Experimental Report ESRF

Experiment code: MA 1171

The aim of this experiment was to correlate the macroscopic properties observed on modified NiO membranes with their structural properties. The samples consisted of nanoporous NiO membranes grown by means of magnetron sputtering with a mixed oxygen-argon plasma, using a nanoporous alumina membrane as substrate. Some of the physical properties of the membranes can be controlled by the oxygen content in the plasma during the growth process. As the oxygen content of the plasma increases, both the electrical conductivity and refraction index of the NiO membranes increas appreciably. To understand the mechanisms involved in these processes, the correlation with the internal structure of the films is of capital importance.

The samples consisted of a series of NiO membranes grown by sputtering RF magnetron with different oxygen content, from 0% to 70%. We have made use of the six-circle diffractometer available at BM25 and measured the samples by grazing incidence x-ray diffraction, to obtain a detailed description of the in-depth lattice parameter of the NiO deposits. Additionally, we measured x-ray reflectivity in order to determine the thickness of the NiO deposits on the membrane. Finally, we also measured carried out a Grazing Incidence Small Angle x-ray Scattering in order to determine the porous distribution size and distances in the samples.

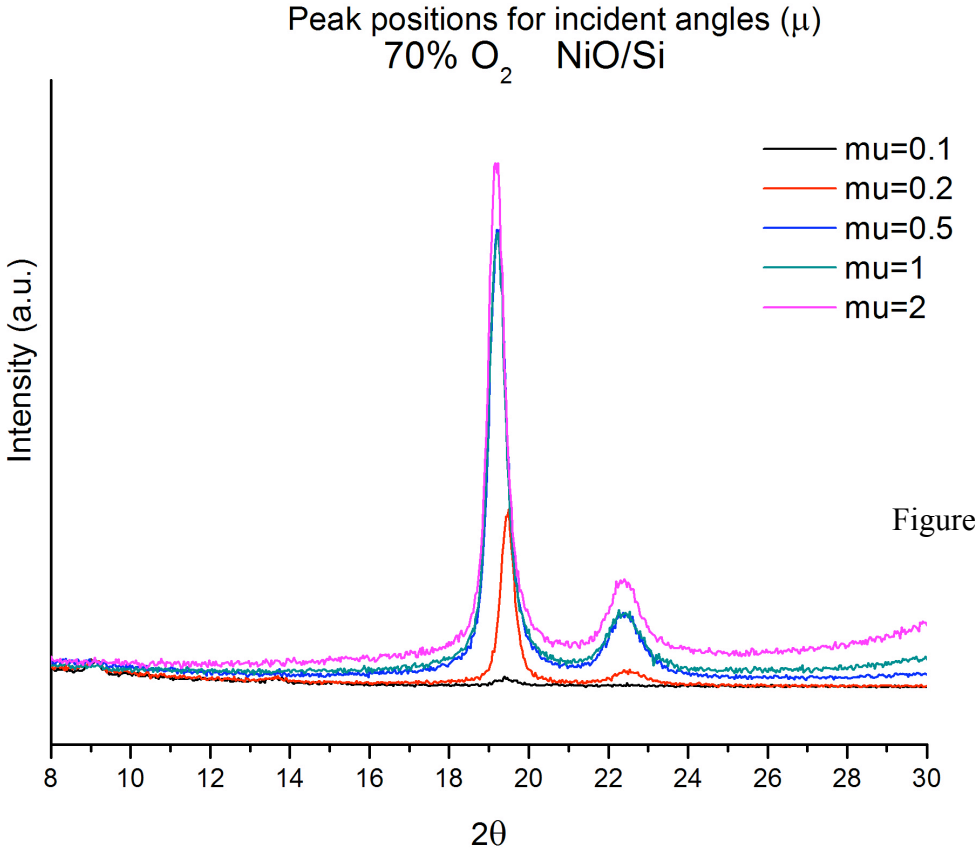

Figure 1

Figure 1 the diffraction pattern, obtained for the sample grown with 70% oxygen in the plasma, on a silicon substrate. There is a clear shift of the (111) peak towards higher angles as the incidence angle decreases, suggesting a change in the lattice constant as with depth: the lattice parameter would be higher closer to the interface.

In Figure 2, a detail of the diffraction pattern of several NiO samples grown on a membrane substrate, with different oxygen content, is shown. The most intense diffraction peak at about 20.5 degrees corresponds to the signal from alumina. The NiO peak is located at about 19.5 degrees. It is clearly observed that the position of the peak is different for different oxygen contents. These changes can be related to the different physical properties observed for the different samples.

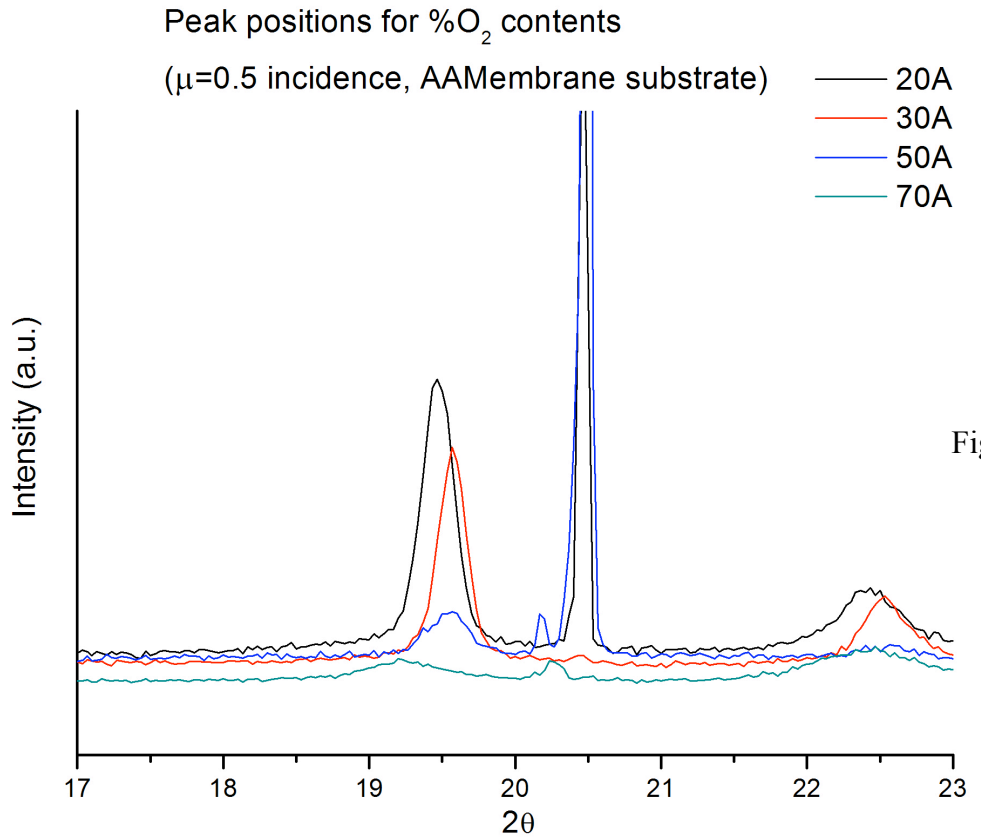

Figure 2

Figure 3 shows a two-dimensional plot with the small angle scattering data. It is possible to see a spot apart from the zero order beam, with a gamma angle of about 0.65. Unfortunately, the pattern is incomplete and the angular range did not allow to obtain any relevant information of the measured samples.

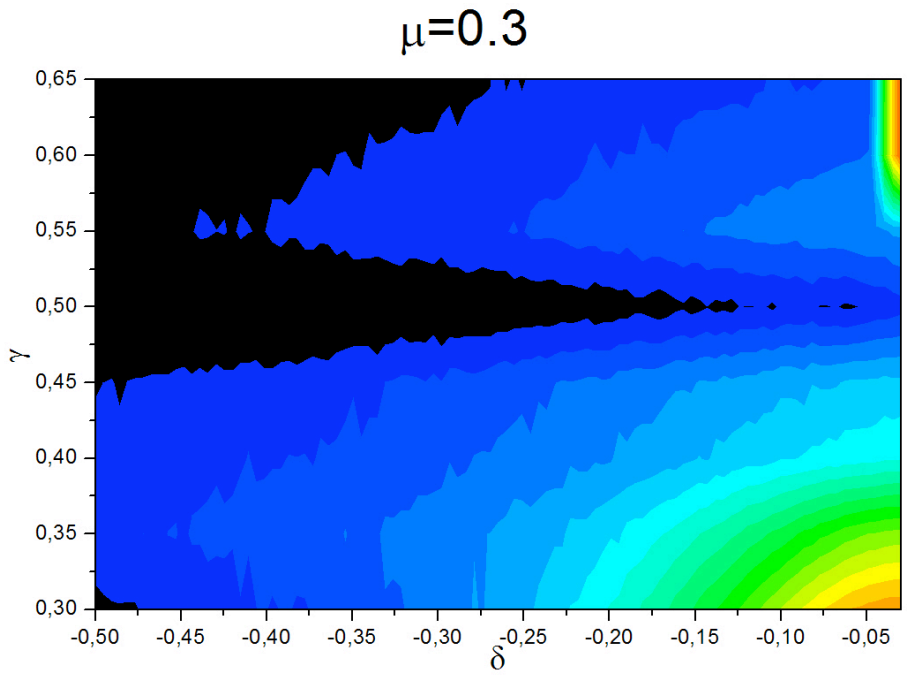

Figure 3