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Structural and Dielectric Properties of BST-BLT Composites

V.C. Malvade[1], N.T. Padal[2] K.V.Rao[3], S.R. Kokare[1]
[1]Post Graduate Department of Physics, Raje Ramrao Mahavidyalaya, Jath, Dist: Sangli (M.S.) INDIA
[2] Willingdon College, Sangli
3 Jawaharlal Nehru Technological University, Hyderabad, INDIA
Corresponding Author: V.C. Malvade

ABSTRACT: *The ferroelectric materials $Ba_{0.7}Sr_{0.3}TiO_3$(BST) and $BaLa_2Ti_4O_{12}$ (BLT) were prepared by hydroxide co precipitation method. Its structural characterization were carried out by X-ray diffraction (XRD) while scanning electron microscopy gives the average grain size in the range of 1μm. The dielectric studies suggest that the BST-BLT composition improves the dielectric properties. As the BLT has the room temperature relative permittivity to be around 30. The relative permittivity at room temperature for BST sample is around 50. However for 75% BST and 25% BLT composites, the room temperature relative permittivity goes beyond 100. The dielectric loss is also comparably low. The paper also gives the frequency dependence of dielectric studies.*

KEYWORDS: *Dielectric Constant, Barium Strontium Titanate, Barium Lanthanum Titanate, composites etc.*

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I INTRODUCTION

In 1920, the ferroelectricity was discovered in Rochelle salt and the first hysteresis loop was published (1). In 1940, the discovery of ferroelectricity in Barium Titanate has brought the greater attention of scientific community due to its practical application (2). However, in 1950, the discovery of Lead Zirconate Titanate ($PbZr_{1-x}Ti_xO_3$) has become an extra ordinary useful candidate for ferroelectric as well as piezoelectric applications. From then a large number of solid solutions were synthesized over the research laboratories of the world. The application and the publication of Lead based Ferroelectric materials was the million dollar question at the end of 20th century. With the rise of new millennium, the environmental hazards, toxicity and polluting agent were the new parameters applied to the Lead based materials. Within last decade, the scientific community is in search of new non lead based materials which gives compatibility to the Lead based materials.The solid solution of BST and BLT are used to form the composites. These non-Lead based solid solutions may have the new compatible candidate for lead based material.

II EXPERIMENTAL

2.1 The raw materials like Barium Nitrate [$Ba(NO_3)_2$], Lanthanum acetate [$C_6H_9LaO_6.H_2O$], Potassium Titanate [$K_2Ti.O(C_2O_4)_2.2H_2O$] and Potassium Hydroxide [KOH] were weighed out in stoichiometric proportions and dissolved in distilled water separately by constant stirring. These solutions are then mixed to form the precipitate of BLT. A similar process is adopted to form the BST material. The detailed synthesis process is explained by P.B. Abhange and et al(3). After making the BLT and BST composites were prepared with different percentage of BST i.e. 25%BST+75%BLT, 50%BST+50%BLT and 75%BST+25%BLT.

2.2 The structural characterization of synthesized samples were made using X-ray diffraction (XRD) pattern. The morphological study of the prepared samples is carried out using Scanning Electron Microscopy (SEM) by exposing the samples with 20 KV as an excitation voltage. The dielectric properties of sintered samples were measured using Precessional Impedance Analyzer (HIOKI- LCR-Q Meter 3532-50)by varying the temperature from room temperature to 500 K. To vary the temperature laboratory made vertical tubular furnace is used.

III RESULT AND DISCUSSION

3.1 XRD: The X-ray diffraction patterns of all samples for pure BST, BLT and Composites. All the sintered samples showed the typical XRD patterns and no additional peaks are observed. The crystal structure and lattice parameters are in good agreement with the reported data (2).For 25% BST+75% BLT the structure is similar to BLT and for 25% BLT+75% BST structure is similar to BST. For 50%BST+50%BLT mixed phases are seen as it is expected for composite materials. X Ray diffraction plots are shown in figure1.

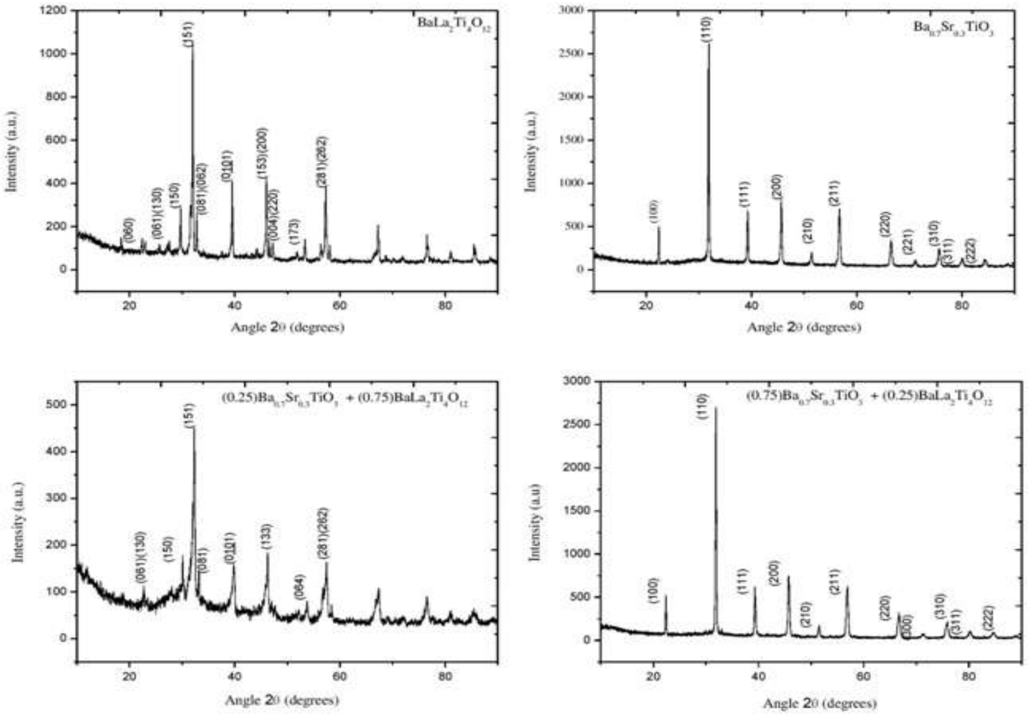

Fig. 1 X-Ray diffraction pattern of BST-BLT composites.

3.2 SEM: Using scanning electron microscopy the surface morphology of the materials synthesized hasbeen studied, which confirms that the material formed is sufficiently dense. Figure 2 shows the microstructure of synthesized samples.Though the high temperature sintering limits the grain size of material in few micrometer, the hydroxide co precipitation has brought down the grain size less than 1μm and it is confirmed by scanning electron microscope.

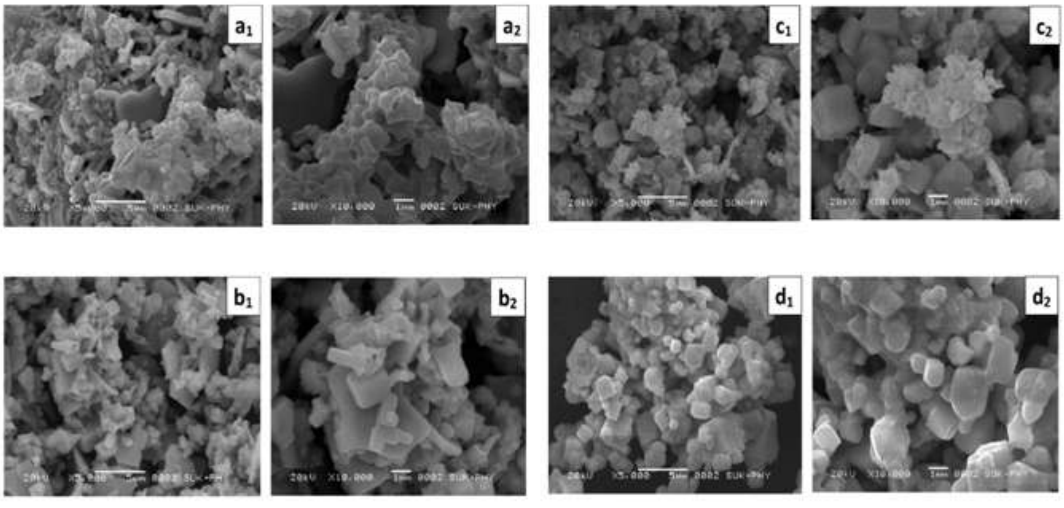

Fig.2- Scanning Electron Micrograph of BST- BLT composites {a_1,a_2: $BaLa_2Ti_4O_{12}$, b_1,b_2:(0.25)Ba0.7Sr0.3TiO3 + (0.75)BaLa2Ti4O12, c_1,c_2:(0.75)Ba0.7Sr0.3TiO3 + (0.25)BaLa2Ti4O12, d_1,d_2:Ba0.7Sr0.3TiO3}

3.3 DIELECTRIC CONSTANT: The Dielectric properties of any solid materials are estimated by electric dipoles having a length of few angstroms. The intensity of dipole is decided by the change in composition and microstructure of the material. Further the dielectric behavior of linear dielectric materials is particle size dependent and it increases with decrease in size of particle (4). Experimental data shows that nanometer sized particle have higher dielectric permittivity than that of for bulk sized particle at least for low frequency. In the case of amorphous SiO_2 particles having average size of about 20nm the dielectric permittivity is about 13 at 1 KHz which is more than three times larger than for the bulk material.(5). A similar dielectric rise was reported for Al_2O_3 (6) and TiO_2(7). This dielectric enhancement is associated with interfaces of the nanometer size particles. However the density of this dipoles can be observed to reduce by thermal annealing without change in particle size, which decreases the dielectric constant again. Thus the dielectric permittivity of nanometer sized particles are calculated using Havriliak-Negami expression (8).

$$\varepsilon^* = \varepsilon_\infty + \frac{(\varepsilon_s - \varepsilon_\infty)}{[\,1 + (i\omega\tau_o)^\beta]^\alpha} \quad [1]$$

Where ε_s is the static permittivity, ε_∞ is the high frequency permittivity, ω is the angular

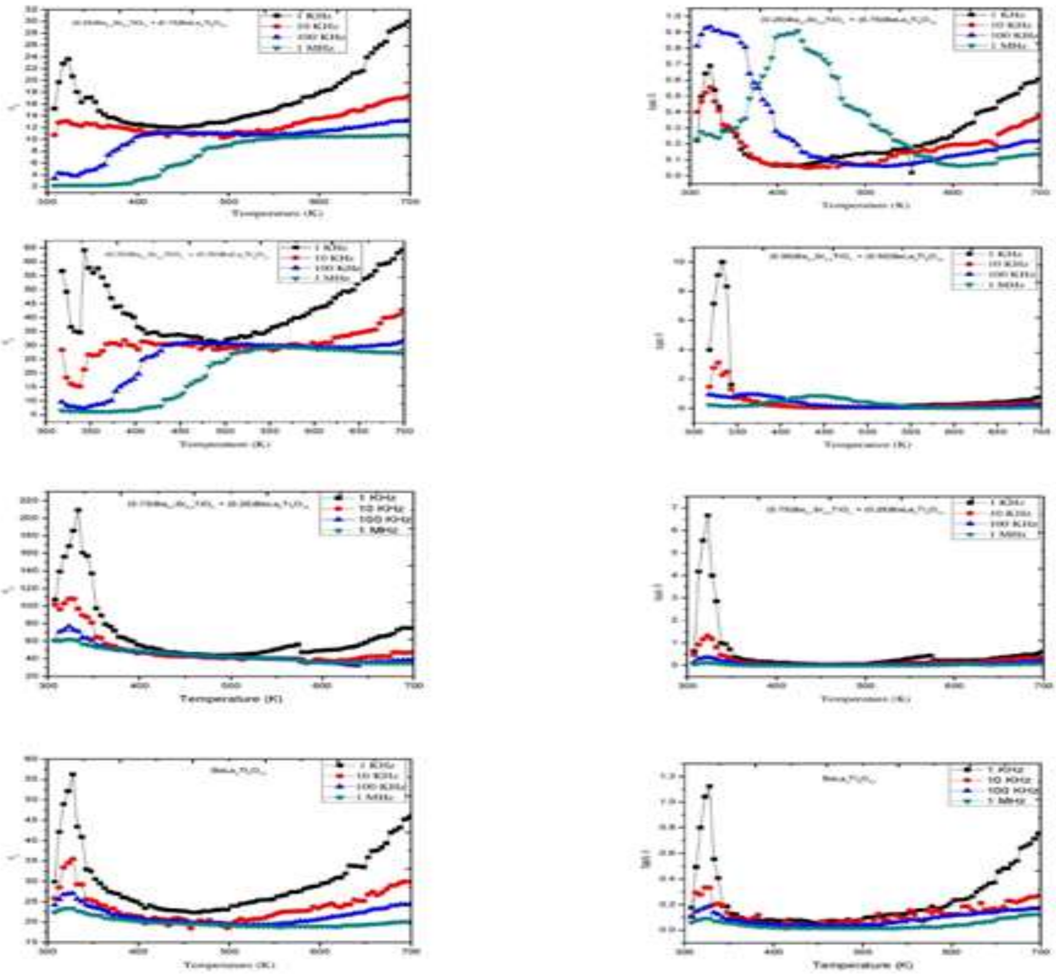

Fig.3 Variation of Dielectric constants with temperature for various frequencies of BST-BLT composites.

frequency, □$_0$ is the mean relaxation time. □ and □ are the parameters for symmetric and antisymmetric broadening of the relaxation time.□ □ and □ □ has the values between 0 and 1.

Figure 3 shows the temperature dependence of relative permittivity.The transition temperature (T_c) for BST is below the room temperature however the BLT gives the transition temperature above the room temperature. The composite materials give the transition temperature above the room temperature. Maximum Tc becomes 343 and it was for 50% addition of BLT. Tc is observed to be decreasing either side of composition and becomes maximum for equal percentage of composition. The very fine tuning in the composite concentration may reveal the further rise in the Transition temperature. The maximum relative permittivity at T_c goes upto 210 for 75% BST. Both this observation suggests that the further study of fine variation of composition is needed to arrive at good results. The figure 4 shows the room temperature relativity for BLT to be around 30 and for BST to be around 50.

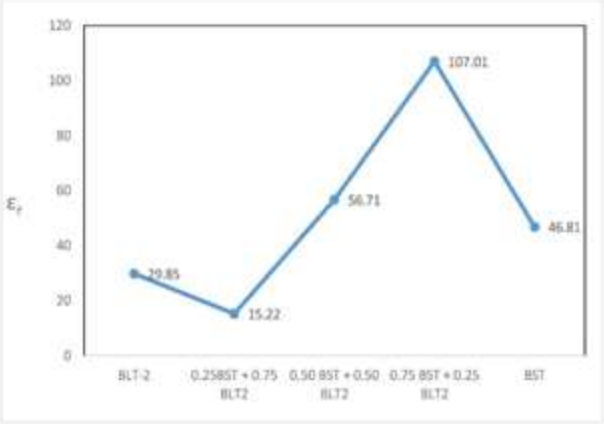

Fig.4- Variation of room temperature dielectric constant for increased concentration of BST.

The composites of these gives increase in relative permittivity and it goes upto 107 for 75% BST. The rise in dielectric constant is attributed to thereduced size of particle which is less than 1□ m. The loss factor is also comparably low, which is an indicative to candidature for microwave device application. The frequency dependence of relative permittivity gives usual and expected behaviour of displacer nature.

IV CONCLUSION

The single phase material of $BaLa_2Ti_4O_{12}$and $Ba_{0.7}Sr_{0.3}TiO_3$havingless than one micron grain size can be synthesized by hydroxide co- precipitation. The composites of above materials gives considerable rise in dielectric properties. For 75% BST and 25% BLT composites, the room temperature relative permittivity goes beyond 100. To have the fine tuning of dielectric rise, one has to study the composite of these materials between 50% to 100% BST with smaller steps.

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Table-1-Variation of □$_r$, tan□ , T$_c$,□ □$_{rmax}$ for various frequencies.

Sr. No.	X	1 KHz					10 KHz					100 KHz					1 MHz				
		Room Temp.		Curie Temp.			Room Temp.		Curie Temp.			Room Temp.		Curie Temp.			Room Temp.		Curie Temp.		
		ε_r	**tanδ**	**T:**	ε_r	**tanδ**	ε_r	**tanδ**	T_c	ε_r	**tanδ**	ε_r	**tanδ**	T_c	ε_r	**tanδ**	ε_r	**tanδ**	T_c	ε_r	**tanδ**
1	**BLT-2**	29.85	0.177	328	56.3	1.12	25.8	0.1	328	35.5	0.33	24.1	0.1	328	27.1	0.19	22.4	0.06	323	23.4	0.09
2	**0.25BST + 0.75 BLT2**	15.22	0.219	323	23.6	0.68	10.7	0.4	323	13.1	0.55	3.37	0.81	318	11.1	0.93	2.19	0.23	313	10.4	0.27
3	**0.50 BST + 0.50 BLT2**	56.71	4	343	64	1.61	28.4	1.47	348	26.6	0.7	9.65	0.95	< R.T.	9.65	0.96	6.57	0.25	< R.T.	6.57	0.25
4	**0.75 BST + 0.25 BLT2**	107	0.632	333	209	2.85	101	0.47	323	108	1.32	60.9	0.14	< R.T.	76.7	0.35	60.4	0.1	< R.T.	62.3	0.13
5	**BST**	46.81	0.238	< R.T.	--	--	34	0.07	< R.T.	--	--	49.1	0.03	< R.T.	--	--	50.6	0.02	< R.T.	--	--

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