

Influence of Sintering Temperature and Mixing Method on the Structure, Microstructure, and Electrical Properties of $\text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3$ Manganites

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ABSTRACT

The electrical behavior in manganites compound is strongly affected by the synthesis routes and methods. Previous works have shown that microstructure formation will affect the electrical transport properties of manganites compound. In this work, the effects of sintering temperature on the structural, microstructural, and electrical properties of $\text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3$ (LBMO) samples prepared by mortar grinding (LBMO(A)) and direct glass rod stirring (LBMO(B)) were explored. TGA analysis of the precursor revealed three main weight-loss stages corresponding to dehydration, decomposition of acetate and nitrate groups, and solid-state reactions, indicating that complete phase formation begins above 650 °C. XRD results showed that LBMO(A) samples sintered at 900-1000 °C contained secondary phases, while LBMO(B) exhibited a single-phase perovskite structure across all sintering temperatures, indicating better crystallinity. SEM observations revealed grain growth and densification with increasing temperature, with LBMO(A) developing gradually and showing partial melting at higher sintering temperature, while LBMO(B) densified more rapidly, forming larger and well-connected grains. Temperature-dependent resistivity measurements showed the typical metal-insulator transition, with LBMO(B) exhibiting lower resistivity and higher T_{MI} , attributed to improved grain connectivity and reduced porosity. Overall, the study highlights the importance of synthesis mixing method and sintering temperature in optimizing phase purity, microstructure, and electrical behavior of LBMO manganites.

Keywords: LBMO; microstructural change; grain boundary effect; electrical behavior

INTRODUCTION

Perovskite-type manganites with the general formula $\text{La}_{1-x}\text{A}_x\text{MnO}_3$ (where A is a divalent alkaline earth metal such as Ba, Ca, or Sr) have attracted significant attention due to their rich physical properties and potential applications in solid oxide fuel cells, magnetic sensors, spintronic devices, and energy conversion systems [1-3]. Among them, $\text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3$ (LBMO) has been widely studied because of its promising electrical transport properties and the occurrence of the metal-insulator transition (T_{MI}), which is strongly influenced by structural and microstructural factors [4]. The double-exchange (DE) mechanism between Mn^{3+} and Mn^{4+} ions plays a crucial role in governing the electrical behavior of LBMO, making its performance highly sensitive to synthesis parameters such as precursor homogeneity, calcination route, and sintering conditions.

Previous studies have demonstrated that the physical properties of manganite compounds are closely correlated with their microstructural characteristics, which in turn depend strongly on the synthesis route [5]. Conventional solid-state reaction methods often suffer from inhomogeneous mixing and require high sintering temperatures, leading to incomplete reactions and unwanted secondary phases [6]. On the other hand, wet chemical methods such as thermal treatment, sol-gel or co-precipitation provide better chemical homogeneity but involve complex procedures and potential contamination issues [7,8]. Thermal decomposition offers a relatively simple yet effective alternative, enabling the preparation of perovskite manganites with controlled particle size and improved phase purity [9]. However, within this method, factors such as precursor mixing strategy and sintering temperature can critically affect the final phase formation, crystallinity, and electrical transport properties of LBMO.

In this work, $\text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3$ compounds were synthesized using the thermal decomposition method to systematically investigate the influence of mixing techniques and sintering temperature on their structural, microstructural, and electrical properties. Two different precursor mixing approaches (i) manual grinding with a mortar and pestle and (ii) gently direct stirring with a glass rod, were employed to assess the role of initial homogeneity. Sintering was conducted over a wide temperature range (900-1400 °C) to study its effect on phase formation, grain growth, and electrical behavior. The resulting samples were characterized using thermogravimetric analysis (TGA), X-ray diffraction (XRD), scanning electron microscopy (SEM), and four-point probe (4PP) measurements. By correlating different processing conditions with the observed structural and electrical behavior, this study provides insights into the optimization of LBMO materials for future functional applications.

MATERIALS AND METHODS

In this study, lanthanum barium manganese oxide ($\text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3$) compounds were synthesized using the thermal decomposition method. The raw materials included lanthanum acetate ($\text{La}(\text{C}_2\text{H}_3\text{O}_2)_3 \cdot x\text{H}_2\text{O}$, 99.9%, Sigma-Aldrich), barium acetate ($\text{Ba}(\text{C}_2\text{H}_3\text{O}_2)_2$, 99.9%, Alfa Aesar), and manganese acetate ($\text{Mn}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 4\text{H}_2\text{O}$, 99.9%, Sigma-Aldrich). The raw materials were weighed according to their molar ratios using an electronic balance and mixed by two different techniques. In method 1, a mortar and pestle were used to grind the starting powders for 30 min, and the resulting sample was designated as LBMO(A). In method 2, a glass rod was used to stir and mix the powders

for 30 min and the resulting sample was designated as LBMO(B). Then, the following steps are similar for both methods. The ground mixture was transferred to an alumina crucible and calcined at 900 °C for 12 h. After that, the material was ground again using a mortar and pestle for 1 h and pressed into pellets using a hydraulic press at 5 tons for 5 min. The pellets were sintered in a tube furnace at various temperatures ranging from 900 °C to 1400 °C for 24 h. Next, the sample characterization of prepared LBMO(A) and LBMO(B) samples were conducted using Thermogravimetric Analysis (STA 8122, Thermo Fisher Scientific), X-ray Diffraction (X'Pert PRO, PANalytical), Scanning Electron Microscopy (Phenom ProX G6, Thermo Fisher Scientific equipped with EDS), and Four Point Probe (Standard Operating Procedure Ver2.211114) techniques. These analysis of crystal structure, surface morphology, and electrical properties provide a better understanding for optimizing the synthesis of desired LBMO compound.

RESULTS AND DISCUSSION

Thermogravimetric Analysis

Thermogravimetric analysis (TGA) of $\text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3$ was conducted to determine the weight change during heating, with the results presented as percentage mass loss versus temperature in Figure 1. The TGA analysis of the $\text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3$ precursor revealed three distinct weight-loss stages, with an overall mass reduction of 54.5%. The initial drop (30-273 °C) corresponds to the evaporation of adsorbed and coordinated water (13.5%) [10,11], while the second stage (273-41 °C), showing the largest mass loss (35.4%), is attributed to decomposition of acetate groups and nitrate complexes [12]. The gradual third stage from 641-1000 °C (5.6%) is due to the solid-state reactions between intermediate oxides [13,14]. These results suggest that the perovskite LBMO phase begins to form above ~650 °C. Therefore, a sintering temperature range of 900-1400 °C was chosen to ensure complete phase formation and promote grain growth.

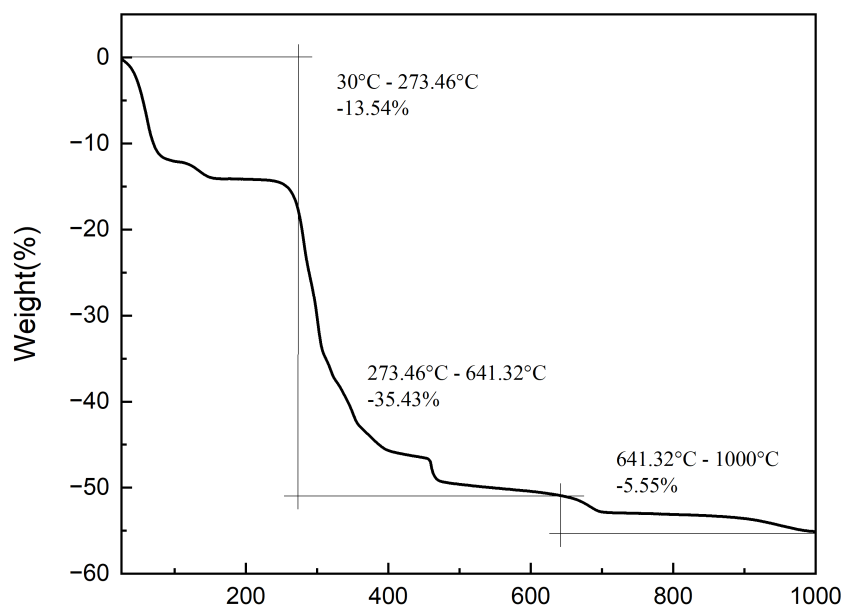


Figure 1. TGA curve of the $\text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3$ precursor

X-ray Diffraction Analysis

The X-ray diffraction (XRD) patterns of the samples, refined using the Rietveld method, are presented in Figure. 2. The XRD results reveal distinct differences in phase formation depending on the grinding techniques and sintering temperature. In the LBMO(A) series, samples sintered at 900 °C and 1000 °C exhibited two different phases corresponding to $\text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3$ (ICSD: 98-004-1043) and BaMnO_3 (ICSD: 98-000-3109). However, at higher sintering temperatures (1100 °C to 1400 °C), only the single $\text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3$ phase with ICSD code of 98-004-1043 was detected. In contrast, all LBMO(B) samples exhibited a single $\text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3$ phase regardless of sintering temperature. This indicates that glass rod stirring technique used in the LBMO(B) series is more effective than mortar mixing in preventing the formation of secondary phases.

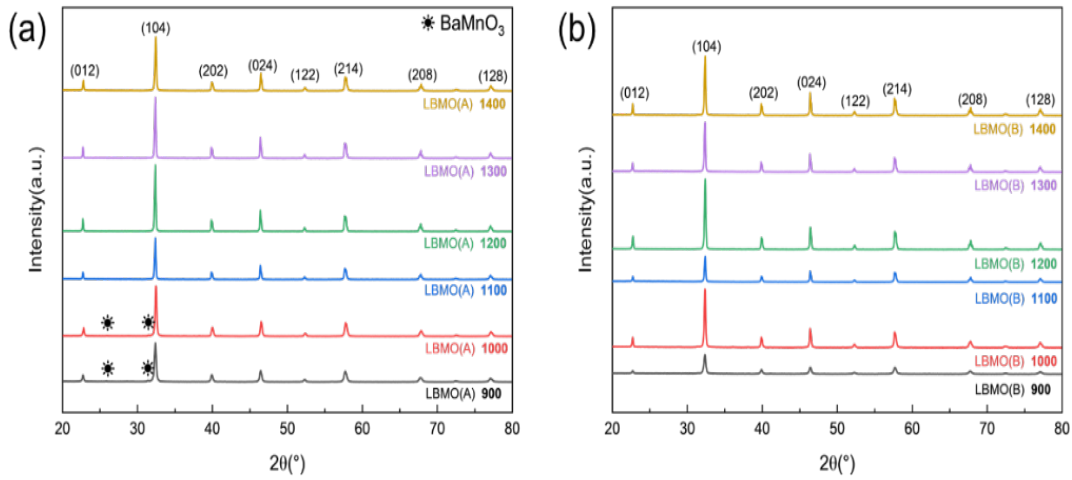


Figure 2. XRD pattern of LBMO samples sintered at temperatures ranging from 900 °C to 1400 °C. (a) LBMO(A) and (b) LBMO(B)

For all samples, the (104) peak was the most intense, suggesting preferred orientation along this plane. As the sintering temperature increases, the diffraction peak intensities became sharper and higher, while the full width at half maximum (FWHM) values decreased. This trend indicates enhanced crystallinity and grain growth at higher sintering temperature [15]. The crystallite size was estimated using Scherrer's equation [16,17]:

$$D = (k \lambda) / (\beta \cos \theta),$$

where D is the crystallite size, λ is the X-ray wavelength for $\text{Cu K}\alpha$ (1.5406 Å), β is the FWHM of the diffraction peak, and θ is the Bragg angle. As shown in Table 1, the crystallite size of LBMO(A) increased from 41.2 nm at 900 °C to 80.3 nm at 1300 °C, before slightly decreasing to 61.7 nm at 1400 °C. Similarly, in Table 2, LBMO(B) samples showed an increase from 37.1 nm to 72.6 nm, followed by a slight reduction to 65.6 nm. This small decrease at 1400 °C may be attributed to abnormal reduce grain growth or slight grain coalescence caused by significant densification at very high sintering temperature [18]

Tables 1 and 2 also show that the lattice parameters (a , b , c), unit cell volume (V), Mn-O bond lengths, and Mn-O-Mn bond angles exhibit negligible variations across all sintering temperatures for both LBMO(A) and LBMO(B) series. This means that the

crystal structure is very stable and does not undergo noticeable distortion within this sintering temperature range.

Table 1. Rietveld refinement data of LBMO(A) samples sintered at different temperatures

Sample code	LBMO(A) 900	LBMO(A) 1000	LBMO(A) 1100	LBMO(A) 1200	LBMO(A) 1300	LBMO(A) 1400
Phase	Lanthanum Barium Manganese Oxide ($\text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3$)					
Reference code	98-004-1043					
Structure type	Hexagonal					
Space group	R-3c (167)					
a (Å)	5.533	5.534	5.534	5.534	5.534	5.534
b (Å)	5.533	5.534	5.534	5.534	5.534	5.534
c (Å)	13.511	13.517	13.512	13.511	13.509	13.507
V (Å ³)	358.249	358.588	358.420	358.381	358.373	358.361
Mn ₁ -O ₁ (Å)	1.961	1.962	1.961	1.961	1.961	1.961
Mn ₁ -O ₁ (Å)	1.961	1.962	1.961	1.961	1.961	1.961
$\angle\text{Mn}_1\text{-O}_1\text{-Mn}_1$ (°)	170.451	170.451	170.451	170.450	170.450	170.449
R_{EXP} (%)	4.133	4.040	4.580	4.220	4.313	4.388
R_{P} (%)	3.871	3.902	5.817	5.333	5.670	4.926
R_{WP} (%)	4.958	4.981	7.517	7.082	7.362	6.527
Goodness of fit, χ^2	1.439	1.520	2.402	2.816	2.912	2.212
Phase	Barium Manganese Oxide (BaMnO_3)					
Reference code	98-000-3109					
Structure type	Hexagonal					
Space group	P63/mmc (194)					
a (Å)	5.695	5.690	-	-	-	-
b (Å)	5.695	5.690	-	-	-	-
c (Å)	4.806	4.808	-	-	-	-
V (Å ³)	135.009	134.860	-	-	-	-
FWHM of (104) peak (°)	0.223	0.189	0.117	0.101	0.098	0.126
Crystallite size (nm)	41.2	48.7	75.9	80.3	77.3	61.7

Table 2. Rietveld refinement data of LBMO(B) samples sintered at different temperatures

Sample code	LBMO(B) 900	LBMO(B) 1000	LBMO(B) 1100	LBMO(B) 1200	LBMO(B) 1300	LBMO(B) 1400
Phase	Lanthanum Barium Manganese Oxide ($\text{La}_{0.7}\text{Ba}_{0.3}\text{MnO}_3$)					
Reference code	98-004-1043					
Structure type	Hexagonal					
Space group	R-3c (167)					
a (Å)	5.532	5.534	5.535	5.534	5.535	5.536
b (Å)	5.532	5.534	5.535	5.534	5.535	5.536
c (Å)	13.516	13.509	13.511	13.508	13.508	13.506
V (Å ³)	358.303	358.333	358.517	358.388	358.401	358.528
Mn ₁ -O ₁ (Å)	1.961	1.961	1.962	1.961	1.961	1.962
Mn ₁ -O ₁ (Å)	1.961	1.961	1.962	1.961	1.961	1.962
$\angle\text{Mn}_1\text{-O}_1\text{-Mn}_1$ (°)	170.452	170.450	170.450	170.449	170.449	170.448
R_{EXP} (%)	4.924	4.403	6.390	4.357	4.547	4.635
R_{P} (%)	5.077	4.935	11.281	4.940	5.545	6.453
R_{WP} (%)	6.352	6.497	14.334	6.44	7.282	8.315
Goodness of fit, χ^2	1.663	2.177	5.031	2.186	2.565	3.217
FWHM of (104) peak (°)	0.242	0.148	0.1317	0.126	0.107	0.103
Crystallite size (nm)	37.1	59.1	70.7	66.2	72.6	65.6

Microstructural analysis

Figures 3 and 4 show the scanning electron microscopy (SEM) micrographs and grain size distributions of LBMO samples sintered between 900 °C and 1400 °C. Both LBMO(A) and LBMO(B) samples exhibit distinct grain growth and improved densification as the sintering temperature increases. However, significant differences in microstructural evolution were observed due to different mixing techniques.

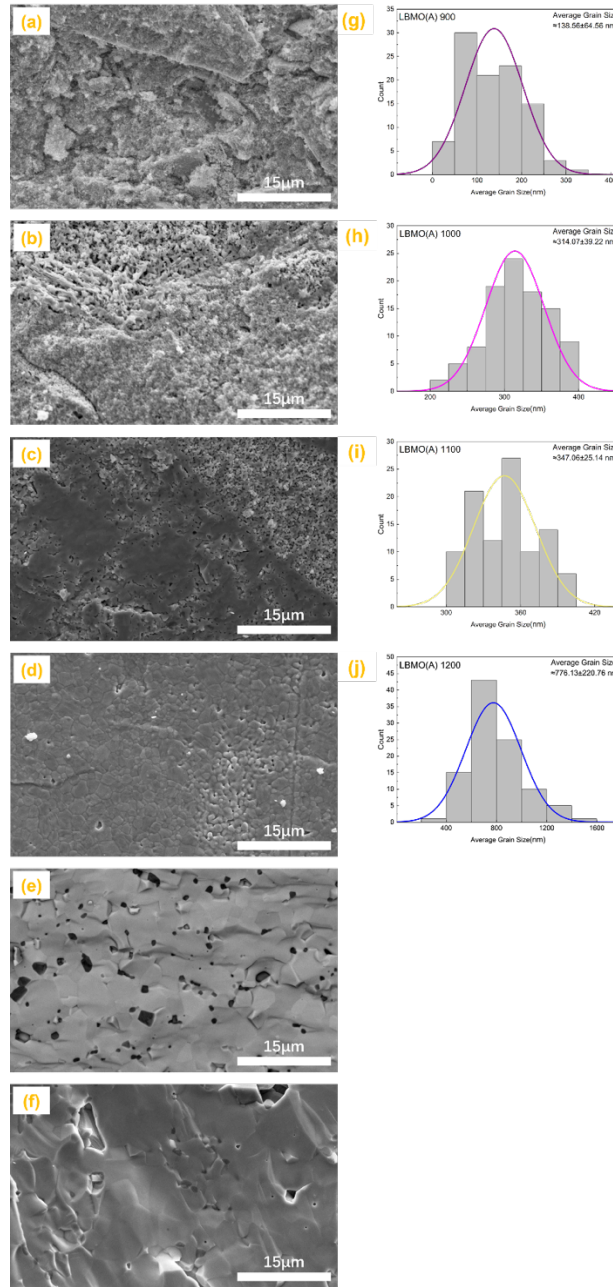


Figure 3. SEM micrographs of LBMO(A) samples sintered at (a) 900 °C, (b) 1000 °C, (c) 1100 °C, (d) 1200 °C, (e) 1300 °C, (f) 1400 °C, and their respective grain size distribution (g, h, i, and j)

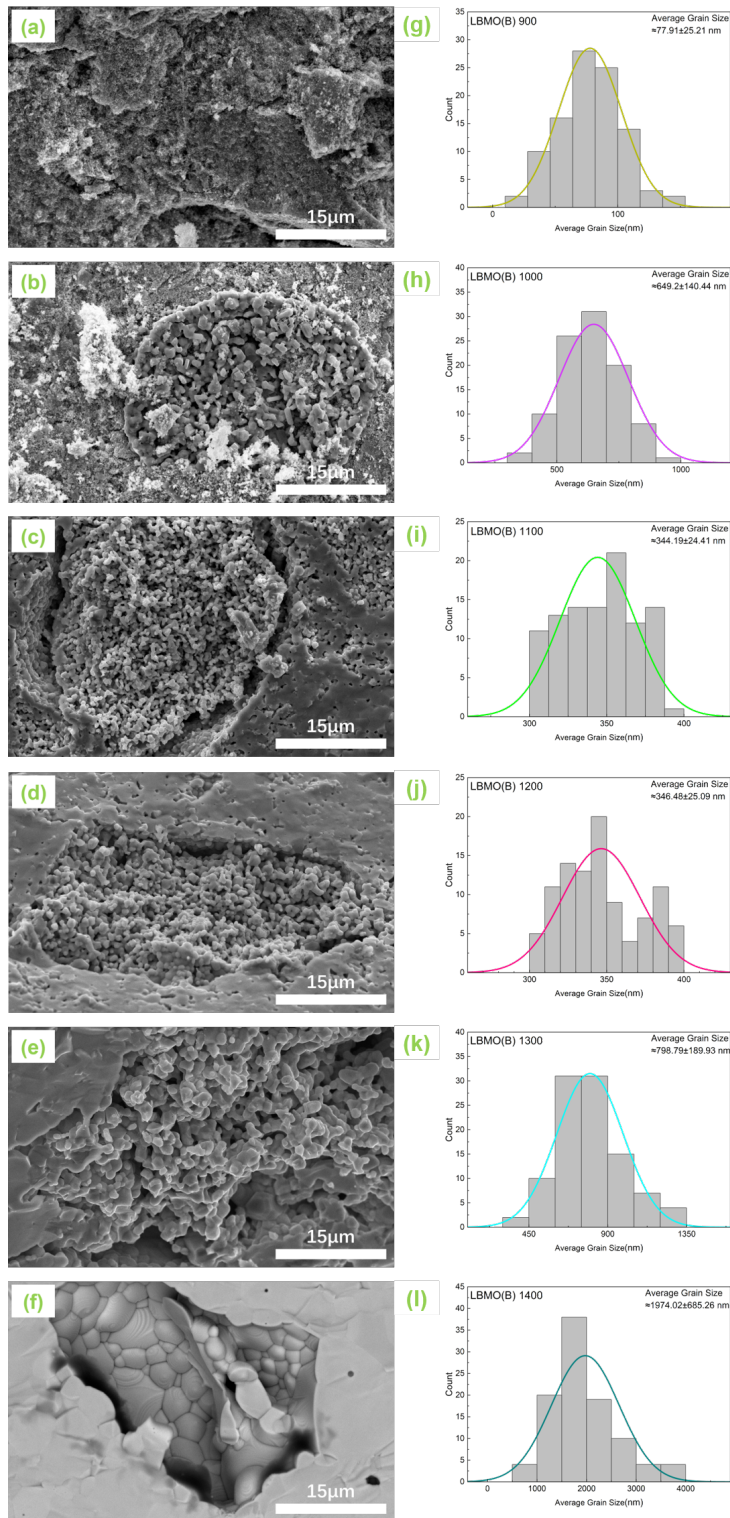


Figure 4. SEM micrographs of LBMO(B) samples sintered at (a) 900 °C, (b) 1000 °C, (c) 1100 °C, (d) 1200 °C, (e) 1300 °C, (f) 1400 °C, and their respective grain size distribution (g, h, i, j, k and l)

In the LBMO(A) series, prepared via mortar grinding, grain growth progresses gradually with increasing sintering temperature. At lower sintering temperatures (Figure 3a-c), the grains are small, irregular and loosely connected, with rough surfaces and high porosity. As the sintering temperature increases (Figure 3d-f), the grains become larger, more uniform, and more compact, with clearer grain boundaries and reduced voids. At the highest temperatures (1300-1400 °C), partial melting occurs, resulting in a dense, interconnected microstructure with smooth surfaces and well-controlled porosity. Overall, higher sintering temperatures promote grain coalescence, densification, and porosity reduction, while excessive sintering temperatures leads to partial melting and further structural consolidation [19].

In contrast, the LBMO(B), mixed by glass rod stirring, demonstrates a different microstructural evolution. When sintered at lower temperatures (Figure 4a-c), the particles remain largely separated, producing a loose, highly porous, and uneven structure. Around 1200 °C, the material begins to soften, creating a mixture of partially melted regions and areas where early grain formation starts, which explains the irregular porosity. At 1400 °C, the sample undergoes a sudden increase in densification, with grains growing much larger and the overall structure becoming significantly more consolidated. However, this rapid densification also introduces large voids or crack-like features, likely caused by thermal stress or excessive grain growth [20].

In summary, LBMO(A) prepared by mortar grinding develops gradually and uniformly, giving a compact and controlled microstructure. Meanwhile, LBMO(B) mixed with glass rod stirring densifies more suddenly, producing larger grains and some irregular features, while still achieving a dense structure at high sintering temperatures.

Resistivity measurement

The temperature-dependent resistivity behavior of LBMO(A) and LBMO(B) samples was measured in the range of 30-300 K, as illustrated in Figure 5. Both series exhibit typical manganite behavior, where resistivity increases with temperature up to a maximum and then decreases, indicating the metal-insulator transition (T_{MI}) [21]. At lower temperatures ($T < T_{MI}$), both LBMO(A) and LBMO(B) show metallic-like conduction, where resistivity decreases as temperature decreases. This behavior is governed by the double-exchange (DE) mechanism [22]. As temperature increases within this metallic region, resistivity gradually rises attributed to enhanced phonon scattering due to increasing thermal energy, which disrupts charge carrier mobility. When the resistivity reaches its maximum at T_{MI} , both series of samples undergo transition into insulating behavior, where resistivity decreases with further temperature increase.

Sintering temperature has a strong influence on the resistivity behavior. In LBMO(A), the sample sintered at 900 °C shows the highest resistivity due to poor grain growth, a high density of grain boundaries, and the presence of structural defects or secondary phases [23,24]. As the sintering temperature increases to 1400 °C, resistivity generally decreases because improved particle connectivity and pore elimination. An exception is LBMO(A) 1300, which shows a slight increase in resistivity, possibly caused by partial melting and grain boundary removal, as supported by the SEM observations in Figure 2. Lastly, for LBMO(A) 1400, the resistivity drops sharply due to the formation of a denser, well-connected microstructure.

A similar trend is observed in the LBMO(B) samples. At 900 °C, the LBMO(B) 900 sample exhibits the highest resistivity and a lower T_{MI} (~263 K), due to strong grain boundary scattering from small, poorly connected grains. Increasing the sintering temperature to 1100 °C improves grain connectivity and crystallinity, lowering resistivity and raising T_{MI} to about 280 K [25]. However, the resistivity rise again at temperatures above 1100 °C. This can be related to microstructural changes observed in SEM analysis, such as partial melting and the formation of large voids that disrupt electron transport.

In summary, across the sintering range, LBMO(B) shows slightly lower resistivity, which can be associated with improved grain connectivity, better crystallinity, and better structural ordering. These microstructural features support more efficient electron transport, suggesting that the glass rod stirring method in LBMO(B) contributes to enhanced electrical performance compared to LBMO(A) prepared by mortar grinding.

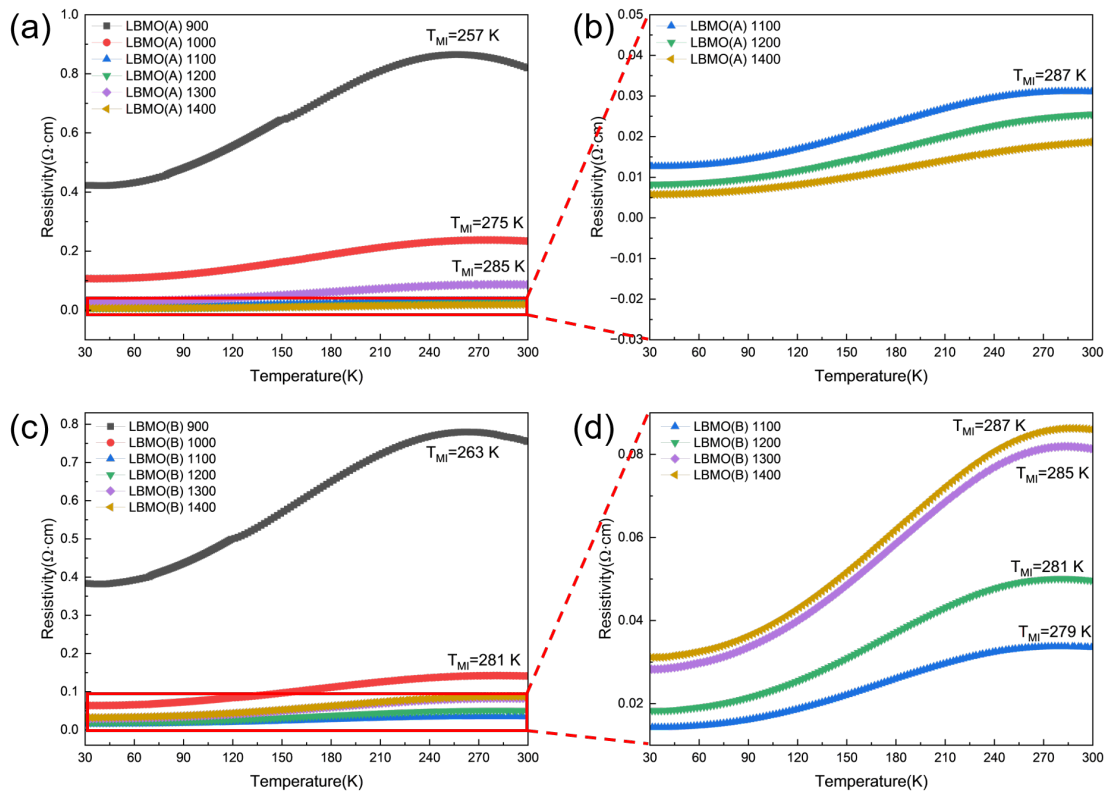


Figure 5. Resistivity vs temperature for (a) LBMO(A) and (c) LBMO(B) samples at different sintering temperatures and zoomed-in resistivity behavior of (b) LBMO(A) and (d) LBMO(B) samples at higher temperatures (1100, 1200, 1300 and 1400 °C)

CONCLUSIONS

The effect of sintering temperature and mixing techniques on the structural, microstructural and electrical properties of LBMO(A) and LBMO(B) samples were systematically investigated. X-ray diffraction (XRD) analysis showed that LBMO(A) sintered at 900-1000 °C contained two different phases, while LBMO(B) exhibited a single LBMO phase across all temperatures, indicating better crystallinity in LBMO(B).

SEM analysis revealed that both series undergo grain growth and densification with increasing sintering temperature, although their microstructural evolution differs due to the mixing methods. LBMO(A), prepared by mortar grinding, exhibits gradual and uniform grain growth, while LBMO(B), mixed by glass rod stirring, shows more rapid densification and larger grain formation at higher temperatures. The temperature-dependent resistivity measurements demonstrate the characteristic metal-insulator transition in both series, with resistivity strongly influenced by sintering temperature. Improved grain connectivity, reduced porosity, and better structural ordering enhance electron transport, particularly in LBMO(B), which shows slightly lower resistivity and higher T_{MI} compared to LBMO(A). Overall, the study highlights that sintering temperature and mixing method play crucial roles in controlling microstructure and electrical performance in LBMO manganites, with LBMO(B) exhibiting a more favorable combination of microstructural and resistivity characteristics.

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AUTHORS' CONTRIBUTIONS STATEMENT

The manuscript was written through the contributions of all authors. L.D, X.T.H, K.P.L, N.R and L.N.L designed and organized this work. L.D, X.T.H and K.P.L writing-review and editing. X.T.H, K.P.L and N.R draft the main ideas of the work. K.P.L, M.M.A.K, S.K.C, M.K.S and A.H.S supervising. L.D, X.T.H, K.P.L, N.R and L.N.L review and editing of the manuscript. All authors have approved the final version of the manuscript.

CONFLICT OF INTEREST

All authors declare no competing interests that could have influenced the work reported in this paper.

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