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Microwave assisted synthesis of nanostructured oxides and their applications

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Abstract – The synthesis method for obtaining nanostructures of oxide compounds is described. Pore wetting of porous polycarbonate templates with the liquid precursor was followed by a two-stage thermal treatment to obtain single phase oxides as hollow and solid structures, with external diameter determined by the template pore size. The first thermal stage, a microwave assisted process, determines the shape of the structures. The second treatment at higher temperature allows to obtain the crystallographic structure of the compound. A variety of techniques are used to characterize the morphology and properties of these nanostructures. Applications of some nanostructures are described.

Several single oxides (tin, silicon, titanium, etc.) and mixed oxides (manganites, cobaltites, etc.) as nanoparticles assembled into hollow or solid nanostructures were synthesized by wetting porous polycarbonate films. The pores were filled with liquid phases obtained from dissolution of metal nitrates or from organic or chlorides precursors. A two stage thermal process was performed to obtain the solid nanostructures, microwave irradiation and conventional thermal treatments [1,2]. Single phase nanostructures were obtained without impurities after completely burning the template.

Cylindrical structures, as wires or tubes, showed the feasibility to be sintered on top of solid substrates, crystalline or non crystalline. We tested the use of SnO_2 nanotubes as catalytic material in a gas sensor, which showed high sensitivity to isopropyl alcohol.

$La_{0.6}Sr_{0.4}CoO_3$ and $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$ nanotubes with different external diameters were tested as cathodes of intermediate-temperature solid oxide fuel cells (IT-SOFCs). These nanostructures were mixed with a commercial ink vehicle (IV, Nextech Materials). The ink was smeared on the electrolyte as thinly as possible with a brush and dried. Then, a fast-firing process was conducted in order to attach the cathode to the electrolyte without losing the hollow structure. The performance of these cathodes was enhanced for decreasing nanostructures diameter and dwell temperature of the fast-firing process [3].

Magnetic properties of $La_{1-x}Sr_xMnO_3$ and $La_{1-x}Ca_xMnO_3$ nanostructures were also studied. Each grain composing the nanostructures was determined to act as a single magnetic domain, with a dead layer (~ 2 nm) on the surface. The magnetic interactions between them are essentially of dipolar character [4].

Microstructural characterization was performed through X-ray diffraction analysis, AFM and electronic microscopy.

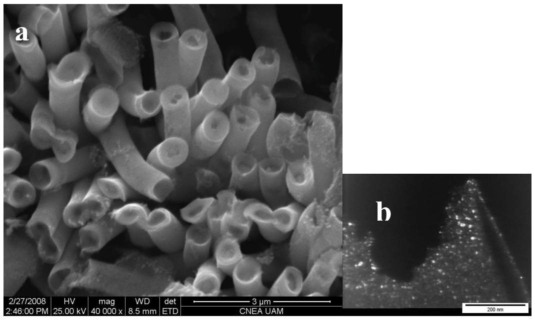

Figure 1: a) SEM image of SnO_2 tubular nanostructures deposited onto silicon nitride surface, b) dark field TEM image showed 7nm crystals forming the walls of the tubes.

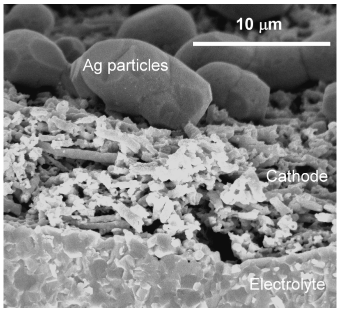

Figure 2: SEM image of a cathode for IT-SOFCs (lateral view).

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