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Synthesis and Performance Evaluation of $Ba_xSr₁$ _{-x}TiO₃ Ceramic-Based MIM Capacitor for Energy Storage Application

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ABSTRACT

Metal-insulator-metal (MIM) capacitor with high-k insulator such as BST enhances specific capacitance. The effects of barium mole fraction (x) of $Ba_xSr_1 = xTiO_3$ ceramic-based MIM capacitor on specific capacitance and leakage current density are reported in this article. $Ba_xSr₁ = xTiO₃$ nanopowder for various x are synthesized using solidstate reaction method and the performance parameters of Ag/Ba_xSr_1 $=$ $_{x}TiO_{3}/Ag$ MIM capacitors are evaluated. The mean crystallite sizes are in 20 to 60 nm range. $Ba_{0.7}Sr_{0.3}$ TiO₃ ceramics show higher relative permittivity owing to its higher mean crystallite size. The insulator with higher crystallite size at dominant orientation shows enhanced leakage performance. $Ba_{0.1}$ Sr_{0.9} TiO₃ ceramic capacitor shows optimum performance altogether.

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Energy storage; ceramic capacitor; crystallite size; specific capacitance; leakage current

1. Introduction

Barium strontium titanate is a continuous solid solution of barium titanate and strontium titanate and is the best ferroelectric material for applications in multilayer ceramic capacitors, ferroelectric random access memories (FRAM), microwave phase shifters, tunable filters, opto-electronic devices and sensors owing to its high dielectric constant, small dielectric loss, high breakdown field strength, pyroelectric properties, ferroelectri-city and good thermal stability [\[1](#page-12-0)–6]. These peculiar properties of $Ba_xSr₁$ _{-x}TiO₃ make it suitable for considering in energy storage devices. Metal-insulator-metal (MIM) capacitors (electrostatic capacitors) are best choice for energy storage devices owing to fast charging–discharging characteristics and very high life-span [[7\]](#page-12-0). Being a very good dielectric material, $Ba_xSr₁ - xTiO₃$ is one of the best candidates for insulator in metalinsulator-metal capacitors.

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The critical performance parameters of MIM capacitors in energy storage application are specific capacitance and leakage current. Specific capacitance enhancement of MIM capacitor can be achieved by using $Ba_xSr₁ = xTiO₃$ as insulator material on account of its high relative permittivity [[8\]](#page-12-0). The leakage current performance can also be improved by synthesizing $Ba_xSr_1 = xTiO_3$ sample with suitable barium mole fraction. Barium strontium titanate have been synthesized by numerous techniques namely solid-state reaction [\[9](#page-12-0),[10\]](#page-12-0), hydrothermal [\[11](#page-12-0),[12\]](#page-12-0), sol-gel [[13](#page-12-0)–15], spray pyrolysis [[16\]](#page-12-0) and RF magnetron sputtering [[17,18](#page-13-0)].

In this work, the Ba_xSr₁ – _xTiO₃ samples were prepared from barium carbonate, strontium carbonate and titanium dioxide using conventional solid-state reaction technique only, but at lower thermal budget and synthesis process duration. $Ba_xSr₁ - xTiO₃$ nanopowder samples for different barium mole fractions $x = 0.1$, 0.3, 0.5, 0.7 and 0.9 were synthesized. Further green pellets of each sample were made using Carver auto series automatic hydraulic laboratory press with enhanced "NE" digital control system. Furthermore silver is painted on both sides of sintered pellets after polishing to form Ag/Ba_xSr_1 – $_xTiO_3/Ag$ MIM capacitors and then evaluated specific capacitance and leakage current density.

2. Experimental Procedure

The $Ba_xSr₁$ – $_xTiO₃$ nanopowder was synthesized using barium carbonate (ACS reagent</sub> grade, 99%, Spectrum), strontium carbonate (ACS reagent grade, 99%, Spectrum) and titanium dioxide (ACS reagent grade, 98%, Spectrum) as precursors for barium, strontium and titanium, respectively. Stoichiometric proportions of barium carbonate and titanium oxide nanopowders were blended in agate mortar for 1 h. Similarly Stoichiometric proportions of strontium carbonate and titanium oxide nanopowders were blended in agate mortar for 1 h. Further calcining both mixtures at 1100° C for 4 h was carried out. The melts were taken out and blended in agate mortar for 1 h which result in barium titanate and strontium titanate powders. Stoichiometric proportions of barium titanate and strontium titanate powders were mixed well in agate mortar for 1 h, and then, calcining the blend at 1100° C for 4 h resulted in $Ba_xSr_{1-x}TiO_3$ nanopowder.

 Ba_xSr_1 - $_xTiO_3$ nanopowder was mixed with 4 wt% aqueous poly vinyl alcohol, the binder in agate mortar for 15 min and then dried up in hot air oven at 60 °C for 20 min. The mixture was taken out and blended again in agate mortar for 15 min. Further the powder was die-pressed into pellets using Carver auto series automatic hydraulic laboratory press with enhanced "NE" digital control system having 11 mm diameter die at 1 ton pressure. The green pellets were then sintered at 1275 °C for 2 h in Nabertherm sintering furnace. The $Ba_xSr_1 = xTiO_3$ nanopowder with $x = 0.1$, 0.3, 0.5, 0.7 and 0.9 were prepared and made into pellets using the above said procedure. The mass, diameter and thickness of each pellet were measured and noted after polishing.

Furthermore silver metal paste was spread over top and bottom surfaces of pellets to act as top and bottom electrodes of MIM capacitor device and kept at $130\,^{\circ}\text{C}$ for 15 min in hot air oven. The electrical continuity of the electrode surfaces were verified using a multimeter. For capacitor leads, copper wires were fixed at top and bottom electrodes with silver paste and dried at $130\,^{\circ}\text{C}$ for 15 min in hot air oven.

The capacitance and dielectric constant were measured using Hioki 3532-50 LCR Hi-Tester. The leakage current was measured using IV meter. X-ray powder diffraction patterns were observed using Bruker D8 Advance equipment.

3. Results and Discussion

The performance evaluation of $Ba_xSr_1 - xTiO_3$ ceramic-based Metal-Insulator-Metal capacitor for different barium mole fractions (x) is discussed in this section.

3.1. Specific Capacitance

During sintering each green pellet undergoes different area shrinkage according to material contents. Hence, it is better to evaluate specific capacitance rather than capacitance density. Specific capacitance is simply capacitance per unit mass. [Figure 1](#page-4-0) shows the specific capacitance of Ba_xSr₁ – _xTiO₃ ceramic-based MIM capacitor for $x = 0.1, 0.3$, 0.5, 0.7, 0.9 and 1.0. From [Figure 1](#page-4-0), it is evident that $Ba_{0.7}Sr_{0.3} TiO₃$ ceramic-based MIM capacitor possesses high specific capacitance and can be used to store more electrical energy.

Relative permittivity can also be measured using LCR meter. Since the specific capacitance is stabilized at a frequency of about 1 MHz, the relative permittivity of $Ba_xSr₁$. $_{x}TiO₃$ ceramic-based MIM capacitor for $x = 0.1, 0.3, 0.5, 0.7, 0.9$ and 1.0 at 1 MHz are measured and is shown in [Figure 2](#page-4-0). It is obvious from [Figure 1](#page-4-0) that $Ba_{0.7}Sr_{0.3}$ TiO₃ ceramic-based MIM capacitor possesses high relative permittivity.

The enhanced specific capacitance, and hence, energy storage capacity of $Ba_{0.7}Sr_{0.3}$ $TiO₃$ ceramic-based MIM capacitor is having direct dependence on mean crystallite size of $Ba_{0.7}Sr_{0.3}$ TiO₃ sample and is explained in subsection 3.3.

3.2. Leakage Current Density

Leakage current density versus bias voltage is shown in [Figure 3.](#page-5-0) The leakage current performance enhances as barium mole fraction decreases except for $x = 0.5$. It is observed that $Ba_{0.5}$ $Sr_{0.5}$ TiO₃ ceramic-based MIM capacitor shows slight improvement in leakage current performance contrary to expected result. The measured leakage current density of Ba_xSr₁ - _xTiO₃ ceramic-based MIM capacitor for $x = 0.1, 0.3, 0.5, 0.7, 0.9$ and 1.0 at 1 V are 7.24×10^{-10} A/cm², 5.44×10^{-9} A/cm², 2.91×10^{-9} A/cm², 1.46×10^{-8} A/cm², 3.24×10^{-8} A/cm² and 9.87×10^{-8} A/cm², respectively.

The pellets are of different thickness, and hence, it is better to analyze leakage current density variation with electric field rather than bias voltage. [Figure 4](#page-5-0) shows leakage current density against electric field of $Ba_xSr₁ = xTiO₃$ ceramic-based MIM capacitor for different barium mole fraction. The average thickness of pellets is about 2 mm, and hence, bias voltage of 1 V corresponds to electric field of 5 V/cm. The measured leakage current density of Ba_xSr_{1 – x}TiO₃ ceramic-based MIM capacitor for $x = 0.1, 0.3, 0.5, 0.7$, 0.9 and 1.0 at 5 V/cm are 6.64×10^{-10} A/cm², 4.32×10^{-9} A/cm², 3.48×10^{-9} A/cm², 1.41×10^{-8} A/cm², 3.61×10^{-8} A/cm² and 7.47×10^{-8} A/cm², respectively. It is observed

Figure 1. Specific capacitance of Ba_xSr_{1 – x}TiO₃ ceramic-based MIM capacitor for $x = 0.1, 0.3, 0.5, 0.7,$ 0.9 and 1.0.

Figure 2. Relative permittivity of Ba_xSr_{1 – x}TiO₃ ceramic-based MIM capacitor for $x = 0.1$, 0.3, 0.5, 0.7, 0.9 and 1.0 at 1 MHz.

that the change in measured leakage current densities with respect to bias voltage and corresponding electric field is marginal.

It is evident from [Figure 4](#page-5-0) that leakage current performance is achieved in $Ba_{0.1}$ $Sr_{0.9}$ TiO3 ceramic-based MIM capacitor is better compared to that with other barium mole fractions. The enhanced leakage current performance of $Ba_{0.1}$ $Sr_{0.9}$ TiO₃ ceramic-based

Figure 3. Leakage current density versus bias voltage of $Ba_xSr_{1 - x}TiO₃$ ceramic-based MIM capacitor for $x = 0.1$, 0.3, 0.5, 0.7, 0.9 and 1.0.

Figure 4. Leakage current density versus electric field of $Ba_xSr_1 = xTiO_3$ ceramic-based MIM capacitor for $x = 0.1$, 0.3, 0.5, 0.7, 0.9 and 1.0.

MIM capacitor has direct dependence on crystallite size of $Ba_{0.1} Sr_{0.9} TiO₃$ sample and is discussed in subsection 3.3.

3.3. X-Ray Powder Diffraction

The specific capacitance and leakage current performance variation with barium mole fraction can be studied using X-ray powder diffraction pattern. XRD patterns of $Ba_xSr₁$

Figure 5. X-ray powder diffraction pattern of Ba_xSr_{1 – x}TiO₃ ceramic-based MIM capacitor for $x = 0.1$, 0.3, 0.5, 0.7 and 0.9.

 $\frac{1}{x}$ xTiO₃ nanopowder for barium mole fraction, $x = 0.1$, 0.3, 0.5, 0.7 and 0.9 are shown in Figure 5. The diffraction peaks are observed at angles $2\theta = 22^{\circ}$, 32° , 40° , 46.5° , 52° , 58° and 68° correspond to BST(100), BST(110), BST(111), BST(200), BST(210), BST(211) and BST(220), respectively [\[19](#page-13-0)–21]. It is also evident from Figure 5 that the prepared samples do not possess any diffraction peaks from impurity phases.

The diffraction peaks corresponding to BST(110) of Ba_xSr₁ – _xTiO₃ nanopowder are higher in all barium mole fractions compared to other orientations of BST such as 100, 111, 200, 210, 211 and 220 [[22](#page-13-0)–24]. It can be observed from Figure 5 that the intensity of diffraction peak of $Ba_{0.1}$ $Sr_{0.9}$ TiO₃ nanopowder at 110 plane is higher compared to that of $Ba_xSr_1 = xTiO_3$ nanopowder with remaining mole fractions. This indicates that the $Ba_{0.1}$ Sr_{0.9} TiO₃ nanopowder sample attained better crystallinity which justifies lower leakage current [\[21](#page-13-0)] in $Ba_{0.1} Sr_{0.9} TiO₃$ -based MIM capacitor ([Figure 4](#page-5-0)).

The crystallite size can be estimated from X-ray powder diffraction pattern using Debey–Scherrer equation [25].

				hkl		
X	2θ (deg)	FWHM (deg)	Crystallite size (nm)		d_{hkl} (Å)	Lattice parameter (Å)
0.1	22.7346	0.1986	40.3648	100	3.9082	3.9082
	32.3806	0.1977	41.4057	110	2.7626	3.9069
	39.9440	0.2160	38.7090	111	2.2552	3.9062
	46.4670	0.2340	36.5522	200	1.9527	3.9054
	57.7627	0.2520	35.6120	211	1.5948	3.9065
	67.8212	0.2856	33.1521	220	1.3807	3.9053
0.3	22.5935	0.2968	27.0008	100	3.9323	3.9323
	32.2470	0.3315	24.6773	110	2.7738	3.9227
	39.7987	0.3833	21.8090	111	2.2631	3.9199
	46.3085	0.4094	20.8810	200	1.9590	3.9180
	57.5834	0.4995	17.9513	211	1.5994	3.9176
	67.5967	0.5106	18.5189	220	1.3848	3.9167
0.5	22.4580	0.5419	14.7878	100	3.9557	3.9557
	32.2154	0.5883	13.9059	110	2.7764	3.9264
	39.5771	0.3151	26.5105	111	2.2753	3.9409
	46.0439	0.3524	24.2347	200	1.9697	3.9393
	57.8265	0.2004	44.8006	211	1.5932	3.9026
	67.8447	0.1259	75.2426	220	1.3803	3.9041
0.7	22.2744	0.4096	19.5577	100	3.9879	3.9879
	31.6499	0.4002	20.4113	110	2.8247	3.9948
	38.9186	0.1641	50.8022	111	2.3123	4.0050
	46.4542	0.1002	85.3672	200	1.9532	3.9064
	57.7585	0.0968	92.6762	211	1.5949	3.9068
	67.7867	0.0924	102.5016	220	1.3813	3.9070
0.9	22.2132	0.2788	28.7281	100	3.9987	3.9987
	31.6011	0.3181	25.6769	110	2.8290	4.0008
	38.9266	0.2206	37.7790	111	2.3118	4.0042
	45.3782	0.1965	43.3488	200	1.9970	3.9940
	56.2870	0.3905	22.8231	211	1.6331	4.0002
	65.9977	0.6733	13.9174	220	1.4144	4.0005

Table 1. Crystallite size and lattice parameter of $Ba_xSr_1 = xTiO_3$ nanopowder for $x = 0.1$, 0.3, 0.5, 0.7 and 0.9 at dominant planes of reflection using X-ray spectrum analysis.

$$
Crystallitesize = \frac{k \times \lambda}{FWHM \times \cos \theta}
$$
 (1)

where $k = 0.89$ (shape factor), $\lambda = 0.15406$ nm (wavelength of X-ray), FWHM is width of diffraction peak at half of maximum intensity of that peak measured in radians and θ is half of diffraction angle.

The interplane spacing (d_{hkl}) can be computed using Bragg's law [[26\]](#page-13-0) which is given as

$$
d_{hkl} = \frac{\lambda}{2 \times \sin \theta} \tag{2}
$$

where h k l is is miller indices of Bragg plane which indicates internal plane of a crystal.

The equation for a cubic crystal [\[27](#page-13-0)] is

$$
\sin^2 \theta = \frac{\lambda^2 \times (h^2 + k^2 + l^2)}{4 \times a^2}
$$
 (3)

The equation for lattice parameter as a function of interplane spacing and miller indices can be derived using Eqs. (2) and (3) which is given as

Figure 6. Crystallite sizes of Ba_xSr_{1 – x}TiO₃ ceramic-based MIM capacitor for $x = 0.1$, 0.3, 0.5, 0.7 and 0.9 at dominant orientation.

$$
LatticeParameter = d_{hkl} \times \sqrt{(h^2 + k^2 + l^2)}
$$
 (4)

The crystallite sizes and lattice parameters of $Ba_xSr_1 = xTiO_3$ nanopowder for $x = 0.1$, 0.3, 0.5, 0.7 and 0.9 at dominant planes of reflection computed using [Eqs. \(1\)](#page-6-0) and [\(4\)](#page-7-0) and is given in [Table 1](#page-7-0).

The crystallite sizes at dominant orientation of $Ba_xSr₁ = xTiO₃$ nanopowder for $x = 0.1, 0.3, 0.5, 0.7$ and 0.9 are shown in Figure 6. The highest grain size is achieved in $Ba_{0.1}$ Sr_{0.9} TiO₃ nanopowder sample and is due to lower XRD pattern broadening at 110 plane. This again indicates sharper XRD pattern and better crystallinity achieved in the $Ba_{0.1}$ $Sr_{0.9}$ TiO_3 nanopowder sample which leads to enhanced leakage current performance.

The average crystallite sizes of samples prepared are in the range of 20 to 60 nm and is comparable with that obtained in the previously reported research works using complex synthesis methods [\[1](#page-12-0),[2,4](#page-12-0),[6,](#page-12-0)[28](#page-13-0)]. Insulator materials with higher mean crystallite size shows increased relative permittivity [\[29\]](#page-13-0). [Figure 7](#page-9-0) shows that $Ba_{0.7}Sr_{0.3}$ TiO₃ sample possesses higher average crystallite size and this substantiate the enhanced specific capacitance of $Ba_{0.7}Sr_{0.3}$ TiO₃ ceramic-based MIM capacitor discussed in subsection 3.1.

It is evident from [Table 1](#page-7-0) that the lattice parameter of $Ba_xSr₁$ – $_xTiO₃$ sample</sub> increases as barium mole fraction (x) increases. This increase in lattice parameter is due to the larger ionic radius of Ba^{2+} than that of Sr^{2+} [[30\]](#page-13-0).

3.4. Performance Evaluation

Specific capacitance and leakage current are equally important as far as an energy storage device concerned. For the best energy storage device, specific capacitance should be

Figure 7. Average Crystallite sizes of Ba_xSr_{1 – x}TiO₃ ceramic-based MIM capacitor for $x = 0.1$, 0.3, 0.5, 0.7 and 0.9.

maximum and leakage current should be minimum. Hence, the performance of proposed Ba_xSr_{1 – x}TiO₃ ceramic-based MIM capacitor for $x = 0.1, 0.3, 0.5, 0.7, 0.9$ and 1.0 can be evaluated with specific capacitance – leakage current ratio. It is defined as the ratio of specific capacitance at a frequency of 1 MHz and leakage current density at an electric field of 5 V/cm. The MIM capacitor with highest value of specific capacitance – leakage current ratio is the one shows optimum performance.

It is evident from [Figure 8](#page-10-0) that $Ba_{0.1}$ $Sr_{0.9}$ TiO_3 ceramic-based MIM capacitor shows optimum performance.

3.5. Synthesis and Performance Comparison with Related Research Works

[Table 2](#page-11-0) gives the comparison of synthesis parameters namely insulator material, synthesis technique, calcining and sintering temperatures as well as performance parameters in particular relative permittivity and leakage current density of Ag/Ba_xSr_1 – $_xTiO_3/Ag$ ceramic-based MIM capacitor proposed in this article with previous research works. The sample synthesis method followed in this work is very simple compared to other related works. The calcination and sintering of prepared samples are done with efficient temperature budget.

Specific capacitances are not mentioned in any of the related works given in [Table 2](#page-11-0), and hence, the comparison is limited to relative permittivity which is directly proportional to specific capacitance. The relative permittivity of $Ba_{0.7}Sr_{0.3}$ TiO₃ sample prepared in this work is better compared to other related works. Choi et al. [[28](#page-13-0)] achieved much better relative permittivity with $Ba_{0.7}Sr_{0.3}$ TiO₃ sample compared to our work at the expense of complex synthesis method. The leakage current study has not been done in many related research works. It is evident from [Table 2](#page-11-0) that the leakage current density of proposed Ag/Ba_xSr₁ - _xTiO₃/Ag ceramic-based MIM capacitor is very low compared to other related works [\[6,14\]](#page-12-0).

Figure 8. Specific capacitance - leakage current ratio $Ba_xSr_1 = xTiO_3$ ceramic-based MIM capacitor for $x = 0.1, 0.3, 0.5, 0.7, 0.9$ and 1.0.

4. Conclusion

The effects of varying barium mole fraction (x) of $Ba_xSr₁ = xTiO₃$ ceramic-based MIM capacitor on specific capacitance and leakage current density are reported in this article. It is observed that $Ba_{0.7}Sr_{0.1}$ TiO₃ ceramic-based MIM capacitor shows higher relative permittivity, and hence, higher specific capacitance compared to $Ba_xSr_1 = xTiO_3$ ceramic-based MIM capacitors with other barium mole fractions. It is also observed that the leakage current decreases as barium mole fraction decreases except for $x = 0.5$ which shows slight improvement in leakage current performance in contrast to the expected result. The leakage current density of $Ba_{0.1}$ $Sr_{0.9}$ TiO₃ ceramic-based MIM capacitor is lower compared to $Ba_xSr_1 = xTiO_3$ ceramic-based MIM capacitor with other barium mole fractions.

The increased specific capacitance of $Ba_{0.7}Sr_{0.3}$ TiO₃ ceramic-based MIM capacitor and enhanced leakage current performance of $Ba_{0.1} Sr_{0.9} TiO₃$ ceramic-based MIM capacitor are explained with X-ray powder diffraction pattern analysis. It is observed that higher mean crystallite size results in increased relative permittivity and sharper diffraction peak at dominant orientation, which leads to enhanced leakage current. Hence, higher relative permittivity of $Ba_{0.7}Sr_{0.3}$ TiO₃ ceramic-based MIM capacitor possesses higher energy storage capacity due to its higher mean crystallite size and $Ba_{0.1}$ $Sr_{0.9}$ $TiO₃$ ceramic-based MIM capacitor can hold stored electrical energy for a long duration due to its higher crystallite size at dominant orientation. Further $Ba_{0.1}$ Sr_{0.9} TiO₃ ceramic-based MIM capacitor is found to possess optimum performance when specific capacitance and leakage current considered together. It is also noted that the lattice parameter increases with barium mole fraction. $Ba_{0.7}Sr_{0.3}$ TiO₃ ceramic-based MIM capacitor can be made best choice for energy storage application by doping $Ba_{0.7}Sr_{0.3}$ TiO₃ ceramic with suitable insulator material to enhance its leakage current performance.

Table 2. Comparison of synthesis and performance parameters with related research works.

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