The Nucleus **48**, No. 1 (2011) 41-43

The Nucleus
A Quarterly Scientific Journal of Pakistan
Atomic Energy Commission
NCLEAM, ISSN 0029-5698

HIGH CURIE TEMPERATURE xBiScO$_3$-(1-x)PbTiO$_3$ CERAMICS

*K. SHAHZAD, M.N. KHAN and J. BASHIR

Physics Division, Directorate of Science, PINSTECH, P.O. Nilore, Islamabad, Pakistan

(Received October 20, 2010 and accepted in revised form January 20, 2011)

Perovskite solid solution of xBiScO$_3$- (1-x)PbTiO$_3$ (BSPT) nano-sized powders were successfully synthesized via sol-gel process and investigated as possible candidates for high temperature piezoelectric applications engines for pressure, force and vibration sensors. The structure and morphology of the powders was investigated with x-ray diffraction (XRD) and scanning electron microscopy (SEM), respectively. The Curie temperature (T_c) observed to be 478°C is higher than any previous reported value.

***Keywords**: Piezoelectric ceramics, BS-PT, Curie temperature*

1. Introduction

Piezoelectric materials are used as variety of sensors such as pressure, force and vibration sensors in automotive and aircraft industry [1]. One of the increased demands over the recent years in these technological applications is the operation of these sensors at high temperature. Recently, Eitel et al. [2] has prepared a system xBi(Me)O$_3$-(1-x)PbTiO$_3$ (Me=Sc) with high Curie temperature (T_c) 450°C from conventional solid-state method which is a strong candidate for high temperature applications. Inguez [3] argued that piezoelectric properties of xBiScO$_3$-(1-x)PbTiO$_3$ (BSPT) can be further improved beyond the present experimental values. Anomalously high piezoelectric and dielectric properties are found in compositions near morphotrophic phase boundaries (MPB). These enhanced properties arise due to the presence of more than one phases at MPB [2]. In this paper, we report our results of sol-gel synthesis of BSPT ceramics which possess higher T_c than the earlier reported temperature.

2. Experimental

The detailed procedure of sol-gel synthesis of BSPT nano powder can be found elsewhere [4]. The finally obtained gel was heated in two steps: first it was dried at 120°C for 24 h to make a stable gel and second heating was done at 200°C for 8h to remove carbonaceous material. The dried gel was calcined at 700°C for 4h and sintered at 1000°C for 2h. The calcined powder was pressed into pellets of 25mm dia and 3mm thickness. Scanning electron microscope (SEM) image of fractured surface of the final sintered ceramic was taken on JEOL JSM-6360LV. The X-ray powder diffraction (XRD) data was collected at RINT 2000 Rigaku diffractometer with Cu K_α radiation using graphite as monochromator. For electrical properties measurements pellets were electroded on both sides with silver paste and poled at 3kV/mm in a heated oil bath at 120°C for 10 minutes. Dielectric measurements were performed at 1kHz, 10kHz and 100kHz with a HP4284A LCR meter equipped with and a furnace from room temperature to 600°C at the heating rate of 3°C/min.

3. Results and Discussion

The powder obtained through sol-gel synthesis possesses better purity and homogeneity which can be seen in Fig. 1 and Fig. 2, respectively, than the conventional solid-state reaction method. All the peaks in the XRD pattern in Fig. 1 are characteristic peaks of perovskite structure. Earlier we reported [4] from the Reitveld refinement of XRD data that two phases namely tetragonal and monoclinic simultaneously exist at this composition. xBiScO$_3$- (1-x)PbTiO$_3$ composition with x = 0.36 is a MPB composition and generally more than one phases exist at MPB composition. Due to the coupling of these phases MPB compositions exhibit better piezoelectric properties than any other composition [2].

* Corresponding author : khurram@pinstech.org.pk

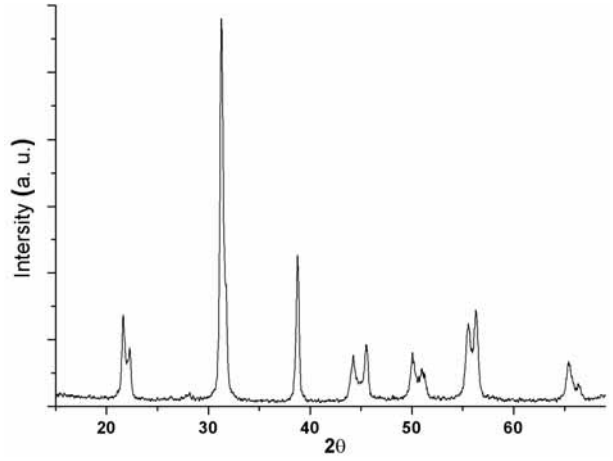

Figure 1. Room temperature XRD pattern of $0.36BiScO_3$-$0.64PbTiO_3$ sol-gel synthesized ceramic calcined at 700°C for 4h and sintered at 1000°C for 2h.

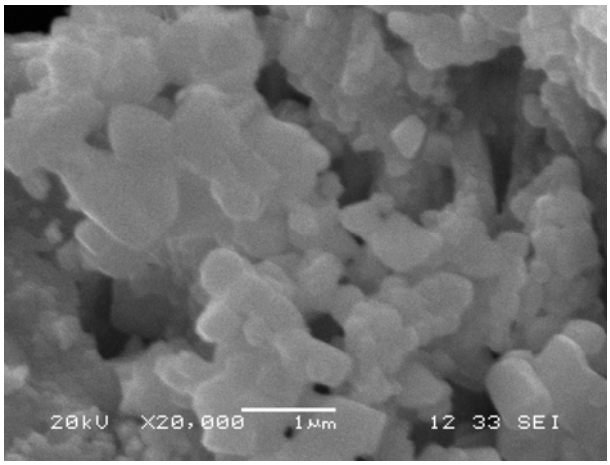

Figure 2. SEM micrograph of BSPT sol-gel powder calcined at 700°C for 4h.

SEM image of the sol-gel synthesized powder calcined at 700°C for 4h is shown in Fig. 2. The micrograph shows agglomeration of the particles. The grain size of the sol-gel synthesized powder is finer than the grain size of the powder prepared through conventional solid-state reaction method. Ceramics prepared with fine grain size exhibit better piezoelectric properties. The change in relative permeability as a function of temperature is presented in Fig. 3. The change has been measured at three different frequencies 1kHz, 10kHz and 100kHz. There is no shift in the peak which suggests that this composition of BSPT does not belong to relaxors [4]. The dielectric constant first increases and then decreases after reaching to

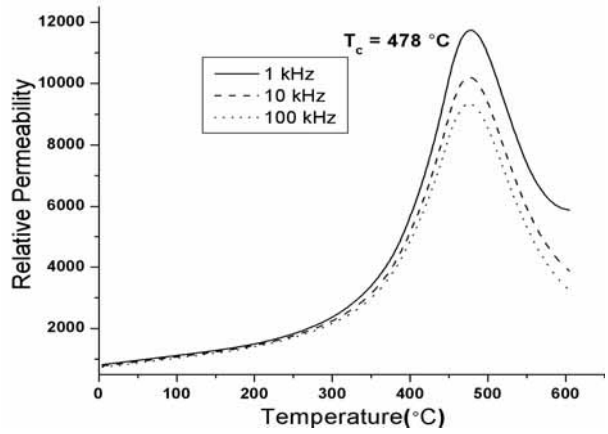

Figure 3. Change in relative permeability as a function of temperature of BSPT ceramics measured at three different frequencies.

a maximum value with increasing temperature. At room temperature the value of relative permeability is 892 whereas its maximum value is 11,700 at 478°C. The Curie Temperature T_c is the temperature at which dielectric constant shows maximum value which corresponds to ferroelectric to paraelectric phase transition. Here T_c is 478°C which is considerably higher than the earlier reported value 450°C [2]. The reason for this higher transition temperature may be attributed to the synthesis route. The ceramics with lower T_c were prepared through solid-state synthesis which resulted in large grain size microstructure whereas in our case sol-gel synthesis produced smaller grain size ceramics. The P-E hysteresis loop is shown in Fig. 4. Symmetry of the hyteresis loop suggests that the ceramic sample is homogenous

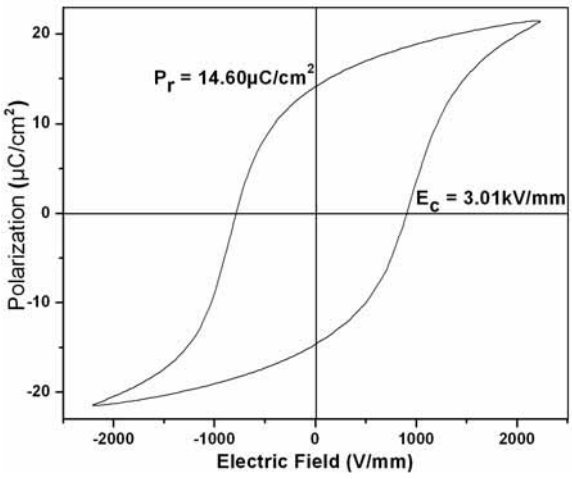

Figure 4. $x BiScO_3$-(1-x)$PbTiO_3$ P-E hysteresis loop measured at room temperature.

and it has no internal stress. The value of remanent polarization (P_r) is 14.60μC/cm^2 whereas coercive energy (E_c) value is 3.01kV/mm. Our fine grain sample has higher E_c value than the earlier reported value of E_c = 2.0kV/mm [2] of coarse grain ceramics. The fine grain size ceramics show high E_c whereas coarse grain size ceramics possess low E_c value [1]. In small grain size domain wall density increases which requires more energy to change its direction [4].

4. Conclusion

Fine grain size BSPT ceramics with pure perovskite structure were successfully synthesized through sol-gel method. The Curie temperature was found to be 478°C which is higher than the earlier reported results which is attributed to the small grain size of the prepared ceramics.

References

[1] G. H. Haertling, J. Am. Ceram. Soc. **82** (1999)797

[2] R. E. Eitel, C. A. Randall, T. R. Shrout, P. W. Rehrig, W. Hackenberger and S.-E. Park, *Jpn. J. Appl. Phys.* Part I **40** (2001) 5999

[3] J. Iniguez, D. Vanderbilt and L. Bellaiche, Phys. Rev. **B67** (2003) 224107

[4] K. Shahzad, Li Hua, Li Zhenrong, G. Shabbir, Li Hui and Xi Yao, Ferroelectrics **402** (2010) 142.