

STRUCTURAL PROPERTIES OF BiFeO₃ - BaTiO₃ CERAMICS

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ABSTRACT

In the present work we have reported the BaTiO₃/xBFO (x= 0, 0.02, 0.04, and 0.06 wt%) ceramics which were successfully synthesized by a solid-state mixed oxide method. X-ray diffraction pattern indicate that the addition of BFO in BaTiO₃ causes a slight change in tetragonality. The result also clearly shows that there are no secondary phases.

Keywords: X-ray diffraction, Scanning Electron Microscope (SEM), BiFeO₃(BFO)

INTRODUCTION

BiFeO₃ is an attractive material because of its multiferroic properties, i.e., ferroelectricity with high Curie temperature ($T_C = 820\text{--}850\text{ }^\circ\text{C}$) and antiferromagnetic properties below Neel temperature ($T_N = 350\text{--}380\text{ }^\circ\text{C}$). BiFeO₃ shows antiferromagnetic G-type spin configuration along the [1 1 1]_c or [0 0 1]_h directions in its pseudocubic or rhombohedral structure. BiFeO₃ has a superimposed incommensurate cycloid spin structure with a periodicity of 620Å along the [1 1 0] axis at room temperature. This structure cancels the macroscopic magnetization and inhibits observation of the linear ME effect. The decrease in particle size has been proved to be effective in suppressing this cycloid structure and enhancing the magnetic moment of BiFeO₃. The aim of the research described in this paper is focused on the preparation and characterization of xBiFeO₃ – (1-x)(BaTiO₃), where x=0, 0.02, 0.04 and 0.06. The basic idea is to enhance the dielectric and ferroelectric properties of these ceramics.

METHODOLOGY

The samples were characterized by powder X-ray diffraction. Stoichiometric amounts of the powders were mixed and milled in an agate mortar for about 20 minutes, with ethanol added as a milling aid. The powders were calcined for 10 hours in alumina crucibles at temperatures between 600 and 800°C. Subsequently, the powders were re-milled and pressed into pellets. The pellets were heated for 40 hours and then the cycle was repeated with milling, pelletizing and firing at raised temperatures. X-ray powder diffraction measurements at room temperature were performed with a Bruker advance D8 diffractometer, using CuK α radiation ($\lambda = 1.54056\text{ \AA}$) in a range of Bragg's angle 2θ (20°-60°) at a scanning rate of 1°min⁻¹. In the present study **LEO-435VP** (operating voltage: 15 to 30 kV) scanning electron microscope has been used. The samples were broken, coated with gold and placed in vacuum (10⁻⁵ Torr) chamber of the electron microscope.

RESULTS AND DISCUSSION

The X-ray diffraction (XRD) patterns of xBiFeO₃ – (1-x)(BaTiO₃) where x=0, 0.02, 0.04 and 0.06

compounds have been shown in (Fig. 1). BiFeO_3 (BFO) and BaTiO_3 (BT) is a member of complex perovskite ABO_3 family with the substitutions of different ions

at A and B site. The diffraction lines were found to be very sharp and single (unsplit) indicating better homogeneity and crystallization of the materials.

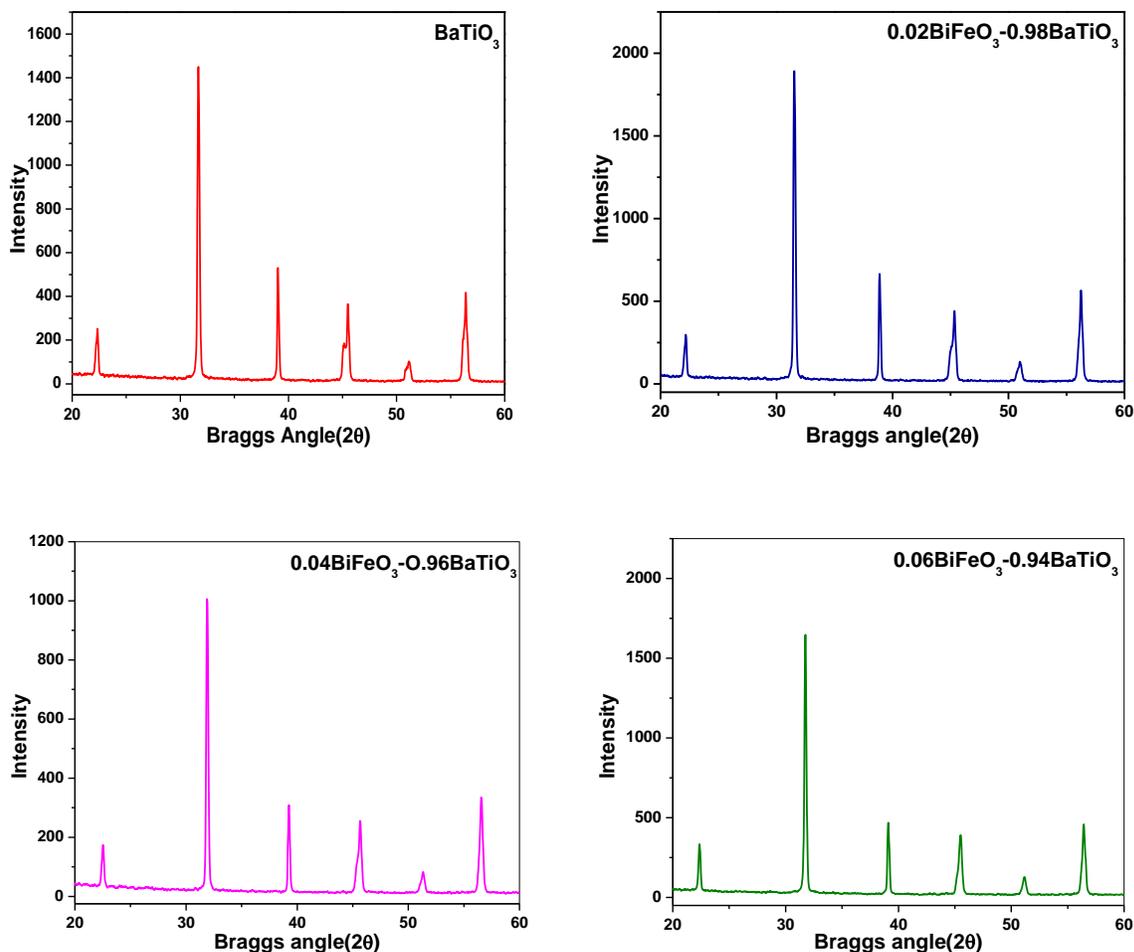


Figure 1 : X-ray Diffraction pattern of $x\text{BiFeO}_3 - (1-x)\text{BaTiO}_3$ where $x=0, x= 0.02, x = 0.04$ and $x=0.06$, sintered at 1100°C , 4 hours.

The crystallite size (P) of all the sample was estimated from the peak broadening β (full width of half maxima) and peak position (2θ) using Scherrer's relation $P = k\lambda/\beta\cos\theta$ ($k = \text{constant} = 0.9$, $\lambda = 0.15407\text{nm}$). The crystallite size calculation using the single intense peak (110) of sintered $\text{BaTiO}_3/x\text{BFO}$ shows that it decreases with increasing percentage of BFO. The crystallite size is of order of $\sim 25\text{nm}$, 22nm , 20nm and 15nm for $x=0, 0.02, 0.04$ and 0.06 respectively. It is clear from the calculation that for $x=0.02$ and $x=0.04$, the decrease in crystallite size is less than that of $x=0.06$. The decrease in

crystallite size results in a decrease of the tetragonal distortion.

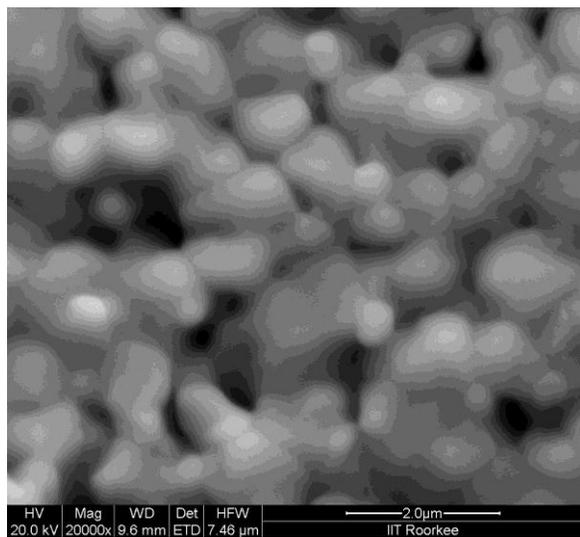
It has been reported in the literature that the splitting of their reflections into triplets takes place in compositional fluctuations leading to the coexistence of tetragonal and rhombohedral phases. Absence of the triplets in the XRD patterns of figures 1, therefore, indicates that there is no coexistence of mixed phases in the entire range of modified compositions. Absence of the coexistence region can be attributed to the greater chemistry homogeneity

of the prepared powders, which restricts compositional fluctuations in the sintered ceramics. The preliminary X-ray analysis indicates that the specimens were of tetragonal structure. By increasing the concentration from $x=0$ to $x=0.06$, a very small difference in the d -value have been observed but keeping the final structure invariant however, a systematic change in intensity of the few reflections have been observed. This change in intensity may be due to (i) variation of particle size and (ii) presence of dopants in different amount.

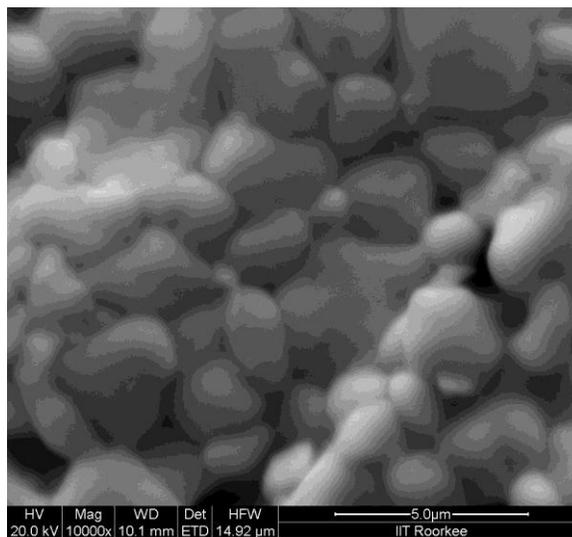
The scanning electron microscope (SEM) is a powerful tool which is capable of producing high-resolution images of a sample surface and used to measure many characteristics of the sample such as microstructure and surface morphology etc. In the present study **LEO-435VP** (operating voltage: 15 to 30 kV) scanning electron microscope has been used.

Surface morphology was studied by scanning electron microscope (SEM); the samples were broken, coated with gold and placed in vacuum (10^{-5} Torr) chamber of the electron microscope. The surface morphology of some of the proposed compounds using SEM technique is show in Fig. 2(a-d). The microstructures of the sintered pellets show spherical grains, which are uniformly and homogeneously distributed, which suggests that the sintered pellets were reasonably dense due to liquid phase sintering.

The average grain size determined by linear interception method ranged from 1300-2400 nm. Since the ionic radii of the elements is different there may be some chance for them to enter Ti^{4+} sites and thus render their ability in grain growth inhibition.



(a)



(b)

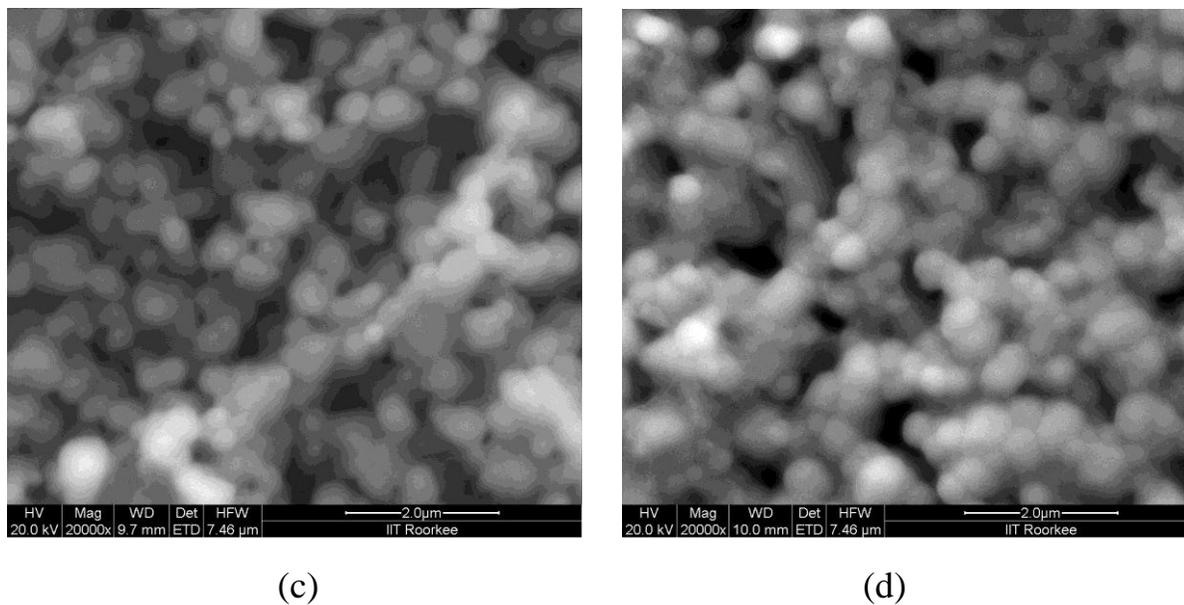


Figure 2 : SEM of $x\text{BiFeO}_3 - (1-x)(\text{BaTiO}_3)$ where (a) $x=0$, (b) $x=0.02$, (c) $x=0.04$ and (d) $x = 0.06$ sintered at 1100°C , 4 hr

CONCLUSION

In the present work we have reported the $\text{BaTiO}_3/x\text{BFO}$ ($x= 0, 0.02, 0.04,$ and 0.06 wt%) ceramics which were successfully synthesized by a solid-state mixed oxide method. X-ray diffraction pattern indicate that the addition of BFO in BaTiO_3 causes a slight change in tetragonality. The result also clearly shows that there are no secondary phases. It shows perfect solubility of BiFeO_3 in BaTiO_3 . The microstructures of the sintered pellets show spherical grains suggests that the sintered pellets were reasonably dense due to liquid phase sintering.

REFERENCES

1. D.C. Jia, J.H. Xu, H.Ke, W. Wang, Y. Zhou, Journal of the European Ceramic Society 29 (2009) 3099.
2. P Uniyal and K L Yadav, J. Phys.: Condens. Matter 21 (2009) 405901.
3. A. K . Pradhan and R.N.P . Choudary , J mat. sc., 22(1987) 2955.
4. K. V. Rao and A . smakula , J . Appl . Phys . , 37 (1066)m 319.
5. C. J. F. Bottcher, "Theory of Electric Polarization" (Elsevier, Amsterdam) 1952.
6. R. N. P. Choudhary and B . K . Choudhary , J . Mat. Sc. Letts., 9 (1990) 394.
7. V.V. Shvartsman, W. Kleemann, R. Haumont , J. Kreisel, Appl. Phys. Lett., 90 (2007).
8. M. Kumar, A. Srinivas and S.V. Suryanarayana; "Structure property relation in $\text{BiFeO}_3/\text{BaTiO}_3$ solid solution", J. Appl. Phys., Vol. 87(2), pp. 855-862, 2000.

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