

Synthesis of Mesoporous Zinc Oxide without Template

B. Murguía-Martínez¹, A. Medina¹, S.E. Borjas-García^{2*}, V. Sayil¹, P. Martínez-Torres², L. Béjar³

¹Instituto de Investigación en Metalurgia y Materiales, UMSNH, Morelia, Michoacán, México.

²Instituto de Física y Matemáticas, UMSNH, Morelia, Michoacán, México.

³Facultad de Ingeniería Mecánica, UMSNH, Morelia, Michoacán, México.

*Corresponding author: sborjas@ifm.umich.mx

Mesoporous materials have pore sizes in the range from 2 to 50 nm. These kinds of materials can be used in applications such as catalysis and molecular sieves [1]. Zinc oxide (ZnO), with a binding energy of 60 meV and a wide electronic band gap of 3.37 eV, has attracted great attention to itself due to its wide range of applications such as varistors, UV absorbers, antibacterial, light emitters, dye degradation, solar cells, gas sensors, photoluminescence materials and transparent conductors [2]. Several chemical methods such as sol-gel, hydrothermal micro emulsion, homogenous self-assembly, microwave-assisted hydrothermal, spray pyrolysis, electrochemical methods and direct precipitation have been reported for the synthesis of ZnO nanostructures [3]. For the synthesis of mesoporous zinc oxide, the sol-gel technique has clear advantages over other techniques due to the excellent control of the composition, the homogeneity at the molecular level due to the mixture of liquid precursors and the lower temperature of crystallization, in addition to this work, a template was not used to obtain mesoporous particles. The mesoporous zinc oxide in this research work, was prepared using a hydrothermal soft treatment. In the first step, two solutions were prepared. The first one was obtained by dissolving 0.020 mol of zinc acetate dihydrate in 1.20 mol of distilled water. For the second solution, 0.02 mol of sodium hydroxide was dissolved in 1.20 mol of distilled water. In a second step, the Na-solution was added slowly (drop by drop) to zinc acetate solution and stirred at 500 rpm for 30 min at room temperature to obtain a material with a molar ratio of Zinc: NaOH: H₂O = 1:1:120. The gel obtained was aged in a stainless-steel autoclave at 120 °C for 1 day. After the hydrothermal treatment, the sample was washed with distilled water and centrifuged at 12000 rpm for 10 min. After that, the material was dried at 80 °C for 1 day. Furthermore, it was necessary to calcine the sample at 560 °C for 1 h to eliminate the organic residues. The microstructural characterization was carried out by using a Bruker D8 X-Ray Diffractometer in order to identify the phases formed after synthesis procedure. The morphology of as-synthesized sample was analyzed by using a high-resolution Scanning Electron Microscope FEG-SEM JEOL JSM 7600.

Figure 1 shows an X-ray diffraction pattern. This figure shows several peaks corresponding to ZnO phase which correspond to wurtzite phase with hexagonal crystal structure.

Figure 2 shows a SEM image; the size of the particles is in the range from 0.2 and 0.7 μm with a hexagonal shape (figure 2a). In addition, the particle size is directly related to the ionic strength. After nucleation, the excess hydroxyl ions in the solution are adsorbed on the polar faces of the growing particles to form of hexagons shape and the water and zinc acetate induced a considerably accelerated growth [4].

On the other hand, in figure 2b it is possible to observe the presence of mesopores on hexagonal crystals, the size of mesopores are less than 50 nm. The concentration of hydroxyl ions played an important role for the shape and size of the hexagonal crystals. The nature hexagonal shape of ZnO could help in the formation of mesopores. From this research work, it was observed that the treatment at higher temperatures did not allow the formation of porosity in the hexagonal crystals of ZnO.

References:

- [1] D. W. Bruce, D. O'Hare and R. I. Walton, "Porous materials", 1st ed. (Wiley, United Kingdom, 2010) et al., p.1.
- [2] L. H. Cheng et al., Elsevier vol. 38 (2012), pp S457-S461.
- [3] J. N. Hasnidawani et al., Elsevier vol. 19 (2016), pp 211-216.
- [4] Y. L. Zhang et al., Springer vol. 51 (2009), pp 198-203.

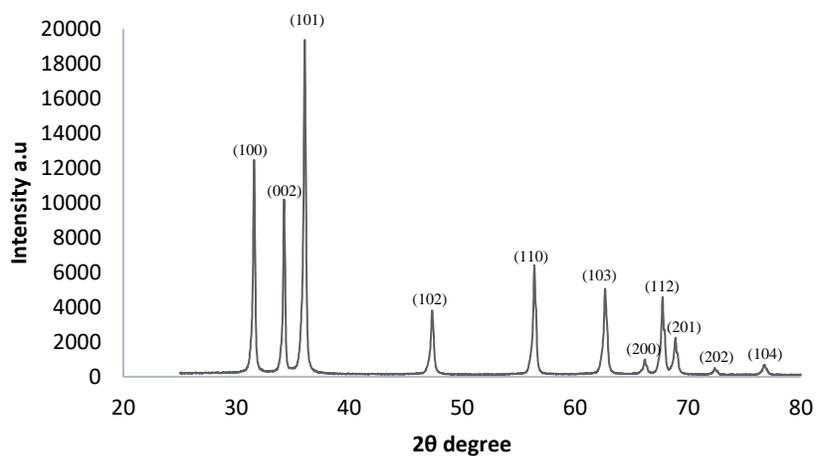


Figure 1. X-ray analysis of mesoporous zinc oxide particles.

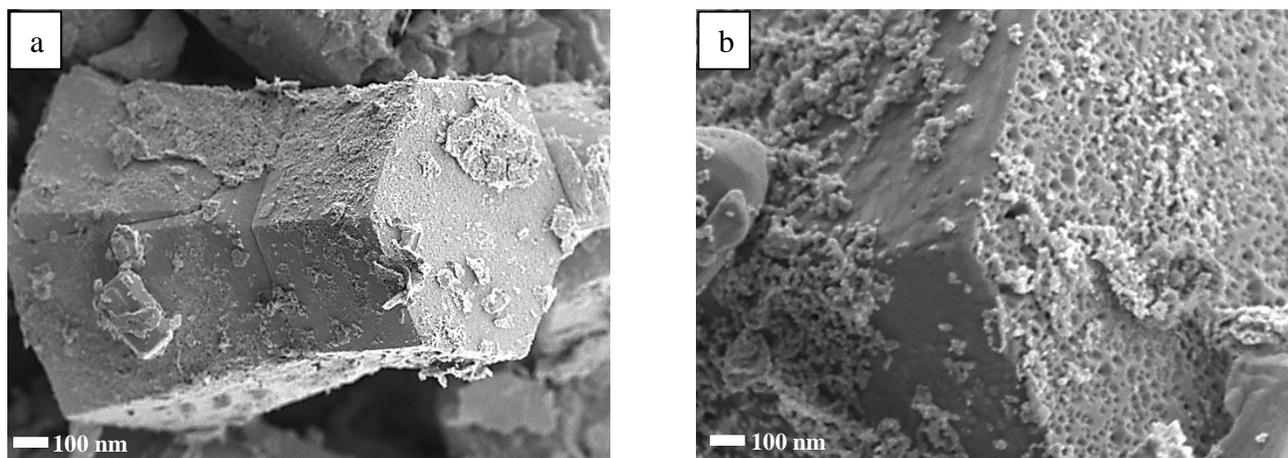


Figure 2. SEM images of zinc oxide sample after calcination at a) 5000 x (hexagonal crystals of ZnO), and b) 40000 x (Mesopores) of magnification.