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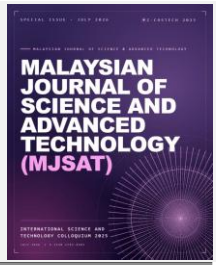

A Study on the Density and Hardness of Y^{3+}-Doped $Ba(Ce,Zr)O_3$ Solid Electrolyte

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ABSTRACT

The Y^{3+}-doped $Ba(Ce,Zr)O_3$ type oxide is a proton-conducting ceramic electrolyte that has been widely studied for application in proton ceramic fuel cells (PCFCs), separation membranes, and other electrochemical devices due to its high proton conductivity, chemical stability, and mechanical durability. The mechanical and physical properties, particularly density and hardness, are crucial to ensuring the performance and durability of the PCFCs. Hence, this study is performed to investigate the density and hardness of BCZY pellets after being sintered at T= 1450 ºC. The two methods, Archimedes and geometrical, were employed to determine the density of the sintered BCZY pellets, while the Vickers Hardness method was used to assess mechanical strength. The results showed relative densities of 90.7% for Archimedes' method and 91.1% for the geometrical method, while the measured Vickers hardness was 508.3 HV (4.99 GPa). These findings show that high-density pellets exhibited acceptable mechanical strength, required for robust electrochemical devices.

1. INTRODUCTION

With the rapid increase in fossil fuel consumption, the development of efficient energy transformation technologies is an urgent task. As the standard fossil fuel-based technology is unable to satisfy the growing demand for energy, fuel cells and hydrogen energy are two examples of efficiency and ecologically beneficial technologies that are used to generate power in the future [1]. As effective, sustainable, and clean energy conversion technologies, solid oxide fuel cells (SOFCs) have attracted a lot of interest [2]. However, its widespread application is hindered by its high operating temperature from 700 to 1000ºC [3]. Hence, proton ceramic fuel cells (PCFC) were proposed as a possible way to lower operating temperatures between 400 to 700ºC due to the higher mobility of proton ions than oxygen ions [4]. Another significant and interesting characteristic of PCFC is that, unlike oxide ion conducting SOFC, PCFC do not experience fuel dilution as water develops on the cathode side.

The PCFC can sustain a greater operating voltage since the anode side fuel dilution does not reduce the Nernst potential, especially for high fuel utilization. A PCFC is one of the SOFC types that use a proton-conducting ceramic as an electrolyte [5].

PCFC components consist of anode, cathode, and electrolyte, where Yttrium-doped Barium Cerium-Zirconium Oxide (BCZY) is one the material in PCFC that has been widely studied as it is the electrolyte that allows protons to pass from the anode to cathode [6]. In contrast of other materials, the BCZY electrolytes exhibit good chemical stability in both H_2O and CO_2 environments, as well as outstanding protonic conductivity at intermediate temperatures, which are often problematic for other materials [7]. Although majority of studies has focused on the densification behaviour and mechanical stability of BCZY electrolytes, there is still lack of knowledge regarding the full correlation between BCZY's microstructure and its mechanical properties which have remained underexplored. Research has primarily focused on the ionic conductivity and phase stability of BCZY. However, its mechanical properties, such as hardness, structural integrity, and resistance to thermal stresses, have not been studied as thoroughly. Despite major studies have investigated densification and microstructure, there is still a lack of consistency regarding the effects of sintering conditions, dopant levels, and sample preparation on mechanical stability [8]. Inconsistencies often arise due to different methodologies and experimental conditions, which highlight the need for more standardized testing to fully understand the material's mechanical properties.

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One of major factor affecting the operational lifespan is the mechanical stability of the proton-conducting electrolyte. While electrochemical properties frequently prioritized, it is important for ceramic membranes to withstand significant thermal and mechanical during high temperature operation. According to current research, high mechanical robustness is essential to prevent micro-cracking and gas leakage. However, detailed studies on the mechanical properties of specific Y^{3+}-doped composition remain limited [9]. The densification of BCZY ceramics is affected by some factor like sintering temperature, atmosphere and the dwell time. Achieving high density in BCZY ceramics often requires high sintering temperatures, which can lead to grain growth, barium evaporation, and phase instability that cause low protonic conductivity, loss of chemical and compromised gas tightness which ultimately degrades the overall electrochemical efficiency of the BCZY electrolyte [10].

These properties are crucial for ensuring the mechanical stability and electrochemical performance of proton-conducting ceramic fuel cells (PCFCs). Achieving high density in BCZY ceramics is essential for minimizing porosity, which improves proton conductivity. Meanwhile, hardness is crucial for structural robustness ensuring the electrolyte's resistance to mechanical failure under thermal cycling and high operational stresses. In this study, BCZY samples were synthesized using a sol-gel method and followed by a two-step sintering process. The primary objective was to evaluate the physical and mechanical properties, specifically the relative density and hardness of the sintered sample and understand the correlation between density, hardness, and sintering conditions. The results of this study will help in improving the electrolyte's reliability and longevity, contributing to the development of more durable and efficient fuel cell systems.

2. METHODOLOGY

2.1 Sample preparations

First, to prepare the samples, stoichiometric amounts of individual metal nitrate salts of barium nitrate, $Ba(NO_3)_2$, cerium(III)nitrate hexahydrate, $Ce(NO_3)_3{\cdot}6H_2O$, zirconium(IV) nitrate hydrate ($Zr(NO_3)_4{\cdot}H_2O$), and yttrium(III) nitrate hydrate ($Y(NO_3)_3{\cdot}H_2O$) were accurately weighed using an analytical balance and were dissolved in 150 mL of deionized water under continuous stirring until a clear homogeneous solution was obtained. Subsequently, the molar ratio of EDTA: citric acid: total metal cations was strictly maintained at 1:2:1 added as chelating agents, followed by the gradual addition of $C_2H_6O_2$, 99.97% ACROS. The pH was adjusted until it reached a pH of 7.0 ± 0.5 by using ammonia hydroxide. Then, this precursor underwent homogeneous heating within the temperature range from 120° to 325°C to reduce the water content and eliminate the hazardous substances. The viscous gel was then continually heated at 325°C in the chamber furnace Carbolite™ at a heating or cooling rate of 10°C min^{-1}. The heating process was done to induce evaporation that resulted in the formation of flakes [11]. The flakes were ground to fine powder, followed by calcination at 1100°C for 10h at a heating or cooling rate of 10°C min^{-1} to obtain green BCZY powder.

2.2 Fabrication of BCZY pellet

The calcined electrolyte powders were dried at 100°C in an oven overnight before the dry-pressing process. This step was taken to eliminate moisture and guarantee no cracks at the time of pellet fabrication. The 2g dried electrolyte powder was compacted in a die mould of 25 mm diameter at a pressure of 9 tons for 7 min using a hydraulic press. The same composition of powder was placed in the alumina boat to cover the green pellet before sintering to prevent delamination, cracks, and reduce barium evaporation. The obtained green pellet (with ~1 mm thickness) was sintered through a two-step sintering process, as shown in Figure 1.

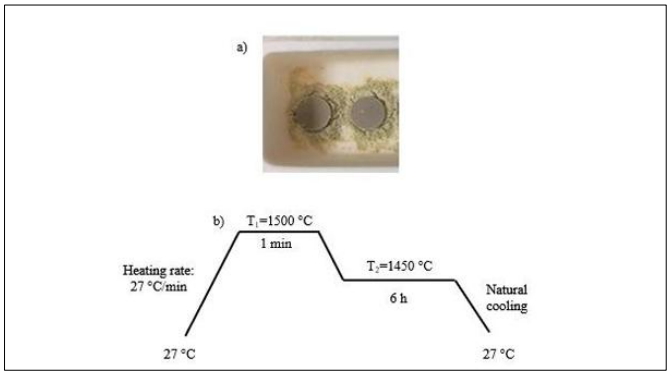

Fig 1. (a) A 25mm BCZY Pellet (b) Two-step Heating Profile to Sinter the Pellet

2.3 Density measurement

The density of the sintered BCZY pellets was determined using two complementary approaches that are Archimedes and the geometrical method. Employing both methods allowed cross-validation of results, as each method has different factors that influence the final density value, especially in terms of how the methods account for the sample's microstructure and porosity.

In the Archimedes method, the buoyancy was applied by weighing the pellets in air (W_{air}) and when submerged in water (W_{water}). The volume of the displaced liquid (V) was determined from the buoyant force, and the experimental density was calculated using Equation (1).

$$\rho = \frac{W_{air}}{W_{air} - W_{water}} \times \rho_{water} \quad (1)$$

This method is highly sensitive to open porosity, as water can penetrate pores, leading to deviations in measured density.

In parallel, the geometrical method, density was obtained by measuring the mass (m) and the geometric dimensions of the cylindrical pellet. The bulk density can be calculated using Equation (2).

$$\rho = \frac{m}{V} \quad (2)$$

This method assumes an ideal solid structure, thus giving the apparent density. Although less sensitive to open porosity compared to Archimedes, dimensional errors during measurement may influence accuracy.

2.4 Vickers Hardness measurement

The hardness of the sintered BCZY pellets was evaluated using a Vickers microhardness tester. A load of 49.03 N which is equivalent to approximately to 5.0 kgf was used with a dwell time of 10 seconds was applied using a VMT-X hardness tester. The polished pellet surfaces were indented with a diamond pyramid indenter under an applied load of 5 kgf and a dwell time of 10 s. For each sample, multiple indents were made at different regions to ensure reproducibility and to minimize the effect of surface defects. Figure 2 shows the indentation mark under the optical microscope of the Vickers hardness test. The diagonals of the indentation (d_1 and d_2) were measured under an optical microscope, and the average diagonal (d) was calculated using Equation (3).

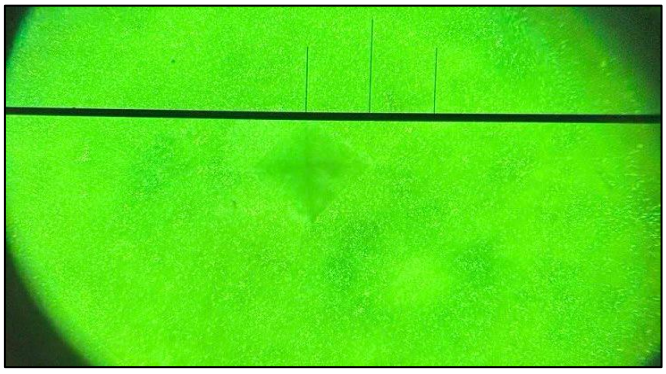

Fig. 2. Indentation Mark Under the Optical Microscope of the Vickers Hardness Test

$$d = \frac{d_1 + d_2}{2} \tag{3}$$

The Vickers hardness value (HV) was then determined using Equation (4).

$$HV = \frac{1.854 \times F}{d^2} \tag{4}$$

3. RESULT AND DISCUSSION

3.1 Density of BCZY pellet

The theoretical density of 6.11 g/cm^3 was calculated using the crystallographic density formula, incorporating a lattice parameter of 4.344 Å as reported by Mazlan et al., to calculate the unit cell volume [12]. The densities of the prepared sample obtained by both methods, as shown in Table 1, demonstrate close agreement, with differences between the two methods generally being less than 2%. This suggests that both methods are consistent in their measurements, but each method has different, distinct factors that influence the final density value, especially in terms of the sample's microstructure and porosity.

Table 1. The Summary of the Density Parameter for BCZY Pellets

Method	Measured density (g/cm^3)	Theoretical density (g/cm^3)	Relative density (%)	Porosity (%)
Archimedes	5.59±0.05	6.11	91.5	8.5
Geometrical	5.61±0.05	6.11	91.8	8.2

The BCZY pellet attains relative densities of approximately 91.5% for the Archimedes method and 91.8% for the geometrical method. As shown, the relative density achieved in this work (91.5% to 91.8%) is comparable to the result reported by Yu et al., who achieved approximately 92% density [13]. Adequate densification of the BCZY electrolyte following the sintering process at 1450 ℃ for 6 hours was achieved in this study, though further densification may be required for ensuring gas-tight electrolyte applications, as more compact structures are essential for long-term operational stability.

Achieving a high relative density, ideally above 90% is important for electrolyte pellets as it represents an excellent electrolyte application for electrochemical characterization and ensures sufficient proton mobility [14]. A high relative density enhances both gas-tightness and the continuity of grain boundaries, which are vital for maintaining the structural integrity and electrochemical performance of the material. While densification does not directly increase proton mobility, it helps enhance the ion transport as it facilitates better ion transport through improved microstructural uniformity and reduced porosity which facilitates the movement of ions through the lattice and result in higher proton conductivity [15]. The observed relative densities being higher than the 90% threshold propose that the sintering conditions used were successful in producing a relatively dense microstructure, which is beneficial for proton transport within the BCZY electrolyte.

The relative density obtained from the Archimedes and geometrical method show a slight difference which were 91.5% and 91.8% respectively. This small variation can be attributed to the fundamental differences in the two techniques. The geometrical method that relies only on the physical fully solid pellet tends to give slightly higher relative density values compared to the Archimedes method as it does not account for any internal or external porosity that may exist within the material [16]. As a result, the geometrical method produces a density value based on the assumption of an ideal, solid structure. In contrast, according to the study by Hall & Hamilton, Archimedes' method is influenced by the presence of open porosity [17]. This technique calculates the volume of displaced liquid when the sample is submerged, the volume calculation is based on the amount of fluid displaced by the sample. Accessible pores in the pellet allow the displaced liquid to enter these pores, resulting in a volume measurement that reflects both the solid material and the voids [18]. Therefore, the Archimedes method yield a lower density value compared to the geometrical method if the sample contains open pores or porosity. In summary, while both methods are effective in measuring density, they are affected by different structural aspects of the sample. The geometrical method tends to overestimate density by neglecting porosity, while the Archimedes method provides a more accurate representation of the sample's true physical structure by considering the existence of open pores.

3.2 Vickers Hardness

Table 2 shows a summary of the Vickers Hardness (HV) Measurement for the BCZY pellet. The value of the measured HV was observed to be 508.3 ± 5.0 kgf/mm^2, which is equivalent to approximately 4.99 ± 0.05 GPa hardness value. This value lies within the expected range for partially dense ceramic electrolytes that vary between 3 to 6 GPa [19]. Other

study by Zuo et al., also shown that, the measured Vickers hardness of 508.3 HV approximately 4.99 aligns well with the range of 4.5-5.5 Gpa typically observed for barium cerate-zirconate ceramics [20]. This shows that the BCZY pellets exhibit sufficient mechanical robustness, which is crucial for structural stability in high-temperature electrochemical applications.

Table 2. Summary of Vickers Hardness Measurement for BCZY Pellets

Load (kgf)	Dwell time (s)	D_1 (mm)	D_2 (mm)	HV (kgf/mm^2)	Hardness (GPa)
5.0	10	0.11481	0.15529	508.3 $\pm$ 5.0	4.99 $\pm$ 0.05

High hardness is important for maintaining structural stability under cyclic thermal loading, which is common in fuel cell environments. This prevents deformation of the electrolyte under operational conditions [21]. The measured hardness reflects efficient densification during the sintering process. Higher sintering temperatures and optimized doping levels result in lower porosity and better grain boundary characteristics, which enhance the mechanical strength.

3.3 Correlation between density and hardness

The electrochemical performance of BCZY electrolytes is directly impacted by the combination of high density and hardness of the electrolyte as high density correlates with the measured hardness. Study stated that, denser materials generally exhibit higher hardness due to reduced porosity and improved grain-to-grain bonding [22]. A dense microstructure reduces porosity, facilitating easier ion movement and enhancing proton conductivity. Simultaneously, higher hardness offers structural stability towards deformation caused by thermal cycling and mechanical stress. This dual benefit is crucial to long-term fuel cell durability.

3.4 Porosity and mechanical stability

Although the relative densities were over 90%, some residual porosity may still exist, as indicated by minor discrepancies between the Archimedes and geometrical methods. Porosity can compromise both ionic conductivity and mechanical strength. Therefore, further optimization of sintering conditions, such as temperature, pressure, and atmosphere, could lead to even higher densities and improved hardness, thus enhancing the overall performance of the electrolyte.

3.5 Experimental error and uncertainties

Several sources of experimental error and uncertainty may have contributed to the discrepancies between the measured densities and the theoretical density value of 6.11 g/cm^3 for the BCZY electrolyte. Both the Archimedes and geometrical methods are prone to inherent measurement errors that can significantly affect the calculation of density. The main cause of error in the Archimedes method is influenced by the accurate determination of the sample. The immersion technique is influenced by the presence of air bubbles or surface roughness, which can lead to an overestimation of the volume of liquid displaced. This is particularly true for the case of porous sample materials where liquid can enter accessible pores, leading to a lower measurement of density. Additionally, any errors in the calibration of the liquid's density, especially if the temperature was not accurately controlled, could have resulted in uncertainties in the volume measurement.

Meanwhile, the accuracy of geometric measurements such as diameter and thickness is very important for the geometrical method. Even small errors in measuring the sample dimensions can result in significant changes in the calculated volume and hence the density. Moreover, this method assumes that the sample has a perfectly regular shape, which is not always true for materials that have negligible surface defects or irregularities. These errors can be classified into measurement errors, material-related uncertainties, and experimental technique limitations. Understanding these factors is crucial for interpreting the results and proposing changes in future studies.

4. CONCLUSIONS

In this study, complementary experimental methods were successfully used to evaluate the density and hardness of BCZY pellets. The small differences between the two density measurement methods show the impact of porosity and sample geometry, emphasizing the significance of selecting appropriate methods based on the microstructural properties of the material. The Vickers Hardness test revealed a hardness value that lies within the expected range for partially dense ceramic electrolytes and confirming its suitability for mechanical stability under operational stress. However, the correlation between density and hardness in this study is inferred and not experimentally demonstrated as only one sintering condition was examined and limiting the scope regarding to direct relationship between these two properties. Future studies should consider varying sintering parameters to further investigate this relationship and validate the findings under different conditions. Additionally, the relationship between density and hardness shows that the densification could be improved to further enhance mechanical strength, which in turn contributes to the longevity and reliability of BCZY-based protonic ceramic fuel cells. Overall, this study contributes to a better understanding of how processing parameters such as sintering conditions affect critical material properties like density and hardness, which are important for optimizing the performance of BCZY electrolytes in advanced energy systems.

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