

**Experiment title:**

STUDY OF THE STABILITY OF THE **NiO(111)** SURFACE  
STRUCTURE BY GRAZING INCIDENCE X-RAY DIFFRACTION  
(GIXD)

**Experiment  
number:**  
SI 265

**Beamline:**

ID 32

**Date of experiment:**

from: March 26, 97 to: March 31, 97

**Date of report:**

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**Shifts:**

18

**Local contact(s):**

A. Stierle

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**Report:**

To our knowledge, no experimental investigation of the NiO(111) single crystal surface was available. Indeed, it is often thought that, like MgO(111), the unreconstructed NiO(111) surface is unstable because it is polar. This has given rise, in the past, to the belief that polar surfaces must decompose in macroscopic (100) facets. However, recent theoretical studies indicate that the NiO(111) surface may be stabilised by a  $p(2 \times 2)$  << octopolar >> reconstruction [1, 2]. Our aim was to provide the first experimental data about NiO(111) single-crystal's surface structure including the nature of the terminating plane and the relaxation between the first atomic planes.

We have investigated the NiO(111) single-crystal surface by GIXD which is not limited by the insulating character of the surface. A first test experiment was performed on the D32 beamline in order to determine the surface preparation conditions [3]. It showed that the NiO(111) surface is relatively stable and not reconstructed after air annealing.

It evidenced also an original behaviour when the surface is UHV annealed: metallic epitaxial Ni segregates in clusters which can be transformed in NiO through oxidation. The metallic Ni shows a  $5 \times 5$  reconstruction which may be a dislocation network. The preparation of other crystals was successfully performed on the ID32 station in a very clean vacuum ( $3.10^{-11}$  mbar). Under defined conditions [3] a  $p(2 \times 2)$  reconstruction takes systematically place. All accessible fractional order reflections were recorded on two different samples as well as several crystal truncation rods. Since thin film experiments indicate that NiO(111) may unreconstruct when water is introduced in the UHV chamber the stability of the reconstruction against several gases ( $H_2O$ , NO) was also investigated.

The reconstruction is surprisingly highly stable but although it is  $p(2 \times 2)$  the proposed theoretical octopolar reconstruction, even when relaxed, is ruled out by the Patterson map analysis (compare fig. 1 and fig. 2). Only a model with a mixed Ni enriched surface plane reproduces the map. Macroscopic (100) facets were not observed. As expected the present experiment is a milestone in the investigation of polar surfaces and the results are of major importance. Moreover we have shown that GIXD is a well adapted tool to investigate polar oxide surfaces. Unfortunately the theoretical predictions are not confirmed and many questions raise about the formation of the real  $p(2 \times 2)$  reconstruction and about the observed Ni clusters. Other experiments will be needed to get a clear description of the physics behind these surfaces. The present results will be presented at the ECOSS-17 conference and should be published in the corresponding proceedings.

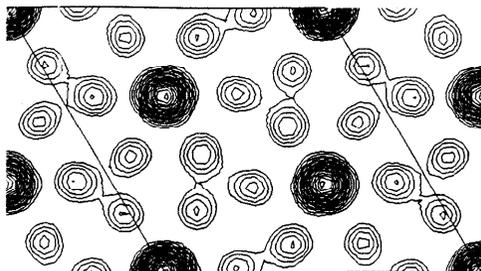


Fig. 1: Experimental Patterson Map.

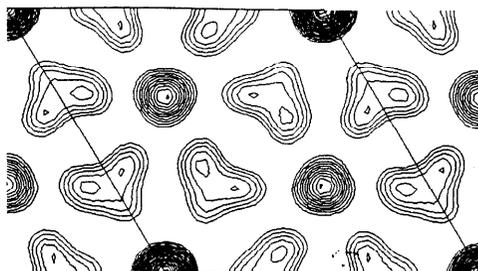


Fig. 2 : Relaxed octopolar structure.

#### References

- [1] D. Wolf, Phys. Rev. Lett. 68,33 15 (1992).
- [2] P.M. Oliver, G.W. Watson and S.C. Parker, Phys. Rev. B 52,5323 (1995)
- [3] A.Barbier, G.Renaud, In press in Surface Science Letters 1997