

Parametric Study and Analysis of Sol-Gel Derived [La/Va] Mg doped Composite PZT Nano Ceramic Material for Electronic Applications.

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Abstract - The presented work in this research paper deals with the fabrication and analysis of Mg, [La/Va] modified thick film PZT composite with an objective of developing Temperature Sensing material in capacitive configuration having higher linearity over a wider range. Using different characterization techniques and laboratory it is established as an excellent remote temperature sensing capability, showing higher reliability, lesser material losses and better homogeneity using modern Sol-Gel fabrication techniques.

Key Words: (Size 10 & Bold) Nanotechnology, sol-gel, PZT, XRD, SEM.

INTRODUCTION

Since the discovery of Lead Zirconium Titanate synthetic ceramic material by Jaffe et.al, it has remained a center of attraction of various researchers and scientists throughout the world due to its highly reconfigurable perovskite structure and coexistence of multiple phases at MPB condition. The change in the perovskite structure by addition of foreign atoms which substitute either a part or whole of the atom thereby changing the entire properties of the parent PZT, is a vital characteristic of the well known nano ceramic system. This property has been used by designers for the design of various ferroelectric actuators and sensors.

With the help of various experimental results and after prolonged investigation, it has been established and illustrated in this research paper that highly homogeneous hard PZT (70/30) derived by Sol-Gel Technology, when re doped in proper stoichiometric ratio of Va and La mixture results in formation of a typical composite PZT capable not only of remote temperature sensing over a wider range but useable as multi-parametric measurement system employing sensor array configuration.

1. FABRICATION AND CHARACTERIZATION

The synthesis of $\text{PbZr}_{0.7}\text{Ti}_{0.3}\text{O}_3$ nanoceramics is carried out by well known Sol-Gel process with the raw materials lead acetate trihydrate $\text{Pb}(\text{CH}_3\text{COO})_2 \cdot 3\text{H}_2\text{O}$ (99.99%, M/s Aldrich Chemicals), zirconium propoxide $\text{Zr}(\text{C}_3\text{H}_7\text{O})_4$ (99.99%, M/s Aldrich Chemicals), titanium iso-propoxide $\text{Ti}[(\text{CH}_3)_2\text{CHO}]_4$ (99.99%, M/s Aldrich Chemicals), mixed together in proper stoichiometric ratio. Glacial acetic acid and water were used as solvents. Ethylene glycol was added as an additive to get the monolithic gel.

First of all lead acetate was dissolved in acetic acid in the ratio of 2gm of salt to 1 ml of acid and was heated at 110°C for $\frac{1}{2}$ hour to remove water and then cooled down to 80°C . With constant stirring using magnetic stirrer zirconium propoxide followed by titanium isopropoxide was added to the above mixture. Ethylene glycol was added to the above mixture in the ratio of 1ml of glycol to 10gm of lead acetate. The initial reaction had to be completed before glycol was added since residual zirconium propoxide and titanium isopropoxide alcolyze with glycol to form a condense solid [1]. A small amount of distilled water was added to get the final sol. The sol was kept at 60°C for 24 hours to get the clear transparent gel. The gel was then dried in a controlled oven at 100°C for 72 hours to get a light brown powder. After proper grinding, the sample powder was divided into three equal parts by weight. To one part La/Va in the ratio of 2.75:1 (obtained from LaF_3 and Vanadium(V) oxytri isopropoxide) was added followed by proper mixing. To one part Mg^{+2} (obtained from MgO)[2]. The third part of the sample is kept undoped for reference characterization purpose. All three samples so obtained were cold pressed at a pressure of $5 \times 10^7 \text{ kg/m}^2$ using a hydraulic press to form disks shaped pallets 2 mm ($\pm 0.0001\text{mm}$) thick and 20 mm ($\pm 0.0001\text{mm}$) diameter These pallets were calcined at 700°C for 5 hours, followed by sintering at 1200°C for 2 hours. PbZrO_3 powder was used as a setter during sintering in order to prevent PbO loss or vaporization at higher temperatures.

High purity silver was coated on flat polished surface of sintered pallets using Vapor phase deposition technique, to work as electrodes in capacitive configuration for various characterization techniques carried out on lab scale.

2. EXPERIMENTAL ANALYSIS

Average grain size was calculated and was found to be 0.1 micron (0.1 μ m), the SEM photograph so obtained revealed the formation of highly homogeneous PZT composite shown in Fig 1.

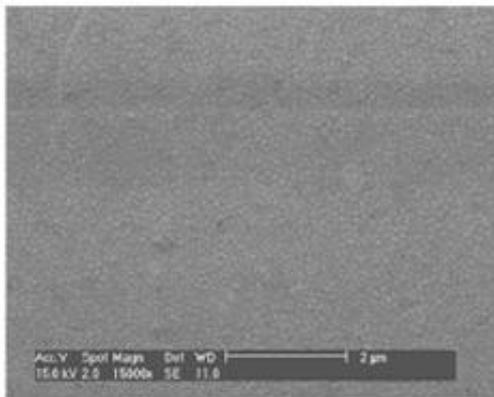


Fig 1 SEM Microphotograph of composite PZT

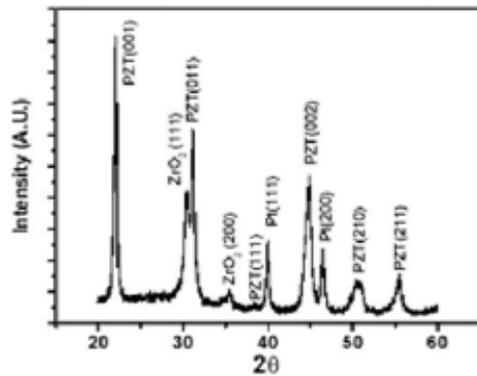


Fig 2 XRD Graph of PZT sample (un doped) fabricated by conventional methods

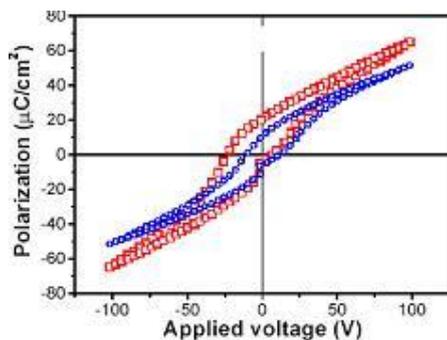


Fig 3 Ferroelectric hysteresis loop of hard doped PZT (red) and the composite PZT with mixed

type composition (blue).

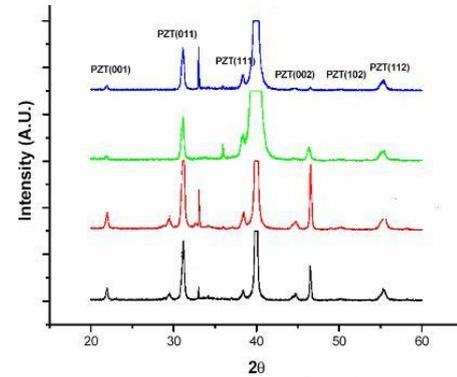


Fig 4 Comparative XRD Graph of PZT sample (un doped) fabricated by Sol-Gel technology. **Blue** (PZT Conventional Method), **Green** (by Sol-Gel Method), **Red** (PZT with 7% Mg Doping), **Black** (PZT with composite composition of 7% Mg, and 2.75:1 La/Va)

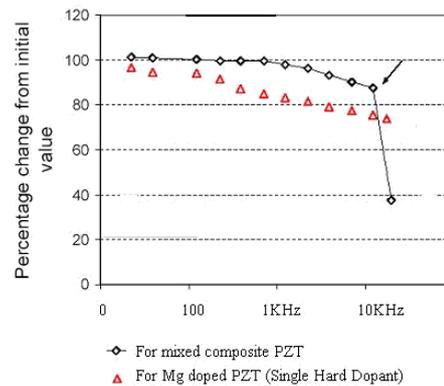


Fig 5 Percentage deviation from linearity for the two doped samples.

The prepared samples were subjected to various tests including ferroelectric hysteresis loop pattern determination, comparative XRD pattern study and graphical comparative illustration of the deviation from linearity as shown in the figures 3, 4 and 5 respectively.

3. RESULTS AND CONCLUSIONS

The conventional XRD graph thus obtained from the PZT (un doped) is seen to have a considerable peak splitting effect which clearly indicated the existence of at least two or more phases simultaneously (tetragonal and rhombohedral). The sol-gel technology adopted for the fabrication reduces not only the peak splitting effect but also introduces a well defined grain domain structure as seen from figure 1[2]. This can be better understood from the comparative XRD graphs as shown in figure 4[3].When observed closely it is well

established from the comparative XRD studies that the composite multi doped PZT has very less peak splitting effect, which affirms the formation of single phase composite PZT(70/30), having better homogeneity and higher dielectric constant values (K) varying linearly with temperature over a wider scale. As shown in the figure 3, the comparative hysteresis loop for the composite com-position PZT is much narrower than the singly Mg hard doped sample [4], making the material even more suitable for sensor fabrication due to very less power consumption, which is one of the most vital design issue of sensor material characterization. Also it has been found after reparative experiments the composite PZT developed on the laboratory scale shows no deviation in the observed value till 1KHz. However the percentage deviation from linearity varies linearly with increasing frequency up to 120 KHz as shown in figure 5, **which is the usable frequency bandwidth of the sensor.**

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REFERENCES

- [1] L.E. Cross, *ferroelectrics*, 76, p241 1987.
- [2] **Roy Kushal**, Effect of Variable MgO Doping on Structural, Electrical and Thermal Properties of PZT $Pb[Zr_{0.7}Ti_{0.3}]O_3$ Nano Ceramics Derived by Sol-Gel Technology, to be Used as Dielectric Material for Temperature Sensitive Variable Capacitors *Proceedings of ISIC 07*, Singapore, pp 155-158, 2007.
- [3] **Roy Kushal**, Effect of variable MgO and Fe_2O_3 on structural, electrical and thermal properties of PZT derived by Sol-Gel technology. *Proceedings of Metamaterials'2007*, pp. 307-310, Rome, Italy, 22-24 October 2007.
- [4] **Kushal Roy**, Research and Reviews: *JoPHY* 2015 4(1), ISSN 2278-2256, pp 31-37.

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