Experimental Report ESRF

Experiment code: SI 2379

The intended experiment consisted in a high-energy photoemission study of NiO surfaces, but due to experimental impediments it was not possible to be carried out. To make use of the beamtime, we decided to measure x-ray diffraction at different grazing angles on NiO films with modified properties. The films were grown by magnetron sputtering, using a mixed O_2/Ar plasma in which the oxygen content was varied between 0% and 70%. Especially interesting are the films grown on alumina nanoporous membranes, since NiO grows maintaining the nanopores of the substrate, creating some kind of antidots structure. The final properties of the NiO films strongly depend on the oxygen content of the plasma. For instance, the electrical conductivity can have a variation between 10^{-12} and 10^{-3} ohm-cm. The aim of the XRD experiment is to relate the observed changes in the macroscopic properties to the internal structure of the films.

The samples consisted of two series of NiO thin films grown by sputtering RF magnetron with two different power outputs: 100 W and 200 W. The substrates used were single-crystal p-type Sillicon wafers (100) and nano-porous anodic alumina membranes. The GIXRD measurements were carried out at the Spline CRG-beamline, branch B, using a photon energy of 14.5 keV. The incidence angle was varied between 0.5 and 0.12 degrees, which, according to the attenuation length curve for NiO, correspond to penetration depths between 200 nm and 5 nm, respectively. The aim of the experiment was to find out whether there were changes in the crystal structure of the films with depth.

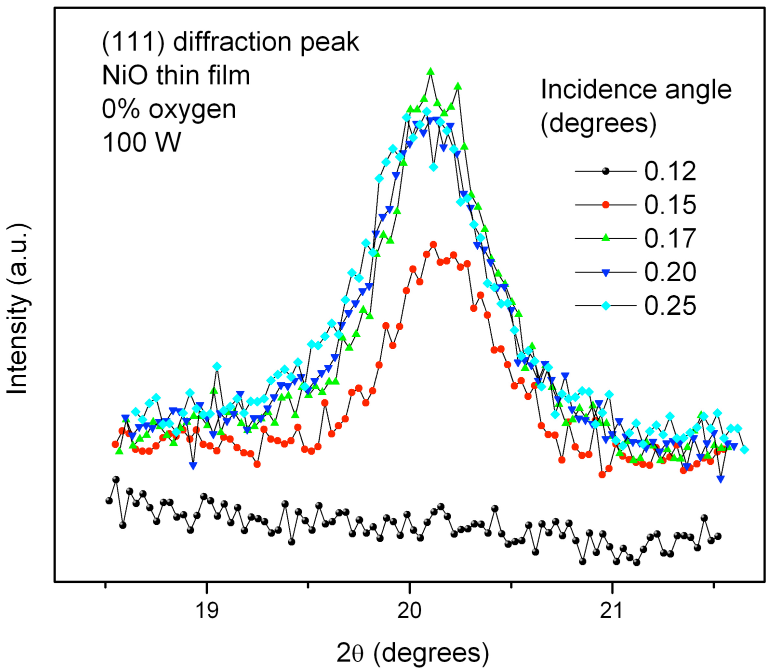

Figure 1

Figure 1 shows a detail of the diffraction pattern, the (111) peak, obtained for the sample grown at 100 W, with no oxygen content in the plasma, on a silicon substrate. At the lowest incidence angle, 0.12 degrees, the intensity of this peak goes to zero. This suggests that the uppermost layers of the NiO film are textured, and the (111) direction is not favoured. This direction is highly polar, with alternation of oxygen and nickel planes.

In Figure 2, the (200) diffraction peak of the same sample is shown. The differences in intensity observed at different incidence angles are due to the deviations in cross section at so extremely low angles. It can be observed that the position of the peak is not constant for all incidence angles, which suggests that the crystal lattice of the film changes with depth.

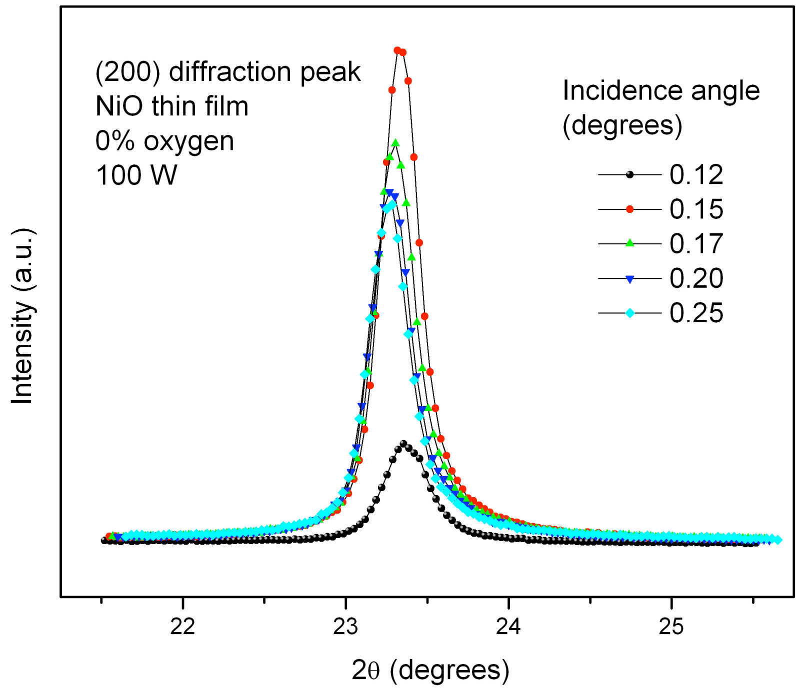

Figure 2

Figure 3 shows the (111) and (200) peaks of a sample grown at 200 W onto a Si substrate kept at 300ºC with no oxygen in the plasma. Samples grown at 200 W have a larger thickness than those grown at 100 W. In this case, the position change of the peaks is more evident than in Figure 2, with a clear trend towards higher angles (lower lattice parameter) when decreasing the incidence angle. This suggests that the lattice parameter of the inner layers is larger than that of the most external ones. This changes observed in depth, both in texture and in lattice parameter, are probably related to the changes in physical properties observed on similar samples.

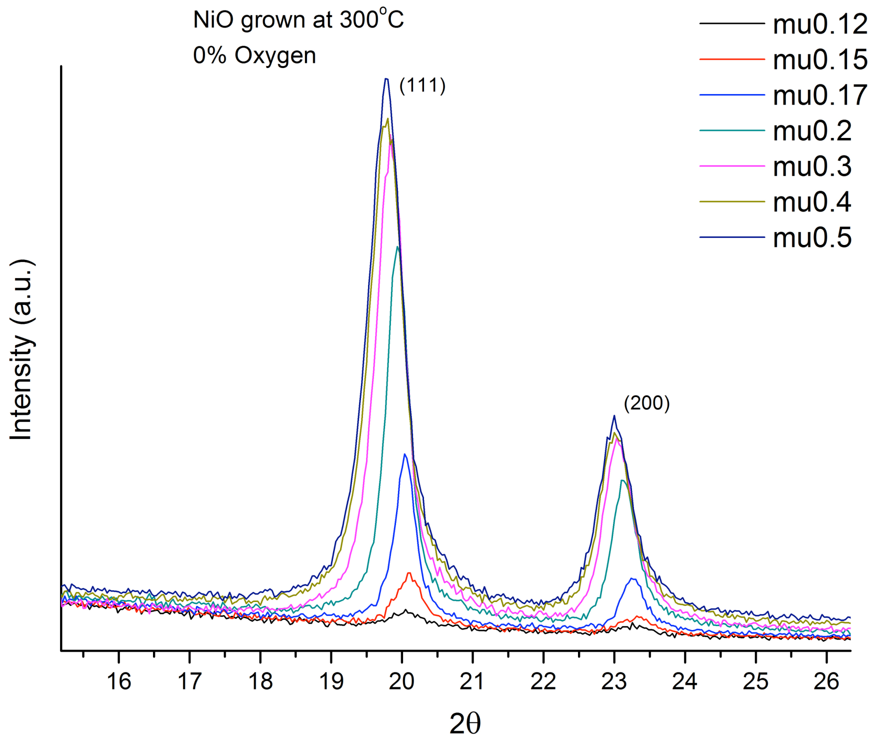

Figure 3

In order to quantify this effect, the diffraction curves have been fitted using a standard Rietveld refinement procedure. The results are shown in Figure 4 for sample grown at 200 W with 0% oxygen in the plasma, and with the substrates kept at 300ºC during growth. Values of the attenuation length have been obtained from B.L. Henke *et al.*, Atomic Data and Nuclear Tables, Vol. **54** (No.2), 181-342 (July 1993) for standard NiO. The lattice parameter clearly increases from the lowest attenuation length, which corresponds to the topmost surface layers, to the highest attenuation length probed in the experiment, which covers the whole film thickness. The values expand from 4.237 Å to 4.295 Å.

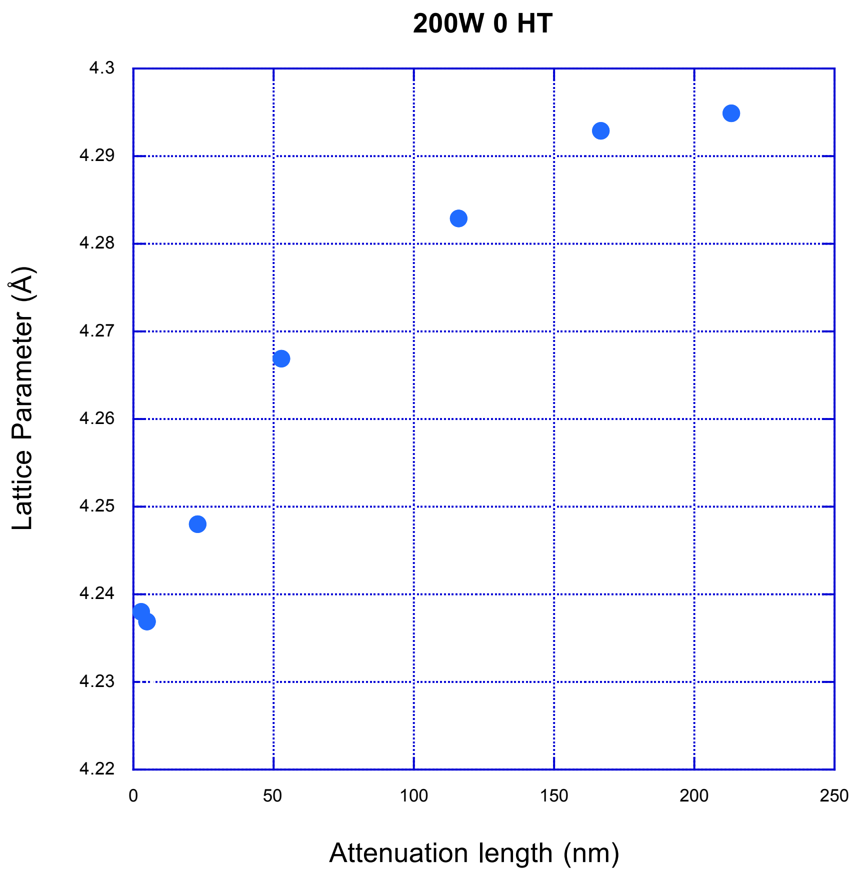

Figure 4