ENHANCED KNOWLEDGE IN SCIENCES AND TECHNOLOGY
e-ISSN: 2773-6385

EKST

Vol. 4 No. 2 (2024) 387-393
https://publisher.uthm.edu.my/periodicals/index.php/ekst

The Effects of Sintering Temperature on CrystallinePhase, Microstructure, and Electrical Properties of Sm0.5Ca0.5MnO3

Viknesvaran Ravinthiran [1], Suhadir Shamsuddin[1*]

[1] *Ceramic and Amorphous Group, Faculty of Applied Sciences and Technology, Pagoh Higher Education Hub, University Tun Hussein Onn Malaysia, 84600 Panchor, Johor, Malaysia*

*Corresponding Author: zaky@uthm.edu.my
DOI: https://doi.org/10.30880/ekst.2024.04.02.042

Article Info

Received: 27 December 2023
Accepted: 15 January 2024
Available online: 12 December 2024

Keywords

Ceramic, Solid state reaction, Sintering temperature, XRD, FTIR, Four point

Abstract

The present status of knowledge in the field of material physics studies and ceramic industry. The article mainly focusing on solid state reaction method and sintering temperature effects on structural, electrical and optical properties of Sm0.5Ca0.5MnO3. There are plenty of studies on doping mechanism using Sm0.5Ca0.5MnO3 but very less focusing on sintering temperature effects using divalent compound. Solid state reaction method used in the making of divalent compounds such as mixing, grinding,and pelleting. The density was calculated utilizing the Archimedes Principle, and the porosity level was calculated using the usual method. As the sintering temperature rises, so does the bulk density and porosity. Scanning electron spectroscopy shows clear increasement in grain size as the sintering temperature increases. As the sintering temperature rises, so does the resistance. Through EDX analysis, the samples were found to have the same composition without any contaminants.

1. Introduction

Researchers have been debating perovskite manganite compounds since the last decade due to their fascinating characteristics. Sintering is a vital processing step in the production of ceramic materials, and understanding its influence on material characteristics is important for the development of sustainable and efficient manufacturing methods. A systematic study of the Sm0.5Ca0.5MnO3 manganite has been conducted, mainly to understand the influence of sintering temperature on structure, electrical and optical properties in the materials.The solid-state reaction technique was used to create the manganese sample Sm0.5Ca0.5MnO3. The Sm0.5Ca0.5MnO3 manganite is synthesized in this work to investigate the influence of sintering temperature on a structural term, surface morphological, electrical, and optical properties of the manganite composition. Further research into the influence of different sintering temperatures on manganite allows for greater comprehension of the alterations in structural properties such as crystalline structure and arrangement, as well as surface morphology including as grain size, boundaries, density, and porosity [1]. This study will use the sample compound Sm0.5Ca0.5MnO3 to evaluate the influence of sintering temperature on the structural, optical, and electrical characteristics of divalent manganite.

The main objective of this study is to prepare Sm0.5Ca0.5MnO3 sample by using solid state method reaction and to investigate the sintering temperature effects on structural properties of Sm0.5Ca0.5MnO3 compound under three different temperatures by using x-ray diffraction analysis. To explore the sintering temperature effects on electrical and optical properties on Sm0.5Ca0.5MnO3 under three different temperatures (1100°C, 1150°C and 1200°C) by using point probe method. Solid state reaction method will be used to prepare the

Sm0.5Ca0.5MnO3 sample. The Sm0.5Ca0.5MnO3 will undergo processes such as mixing, calcination, grinding, pelleting, and sintering.

2. Method

Sample with chemical formula, Sm0.5Ca0.5MnO3 has been prepared using solid state reaction method. The preparation of the sample process has been described extensively elsewhere. Synthesized of Sm0.5Ca0.5MnO3 in this research, contains processes such as grinding, calcination, palletization, and sintering [2]. The first step in sample preparation involved weighing the mass of each of the elements accurately, which is Samarium Oxide III, Calcium Carbonate and Manganese IV Oxide. The weighing process repeated twice to obtain the accurate mass.

The chemical powders mixed homogenously and undergo calcination process at 100°C to remove impurities and volatile substances to cause thermal composition. Followed by grinding process topromotes homogeneity, increases surface area, and reduces particle size. The powder undergoes calcination process once again at 100°C to increase the quality of the sample. Next is the palletization process with a scale of 13mm diameter and 2mm to 3mm thickness. In the last phase of the sample preparation technique, the taken samples had been sintered for twenty-four hours at three different temperatures which is 1100 °C, 1150 °C, and 1200 °C.

3. Results and Discussion

The chemicals used to synthesize the sample were Samarium (III) Oxide, Calcium Carbonate and Manganese (IV) Oxide powder of high purity (99.9%). Agate mortar, crucible, spatula, digital weighing scale preterm furnace, Carver model 3851-0 hydraulic press, Coxem EM-30AX Plus (SEM), four-point probe.

4. Sample characterization

To study structural analysis, surface morphology imaging was performed using X-ray diffraction (XRD) and a scanning electron microscope (SEM) equipped with energy dispersion x-ray spectroscopy (EDX). The electrical and optical properties were analyzed using the four-point probe method and Fourier transform infrared spectroscopy analysis respectively. The bulk density of the materials was calculated using the Archimedes principle with acetone as the buoyant medium. The bulk density was then calculated using the equation below.

$$\rho_{\text{bulk}} = \frac{w_{\text{air}}}{w_{\text{air}} - w_{\text{acetone}}} \rho_{acetone} \qquad Eq. 1$$

where w_{air} represents the weight of the sample in air, $W_{acetone}$ represents its weight of the sample immersed into acetone (buoyant medium), and acetone represents the density of acetone. The formula to calculate porosity is shown in equation 2.

$$\text{Porosity (\%)} = (1 - \frac{\rho_{\text{bulk}}}{\rho_{theoretical}}) \times 100\% \qquad Eq. 2$$

on which ρ_{bulk} is the bulk volumetric density of the sample's material derived utilizing Equation 1 and $\rho_{\text{theoretical}}$ is the theoretical density of the material computed applying Equation 3.

$$\rho_{theoretical} = \frac{n'(\sum A_c + \sum A_A)}{V_c N_A} \qquad Eq. 3$$

while n' indicates the approximate number of formula units/unit cell, ΣAc represents the total atomic weights of cations inside the formula units, AA indicates the entire atomic weights of anions inside the formula units, Vc provides the unit volume of the cell, and NA indicates Avogadro's numbers. To assess the crystallized state for the samples, the information on lattice parameter and unit cell volume was examined using the Sigmaplot application.

Following that, the crystalline structures were identified and designated with miller indices (h, k, l), which indicate Bragg's plane. The lattice parameters a, b, and c were calculated using the methoddescribed in Equation 4 [3].

$$\frac{1}{{d_{hkl}}^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \qquad Eq. 4$$

Enhanced Knowledge in Sciences and Technology Vol. 4 No. 2 (2024) p. 387-393

5. Result and Discussion

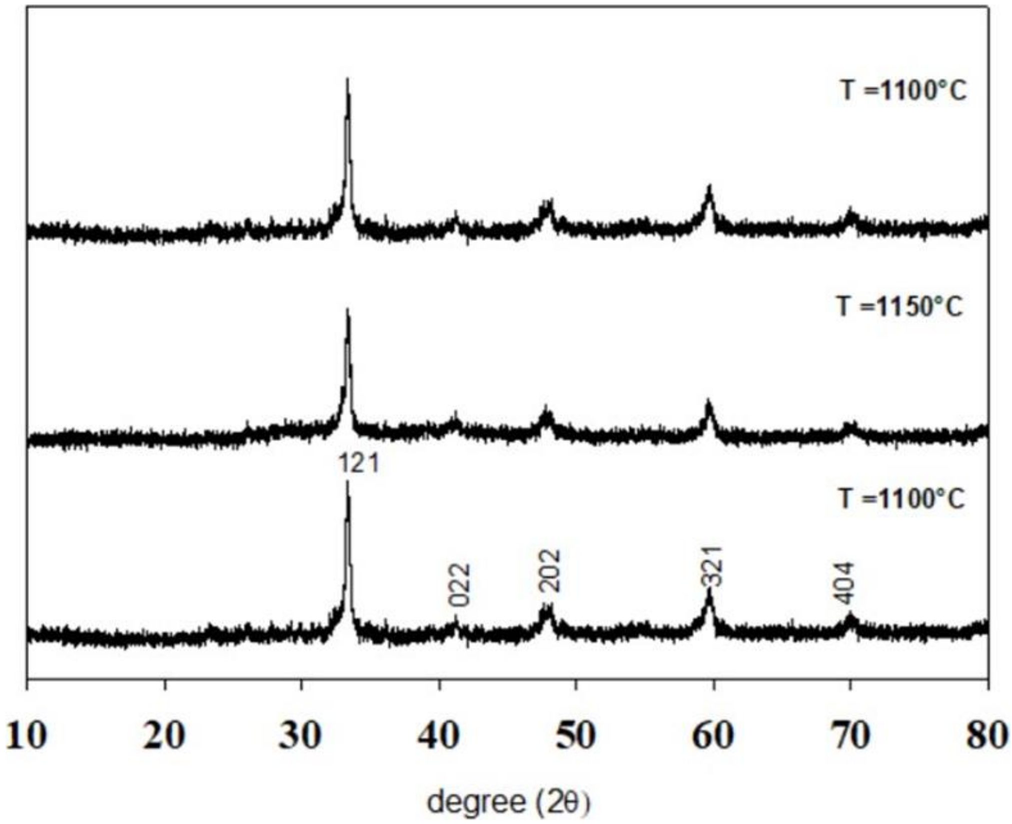


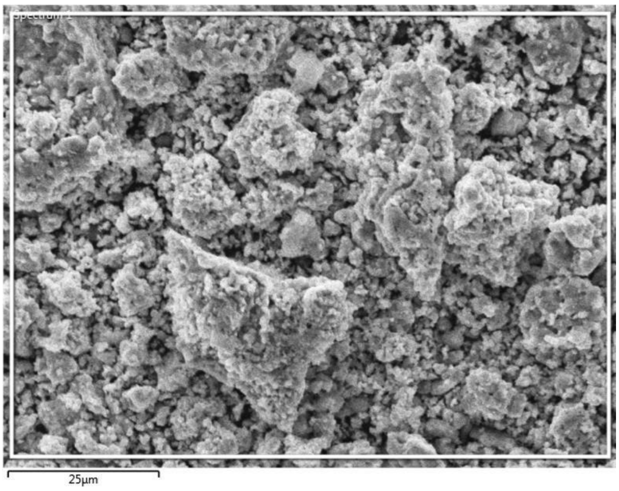
Fig. 1 *X-ray Diffraction (XRD) analysis for the sample Sm0.5Ca0.5MnO3 sintered at 1100°C, 1150°C and 1200°C.*

Table 1 *bulk density (ρbulk) and porosity of the sample Sm0.5Ca0.5MnO3 sintered at 1100°C, 1150°C and 1200°C*

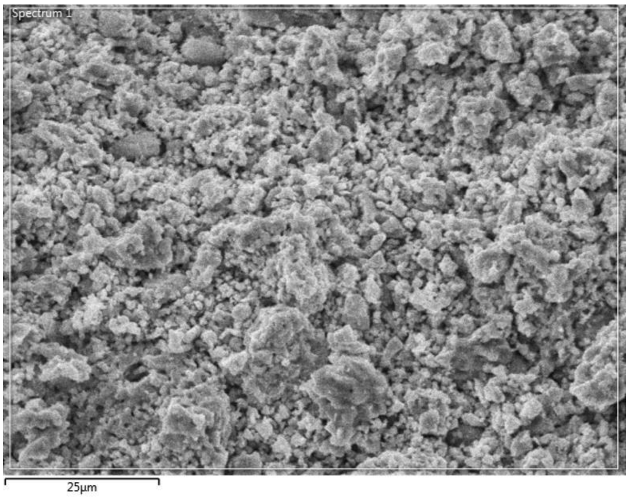
Sintering temperature, °C	a (Å)	b (Å)	c (Å)	ρ_{bulk}, g/cm^3 (± 0.001)	Porosity, % (± 0.01)	Resistivity Ohm m
1100	5.458	7.720	5.454	4.936	21.89	117.443
1150	5.459	7.705	5.458	5.287	16.43	117.658
1200	5.453	7.708	5.451	6.082	4.03	117.870

Higher sintering temperatures gives more ordered structure and improved crystallinity, which may result in improved magnetic and electrical properties. Excessive sintering temperatures, on the other hand, may result in the formation of secondary phases, which can have a negative impact on the material properties (Teoh, 2022). The peak in the graph shows the compound is ceramic. Ceramics are crystalline due to their highly organized atomic structure, which is structured in a repeating pattern known as a crystal lattice. The atoms in a ceramic substance are densely packed together in a regular and symmetrical pattern.

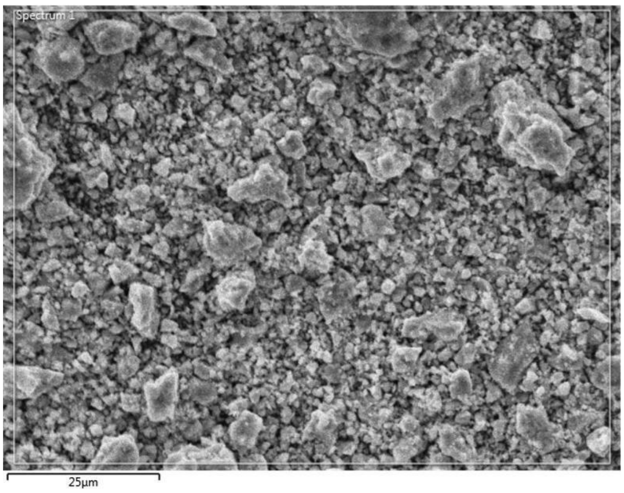
This arrangement produces discrete crystal grains, which gives ceramics their crystalline appearance. Sm0.5Ca0.05MnO3 is an orthorhombicstructure. The orthorhombic unit cell is identified by three lines of double levelness around and this the phone may be turned by 180° not affecting its appearance. The trademark requires the points connecting both edges of the unit cell to be accurate points, even though these edges could vary in any length. The resistivity increases as the sintering temperature increases. The four-point probe analysis shows 117.443, 117.658 and 117.870 for 1100°C, 1150°C and 1200°C respectively [4].



1100°C



1150°C



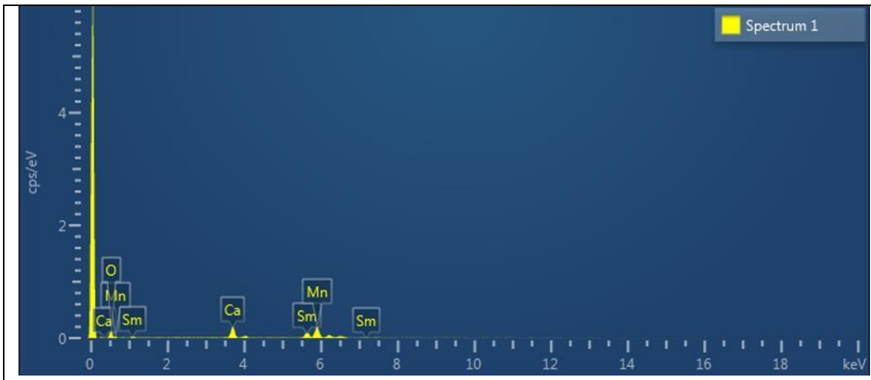
1200°C

Fig. 3 *SEM images of Sm0.5Ca0.5MnO3 for 1100°C, 1150°C and 1200°C with 5kX magnification.*

Enhanced Knowledge in Sciences and Technology Vol. 4 No. 2 (2024) p. 387-393

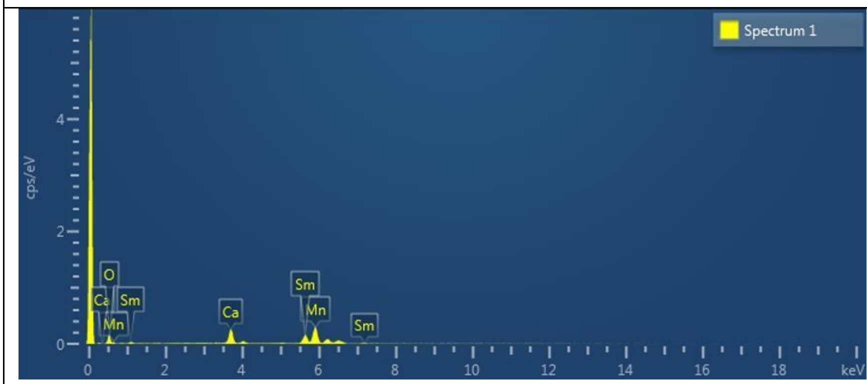
Fig. 3 shows SEM imaging for each specimen at a magnification of 5kX. The SEM pictures indicated that grain size increases as the sintering temperature rises, whereas the boundaries of the samples become compact and linked, achieving a well-defined grain boundary at 1200°C. Indeed, it may be suggested that increasing sintering temperature might enhance particle size and agglomeration because at higher temperatures, grain microstructures are decreased, resulting in tightly packed grains. These properties can be seen in the photos where there are apparent porosities, this decreases as the sintering temperature increases from 1100°C to 1150°C.

The grain agglomerate rate increases as well until no obvious pore exists that can be seen in the sintered sample at 1200°C. These might be explained by grain size rising as temperature rises, as grains try to create larger particles. While the information collected through SEM imaging on the number of pores in the sample corresponds to the porosity determined by XRD analysis according to the bulk density and lattice parameter, it might be assumed that the XRD analysis corresponds with the SEM imaging, as both results show that porosity decreases as the sintering temperature rises. This suggested a link between the two analytic approaches, which showed a high level of the agreement.



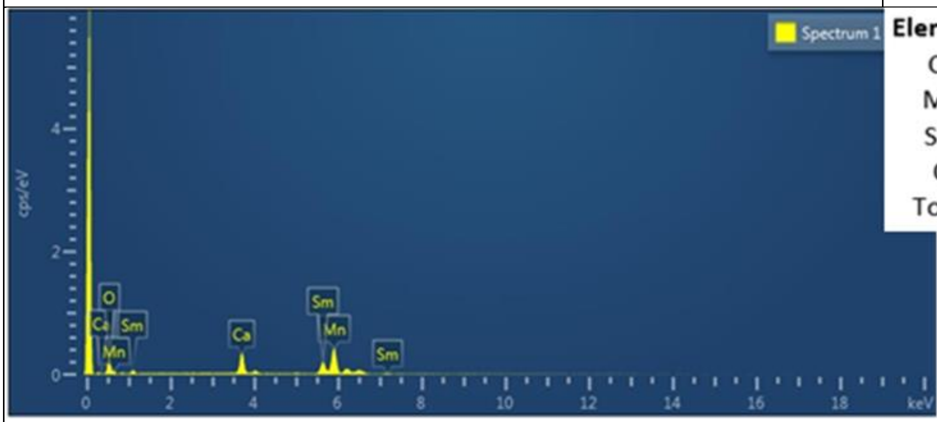
1100°C

Element	Weight %	Atomic %
Ca	13.34	12.01
O	27.29	61.56
Mn	29.20	19.18
Sm	30.18	7.25
Total	100.00	100.00



1150°C

Element	Weight %	Atomic %
Mn	31.53	23.52
Ca	11.66	11.92
Sm	35.38	9.64
O	21.44	54.92
Total	100.00	100.00



Element	Weight %	Atomic %
Ca	10.82	9.70
Mn	31.04	20.30
Sm	30.17	7.21
O	27.96	62.79
Total	100.00	100.00

1200°C

Fig. 4 The analysis spectrum EDX

Fig. 4 depicts the analysis spectrum EDX for each sample. Based on the spectra, all the constituent components (Mn, Ca, Sm, O) were confirmed to be free of impurities, indicating that the sample was properly synthetized. The samples were undergoing three different sintering temperatures are estimated as per to the stoichiometry proportion calculated during sample preparation based on the elemental composition weight percentage. when a result, when the sintering temperature rises, the atomic weight of Sm increases [3]. The other studies have been done in material physics and ceramic industry [5-11].

6. Conclusion

In general, increased sintering temperature improves the crystalline structure and surface appearance of the manganite complex Sm0.5Ca0.5MnO3. Despite a change in sintering temperature having no influence on the crystalline structure, it has previously been seen to modify the specimens' density, grain size, porosity, and resistivity. The material's constituent makeup demonstrates that altering the sintering temperature has no influence on the sample. The influence of sintering temperature has offered some insight on the structural as well as surface modifications to the sample manganite compound during this inquiry. To acquire a better knowledge of the influence of sintering temperature on manganite, more inquiry and characterization, such as magnetic characteristics and ultrasonic anomaly characterization, are advised.

Acknowledgement

The author would thank to Ceramic and Amorphous Group, Faculty of Applied Sciences and Technology, Pagoh Higher Education Hub, Universiti Tun Hussein Onn Malaysia, 84600 Panchor, Johor for the provided facilities. A special thanks supervisor and to all the lab assistance that support this project.

Conflict of Interest

There is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design, data collection, methodology, analysis and interpretation of results:*** *Viknesvaran Ravinthiran and Suhadir Shamsuddin. All authors reviewed the results and approved the final version of the manuscript.*

References

[1] Yan, F., Zhang, X., Yang, C., Hu, B., Qian, W., & Song, Z. (2023). Data ‐ driven modelling methods in sintering process: Current research status and perspectives. The Canadian Journal of Chemical Engineering, 101(8), 4506-4522.

[2] Neves, G. O., Araya, N., de Mello, J. D. B., Binder, C., Klein, A. N., & Aguilar, C. (2022). Synthesis of nanostructured carbon derived from the solid-state reaction between iron and boron carbide. Materials Chemistry and Physics, 276, 125396.

[3] Teoh, W., & Shamsuddin, S. (2022). Effect of Sintering Temperature on Structural and Surface Morphology of Manganite: Pr0. 75Na0. 25-xAgxMnO3. Enhanced Knowledge in Sciences and Technology, 2(2), 052-059.

[4] Razaq, D. S., Kurniawan, B., & Imaduddin, A. (2019, December). Influence of sintering temperature on the structure and electrical transport properties of La0. 7Ba0. 1Sr0. 2Mn0. 85Cu0. 15O3 manganites. In Journal of Physics: Conference Series (Vol. 1402, No. 6, p. 066013). IOP Publishing

[5] Karanam, S. A. K., Rathinam, R., Mouria, P. K., Alguno, A. C., Capangpangan, R. Y., Achamyeleh, T., ... & Livingston, T. S. (2023). Optimizing the Parameters of Spark Plasma Sintering to Enhance the Hardness of MgO/TiC Composites. Advances in Materials Science and Engineering, 2023.

[6] Gu, J., Zhong, H., Chen, Z., Shi, J., Gong, Z., & Yang, Y. (2023). Advances in sulfide-based all-solid-state lithium-sulfur battery: Materials, composite electrodes and electrochemo-mechanical effects. Chemical Engineering Journal, 454, 139923.

[7] Lucero, M., Qiu, S., & Feng, Z. (2021). In situ characterizations of solid–solid interfaces in solid ‐ state batteries using synchrotron X ‐ ray techniques. Carbon Energy, 3(5), 762-783.

[8] Banerjee, A., Wang, X., Fang, C., Wu, E. A., & Meng, Y. S. (2020). Interfaces and interphases in all-solid- state batteries with inorganic solid electrolytes. Chemical reviews, 120(14), 6878-6933.

[9] Mendive-Tapia, E., Paudyal, D., Petit, L., & Staunton, J. B. (2020). First-order ferromagnetic transitions of lanthanide local moments in divalent compounds: An itinerant electron positive feedback mechanism and Fermi surface topological change. Physical Review B, 101(17), 174437.

[10] Surekha, G., Krishnaiah, K. V., Ravi, N., & Suvarna, R. P. (2020, March). FTIR, Raman and XRD analysis of graphene oxide films prepared by modified Hummers method. In Journal of Physics: Conference Series (Vol. 1495, No. 1, p. 012012). IOP Publishing.

[11] Marangoni, T. A., Guralnik, B., Borup, K. A., Hansen, O., & Petersen, D. H. (2021). Determination of the temperature coefficient of resistance from micro four-point probe measurements. Journal of Applied Physics, 129(16).