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Nano-powders of PZT obtained by high-energy ball milling.

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Abstract – Lead zirconate titanate (PZT) is one of the most studied ceramics by its dielectrics properties. In this paper is investigate the phase evolution of nanocrystalline $Pb(Zr_{0.6}Ti_{0.4})O_3$ powder obtained by high energy planetary mill. X-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques were used. The bulk density (97%) was obtained for the compressed nano-powder, fast fired at 950°C for 30, 45, 60 and 90 hours. The grain size in the ceramic samples was less than 100 nm. This result was obtained using atomic force microscopy (AFM).

Lead zirconate titanate (PZT) ceramics, are commonly used in numerous piezoelectric transducers and is well known for its dielectric, electro-optic and piezoelectric properties. At temperatures below the Curie temperature, PZT crystallites may exhibit tetragonal or rhombohedric structure [1]. The size of the particles of the PZT powders has a direct influence on the piezoelectric response of these materials. The preparation of this compound is normally based on conventional ceramic route or various chemical routes. Many methods have been developed to obtain PZT nanopowders. The high-energy ball milling technique has been employed successfully to obtain ferroelectric powders to decrease its sintering temperature.

The enhancement in the sinterability of the commercial PZT powder through this technique was evident by the experimental results. The milled PZT powder achieves its maximum sintering temperature at about 800° C, while the sintering temperature, for unmilled powder, peaks at about 1150 ºC. The samples were prepared using a planetary mill and under the following conditions: 400 rpm, ball to powder weight ratio (BPR) of 15 and 20, 40, 60 and 80 hrs of milling time.

Figure 1 shows the behavior of the d and η with milling time. As is observed, the rate of refinement of the internal structure (particle size, crystallite size, lamellar spacing, etc.) is roughly logarithmic with milling time. The crystallite size decreases when the milling time is increased and near to 40 hrs it reaches the saturation point. The strain decreases too with milling time, however continuing for more than 60 hrs, it increases, due to the increase in the deformation in the crystalline lattice. Nanocrystalline structure of PZT was expected, since the earlier investigation, performed on other materials under the same conditions of milling, always resulted in a significant reduction in crystallite size [2].

A small amount of excess PbO (3 wt.%) and a fast firing schedule are required to achieve the desired sintering. It was determined; using atomic force microscopy (AFM), that the grain size obtained in the ceramic samples was less than 100 nm.

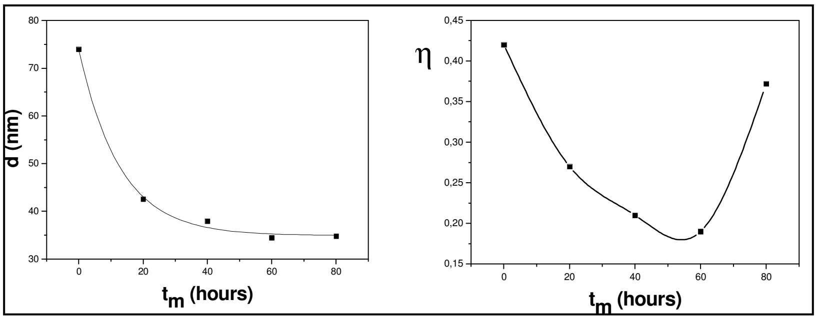

Figure 1. The milling time dependence of the crystallite size and strain for the unmilled and milled samples at 400 rpm during 20, 40, 60, 80 and 100 h and BPR = 15

References

[1] L.B. Kong, T.S. Zhang, J. Ma, F. Boey. Progress in Materials Science 53 (2008) 207–322.

[2] Haertling GH. J Am Ceram Soc 1999; 82(4), 797–818.