Synthesis and Characterization of ZnO nanotubes

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Abstract – ZnO regular shaped nanotubes coexisting with a structure of thinner nanotubes in bundles have been synthesized from $Zn(CH_3COO)_2.H_2O$ in CH_3OH by sol-gel method, using polycarbonate membranes (holes: 800 and 200nm), following with drying and calcination (550-750 °C). Characterization was performed by TEM, SEM and XRD. The straight nanotubes exhibit regular polyhedric forms and hollow cores (external diameter: 0.5-1 μ m, mean inner diameter: ~0.23 μ m and mean length: ~8,5 μ m). The nanotubular structure is tightly related to the hexagonal crystallography of ZnO. The bundles of thinner nanotubes (usually terminated in sharp tips) appear to self-assemble into various different microscopic structures.

The nanotubes represent a unique type of materials in which all atoms are located near the surface so, they exhibit amazing fundamental physical properties enabling important and wide possible applications in optics and optoelectronics as well as in biology. ZnO nanotubes or other nanostructured forms (nanowires, nanoribbons or nanoparticles) require an intensive research since their production yield, their morphology and crystalline quality are still usually poor.

Not aligned and well crystallized ZnO nanotubes and coexisting bundles of thin nanotubes terminating in sharp tips were synthesized from the $Zn(CH_3COO)_2$.H₂O precursor in a 15w/% CH₃OH (~0,6 M) solution by the sol-gel method, with impregnation by pressure difference, using polycarbonate membranes (holes: 800 and 200 nm), followed by IR-drying and a calcination (at 550-750 °C). The authors describe an original synthesis technique in this work, however sol-gel was technique has been used before [1, 2] to prepare nanostructured ZnO.

The XRD analysis indicated that nanotubes were hexagonal wurzite ZnO (space group P6₃mc, a = 0.325 nm and c = 0.5021 nm).

The nanotubes exhibit regular polyhedric shape with hollow core. The nanotubes external diameter resulted: $0.5-1\mu$ m, their mean inner diameter ~ 0.23μ m, mean wall thickness: ~ 0.07μ m, maximal lenght: $3.5 -13 \mu$ m and a mean lenght: ~ 8.5μ m. With regards to morphology, nanotubes are straight or slightly bended. The nanotubular ZnO structure is tightly related to the hexagonal crystallography of this oxide (**Figure 1**). Nanotube walls tend to grow along the c-axis [0001] and in the open extremes of tubes, hexagonal forms are typically observed. Directions on the hexagonal structure are depicted in **Figure 1**. There are also open ends of nanotubes showing rectangular or round shapes. Several nanotubes are broken along the c-axis.

The other coexisting structure shows bundles of thinner nanotubes (**Figure 2**) appearing to selfassembly into various different microscopic structures. They are formed by bended and twisted thin nanotubes which terminate in sharp tips, being only possible to observe the hollow core if nanotubes are broken. Different morphological coexisting forms in the same specimen result extremely interesting, however, to clarify their growth mechanisms, intense research is still required.



Figure 1: SEM image revealing the nanotubes, morphology related with the hexagonal ZnO structure is shown.



Figure 2: SEM image revealing thin nanotubes in bundles.

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