ESRF	**Experiment title:** **THE STRUCTURE OF EPITAXIAL Ni_xO_y GROWN ON Cu(111)**	**Experiment number**: SI-1133
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Preliminary Report:

We studied in detail the growth of nickel oxide on the Cu(111) surface, under different preparation conditions (i.e. different oxygen partial pressures during Ni deposition). We wanted to address in detail the low thickness regime for which interesting LEED patterns were observed [1].

The Cu(111) single crystal was mounted in a portable UHV chamber on the ID1 diffractometer, using horizontal surface scattering geometry. The typical Ni deposition rates were of 0.35 Å/min. (substrate temperature of 250°C, in partial oxygen pressure of 1 to $6*10^{-6}$ mbar), yielding to NiO layer thicknesses in the 5-15 Å range. The base pressure in the chamber was $1.1*10^{-9}$ mbar ($1*10^{-8}$ mbar base presure during Ni deposition). Prior NiO deposition the Cu(111) single crystal was cleaned by Ar^+ sputtering. NiO is known to be un-sensitive to contaminants, which made possible the study of its structure (measurements spanning over 1 day) in a base presure of 10^{-9} mbar.

The Cu(111) single-crystal sample is aligned in the x-ray beam using a hexagonal surface orientation matrix, i.e. the in-plane and out-of-plane unit vectors are related to the cubic ones by:

$$a_s = [\bar{1}\,10]_{cube}/2; b_s = [0\,\bar{1}\,1]_{cube}/2; c_s = [111]_{cube}$$

For large NiO thicknesses our data show the formation of the expected NiO(√3 x √3)R30° layer [1], with good epitaxy and crystalline quality. During this experiment the data previously measured for such situations were completed, especially with out of the surface plane measurements. We evidenced the presence of the 2 variants NiO(√3 x √3)R±30° in equal quantities.

Surprisingly for the low thickness regime, at any probed oxygen pressures, part of the NiO structure was found to be very different from the structure measured in LEED, i.e. the specific surface superstructures were found different [1]. Although extra peaks were observed as a function of the preparation conditions (Figure 1), their symetry and position around the Bragg peaks is not compatible with the ±18° rotation used to simulate the LEED images. Also, at higher O_2 pressures, a noticable change in the peak shape (larger width) of the NiO(110) Bragg peak has been observed. In fact, a carefull analysis of the shape of the rocking curves reveals the existence of a sharp contribution on top of a broad (few degrees wide) peak.

During the Ni deposition under O_2, the Cu substrate surface is roughening (expected 3-dimmensional growth) as proved by the decrease of the intensity along the substrate crystal truncation rods (CTR).

Moreover, interference effects appear on the CTRs (figure 2a). These measurements will be used to determine the registry of the NiO film with respect to the Cu substrate. The in-plane scans (L≈0) along high symmetry directions (figure 2b) show the appearance of the expected NiO(111)R30° structure (peaks at half-integer values), but also of low-intensity peaks corresponding to a different NiO epitaxy. Although this last mentionned structure is not the one measured in LEED, it is clearly different from the R30° one, and seems to be present in small quantities only.

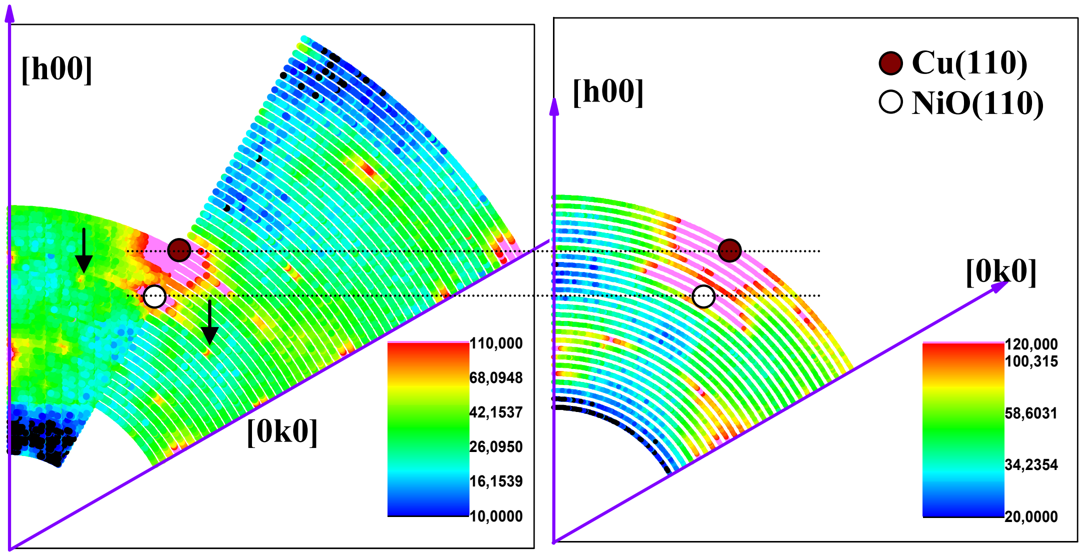

***Figure 1 (color)**: Comparison of in-surface-plane reciprocal space maps (similar intensity color scales) for NiO films prepared at 10^{-6} mbar (left) and $6*10^{-6}$ mbar (right) O_2 pressure respectively. The positions of the Bragg peaks of the substrate and of the NiO film are indicated (full and open circle respectively). Arrows highlight peaks which disappear when a higher O_2 pressure is used.*

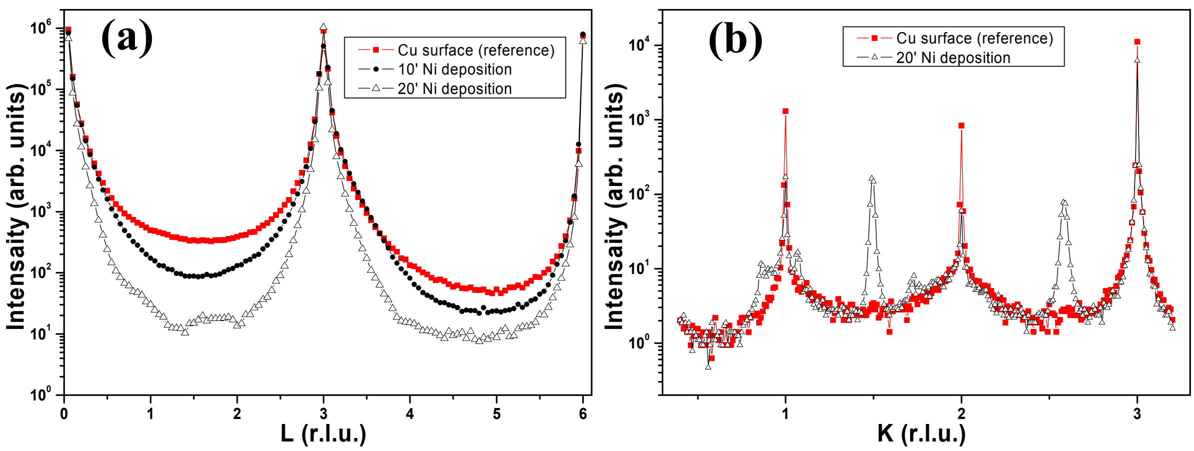

***Figure 2**: (a) CTRs measured for different NiO film thicknesses. The L-axis is given in reciprocal space lattice units (r.l.u.). Beside a roughening of the surface (decrease of the CTR signal), interferences (oscillations) are also evidenced. (b) In-plane scans along the [0k0] direction for different NiO film thicknesses. The half-integer peaks are attributed to the expitaxial NiOR30° structure.*

These results will allow to determine and better understand the epitaxy between NiO and Cu(111). The above mentionned measurements were performed for different thicknesses and O_2 partial pressures and will define the effect of the O_2 pressure during the NiO growth in the ultra-thin layer regime, in corroboration with existing XPS measurements.

Refereneces:

[1] S. Stanescu, C.Boeglin, A.Barbier, J.-P.Deville, Epitaxial growth of ultra-thin NiO films on Cu(111), Surf.Sci. **549** (2004) 172.