A correlative microscopy study of nickel and copper oxide nanostructures grown by a vapor-solid process

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Nickel and copper based oxides belong to the family of p-type wide band gap semiconductor oxides, demonstrating potential applicability in electrochemical capacitors, alkaline batteries, smart windows and gas sensing. They are commonly synthesized in form of nanoparticles, ceramic or thin films by sol-gel, electro-spinning or hydrothermal methods, and less has been done in the fabrication of micro- and nanostructures with elongated morphology such as nanowires, so far. Morphology engineering can lead to optimize most of these applications by reducing the size and selecting proper shapes. Alternatively, doping is an additional parameter, which can induce property improvements and originate new applications.

A catalyst free vapor-solid method, using commercial Cu and Ni powders of 99% purity were used as starting materials. The powder was pressed to form pellets, which were annealed at temperatures ranging from 600 to 1500 °C under controlled argon flow for durations of 5 - 15 h. For the Sn doped samples, the Cu or Ni precursor powder was mixed and milled using different amounts of Sn or SnO_2 in the initial mixture. Surface reconstruction and texturing of the precursor pellet is achieved for Ni containing samples treated at temperatures lower than 1200 ºC, exhibiting ordered hollow cavities which present square sections of hundreds of nm, resembling inverted square pyramids. Besides, changing the temperature conditions whether Ni or Cu based, elongated nanostructures with different kind of morphologies grew on the doped pellet surface, which acts simultaneously as the source of the precursor material and as a substrate. Studies of the morphology and characterization of the as grown nanostructures with SEM, TEM, EDS, CL and Raman in a confocal microscope were performed. XPS results measured at the ESCA microscopy beamline at the Elettra synchrotron (Trieste, Italy) will be also discussed. Sn doping not only induces the growing of nanostructures, but also induces changes in morphology, size and the Raman and CL signals. By adding Sn or SnO_2 to the precursors, both Sn doping of nickel or copper oxides or heterostructure SnO_2/MO_x (M=Cu, Ni) formation could be induced depending on the precursor, mixture ratio and temperatures used.