Experimental Report ESRF

Experiment code: PM25-732

The aim of this experiment was to measure the interatomic distances and coordination numbers around Ni in metastable NiO thin films. For this we used EXAFS technique at the Ni-K edge, which offers a very accurate information on distances between the emitting atom and its neighbours.

The samples consisted of a series of NiO thin films grown by Sputtering RF Magnetron with a gradient of increasing Oxygen content in the Plasma from 0% to 70% using a NiO magnetron target. The substrates used were single-crystal p-type Sillicon wafers (100) and nano-porous anodic alumina membranes.

31 samples were successfully measured by EXAFS in BM25 SpLine beamline, branch A. The Ni K absorption edge (8200-9200 eV) was measured with a solid-state multichannel detector in fluorescence mode. The EXAFS results show a double structure both at the Ni-O and Ni-Ni bond distances for all samples. This could be an indication of either a modified cell in the crystal structure, or a mixture of two different phases. The obtained data have quite good statistics so as to obtain reliable information until the second peak in the FT of the absorption EXAFS oscillations.

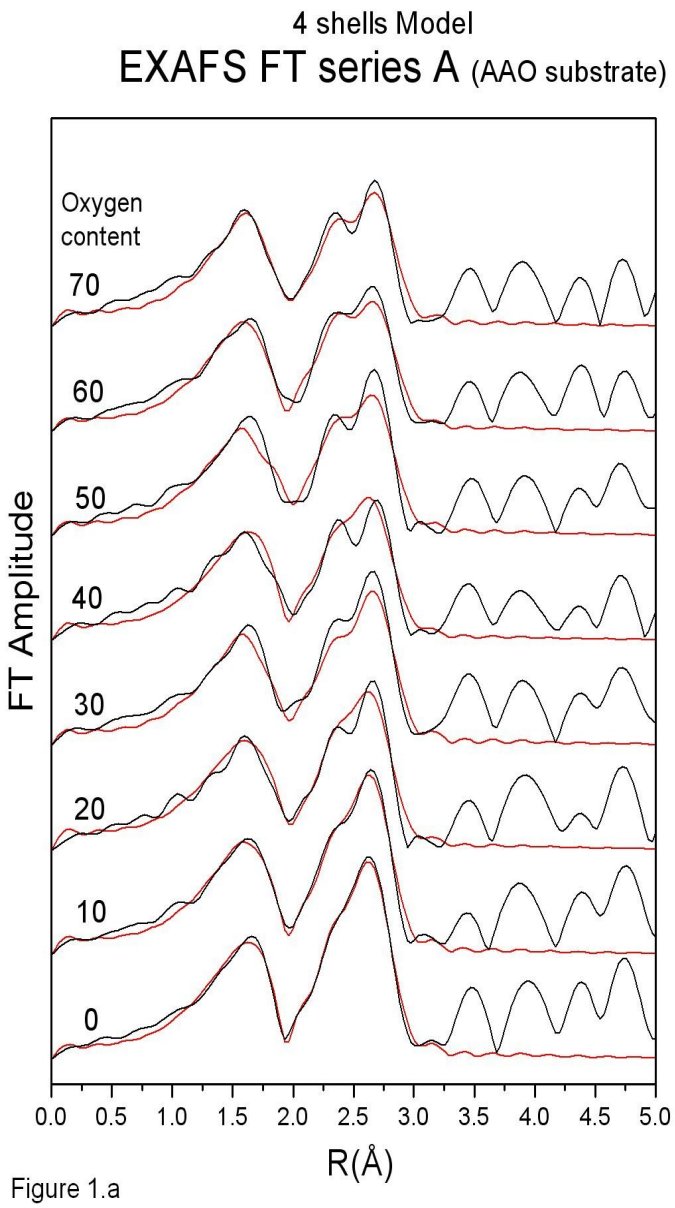

Figure 1.a

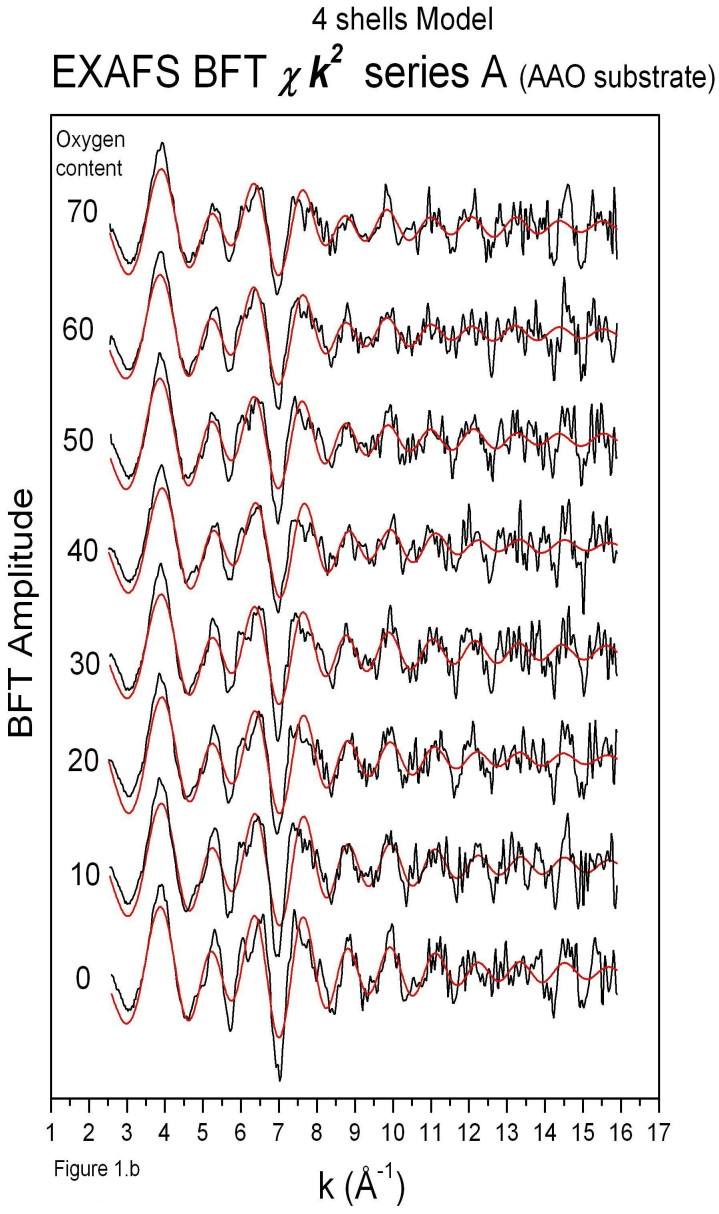

Figure 1.b

Figure 1.a shows the Fourier transform (FT) of the original data obtained through a standard EXAFS analysis procedure (black). The red lines correspond to the fitting of the FT using the VIPER package extended to the first four peaks. Fig. 1b shows the results of this fit on the original data in the reciprocal space. EXAFS data have been analysed using several computing programs: FEFF, Atoms, Athena and Viper. Two models were used to simulate the distances of the Nickel and Oxygen atoms in the crystal structure using the Atoms program to generate phase shifts and amplitudes of the most probable electron interference paths and fitting them with the Viper program.

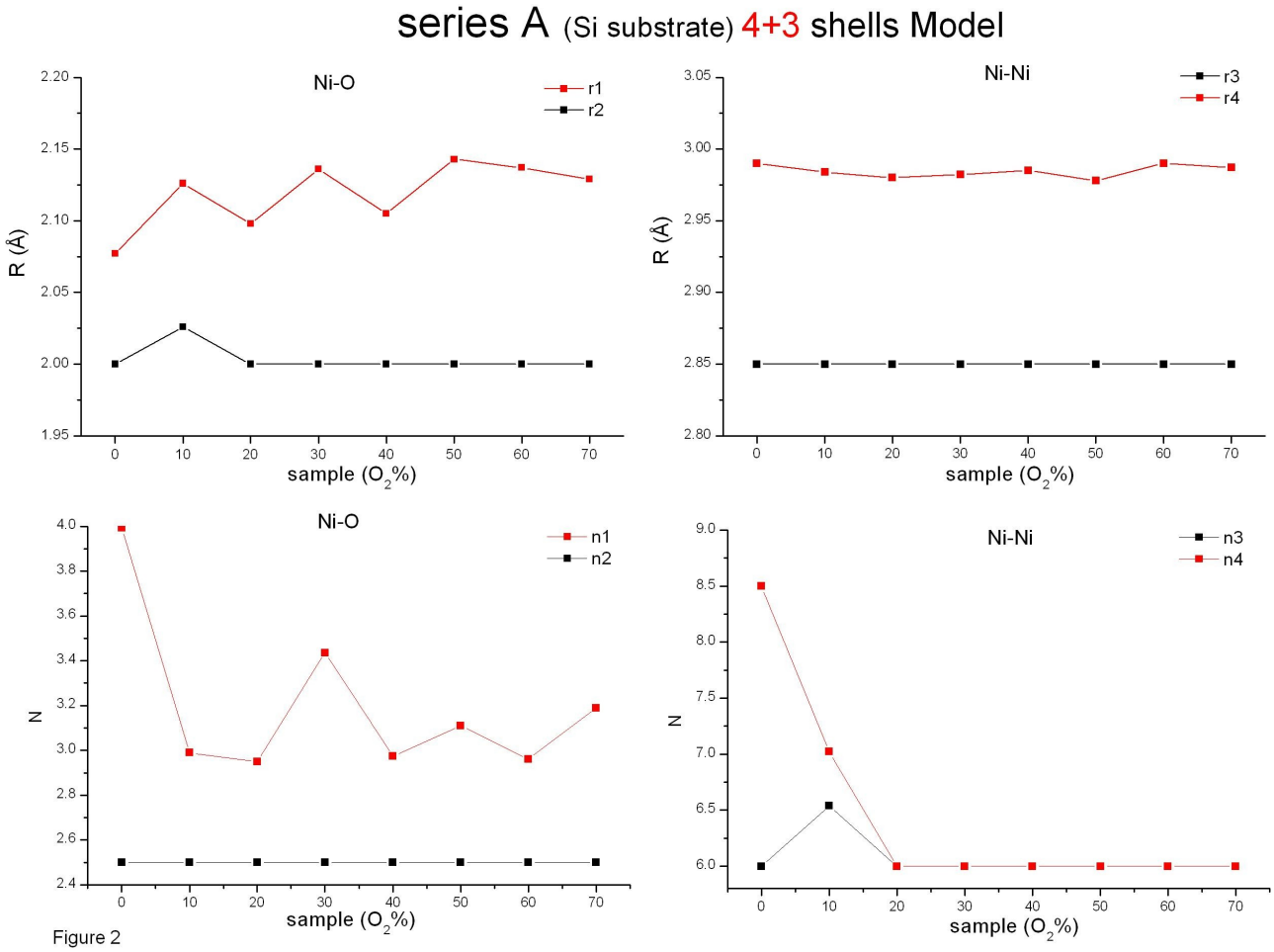

Figure 2

Figure 2 shows the summary of the fittings for each of the four peaks in all samples grown on the Si substrates. The upper figures show the adjusted distances, and the lower figures the coordination numbers. As it can be seen, atomic distances Ni-O and Ni-Ni show two differentiated positions and different with respect to the NiO reference atomic distance also measured in the experiment: Ni-O ref.: 2.11 Å and Ni-Ni ref.: 2,98 Å. It would be possible to obtain valuable information of the EXAFS data and correlate the bond distances to the Oxygen content in the samples. Future simulations and studies of the data will show a complete understanding of this behaviour in the NiO crystal structure. Additionally, X-ray diffraction experiments can help to understand the origin of the double features observed in EXAFS.