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P.G.R. Achary

Siksha O Anusandhan University Institute of Technical Education and Research

Soumakanta Khandai

Siksha O Anusandhan University Institute of Technical Education and Research Department of Physics

Priyabrata Sahoo

Siksha O Anusandhan University Institute of Technical Education and Research Department of Physics

R.K. Bhuyan

Government Auto College Angul

R.N.P. Choudhary

Siksha O Anusandhan Deemed to be University

S.K Parida (✉ santoshparida@soa.ac.in)

Department of Physics, Siksha O Anusandhan, Deemed to be University, Khandagiri Square,
Bhubaneswar <https://orcid.org/0000-0001-8875-4816>

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Structural, dielectric and impedance spectroscopy of cerium doped BaMnO₃ single perovskite

P.G.R. Achary¹, Soumakanta Khandai², Priyabrata Sahoo², R.K. Bhuyan³,
R.N.P. Choudhary² and S. K. Parida^{2,*}

¹Department of Chemistry, Siksha O Anusandhan, Deemed to be University, Khandagiri
Square, Bhubaneswar

²Department of Physics, Siksha O Anusandhan, Deemed to be University, Khandagiri
Square, Bhubaneswar

³P.G. Department of Physics Government (Auto) College Angul, Angul-759143, India

*Corresponding author (email: santoshparida@soa.ac.in)

Abstract

The synthesis of barium cerium manganite (BaMn_{0.9}Ce_{0.1}O₃) perovskite compound by using a high-temperature oxide mixing method is reported. Structural analysis suggests a hexagonal crystal structure with space group P6₃/mmc. The average crystallite size and micro-lattice strain of the sample are about 74.4 nm and 0.107% respectively. The SEM micrograph suggests the uniform distribution of grains through well-defined grain boundaries and average grain size is about 11.9 μ m. The relaxation time of the electrical process is calculated using the analysis of the real and imaginary parts of the complex impedance data, which suggests that the grain plays a dominant role compare to grain boundaries in defining the conductivity in the sample. Increase of activation energy from 96.8 meV to 689.9 meV with rise of temperature supports thermally activated conduction process. The modulus study suggests the relaxation process is controlled by the immobile charge carriers in the host materials at low-temperature range while controlled by defects at higher temperatures. The presence of semicircular arcs in both Nyquist and Cole-Cole plots confirms the semiconducting nature of the sample and find applications in solid oxide fuel cells and scintillators.

Keywords: Hexagonal; Lattice strain; Grain boundaries; Activation energy; Relaxation process; Cole-Cole plots

1. Introduction

The members of the perovskite family that include BaTiO_3 , PbTiO_3 , SrTiO_3 , BaMnO_3 , and SrMnO_3 have been constantly attracting the material scientists because of phase transition and strong ferroelectric behavior [1]. The study of ferroelectric properties of the nanocrystalline materials reports many anomalous physical, chemical, optical, and magnetic behavior compared to their bulk counterparts [2]. Many of the research papers, it has been mentioned that manganite properties depend on the surface effect and more strongly on the nanosize particles [3]. BaMnO_3 is an important member of this family due to high Curie temperature, high pyroelectric coefficient, low dielectric constant, and high spontaneous polarization [4]. The BaMnO_3 single perovskite shows the elevated electromechanical properties in both thin-film and bulk form for technological applications after the addition of the foreign dopants [5-8]. Moreover, the nature of dopant and growth rate of the grain boundaries during the process of high-temperature sintering are two important controlling factors in the study of electrical properties, particularly dielectric properties [9-11]. The presence of strong ferroelectric properties and magnetic orders at room temperature makes the manganite materials as special for investigation. The use of manganite materials has the main advantage in the electric and magnetic devices such as tunable transition temperature, minimized eddy current loss and low cost, etc. [12-15]. The barium manganite (BaMnO_3) is a perovskite material having general formula ABX_3 , where A and B are the cations and X is anion [16]. It is possible to dope in A-side with mono-, di- or trivalent substitution whereas, on B-side, tri-, tetra- or pentavalent metals can be used. Both A- and B-site in the BaMnO_3 can be doped simultaneously depending upon the valency and nature of the dopants [17]. In the present study, cerium dioxide (CeO_2) is used as a stable oxide dopant having an fcc crystal structure with space group $\text{Fm}\bar{3}\text{m}$. The dopant CeO_2 has two oxidation states Ce^{3+} and Ce^{4+} , out of which the Ce^{4+} oxidation state is more stable than Ce^{3+} due to its electronic structure [18]. It is reported that Ce is mostly preferred to incorporate in +4 ionic states on the B-site, whereas it prefers to incorporate in +3 ionic states on the A-site [19]. In the Mn-rich phase of the BaMnO_3 perovskite, it is commonly observed that Ce^{3+} is preferred to be incorporated in the A-site ion, whereas, in Ba-rich case, Ce^{3+} is preferred to incorporate in the B-site ion with either 3+ or 4+ valence [20]. In this present investigation, Ce is incorporated as 4+ ionic states at the Mn^{4+} site rather than at the Ba^{2+} site. When Ce enters into the host BaMnO_3 to incorporate Mn, both lattice strain and grain size increase because of Ce has a larger ionic

radius (248 pm) compared to that of Mn^{4+} ionic radius (108pm) in the B-site occupancy. Ferroelectric materials with cerium oxide (CeO_2) doped attract both material scientists and engineers because of the advanced applications in different fields of science and technology like scintillators, solid oxide fuel cells, biosensors and dielectric, etc. [21].

Because of this, the study of electrical properties of Ce doped barium manganite ceramics is done using complex impedance spectroscopy techniques. The frequency and temperature-dependent properties of a prepared sample are described through the complex permittivity (ϵ^*), complex impedance (Z^*), and dielectric loss or dissipation factor ($\tan\delta$). The inter-relation among these complex parameters can be defined as

$$Z^* = Z' - jZ'' = \frac{1}{j\omega C_0 \epsilon^*} \quad \text{..... (i)}$$

$$\epsilon^* = \epsilon' - j\epsilon'' \quad \text{..... (ii)}$$

$$\epsilon' = \frac{Z''}{\omega C_0 (Z'^2 + Z''^2)} \quad \text{..... (iii)}$$

$$\epsilon'' = \frac{Z'}{\omega C_0 (Z'^2 + Z''^2)} \quad \text{..... (iv)}$$

$$\tan\delta = \frac{\epsilon''}{\epsilon'} = \frac{Z'}{Z''} \quad \text{..... (v)}$$

$$\left[\begin{array}{l} \omega\tau = 1 \\ 2\pi f(R_b C_b) = 1 \\ f = \frac{1}{2\pi R_b C_b} = \\ \sigma_{ac} = \omega\epsilon_r\epsilon_0 \tan\delta \end{array} \right] \quad \text{..... (vi)}$$

Where $\omega = 2\pi f$ is the angular frequency, C_0 is the geometrical capacitance, and $j = \sqrt{-1}$. The relaxation frequency ($f_{\max}$) of the prepared sample can be calculated by finding the peak of the semicircle in the complex plane using the relation: $f = \frac{1}{2\pi R_b C_b}$, where R_b is called bulk resistance, C_b is called as bulk capacitance, and f is the relaxation frequency. The calculated relaxation frequency (f) and relaxation time (τ) depend on the intrinsic properties, not on the sample geometrical factors. The intrinsic properties of the sample under study are the results of the structure and microstructure (i.e., grain interior or bulk, grain boundary, etc.) analysis. The intrinsic properties control the possible distribution of resistive and capacitive components in the prepared sample on which the relaxation time ultimately depends [22]. Here, we made a small attempt to explain the structural, microstructural, and electrical properties of the sample.

The structural, mechanical, dielectric, conductivity, optical, and magnetic properties of barium cerium manganite ($\text{BaMn}_{0.9}\text{Ce}_{0.1}\text{O}_3$) single perovskite are available in plenty in the literature [23-25], but study with a low percentage of Ce dopant ($x \leq 0.1$) in the host barium manganite is scarce. The addition of Ce dopant in the host barium manganite matrix may be responsible for elevated structural and electrical properties of the prepared sample. So, we decided to prepare $\text{BaMn}_{0.9}\text{Ce}_{0.1}\text{O}_3$ nanoparticles with Ce as a dopant to study structural and electrical properties. The X-ray diffraction (XRD) pattern is used to study structural properties whereas scanning electron microscope (SEM) is used to study the microstructure of the sample. LCR meter measurements are used to study the electrical properties of the prepared sample. The main highlight points of the present study are phase transition from hexagonal to orthorhombic along with elevated electrical properties i.e. changing from dielectric to semiconducting material.

2. Experimental details

$\text{BaMn}_{0.9}\text{Ce}_{0.1}\text{O}_3$ perovskite compound is prepared at high temperatures by a mixed oxide method. The starting pure metal oxides BaCO_3 , MnO_2 (99.9% purity, Loba Chemie Co. Ltd), and CeO_2 (99.9% purity, Himedia) have been taken in the stoichiometry ratio by using a highly accurate digital spring balance. The average particle size of the above three oxide powders shows in the range of 2 – 5 μm . The above starting pure metal oxides are mixed thoroughly with high purity (99%) mortar pestle about 5 hrs. For uniformity of the constituent elements, some methanol is added to the dry powder and again mix for 2-3 hrs. For calcination, high-temperature silica crucible with a sample placed inside the furnace from 700 – 1150 $^{\circ}\text{C}$ with an interval of 50 $^{\circ}\text{C}$. To confirm the completion of the chemical reaction and formation of the crystal sample, the grinding process may repeat 2 – 5 times depending upon constituent fused elements. A number of cylindrical pellets of diameter 10.70 mm and thickness 1.65 mm are prepared using polyvinyl alcohol (PVA) as a binder under the pressure of about $4 \times 10^6 \text{ N/m}^2$ using a hydraulic press. For the removal of carbon traces, oxygen, and volatile impurities, the prepared cylindrical pellets are sintered at 1200 $^{\circ}\text{C}$. For phase identification and quantification, calcinated samples are being to the XRD chamber. X-Ray Diffractometer (D-8 Advance Bruker axis) scan on the sample over a wide range of Bragg angles ($10^{\circ} \leq \theta \leq 90^{\circ}$) at room temperature. X'Pert HighScore Plus software is used to analyze the XRD data. The average crystallite size and lattice strain are calculated by using

the Scherer and W-H method. The scanning electron micrograph is used to identify the distribution of the grains and the effect of the grain boundaries and show the uniform distribution and densely packed grains over the entire surface. The electrical properties including dielectric, impedance, conductivity, modulus, etc. of the prepared sample were calculated by using an LCR meter (N4L PSM, 1735) in a range of the temperature (25 - 500°C) and frequency range (1 kHz to 5 MHz)

3. Results and discussion

The ABO_3 is known as standard formula for single perovskite ceramics, where A-site is substituted by divalent elements and B-site is substituted by tetravalent elements. The tolerance factor of the $BaMn_{0.9}Ce_{0.1}O_3$ perovskite can be calculated by $t = \frac{r_A + r_O}{\sqrt{2}(r_B + r_O)}$, where r_A = ionic radius of A, r_B = ionic radius of B and r_O = ionic radius of oxygen. The standard tolerance factor should lie within the range from 0.75 to 1.0 with addition condition $r_A > 0.09$ nm and $r_B > 0.051$ nm [26]. Robert Ian Hines solely suggested a method of evaluation of the perovskite structure using tolerance factor. The possible value of t for different crystal structure are given like hexagonal crystal structure; $1.00 < t < 1.13$, for cubic structure; $0.9 < t < 1.0$, for orthorhombic structure $0.75 < t < 0.9$ and for $t < 0.75$, the structure was adopted to hexagonal ilmenite structure ($FeTiO_3$) [27]. The ionic radii of Ba^{2+} , Mn^{4+} and Ce^{4+} are 0.149, 0.053 nm and 0.097 nm, respectively [28]. For calculation, $r_A = 0.149$ nm, $r_B = Mn_{0.9}Ce_{0.1} = 0.9(0.053) + 0.1(0.097) = 0.0574$ nm and $r_O = 0.140$ nm. Therefore, tolerance factor of cerium doped barium manganite is 1.03, which demands hexagonal crystal structure.

Figure 1 (a) XRD pattern of $\text{BaMn}_{0.9}\text{Ce}_{0.1}\text{O}_3$ single perovskite

Figure. 1(b) Rietveld analysis of $\text{BaMn}_{0.9}\text{Ce}_{0.1}\text{O}_3$ single perovskite

Figure1 (c) Space filling structure (d) Ball-and-stick structure of $\text{BaMn}_{0.9}\text{Ce}_{0.1}\text{O}_3$ single perovskite

Figure 1 (e) wire frame dot structure (f) polyhedral structure of $\text{BaMn}_{0.9}\text{Ce}_{0.1}\text{O}_3$ single perovskite taking 62 atoms, 96 bonds, 14 polyhedral.

Figure 1. (g) SEM micrograph of $\text{BaMn}_{0.9}\text{Ce}_{0.1}\text{O}_3$ single perovskite

Fig. 1 (a) represents the XRD pattern of the $\text{BaMn}_{0.9}\text{Ce}_{0.1}\text{O}_3$ single perovskite. The X-ray Diffractometer having CuK_α radiation source ($\lambda=1.54056 \text{ \AA}$) is used to scan the sample from $2\theta = 20^\circ$ to 80° with a small step size of 0.005° at room temperature. The presence of sharp and distinct intense peaks suggests that the sample is a single-phase. The X'Pert HighScore Plus software is used to complete XRD data analysis [29]. The JCPDS file No. 01-071-1594 is matched very closely to all peaks in the sample. Our XRD analysis suggests that the sample has a hexagonal crystal structure with the $P6_3/mmc$ space group. Fig. 1(b) shows the Rietveld refinement of XRD data of the $\text{BaMn}_{0.9}\text{Ce}_{0.1}\text{O}_3$ single perovskite using Pseudo-Voigt

function. The XRD data analysis is done by using MAUD software to complete the Rietveld refinement. The XRD Rietveld analysis suggests a hexagonal crystal structure with space group P6₃/mmc (#194-1). The refined cell parameters are ; $a = b = 5.82740 \text{ \AA}$, $c = 4.83410 \text{ \AA}$, $\alpha = 90^\circ$, $\beta = 90^\circ$, $\gamma = 120^\circ$ and $V = 142.166061 \text{ \AA}^3$ [Refer-material project ID: mp-19156BaMnO₃.cif]. Rietveld refinement provides reliability factors like $R_p(\%) = 13.79$, $R_{exp}(\%) = 5.36$, $R_B(\%) = 17.6$, $\sigma = 4.03$. The XRD pattern of pure host matrix BaMnO₃ perovskite confirms hexagonal crystal structure (P6₃/mmc) with lattice constants $a = b = 5.694 \text{ \AA}$ and $c = 4.817 \text{ \AA}$ [30]. Therefore, there is a modification of cell parameters after the doping of Ce into the host BaMnO₃ matrix. Figure.1 (c) represents the space filling structure; Figure.1 (d) represents ball-and-stick structure; Figure.1 (e) represent wire frame dot structure; and Figure.1 (f) represents polyhedral structure image of BaMn_{0.9}Ce_{0.1}O₃ single perovskite. These structures were obtained from the VESTA crystal software code. The structural parameter of the barium manganite crystals is listed in Table 1. The structure of BaMn_{0.9}Ce_{0.1}O₃ is built using 62 atoms, 96 bonds, and 14 polyhedral.

Table 1. Structural parameters of the BaMn_{0.9}Ce_{0.1}O₃ single perovskite

Number	Element	Label	x	y	z	Occ.	B	Site	Sym.
1	Ba	BaO	0.66667	0.33333	0.75000	1.000	1.000	2c	-6m2
2	Ba	Ba1	0.33333	0.66667	0.25000	1.000	1.000	0	
3	Mn	Mn2	0.00000	0.00000	0.00000	0.900	1.000	2a	-3m
4	O	O5	0.14852	0.85148	0.75000	1.000	1.000	6h	mm2
5	O	O4	0.85148	0.70295	0.25000	1.000	1.000	0	
6	Mn	Mn3	0.00000	0.50000	0.900	1.000	0		
7	O	O9	0.85148	0.14852	0.25000	1.000	1.000	0	
8	Ce	Ce1	0.00000	0.00000	0.50000	0.100	1.000	0	
9	O	O8	0.14852	0.29705	0.75000	1.000	1.000	0	
10	O	O7	0.29705	0.14852	0.25000	1.000	1.000	0	
11	O	O6	0.70295	0.85148	0.75000	1.000	1.000	0	

The study of the SEM micrograph suggests that grains are closely packed, which confirms that the density of the sintered pellet is very high and having minimum porosity as shown in Fig.1 (g). It is also observed that a large number of grains are uniformly distributed through clear and distinct grain boundaries over the sample surface. This may conclude that the charge carriers due to Ce⁺³/Ce⁺⁴ or evaporation of the Mn⁺⁴ generate the leakage current in the material. The above-observed properties in the sample will be further confirmed from the frequency and temperature-dependent dielectric study in the later section. The average grain

size of the prepared sample is about 11.9 μm calculated from the intercept method. The presence of small size grains suggests that doping hinders the grain growth and guides to the larger surface area, which is efficient to upgrade the performance of the sample.

The Scherrer formula for average crystalline size (D) = $\frac{k\lambda}{\beta \cos\theta}$, where $\lambda = 1.54 \text{ \AA}$, $k \sim 0.89$, θ = peak position and β = FWHM [31]. After rearranging the Scherrer formula, we get, $\cos\theta = \left(\frac{k\lambda}{D}\right)\frac{1}{\beta}$. A graph is plotted taking $1/\beta$ along X-axis and $\cos\theta$ along Y-axis shown in Fig. 2(a). The average crystalline size of the sample is about 74.4 nm, which is calculated from the slope of the linear fit to the experimental data [32].

Figure 2. (a) Scherrer plot (b) Williamson-Hall plot of the $\text{BaMn}_{0.9}\text{Ce}_{0.1}\text{O}_3$ single perovskite

The Williamson-Hall (W-H) method calculates the lattice strain generated due to the addition of Ce dopant to the host matrix. A relation between FWHM (β) and lattice strain (ϵ) is derived by Williamson-Hall as $\cos\theta = \frac{k\lambda}{D} + 4\epsilon \sin\theta$. A graph is plotted by taking $4\sin\theta$ along X-axis and $\beta \cos\theta$ along Y-axis shown in Fig. 2(b). The average crystalline size of the sample is calculated from the Y-intercept and the slope of the linear fit gives the lattice strain. The average crystalline size of the sample is about 74.4 nm. During the synthesis of the $\text{BaMn}_{0.9}\text{Ce}_{0.1}\text{O}_3$ single perovskite, Ce atoms enter into the host BaMnO_3 matrix. Due to strong interaction and bond stretching in the sample, a mechanical lattice strain (ϵ) is produced. The value of the lattice strain in the sample is 0.00107 [33].

Figure 3. Frequency response curves of (a) Z' and (b) Z'' of the $\text{BaMn}_{0.9}\text{Ce}_{0.1}\text{O}_3$ single perovskite

Fig. 3 (a) represents the variation of the frequency with the real component of impedance (Z') at a wide range of temperatures (25°C to 500°C). The complex impedance of an electrical network can be written as $Z^*(\omega) = Z'(\omega) - jZ''(\omega)$, where Z' is the real component and Z'' is the imaginary component. It is observed from the graph that Z' has a higher value at both the lower value of frequency and temperature. With an increase in frequency and temperature, the real component (Z') decreases, which confirms that the conduction mechanism is enhanced in the sample [34]. The release of the space charge polarization is confirmed, as all the curves of Z' merge into one at a high-frequency range and also due to higher temperature effect [35]. Fig. 3 (b) shows the frequency response curves of imaginary part (Z'') a wide range of temperatures (25°C to 500°C). Here, we observed six peaks at a different value of low frequencies. Out of which two small peaks at high frequency developed due to bulk effect, which is partially dominated by four peaks at low frequency due to the grain boundaries effect. The microstructure study suggests that the relaxation phenomenon is developed due to the shifting of peaks to the higher frequencies with the rise of temperature. The height of peaks decreases with the rise of temperature confirms that thermally activated charge carriers are developed and responsible for the conduction mechanism in the sample [36-37].

Figure 4. (a) Frequency response of Z' and Z'' of the complex impedance at $T = 250^\circ\text{C}$ and (b) Logarithm of relaxation time versus temperature of the $\text{BaMn}_{0.9}\text{Ce}_{0.1}\text{O}_3$ single perovskite

Fig. 4 (a) represents the frequency response curve of the real and imaginary part of the complex impedance at fixed temperature 200°C . The nature of the two curves suggests that relaxation frequencies of Z' and Z'' are very close to the same value and at the same time, curves show a deviation from the Debye model [38]. The relaxation time is calculated using the standard formula $\tau = \frac{1}{2\pi f_r}$, where f_r is called relaxation frequency. Arrhenius model is used to calculate the activation energy by plotting $\ln(\tau)$ versus $1000/T$ ($^\circ\text{C}^{-1}$) as shown in fig. 6 (b) [39]. The observed two activation energies are $E_{a1} = 173$ meV and $E_{a2} = 0.43$ meV at high and low-temperature range respectively. Therefore, the relaxation process is controlled by the immobile charge carriers in the host materials at low-temperature range while controlled by defects at higher temperatures [40].

Figure 5 (a) Nyquist and (b) Cole-Cole plots of the BaMn_{0.9}Ce_{0.1}O₃ single perovskite

The effect of grain and grain boundaries are two important factors that need to address to understand the conduction mechanism in the sample. Fig. 7 (a) presents Nyquist plots and Fig. 7 (b) shows Cole-Cole plots of BaMn_{0.9}Ce_{0.1}O₃ single perovskite in a wide range of temperatures from 25^oC – 500^oC. Nyquist semicircular plots explain grain and grain boundaries affect by resistive and capacitive data in the sample. An electrical circuit is designed taking the resistive and capacitive passive elements (R, C, Q) to calculate the theoretical value of the frequency power (n), bulk resistance (R_b), bulk capacitance (C_b) constant phase factor (Q), grain boundary capacitance (C_{gb}) and grain boundary resistance (R_{gb}). The {CQR} circuit works at low temperature whereas {(CQR) × (CR)} circuit works at high temperature to calculate different electrical parameters, where, $C = A(j\omega)^{m-1}$ and $Q = A(j\omega)^{n-1}$ are Jonscher's universal components. A software package called ZSIMP WIN is used to model with above-designed circuits. The semicircular curves with their centers lying below or on the Z' axis suggest a homogeneity mixture of the constituents with very small imperfection/defects (if any) in the sample. For non-Debye type relaxation in the sample, a new model is designed by adding a constant phase element (CPE) parallel to the RC network. The value of the electrical admittance (Y) of the CPE can be written as; $Y_{CPE} = X_0(j\omega)^n = M\omega^m + jN\omega^m$ where $M = X_0 \cos(n\pi/2)$ and $N = X_0 \sin(n\pi/2)$. Here, m and X₀ are temperature-dependent constants and dispersion measurement factors respectively.

Table 1. represents the calculated value of experimental and fitting data of the grain/bulk that include bulk resistance (R_b), bulk capacitance (C_b), grain boundary resistance (R_{gb}), grain boundary capacitance (C_{gb}), and power frequency (n) from 25⁰C to 500⁰C. It is observed from Table.1 data that the bulk capacitance (C_b) decreases from 2.253×10^{-16} F/cm² at 25⁰C to 6.242×10^{-20} F/cm² at 500⁰C and also bulk resistance (R_b) decreases from $7.236 \times 10^3 \Omega$ at 25⁰C to $9.432 \times 10^2 \Omega$ at 500⁰C confirm semiconducting nature i.e. negative temperature coefficient of resistance (NTCR) behavior of the sample. The larger Nyquist semicircles at low frequencies are due to the effect of grain boundaries while smaller Nyquist semicircles at higher frequencies are due to the effect of bulk grains [41]. The presence of both semicircular Nyquist and Cole-Cole plots support the semiconducting nature of the BaMn_{0.9}Ce_{0.1}O₃ single perovskite [42].

Table 2: List of the fitted data of equivalent circuit (CQR) (CR): bulk capacitance (C_b) constant phase factor (Q), bulk resistance (R_b), boundary resistance-capacitance (C_{gb}), grain boundary resistance (R_{gb}) and frequency power at 25⁰C, 50⁰C, 100⁰C, 150⁰C 200⁰C, 250⁰C, 300⁰C , 350⁰C, 400⁰C , 450⁰C and 500⁰C

Temp.	C_b (F/cm ²)	Q (S-sec ⁵ /cm ²)	R_b (Ω -cm ²)	C_{gb} (F/cm ²)	R_{gb} (Ω -cm ²)	Frequency power (n)
25 ⁰ C	2.253×10^{-16} (Expt.)	7.793×10^{-9} (Expt.)	7.236×10^3 (Expt.)	2.012×10^{-10} (Expt.)	1.301×10^7 (expt.)	34.10
	2.201×10^{-16} (fitting)	7.778×10^{-9} (fitting)	7.241×10^3 (fitting)	2.012×10^{-10} (fitting)	1.302×10^7 (fitting)	
50 ⁰ C	2.135×10^{-18} (Expt.)	7.439×10^{-9} (Expt.)	1.501×10^3 (Expt.)	2.009×10^{-10} (Expt.)	2.082×10^7 (Expt.)	14.40
	6.011×10^{-17} (fitting)	7.433×10^{-9} (fitting)	1.500×10^4 (fitting)	2.009×10^{-10} (fitting)	2.081×10^7 (fitting)	
100 ⁰ C	1.848×10^{-10} (Expt.)	1.326×10^{-9} (Expt.)	1.000×10^{20} (Expt.)	1.002×10^{-20} (Expt.)	1.093×10^1 (expt.)	7.98
	1.848×10^{-10} (fitting)	1.327×10^{-9} (fitting)	1.004×10^{20} (fitting)	1.005×10^{-20} (fitting)	1.093×10^1 (fitting)	
150 ⁰ C	3.835×10^{-17} (Expt.)	2.895×10^{-8} (Expt.)	2.867×10^4 (Expt.)	2.239×10^{-10} (Expt.)	3.533×10^6 (Expt.)	10.30
	4.806×10^{-17} (fitting)	2.894×10^{-9} (fitting)	2.869×10^4 (fitting)	2.240×10^{-10} (fitting)	3.533×10^6 (fitting)	
200 ⁰ C	5.408×10^{-10} (Expt.)	3.880×10^{-9} (Expt.)	4.423×10^4 (Expt.)	2.609×10^{-10} (Expt.)	8.984×10^5 (Expt.)	9.44
	5.408×10^{-10} (fitting)	3.880×10^{-9} (fitting)	4.423×10^4 (fitting)	2.609×10^{-10} (fitting)	8.984×10^5 (fitting)	
250 ⁰ C	1.843×10^{-10} (Expt.)	2.093×10^{-8} (Expt.)	2.311×10^5 (Expt.)	1.000×10^{-20} (Expt.)	9.987×10^0 (Expt.)	3.55
	1.843×10^{-10} (fitting)	2.095×10^{-9} (fitting)	2.311×10^5 (fitting)	1.208×10^{-18} (fitting)	9.992×10^0 (fitting)	

	(fitting)	(fitting)	(fitting)	(fitting)	(fitting)	
300 ⁰ C	1.000 x10 ⁻²⁰ (Expt.) 3.507 x10 ⁻²³ (fitting)	3.618 x10 ⁻¹⁰ (Expt.) 3.618 x10 ⁻¹⁰ (fitting)	1.099 x10 ⁷ (Expt.) 1.099 x10 ⁵ (fitting)	1.286 x10 ⁻⁹ (Expt.) 1.286 x10 ⁻⁹ (fitting)	4.868 x10 ³ (Expt.) 4.868 x10 ³ (fitting)	11.80
350 ⁰ C	1.000 x10 ⁻²³ (Expt.) 1.000 x10 ⁻²³ (fitting)	3.901 x10 ⁻¹⁰ (Expt.) 3.901 x10 ⁻¹⁰ (fitting)	4.478 x10 ⁴ (Expt.) 4.478 x10 ⁴ (fitting)	1.543 x10 ⁻⁹ (Expt.) 1.543 x10 ⁻⁹ (fitting)	2.211 x10 ³ (Expt.) 2.211 x10 ³ (fitting)	7.87
400 ⁰ C	3.182 x10 ⁻²² (Expt.) 3.182 x10 ⁻²² (fitting)	1.011 x10 ⁻⁹ (Expt.) 1.011 x10 ⁻⁹ (fitting)	1.277 x10 ⁴ (Expt.) 1.277 x10 ⁴ (fitting)	4.888 x10 ⁻¹⁰ (Expt.) 4.886 x10 ⁻¹⁰ (fitting)	6.605 x10 ³ (Expt.) 6.607 x10 ³ (fitting)	7.52
450 ⁰ C	1.033 x10 ⁻¹⁹ (Expt.) 5.120 x10 ⁻¹⁹ (fitting)	1.047 x10 ⁻⁶ (Expt.) 1.043 x10 ⁻⁸ (fitting)	2.677 x10 ³ (Expt.) 2.680 x10 ³ (fitting)	2.948 x10 ⁻¹⁰ (Expt.) 2.949 x10 ⁻¹⁰ (fitting)	5.950 x10 ³ (Expt.) 5.947 x10 ³ (fitting)	8.78
500 ⁰ C	6.242 x10 ⁻²⁰ (Expt.) 9.280 x10 ⁻²⁰ (fitting)	8.782 x10 ⁻⁷ (Expt.) 8.795 x10 ⁻⁸ (fitting)	9.432 x10 ² (Expt.) 9.430 x10 ² (fitting)	2.724 x10 ⁻¹⁰ (Expt.) 2.723 x10 ⁻¹⁰ (fitting)	3.522 x10 ³ (Expt.) 3.522 x10 ³ (fitting)	9.79

According to the Jonscher's power law, the total electrical conductivity (σ_t) in a material can be written as $\sigma_t = \sigma_{dc} + A \omega^n$, Where σ_{dc} is the dc part of the conductivity comes from the low-frequency static region, A is temperature dependant constant related to the intrinsic property of the material, ω is the angular frequency, and the exponent n is the degree of interaction of mobile ions with the lattice. Fig. 6(a) represents the variation of ac conductivity with frequency at different temperatures (25⁰C to 500⁰C). It is observed that at a fixed temperature, a low-frequency steady behavior of conductivity is called σ_{dc} and high-frequency dispersive conductivity is called σ_{ac} . At higher frequency, the value of σ_{ac} gradually increases and suddenly rises after a certain frequency is known as hopping frequency (ω_p) which is also temperature-dependent. This confirms the motion of charge carriers hopping type in between two potential minimum and sudden hopping.

Figure 6. (a) Frequency response curves of ac conductivity (b) represent temperature response curves of ac conductivity of the BaMn_{0.9}Ce_{0.1}O₃ single perovskite

The electrical ac conductivity of a sample can be calculated using a relation; $\sigma_{ac} = \omega \epsilon_r \epsilon_0 \tan \delta$, all the symbols have their usual meaning. Fig.6 (b) represents the temperature response curves of the ac conductivity in the sample. It is observed that the value of ac conductivity increases in both a higher range of frequency and temperature. The activation energy (E_a) in the sample can be calculated by using a relation; $\sigma_{ac} = \sigma_0 e^{-\frac{E_a}{k_B T}}$, where k_B is the Boltzmann constant. The calculated activation energies are 689.9 meV, 512.2 meV, 356.1 meV, 221.2 meV and 96.8 meV at 1 kHz, 10 kHz, 100 kHz, 1 MHz and 5 MHz with varying temperature. It is observed that activation energy increases with an increase in temperature suggesting that conductivity in the sample is thermally activated. The presence of elevated activation energy help in the conduction process because of the hopping of charge carriers due to the defects and oxygen vacancies at the higher range of temperature and frequency [43]. Therefore, the increase of conductivity from 25°C to 500°C confirms the semiconducting nature of the BaMn_{0.9}Ce_{0.1}O₃ single perovskite [44]

Figure. 7 Frequency response curves (a) M' and (b) M'' of the $\text{BaMn}_{0.94}\text{Ce}_{0.06}\text{O}_3$ single perovskite

The real component of modulus (M') and imaginary components (M'') is given as $M' = -\omega C_0 Z''$, $M'' = \omega C_0 Z'$, and $C_0 = \frac{\epsilon_0 A}{d}$, where $\epsilon_0 = 8.85 \times 10^{-12} \text{ F/m}$, A = surface area of the pellet sample, d = thickness of the pellet. Fig. 7 (a) and (b) show variation frequency with the real modulus (M') and imaginary modulus (M'') of the $\text{BaMn}_{0.9}\text{Ce}_{0.1}\text{O}_3$ single perovskite. Fig. 7(a) shows that increase in frequency, M' increases and get merged into one at all temperature confirming that the conduction mechanism is controlled by thermally activated charge carriers in the sample [45]. The Fig 7 (b) shows that imaginary modulus M'' increases with frequency got maximum value and then decreases in the $\text{BaMn}_{0.9}\text{Ce}_{0.1}\text{O}_3$ single perovskite [46]. The presence of the symmetrical curves of the M'' plots confirms the Debye-type behavior in the sample. Therefore, we may conclude that the prepared $\text{BaMn}_{0.94}\text{Ce}_{0.06}\text{O}_3$ single perovskite must undergo to thermally activated relaxation process due to the hopping of charge carriers [47].

Figure 8. (a) Frequency response curves of dielectric constant (b) tangent loss of BaMn_{0.9}Ce_{0.1}O₃ single perovskite

The electrical complex permittivity of a material can be written as; $\varepsilon^*(\omega) = \varepsilon'(\omega) - j\varepsilon''(\omega)$, where ε' and ε'' are the real and imaginary components of the complex permittivity respectively [48]. The role of the real part $\varepsilon'(\omega) = \frac{Z''}{\omega C_0(Z'^2 + Z''^2)}$ provides the amount of energy stored whereas the complex part $\varepsilon''(\omega) = \frac{Z'}{\omega C_0(Z'^2 + Z''^2)}$ provides the quality and quantitative information of the dissipation energy in the material under study. The complex permittivity can be calculated from the complex impedance using the relation; $\varepsilon^* = \frac{1}{j\omega C_0 Z^*}$, all symbols have their usual meaning [49]. Fig. 8 (a) & (b) show frequency response curves of dielectric constant and tangent loss at a wide range of temperature from 25°C to 500°C whereas Fig. 9(a) and (b) temperature response curves of dielectric and tangent loss at 1kHz, 10 kHz, 100 kHz, 1MHz and 5 MHz. It is observed that at low frequencies, dielectric constants are very large value and decrease with both frequency and temperature. This tendency of the variation of the curve indicates a change in dielectric properties in the prepared BaMn_{0.9}Ce_{0.1}O₃ single perovskite. To explain the nature of the curves, it is observed at low frequencies and for each temperature, both dielectric and tangent loss start their maximum value and then decreases towards high frequencies and merge with all temperature. The development of the electric polarization in the sample can help to understand the real cause of change in dielectric properties. The polarization present in the sample is the combination of the electronic, ionic, interfacial, and dipolar polarization [50].

Figure 9. (a) Temperature response curves of dielectric constant (b) tangent loss of BaMn_{0.9}Ce_{0.1}O₃ single perovskite

The reason for the maximum value of the dielectric constant at low frequencies is due to the application of an external *ac* electric field which aligned all the electric dipoles into the same direction of the field. But with an increase of frequency, comparatively heavy particles cannot follow with the applied electric field, by which both ionic and dipolar polarization contribution decrease in the calculation of total polarization. However, at the higher frequencies, the contribution of grain boundaries becomes significant by accumulating the thermally activated charge carriers and defects including impurities and deficiencies, and summing together to raise the value of interfacial polarization introduced by Maxwell-Wagner [51-52]. They proposed a two-layer model, formed by good conducting grains and poorly conducting grain boundaries. The electrons are more active at the grains in higher frequencies while less active at grain boundaries in low frequencies. This implies that grain boundaries are high resistance zone. Therefore, dielectric constant and $\tan\delta$ have a minimum value at higher frequencies resulting in a small dielectric loss.

4. Conclusion

BaMn_{0.9}Ce_{0.1}O₃ single perovskite is prepared by using a high-temperature oxide mixing method. XRD analysis suggests a hexagonal crystal structure with space group P6₃/mmc. Scherer's relation is used to the average crystallite size of the sample, which is about 74.4 nm. The micro-lattice strain developed because of Ce dopant in the pure BaMnO₃ matrix, which is about 0.142% calculated by using the W-H method. The SEM micrograph suggests that the sample has a uniform distribution of the grains and distinguishable grain boundaries and also average grain size is about 11.9 μm. The impedance spectroscopy technique is the prime method used to calibrate the electrical properties of the prepared sample. It is observed that the grain plays a dominant role compare to grain boundaries in defining the conductivity in the sample. The study of dielectric data concludes that two conduction mechanisms are observed at low and high temperatures. Shifting of potential height and calculated activation energy, it may conclude that conductivity in the prepared sample is thermally activated. The thermally activated relaxation process is controlled by the immobile charge carriers in the host materials at low-temperature range while controlled by defects at higher temperatures. From the above discussion, it is confirmed that the prepared sample is dominated by the hopping process. The presence of semicircular arcs in both Nyquist and Cole-Cole plots confirms the semiconducting nature of the sample and find applications in solid oxide fuel cells and scintillators.

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Figures

Figure 1

(a) XRD pattern of BaMn_{0.9}Ce_{0.1}O₃ single perovskite (b) Rietveld analysis of BaMn_{0.9}Ce_{0.1}O₃ single perovskite (c) Space filling structure (d) Ball-and-stick structure of BaMn_{0.9}Ce_{0.1}O₃ single perovskite (e) wire frame dot structure (f) polyhedral structure of BaMn_{0.9}Ce_{0.1}O₃ single perovskite taking 62 atoms, 96 bonds, 14 polyhedral. (g) SEM micrograph of BaMn_{0.9}Ce_{0.1}O₃ single perovskite

Figure 2

(a) Scherrer plot (b) Williamson-Hall plot of the BaMn_{0.9}Ce_{0.1}O₃ single perovskite

Figure 3

Frequency response curves of (a) Z' and (b) Z'' of the $\text{BaMn}_{0.9}\text{Ce}_{0.1}\text{O}_3$ single perovskite

Figure 4

(a) Frequency response of Z' and Z'' of the complex impedance at $T = 250\text{ }^{\circ}\text{C}$ and (b) Logarithm of relaxation time versus temperature of the $\text{BaMn}_{0.9}\text{Ce}_{0.1}\text{O}_3$ single perovskite

Figure 5

(a) Nyquist and (b) Cole-Cole plots of the BaMn_{0.9}Ce_{0.1}O₃ single perovskite

Figure 6

(a) Frequency response curves of ac conductivity (b) represent temperature response curves of ac conductivity of the BaMn_{0.9}Ce_{0.1}O₃ single perovskite

Figure 7

Frequency response curves (a) M' and (b) M'' of the $\text{BaMn}_{0.94}\text{Ce}_{0.06}\text{O}_3$ single perovskite

Figure 8

(a) Frequency response curves of dielectric constant (b) tangent loss of $\text{BaMn}_{0.9}\text{Ce}_{0.1}\text{O}_3$ single perovskite

Figure 9

(a) Temperature response curves of dielectric constant (b) tangent loss of BaMn_{0.9}Ce_{0.1}O₃ single perovskite