

Type of presentation: Poster

MS-1-P-3446 Structure and Morphology Study of Pure and Mixed ZnO and ZnO₂ Nanoparticles

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Synthesis of ZnO₂ nanoparticles was performed via a sol-gel technique assisted with UV irradiation. One gram of zinc acetate dehydrate, Zn(CH₃COO)₂·2H₂O, was dissolved under vigorous stirring in a mixture of 50 ml distilled water and 5 ml of 30% H₂O₂. The resulting solution was then irradiated with a 300W Ultra-Vitalux lamp (Osram), positioned 10 cm above the solution, for 30 min at ambient temperature. This procedure resulted in the formation of a white zinc peroxide colloidal suspension. The ZnO₂ nanoparticles were precipitated by centrifugation. The precipitate was then washed using distilled water until a pH of 8 was reached. Finally the resultant white solid was dried at 80 °C for 12 h, similar to follow in the reference [1]. The resultant powder was annealed between 100 and 220°C for 1 h in an oven with air atmosphere. The morphology, structure and domain size of the nanoparticles were determined by X-ray diffraction, and scanning transmission electron microscopy. By X-ray diffraction, all patterns can be indexed to the zinc peroxide phase for samples prepared up to 120°C. For a sample prepared at 160°C we had a mixture of ZnO₂ and ZnO, while for particles treated at 220°C all the material was pure ZnO.

Micrographs shows STEM images for zinc oxide and zinc peroxide nanoparticles. Fig 1 shows rounded ZnO particles, with an average grain size of 18±5 nm. The inset displays that the ZnO d-space was 2.8 Å. Fig 2 shows an image of ZnO and ZnO₂ mixture, in the inset figure can be appreciated round conglomerated particles. There are two types of particles, the bigger ones belong to ZnO and the smaller ones belong to ZnO₂. The information of the atomic columns acquired by HAADF detector indicated that ZnO d-spaces were between 2.8 Å and 2.6 Å. This parameter must be connected to synthesis conditions of the material. In any case the average diameter size was 145 ± 55 nm.

Figures 3 and 4 belong to images of pure ZnO₂ particles acquired by HAADF and BF detectors respectively. At low magnification can be observed spherical shapes with broad size dispersion between 40 and 287 nm. The average diameter was 130±64 nm. At higher magnification these conglomerates displays small grains (≈ 5 nm). Figure 1d confirmed that each small grain had d-space values which belong to ZnO₂.

Using electron microscope techniques we have studied in detail the morphology and the structure of ZnO nanoparticles, ZnO₂ nanoparticles and a mixture of both. The ZnO₂ nanoparticle are of great interest, because they had interesting microbiological characteristics [2].

References

[1] R Colonia, J L Solís and M Gómez, Adv. Mat. Sci.: Nanotechnol 5 (2014) 015008 (4pp).

[2] R Colonia, V. Martinez, J. L. Solís and M. M.Gómez, Rev. Soc. Quim. Peru 79(2)2013 126 (10pp)

Acknowledgement: Thanks to P. Pisa and E. Torres for their technical help, at nanotech Cimav.

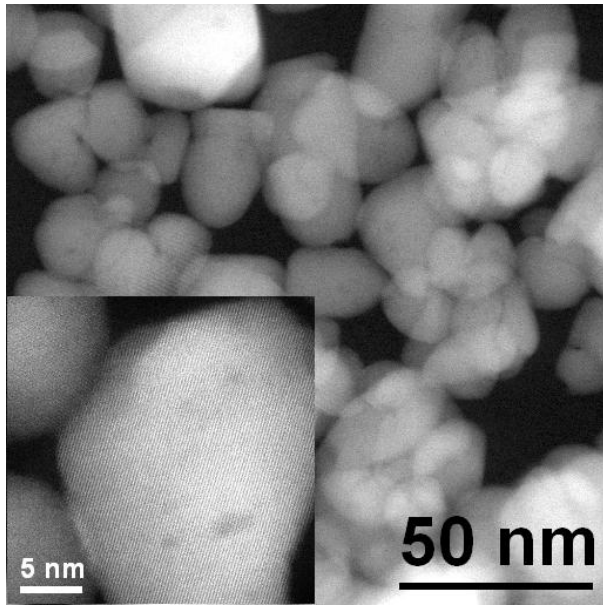


Fig. 1: STEM image by HAADF and BF images for ZnO particles, inset image show d space of ZnO.

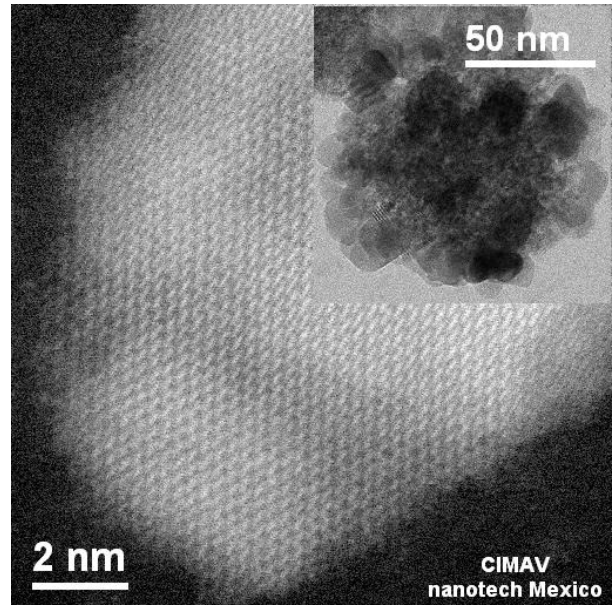


Fig. 2: ZnO-ZnO2 particles, d-space belongs to ZnO.

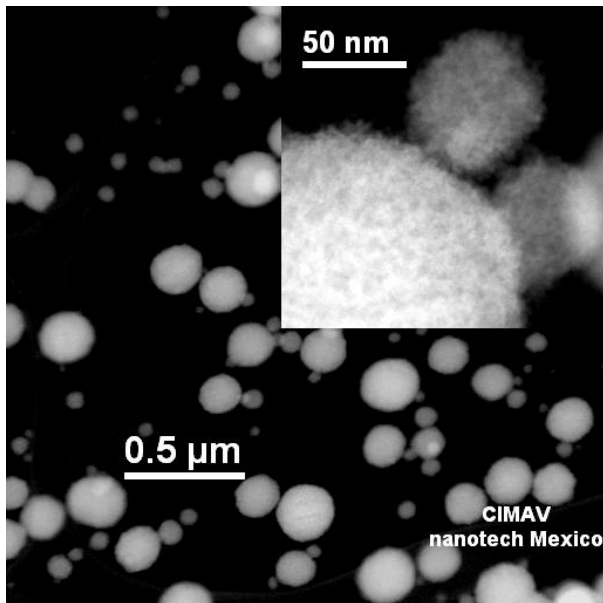


Fig. 3: ZnO₂ particles conglomerate in a spherical shape, inset can be notice the nanoparticles.

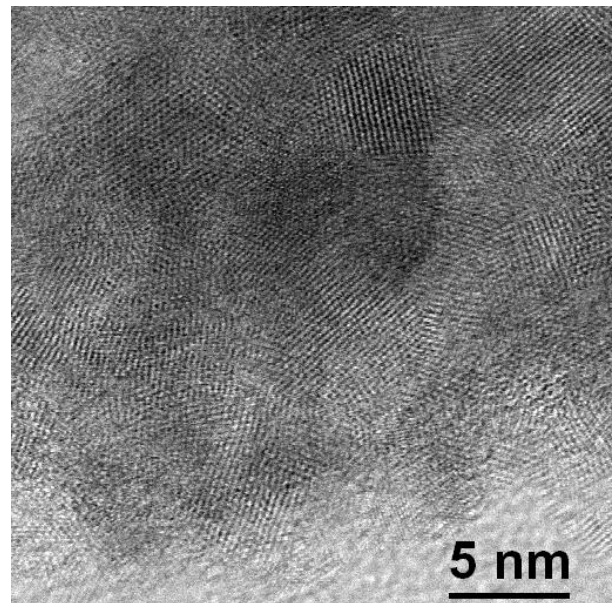


Fig. 4: BF image showing d-spaces of ZnO₂.