

# SYNTHESIS OF NEW 0.6PZS-0.4PLZT CERAMICS SYSTEM, SINTERING AND MICROSTRUCTURE PROPERTIES CHARACTERIZATION

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## Resume

The composition of 0.6PZS-0.4PLZT system was 0.6Pb (Zn<sub>1/3</sub>, Sb<sub>2/3</sub>) O<sub>3</sub>-0.4Pb<sub>0.98</sub>La<sub>0.02</sub> (Zr<sub>0.48</sub>, Ti<sub>0.52</sub>) O<sub>3</sub> were synthesized by a solid mixed-oxide solution method. The specific weight and porosity of the sintered component was measured from its mass and dimensions. The phase development and the lattice parameters of the sintered samples at 1050°C and 1200°C were analyzed and estimated by X-ray diffraction (XRD) (High score) and Celref software. The microstructure properties and morphology were characterized by scanning electron microscopy (SEM). The increasing in sintering temperature from 1050°C to 1200°C causes change for compound formation from Pb<sub>2</sub>Sb<sub>2</sub>O<sub>7</sub> with cubic phase structure (FD3M) ,a= 10.4825°Å; to another compound Zn<sub>2.3</sub>Sb<sub>0.67</sub>O<sub>4</sub> with cubic phase structure (FD3M) ,a= 8.5354°Å and slightly porous morphology.

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## 1. Introduction

Research on ferroelectric (FE) materials, motivated by the latest advance in thin-film growth technology, has significantly increased in recent years because of the anticipated applications for decoupling capacitors, micro-actuators, electro-optical components, and digital memories [1 - 6].

PbLa(Zr,Ti)O<sub>3</sub> is a well-known ceramic material that, depending on its composition, exhibits a wide range of properties at room temperature, including ferroelectricity, antiferroelectricity, pyroelectricity, and paraelectricity [7 - 8]. The conventional way of synthesis PLZT ceramic powders is the solid state reaction process using oxides as the starting materials [9, 10, 11]. There are other methods of preparation that recently developed as chemical coprecipitation [12], sol-gel process [13, 14], and hydrothermal reaction [15].

The conventional method requires a high calcination temperature, leading to hard particle agglomerates of the powders and subsequent poor microstructure and properties on the one hand and evaporation of lead component on the other. Wet chemistry-based methodologies mostly use expensive and environment-sensitive chemicals, making the process difficult to deal with [11].

Zinc oxide (ZnO) is an important II - IV group semiconducting material. Due to its versatile properties, it has been applied in electronic and electro-optic devices. It is an important wide and direct band gap semiconductor with  $E_g=3.3$  eV at room temperature and has a wurtzite structure ( $a = 3.249$  and  $c = 5.206$  Å) and  $P63mc$  (1 8 6) space group [16, 17, 18].

The addition of Sb<sub>2</sub>O<sub>3</sub> to ZnO resulted in a decrease in both the grain size and the density,

indicating that  $\text{Sb}_2\text{O}_3$  inhibits the grain growth of ZnO [18].

The purpose of present work is to prepare a new ceramic material 0.6PZS-0.4PLZT and undertake investigation of the effect of sintering temperature on crystallographic phase of a formed compound, morphology and microstructure properties.

## 2. Experimental

The composition of PZS-PLZT system was  $0.6\text{Pb}(\text{Zn}_{1/3}, \text{Sb}_{2/3})\text{O}_3-0.4\text{Pb}_{0.98}\text{La}_{0.02}(\text{Zr}_{0.48}, \text{Ti}_{0.52})\text{O}_3$  were synthesized from commercially available  $\text{PbO}$ ,  $\text{ZrO}_2$ ,  $\text{TiO}_2$ ,  $\text{Sb}_2\text{O}_3$ ,  $\text{ZnO}$ , and  $\text{La}_2\text{O}_3$  powders, the sample were prepared by the conventional ceramic procedure. Stoichiometric amount of metal for the designated PZS-PLZT composition were mixed for 2 hours as the grinding media and ethanol as the solvent. After milling for 6 hours, the resultant slurry was dried in an oven and then the powders were calcined in a furnace at  $850^\circ\text{C}$  for 120 min.

Then, the calcined powder was ball-milled for 6 hours to ensure a fine particle size. After drying, the powder was pressed as disks and sintered at two different temperature  $1050^\circ\text{C}$  and  $1200^\circ\text{C}$  in a closed alumina crucible, the atmosphere was enriched in  $\text{PbO}$  vapor using  $\text{PbZrO}_3$  powder, the density of the sintered component was measured from its mass and dimensions [19].

An X-ray diffractometer with  $\text{CuK}$  radiation was used to reveal the phases of the sintered samples. The X-ray diffraction data was used to evaluate the theoretical density ( $\rho$ ) of the sample using the formula:

$$\rho = \frac{\sum A/N}{V} \quad (1)$$

where  $\rho$  is the density ( $\text{g}\cdot\text{cm}^{-3}$ ),  $A$  is the sum of the atomic weights of all the atoms in the unit cell,  $N$  is the Avogadro's number, and  $V$  is the volume of unit cell ( $\text{cm}^3$ ) [19].

## 3. Results and Discussion

The XRD patterns of 0.6PZS-0.4PLZT samples sintered at  $1050^\circ\text{C}$  and  $1200^\circ\text{C}$  shown in Fig.1 were identified as a material with perovskite structure having cubic symmetry, it is observed that there is a formation of compound  $\text{Pb}_2\text{Sb}_2\text{O}_7$  (Bindheimite) with a cubic geometry and  $\text{FD}3\text{M}$  group symmetry, for sample sintered at  $1050^\circ\text{C}$  according to JCPDS card No. 00-042-1355, on the other hand, we note that for the sintered sample at  $1200^\circ\text{C}$  there is a formation of compound  $\text{Zn}_{2.33}\text{Sb}_{0.67}\text{O}_4$  with a cubic geometry and  $\text{FD}3\text{M}$  group symmetry, according to JCPDS card No. 00-015-0687 (Zinc Antimony Oxide). The lattice parameters for phase structure were determined from the evolution of the phase peaks by using *CELREF* software. The results given in Table 1 revealed the cubic cell parameters with some physical properties of the sintered samples. The specific weight increased with increase the sintering temperature.

Fig.2 showed the scanning electron microscopy (SEM) microstructure of the 0.6PZS-0.4PLZT prepared samples sintered at  $1050^\circ\text{C}$  and  $1200^\circ\text{C}$ . The increase of grain size is observed significantly with the increasing of sintering temperature, and it can clearly be seen that the prepared 0.6PZS-0.4PLZT ceramics and for the sample sintered at  $1200^\circ\text{C}$  is observed that the obtained product ( $\text{Zn}_{2.33}\text{Sb}_{0.67}\text{O}_4$ ) is slightly porous with a uniform distributions of grain shape and an approximate average size of  $6.93\ \mu\text{m}$  and  $1.43\ \mu\text{m}$  for ( $\text{Pb}_2\text{Sb}_2\text{O}_7$ ) obtained by sintering at  $1050^\circ\text{C}$ .

## 4. Conclusions

0.6PZS-0.4PLZT was successfully prepared by a solid mixed-oxide solution method; The X-ray diffraction measurements for all  $0.6\text{Pb}(\text{Zn}_{1/3}, \text{Sb}_{2/3})\text{O}_3-0.4\text{Pb}_{0.98}\text{La}_{0.02}(\text{Zr}_{0.48}, \text{Ti}_{0.52})\text{O}_3$  sintered samples indicated the presence of cubic phase. The influence of the sintering temperature on the structural properties of 0.6PZS-0.4PLZT ceramics was studied.

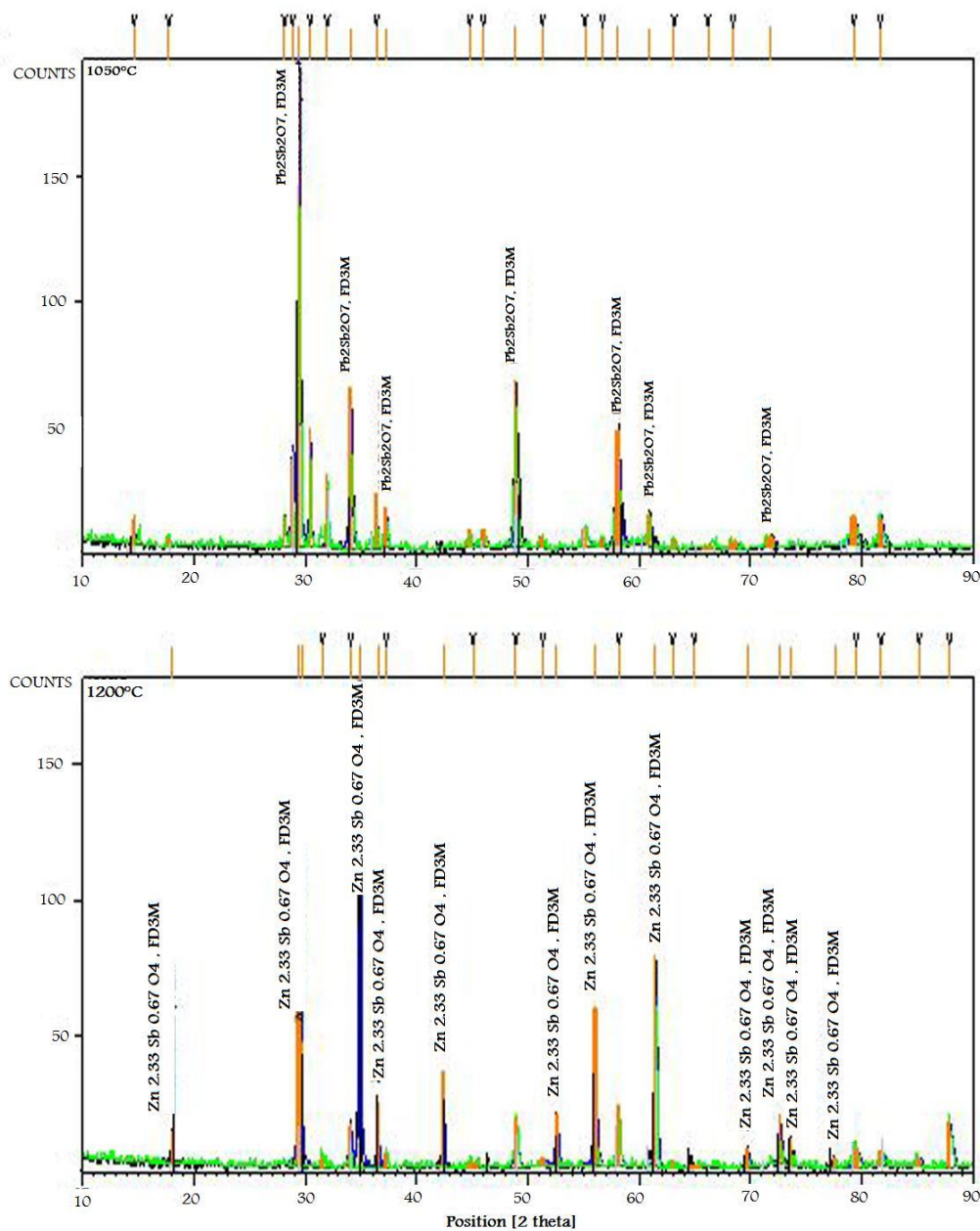


Fig. 1. X-ray diffraction pattern of 0.6PZS-0.4PLZT samples sintered at 1050°C and 1200°C. (full colour version available online)

Table 1

Physical properties of the sintered 0.6PZS-0.4PLZT samples

Sintering temperature (°C)	Thickness (cm)	Diameter (cm)	Specific weight (g.cm <sup>-3</sup> )	Compound obtained	Theoretical density (g.cm <sup>-3</sup> )	Phase structure	Relative density	Lattice parameters a (°Å)
1050°C	0.20	1.12	7.210	Pb <sub>2</sub> Sb <sub>2</sub> O <sub>7</sub>	8.872	Cubic	0.812	10.4825
1200°C	0.25	1.18	6.294	Zn <sub>2.33</sub> Sb <sub>0.67</sub> O <sub>4</sub>	6.363	Cubic	0.989	8.5354

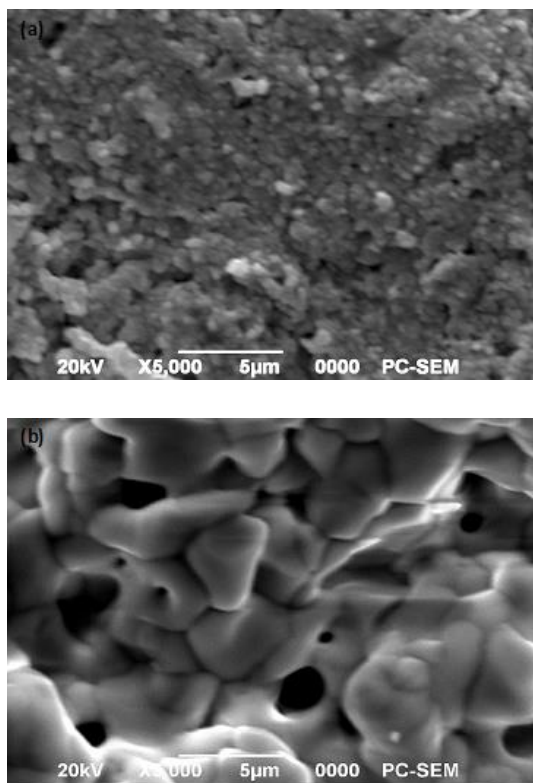


Fig. 2. SEM microscopy of PZS-PLZT ceramics sintered at: a) 1050°C b) 1200°C for 2 hours.

The Lattice parameter and specific weight are measured with increase the sintering temperature. This increase from 1050°C to 1200°C causes change for compound formation from  $Pb_2Sb_2O_7$  with cubic phase structure (FD3M),  $a=10.4825\text{Å}$ ; to another compound  $Zn_{2.3}Sb_{0.67}O_4$  with cubic phase structure (FD3M),  $a=8.5354\text{Å}$  and slightly porous morphology.

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