changes at the buried interface between Ni and NiO. Some truncations rods were quantitatively measured. Finally the metallic Ni layer was investigated and characterized. Again, several diffraction rods were measured quantitatively.

The present experiment yields a number of new and very interesting results on NiO(111) single crystal surfaces. Although these are strong experimental evidences they seem to be in contradiction with the literature about NiO(111) thin films. Work will still be needed to get a clear description of the physical rules governing polar surfaces.

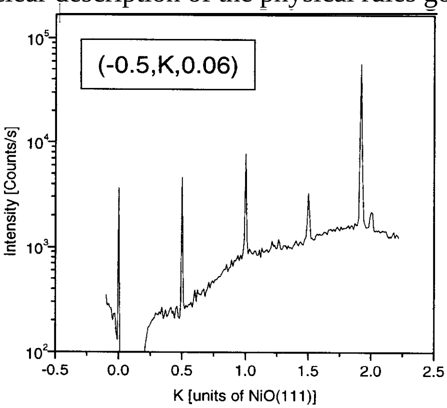

Figure 1 : in plane scan along the (-0.5, K, 0.06) direction on a as introduced NiO(111) crystal.

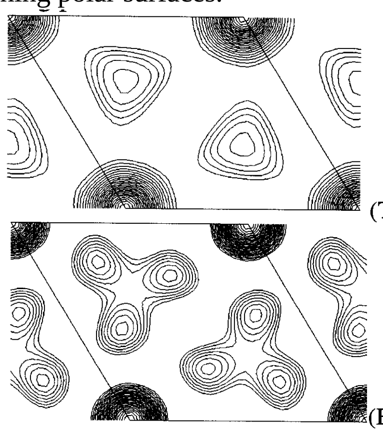

Figure 2 : Patterson maps measured on the t
of the NiO(111) single crystal.