Structural Study by High Resolution Transmission Electron Microscopy of Zirconia Nanoclusters.

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Abstract. The recent development of nanostructured materials, needs processing technologies that allow to obtain physical-chemical stable nanoparticles^[1], the characterization techniques of this kind of materials needs the use of high resolution Electron Microscopy that allow to study the nanostructural morphology and defects, and to correlate them with the processing parameters. In this work, chemical techniques of solution-precipitation have been used, they allow to obtain molecular structured precipitates from which nanometric particles are easily obtained, keeping strictly their stability during the subsequent separation process. In this process is extremely important to employ methods that allow the best particles dispersion, so in this study, it has been proposed the use of microwaves, that change the kinetics of hydrolysis and condensation reactions enhancing the reactions rates and also helping the dispersion process^[2]. It has been proved that the use of microwaves is highly beneficial for helping the sol-gel processing, principally because to its contribution to the mixed dispersion and thermal effects of the electric and magnetic fields applied to the sample by the microwave treatment^[3]. It has been shown that those effects are evident in the microstructures obtained by TEM (Field Emission, Phillips TECHNAI 200 Microscope) of the ZrO₂ nanoclusters which are uniformly spherical as it is shown in fig.1, the sizes of them are around the 300nm. The high resolution image fig.2, shows that the nanoclusters are polycrystalline and the size of these crystallites is about of the order of 6-8 nm. Some of them presented dislocations Fig.3. probably originated by the zirconia phase transformation during the thermal treatment of the obtained clusters. Fig. 4, shows the EDX microanalysis of the sample with only zirconia signals (Cu and C signals correspond to the grid composition).

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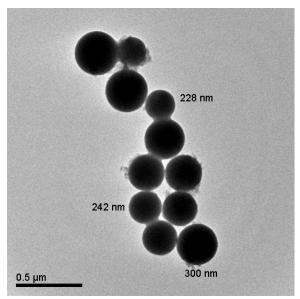


Fig.1. TEM image of ZrO₂ nanoclusters.

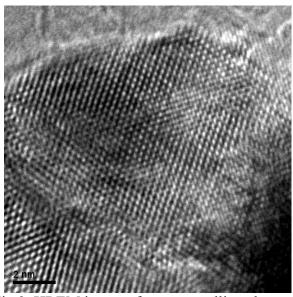


Fig.2. HREM image of nanocrystalline cluster.

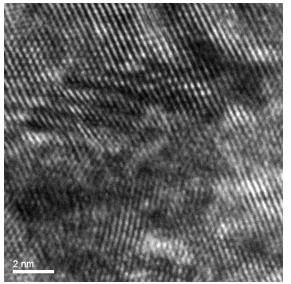


Fig.3. HREM showing dislocation defects.

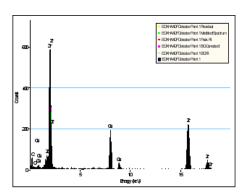


Fig.4. EDX microanalysis of ZrO₂ nanocrystal.