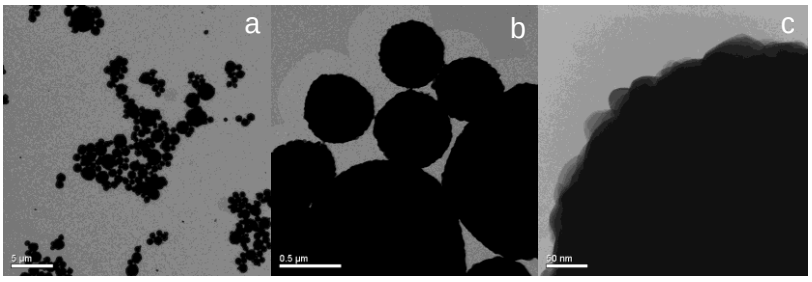
Synthesis and characterization of SnO_2 sphere nanostructures

Abstract - SnO_2 microspheres were synthesized by a chemical route, heating the mixed Sn^{2+} and sulfuric acid solution in the presence of pressurized oxygen at 900°C. Phase analysis was carried out by XRD and the results confirmed the SnO_2 microspheres as a single-phase tetragonal structure. SEM images indicated that these microspheres with average diameters of 0.9 μm were composed of SnO_2 nanoparticles with a diameter of about 4.7 nm and a crystallite size of 3.7 nm, as observed by TEM and calculated by Scherrer's equation. Formation mechanism of SnO_2 sphere nanostructures and the influence of changes in processing are proposed and explained.

Tin dioxide, an important n-type semiconductor with a wide band gap, is a key functional material that has a been extensively used for optoelectronic devices, gas sensors for detecting leakages, transparent conducting electrodes, catalyst supports, electrochemical modifiers on electrodes, solar cells, etc [1]. Over the last several years, considerable efforts have focused on exploring new routes to synthesize SnO_2 nanorods, nanowires, nanowire arrays, and nanobelts or nanoribbons [2]. SnO_2 spheres have also been fabricated by hydrothermal-based methods [3]. The experimental set-up used for the synthesis consisted of a horizontal tube furnace with the injection of a controlled atmosphere. SnO_2 powder was formed by a vapor-solid mechanism from a sulfuric solution with pH controlled between 3 and 4, in the presence of Sn^{+2} 0.25 $mol.L^{-1}$. This solution was deposited through a horizontal tube furnace at 900°C into a designed calorimetric pump capable of reaching the temperature required for the formation of the desired oxide, under pressurized oxygen gas (2 atm) at an amount exceeding the stoichiometric. The as-synthesized oxide was solubilized in an alcohol solution at room temperature and oven-dried at 80°C.

The resulting powder was characterized by XRD. All the peaks were fully indexed to the tetragonal rutile SnO_2 phase. The lattice parameters were calculated using the least-squares refinement of the UNITCELL-97 curso de python program. The parameters were: a = 4.733 °A and c = 3.161 °A with volume cell of 70.822 °A. These values are consistent with those reported in the literature and with the respective JCPDS card No. 41-1445. No characteristic peaks for other impurities were detected. However, the diffraction peak of the SnO_2 microspheres was broadened due to the small size of the crystallites, which was later confirmed by TEM (figure 1). Figure 1 shows three images by TEM with 4000 x (a), 60000 x (b) and 400000 x (c) magnification. The crystallite average size calculated by Scherrer's method was d_{110} = 3.7 nm.

Comparing the data of SnO_2 microspheres obtained by different methods, it is evident that the chemical route proposed here, which involves heating the mixed Sn^{+2} and sulfuric acid solution in the presence of pressurized oxygen at 900°C in a calorimetric pump, leads to small diameters at an instantaneous annealing time. This is an interesting result, since this method minimizes electrical energy costs, which is something that cannot be achieved by conventional hydrothermal-based methods. These SnO_2 microspheres are promising candidates for potential application in low cost nanocatalysis involving low temperatures and short preparation times.



References

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