

SYNTHESIS AND CHARACTERIZATION OF STRONTIUM DOPED ZINC MANGANESE TITANATE CERAMICS

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In the present Study, the Strontium doped Zinc Manganese Titanate($\text{Sr}_x\text{Zn}_{1-x}\text{MnTiO}_5$, $x=0.1, 0.3, 0.5, 0.7$ & 0.9) Ceramics were prepared by conventional solid state reaction route. The raw powders were mixed according to molar ratio and the mixed powders were grained with Ball Mill for 12h. The grained samples were calcined at 1150°C and sintered at temperature 1250°C . These samples were characterized by XRD, SEM, and HIOKI 3532-50 LCR Hitester for structural, Micro structural and Dielectric studies respectively. From the XRD studies the structure of the compound were found as Cubic. The SEM with EDAX results shows that the compound well stoichiometric and high porous. The activation energies were increased from 0.78eV - 1.25eV with addition of Sr composition. The dielectric constant was increased with temperature and decreased with frequency.

(Received November 11, 2014; Accepted February 7, 2015)

Keywords: Solid state reaction route, XRD, SEM, Dielectric constant, Activation Energy

1. Introduction

Titanium dioxide [1] has attracted much attention due to its promising applications in the environmental photocatalytic degradation of pollutants of organic compound in waste water [2,3] and utilization of solar energy[4,5]. Zinc Manganese Titanates are promising candidates for low temperature sintering dielectrics [6, 7]. Hexagonal ZnTiO_3 is unstable at higher temperatures and much work has been devoted to the synthesis of ZnTiO_3 powder [8]. Dopents have been add to the reduced sintering temperature, but practical applications of partly restricted due to unstable dielectric properties originating from complex phase transitions [9]. Therefore many efforts have been made to prepare ZnMnTiO_3 improved stability. To achieve high stability divalent cations such as Mn^{2+} , Mg^{2+} , Sr^{2+} etc., were introduced to ZnMnTiO_3 and their stability were much better than that of ZnTiO_3 . It is possible to substitute Sr ion and Zn ion for each other to form $\text{Sr}_x\text{Zn}_{1-x}\text{MnTiO}_5$ ($x=0.1, 0.3, 0.5, 0.7$ & 0.9) by solid state reaction method, which might improve the stability and dielectric properties. In the present work nominal compound of ZnMnTiO_3 were prepared by solid state reaction method [10], and observed that the Dielectric properties were very poor in this case. So to enhancing the dielectric properties with the doping of divalent Strontium ion.

2. Experimental details

2.1. Materials

Titanium dioxide (TiO_2) powder (P-25), Zinc Oxide (ZnO), Manganese Carbonate (MnCO_3) and Strontium Carbonate (SrCO_3) were purchased from Aldrich Chemical (USA) with purities above 99%. Poly Vinyl Alcohol (PVA) used as a binder to prepare the pellets. $\text{Sr}_x\text{Zn}_{1-x}$

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$x\text{MnTiO}_5$ ($x=0.1, 0.3, 0.5, 0.7$ & 0.9) Ceramic samples were prepared by the conventional Solid State Reaction Route from oxide powders with purities above 99%. To obtain the single phase of each composition, grained by Ball Milling for 12h with Zirconia balls in polyethylene jars. The mixed powders were calcined at 1150°C for 24h. The obtained grained powders were mixed with Polyvinyl Alcohol (PVA) solution as binder and then pressed into discs 10mm diameter and thickness of 2mm under hydrostatic pressure about $10\text{Kg}/\text{cm}^2$. The resultant pellets were sintered at 1250°C for 4h. The structural studies were carried out by X-ray Diffraction [11] Unit. The surface morphology [12] was examined by scanning electron microscopy (JEOL J. SM-35, Japan). The porosity were measure by the Archimedes techniques [13] using water immersion. The dielectric constant, loss tangent, ac conductivity were measured using HIOKI 3532-50 LCR HiTester (imported from Japan) with variation of temperature and frequency (up to 1MHz).

3. Results and discussion

3.1. Structure analysis with xrd

From the below figures the XRD patterns of the samples at $x= 0.1, 0.3, 0.5, 0.7$ & 0.9 respectively. As the Sr composition was increased the maximum intensity peaks shifted towards the lower angles because of the substitution of the Sr ion in to the Zinc ion with the increase of the ion radius. From the XRD studies the structure of the samples were confirmed as cubic with the entire composition and with the help of Lattice parameters. The average grain size of the samples were calculated from the Scherrer's equation as varied from $11.6 - 9.01\text{\AA}$

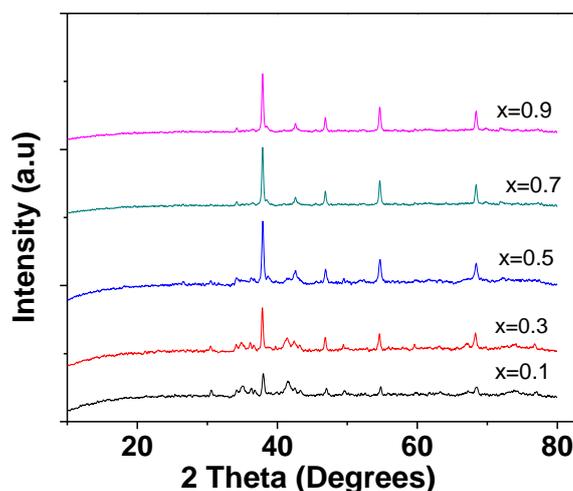


Fig. 1. The XRD Patterns for the $\text{Sr}_x\text{Zn}_{1-x}\text{MnTiO}_5$ ($x=0.1, 0.3, 0.5, 0.7$ & 0.9)

3.2. Micro structure analysis with SEM and EDAX:

The following fig. 2 shows the Scanning electron micrographs of $\text{Sr}_x\text{Zn}_{1-x}\text{MnTiO}_5$ ($x=0.1, 0.3, 0.5, 0.7$ & 0.9). The micrographs shows the particle size is going to be increased when change the Sr composition from 0.1- 0.9, but we observed at very clear formation of grains at $x=0.5$ because of the stiochiometric ratio of the sample and also the particles are arranged uniformly. So we get the better results at $x=0.5$ i.e. in terms of Dielectric measurements (K). The Strontium composition is further increased the uniformity goes decreased because of Sr^{2+} ions dominates.

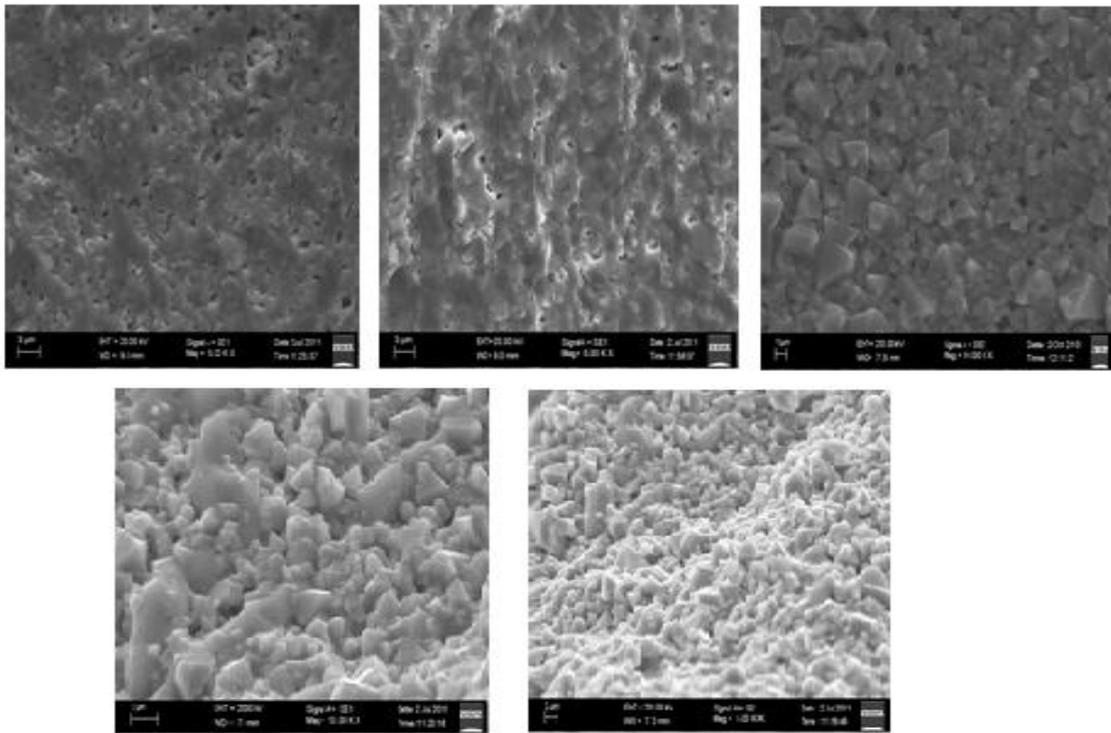


Fig.2. The Scanning Electron Micrographs of $Sr_xZn_{1-x}MnTiO_5$ ($x=0.1, 0.3, 0.5, 0.7$ & 0.9)

3.3. Dielectric measurements

For dielectric measurements the powder form of the sample is grained for 12h and taken 1 gram of the sample then add small amount of PVA as a binder and prepare the pellet by pellet making machine with diameter 1.0cm and thickness 2mm with the application of hydraulic pressure of 10 Tons. These pellets were sintered at 1250°C for 4h. Then these pellets were used for the dielectric measurements with the help of HIOKI 3532-50 LCR HiTester (Imported from Japan) at different frequencies and different temperatures

The following Fig.3 plots shows the variation of dielectric constant with temperature at different frequencies of $Sr_xZn_{1-x}MnTiO_5$ ($x=0.1, 0.3, 0.5, 0.7$ & 0.9). From these plots, we found the maximum dielectric constant was observed at $x=0.5$ with all frequencies (100, 1K, 10K, 100K, 1M Hz). The dielectric constant is further decreased because of the smaller ionic polarizability of the Zinc when compare with the Strontium.

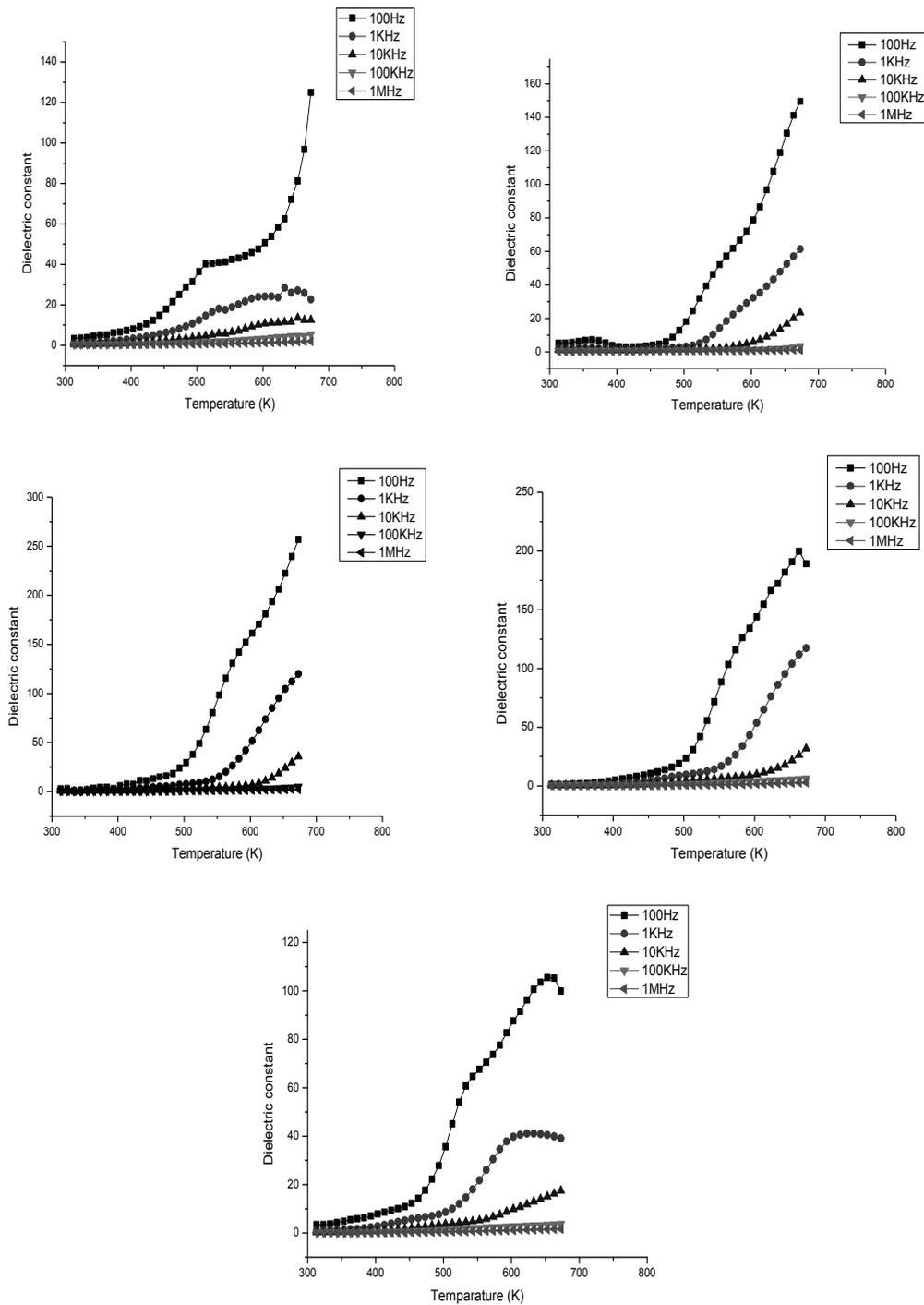


Fig.4. The Dielectric constant Vs Temperature of $Sr_xZn_{1-x}MnTiO_5$ ($x=0.1, 0.3, 0.5, 0.7$ & 0.9) at different frequencies (100-1MHz)

4. Conclusions

The Sr doped Zinc Manganese Titanate ceramics were prepared with solid state reaction route. We confirm that the structure as a cubic from X-ray Diffraction analysis. From SEM micrographs, we found the clear arrangement of grains at $x=0.5$ because of the stoichiometric composition of Strontium and Zinc. The EDAX analysis gives the percentage of Sr, Zn in the composition and also the compound is well stoichiometric. The Dielectric measurements gives the

dielectric constant is temperature and frequency dependent. As the temperature increases the dielectric constant is also increases and get good dielectric constant value is observed at $x=0.5$ for all the frequencies when compare with previous literature.

References

- [1]. Guo Wei Zhou, Young Soo Kang, J. Mat. Sci. & Eng., **C 24**, 71 (2004).
- [2]. T. Ohno, K. Sarukawa, K. Tokieda, M. Matsumura, J. Catal., **203**, 82 (2001).
- [3]. L. Arslan, L.A. Balcioglu, D.W. Bahnemann, Appl. Catal., **B 26**, 193 (2000).
- [4]. T. Takaca, Y. Furumi, K. Shinohara, A. Tanaka, M. Hara, J.N. Kondo, K. Domen, Chem. Mater., **9**, 1063 (1997).
- [5]. T. Ohno, D. Haga, K. Fujihara, K. Kaizaki, M. Matsumura, J. Phys.Chem., **B 101**, 6415 (1997).
- [6]. Alexander Tkach, Paula M. Vilarinho, Andrei L. Kholkin, Acta Materialia, **53**, 5061 (2005).
- [7]. Tkach A, Vilarinho PM, Kholkin AL., ApplPhys Lett., **86**, 172902 (2005).
- [8]. Eung Soo Kim, Chang Jun Jeon, J.of EuroCer.Society, **30**, 341 (2010).
- [9]. M.V. Nikolic, N. Labus, M.M. Ristic, Cer. Int., **35**, 3217 (2009).
- [10]. Anthony R. West, "Solid State Chemistry and its Applications", Third Ed., Wiley and Sons, NewYork, 2005.
- [11]. Cullity, B.D. and Stock, S.R., "Elements of X-Ray Diffraction", Thitd Ed., Addison- Wesley, NewYork, 2001.
- [12]. L. Reimer, "Scanning electron microscopy: physics of image formation and Microanalysis", Springer, Germany, 1998.
- [13]. S.K. Manik, S.K. Pradhan, Physica E, **33**, 69 (2006).