ESRF	**Experiment title:** Crystallographic study of epitaxial (111) NiCo2O4 thin films with spinel structure and with coexisting rock salt and spinel structural phases	**Experiment number**: MA4467
Beamline:	**Date of experiment**: from: 03/02/2021 to: 09/02/2021	**Date of report**: 12/02/2023
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Report:

The effect of the post-annealing temperature $NiCo_2O_4$ thin films grown by Ion beam sputtering on Al_2O_3(0001) substrates have been studied by Grazing Incidence Surface X-Ray Diffraction (GIXRD) experiments at the BM25-SpLine beamline at 13 keV at the ESRF (Grenoble, France). Several crystallographic measurements (CTRs, RODs, low and high angle XRR, among others) have been performed in order to determine the crystalline structure of the films, elucidate the presence of strains generate during the epitaxial grown and determine the presence of the different phases present in the films as well the possible chemical inter-diffusion at the interface in the post-annealing processes.

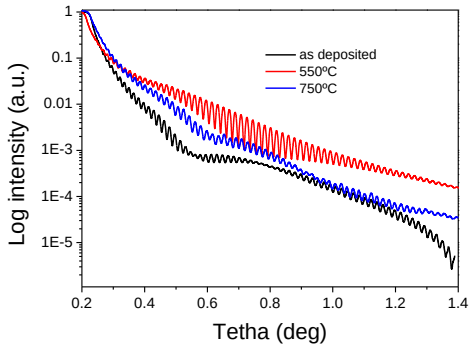

Figure 1. Low angle XRRs of $NiCo_2O_4$ epitaxial films as deposited and after annealing at 550 ºC and 750 ºC

Figure 1 displays the low angle XRR result for a $NiCo_2O_4$ thin film as deposited and after post thermal annealing a 550 ºC and 750 ºC. It can be observed the presence of two contributions in the case of the as deposited film, one due to the thickness of the film and other due to an intermediate layer that disappears after post-annealing at 550ºC and emerge again during post-annealing at 750C. This result is in agreement with high angle XRR results and the electrical properties of the films. The film obtained after post-annealing a 550 ºC shows the lowest crystallinity but also the lowest resistivity.

From the Reciprocal Space Maps (RSM) measurements, it is determine the crystallographic axes of the different lattices that appear in the films. It is possible to determine that the as deposited $NiCo_2O_4$ thin film grows incommensurate, based on the noncoincidence of the in-plane diffraction maxima from the film and substrate. This behaviour remains also in the films obtained after annealing temperatures between 450 ºC and 850 ºC, however the main effect observed in the post-annealing processes is a structural transformation from spinel structure to rock-salt structure. These findings can observe in Figure 2 where representative LH reciprocal space maps (RMS) are displayed for the as deposited film and after post-annealing at different temperatures indicating the coexistence of insulating rock-salt and conductive spinel structures after annealing at 650ºC or higher temperatures.