



Characterizations of Synthetic 8mol% YSZ with Comparison to 3mol %YSZ for HT-SOFC

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ABSTRACT

In this work, Yttria (Y₂O₃) was successfully doped into tetragonal 3mol% yttria stabilized Zirconia (3YSZ) by high energy-mechanical milling to synthesize 8mol% yttria stabilized Zirconia (8YSZ) used as an electrolyte for high temperature solid oxide fuel cells (HT-SOFC). This work aims to evaluate the densification and ionic conductivity of the sintered electrolytes at 1650°C. The bulk density was measured according to ASTM C373-17. The powder morphology and the microstructure of the sintered electrolytes were analyzed via Field Emission Scanning Electron Microscopy (FESEM). The chemical analysis was obtained with Energy-dispersive X-ray spectroscopy (EDS). Also, X-ray diffraction (XRD) was used to obtain structural information of the starting materials and the sintered electrolytes. The ionic conductivity was obtained through electrochemical impedance spectroscopy (EIS) in the air as a function of temperatures at a frequency range of 100(mHz)-100(kHz). It is found that the 3YSZ has a higher density than the 8YSZ. The impedance analysis showed that the ionic conductivity of the prepared 8YSZ at 800°C is 0.906 (S.cm) and it was 0.214(S.cm) of the 3YSZ. Besides, 8YSZ has a lower activation energy 0.774(eV) than that of the 3YSZ 0.901(eV). Thus, the prepared 8YSZ can be nominated as an electrolyte for the HT-SOFC.

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1. Introduction

There are three Zirconia polymorphisms: monoclinic (m), tetragonal (t), and cubic (c) as shown in the scheme Figure 1. On cooling, ZrO₂ undergoes a martensitic transformation c → t accompanied with volume changes of approximately 2.31% and t → m transformation with the volume change of 4.5%. Thus, the sintering of pure Zirconia ceramic is not possible [1].

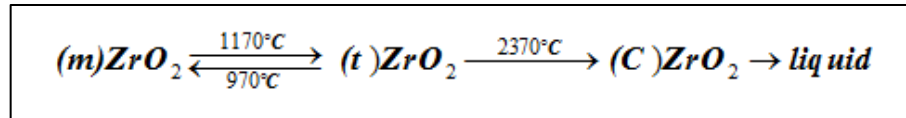


Figure 1: Scheme of zirconia phase transformation after [2]

Therefore, it is necessary to use stabilization agents for ZrO₂ like MgO, CaO, Y₂O₃, or other rare earth oxides [3]. In this work, yttria (Y₂O₃) is used for the synthesis of 8mol% yttria stabilized Zirconia (8YSZ) due to its known excellent oxide ion conductivity above 800°C, chemical and thermodynamic stability, and mechanical strength [4]. The ionic conductivity of the stabilized Zirconia qualifies it as an electrolyte for the synthesis of SOFC as an alternative and clean energy source [5]. Thus, SOFC responds to growing energy demands and sustainability [6]. Accordingly, the electrochemical energy converters that transform chemical energy into heat and electricity, known as fuel cells (FCs), which represent promising candidates for clean energy supply [7]. FCs are characterized by high efficiency and clean energy generating technologies with high performance which doesn't depend on system size, fuel flexibility. They also have a low cost of maintenance due to the absence of moving parts in the stacks. It can be used in portable power, transportation, and stationary power greater power [8]. In general, FCs can be classified according to its shape, type and operation temperatures. Amongst these different types of FCs, Solid Oxide Fuel Cell (SOFC) is a premium technology in this field due to inexpensive materials, high efficiency, with fuel flexibility [9]. SOFC dictated by their operating temperature, which classified into High-temperature Solid Oxide Fuel Cell (HT-SOFC) operated between 700-1000°C [10]. Intermediate temperature Solid Oxide Fuel Cell (ITSOFC) operated between 500-700°C [11,12,13,14], and Low temperature Solid Oxide Fuel Cell (LT-SOFC) operated below 600°C [15]. The objective of electrolyte is to achieve the major roles to give ions transportation from cathode to anode and to prevent gas diffusion or leakage from both cathode and anode side [16, 17]. A typical SOFC consists of three essential parts: anode, cathode, and electrolyte [18]. A few attempts have been done to prepare 8YSZ by the solid-state reaction method. Budiana et al. successfully prepared 8YSZ and measured its ionic conductivity to get activation energy 0.96 eV and relative density of 96.70% [19]. Tong et al. characterized the submicron-grain size of YSZ electrolyte for SOFC application. It obtained the greatest variation between the 3 mol% YSZ and 8 mol% YSZ. Thus, the conductivity varied by ~65% at 850 °C [20]. The utilized 8YSZ in the early works of SOFC was in the micro-range. When nanotechnology emerged, 3YSZ became popular and, thus, utilized for SOFC. In this work, a nano-sized 8YSZ was prepared and examined for the requisite of HT-SOFC electrolyte to study their electrical characterizations and physical properties to achieve better ionic conductivity.

2. Experimental Work

Commercially available starting powders of Y₂O₃ with purity (99.995) % supplied by Sky Spring Nanomaterials, USA, the 3mol% YSZ nanopowders supplied by Hongwu International Group Ltd., China, and stearic acid supplied by Merck, Germany used as starting materials to synthesize 8mol% yttria stabilized Zirconia (8YSZ) powder used to produce (ZrO₂)_{0.92}-(Y₂O₃)_{0.08} electrolyte. Also, another type was of electrolytes (ZrO₂)_{0.97}-(Y₂O₃)_{0.03} (3YSZ) was manufactured. The starting powders of electrolyte type (8YSZ) produced by high energy-mechanical milling method via planetary milling type Retsch PM400 with Zirconia balls in different diameters range between Ø (30-10) mm were used in the ratio of ball to powders 10:1 with 2wt% additives of stearic acid as the process control agent. The milling process achieved after (30) h with speed (300) RPM. The particle size investigated via scanning electron microscope (SEM) type TESCAN VegaIII Czech Republic with 5 KV. Moreover, the sintered electrolyte investigated by field emission scanning electron microscope (FESEM) type TESCAN VegaIII Czech Republic with 5 KV due to its better z-field, thus, the focus is better.

The HT-SOFC electrolyte compacted using the cold press method pressed at 80 MPa to get pellets with a diameter of 30 mm and 10 mm thickness. After this, the electrolytes sintered at 1650°C with 3h soaking time using box furnace type Retsch in the range of previous work [21]. The X-ray diffraction (XRD) type Philips analytical diffraction model PW1930 with cobalt $\lambda\alpha_1=1.78901 \text{ \AA}$ radiant tube operating at 40 kV and 30 MA. It used to identify the sintered electrolyte. The physical

properties comprised of bulk density and porosity according to ASTM (C373-17) and relative density (RD) measured according to Archimedes' method [22].

The ionic conductivity of the sintered electrolyte measured after the heat treatment of silver paste (Ag) as collect current at both sides of electrolyte. Thus, the electrical properties evaluate with electrochemical impedance spectroscopy (EIS) at a frequency range from 100mHz to 100KHz as shown in Figure 2.

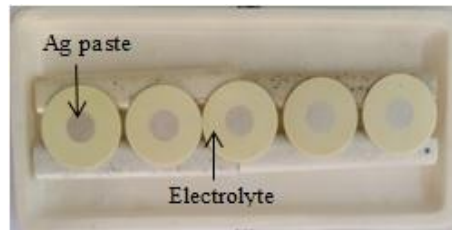


Figure 2: Sintered electrolyte for EIS measurement

3. Results and Discussion

The average particle size of sintered pellets displayed about 100 nm for 3mol% YSZ powder as shown in Figure 3 while the particle size of 8mol% YSZ was about 120 nm as seen in Figure 4.

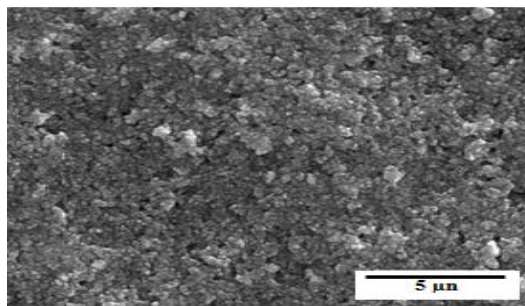


Figure 3: SEM of 3mol% YSZ powders

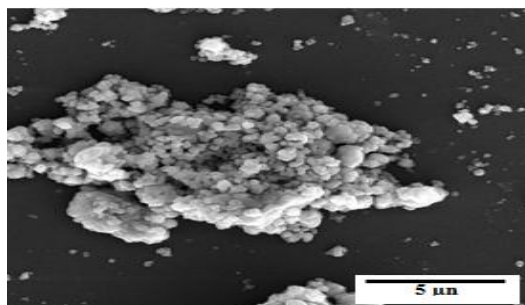


Figure 4: SEM of 8mol% YSZ powders

Figure 5 obtained FESEM of sintered 3mol% YSZ electrolyte. Thus, the 3mol% YSZ electrolyte obtained grain size of 978 nm with some grain growth in heterogeneous grain shape. While the sintered 8mol% YSZ electrolyte offered homogenous grain shape with clear grain boundaries. Also, the 8YSZ electrolyte gave a grain size of 13568 nm (Figure 6). The weight% of the powders obtained from EDS in Table 1.

Table 1: Element Wt% with Spectra of EDX of powders of raw materials

Element (Wt%)	3mol% YSZ	8mol% YSZ
Zr	32.78	29.03
O	65.20	65.91
Y	2.02	5.06

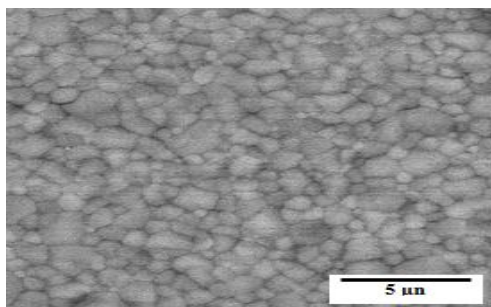


Figure 5: FESEM of sintered 3mol%YSZ electrolyte

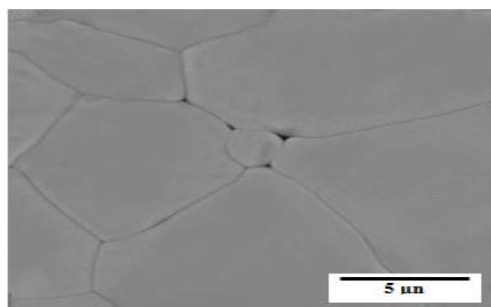


Figure 6: FESEM of sintered 8mol%YSZ electrolyte

The XRD of 3mol%YSZ and 8mol%YSZ powders are shown in Figures 7 and 8 respectively.

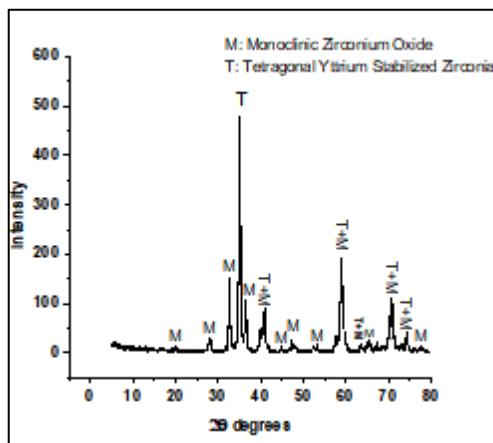


Figure 7: XRD pattern of 3YSZ powders

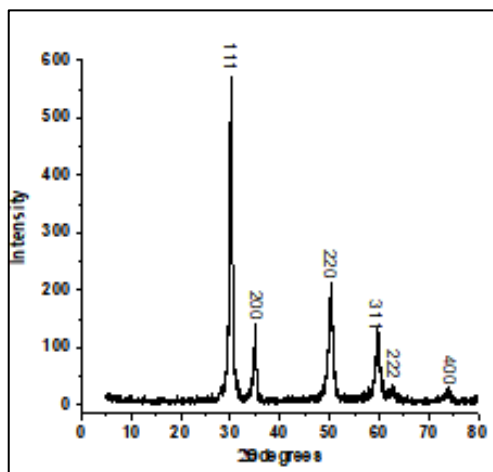


Figure 8: XRD pattern of 8mol%YSZ powders

It can be seen from XRD results in Figure 7 that the powder of 3mol%YSZ consisted of monoclinic Zirconia oxide and tetragonal yttria stabilized zirconia phases. It can be indicated from XRD results that all peaks of synthesized 8mol%YSZ powders in Figure 8 obtained cubic stabilized zirconia phases. The XRD patterns of both types of electrolytes sintered at 1650°C were shown in Figure 9 and Figure 10.

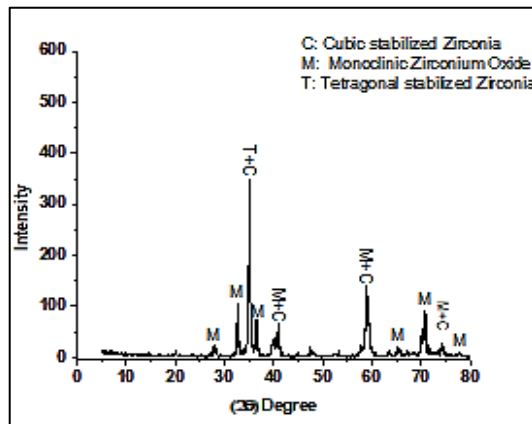


Figure 9: XRD pattern of sintered 3YSZ electrolyte

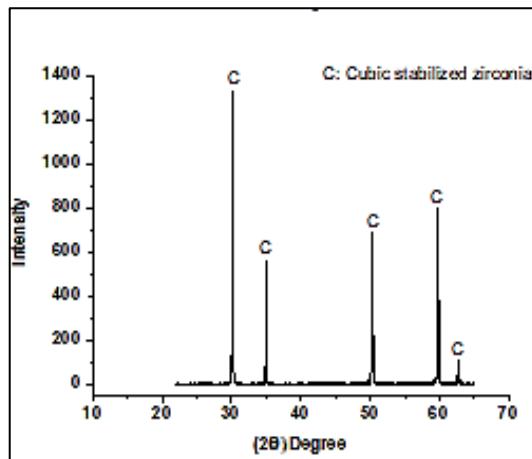


Figure 10: XRD pattern of sintered 8YSZ electrolyte

From Figure 9, it can be seen that the main phase of the sintered 3YSZ electrolyte was the cubic and tetragonal Zirconia phase. Moreover, the minor phase was tetragonal Zirconia with monoclinic Zirconia oxide and a little portion of monoclinic Zirconia oxide. In Figure 10, it can be noticed that there is a single phase of cubic stabilized Zirconia from the XRD plot 8YSZ.

The physical properties of sintered electrolytes calculated according to ASTM C373-17 and are listed in Table 20. As illustrated, the density of 3YSZ is higher than 8YSZ due to the lower porosity (2.46%) than 8YSZ sintered electrolyte (3.85)%. The presence of larger grains with less porosity% will increase the total ionic conductivity in the electrolyte [23]. RD% of the sintered electrolyte was calculated by the Archimedes method [24]. The relative density considered as an essential parameter of linear shrinkage and a degree of electrolyte densification. Moreover, it's an essential function of the gas-tight of electrolyte [25,26].

Table 2: Physical characterizations of sintered 3YSZ and 8YSZ electrolytes

Y ₂ O ₃ (mol%)	3mol% YSZ (3YSZ)	8mol% YSZ (8YSZ)
Density (g/cm ³)	5.917	5.521
Porosity (%)	3	2
Relative density (RD)%	97	98

The electrical properties of two types of electrolyte pellets measured with electrochemical impedance spectroscopy (EIS) type potentiostat model parstat 2273 made in the USA are shown in Figure 11. The electrolytes tested at different temperatures ranging from (600,650, 700, 750, 800) °C. The impedance plots (Z''/Z') with Nyquist standard plot analysis of (EIS) obtained bulk conductivity data from the low-frequency intercept of the high-frequency arc semicircle on the real Z' axis [27]. It can be seen from Figure 11 that there is a lite distortion in semicircle arc with low frequency and lower temperature at 600 °C with a clear semicircle at high temperature 800°C. While in Figure 13, it can be seen from impedance plots of 8YSZ electrolytes that there is a clear semicircle arc at the high and low used frequency with all testing temperatures. From Figures 12 and 14, the activation energy calculated for 3YSZ and 8YSZ electrolytes. It saw an increase of ionic conductivity with increasing temperatures as a result of raising the number of ions charger causing higher mobility of the ions [28,29].

The sintered electrolyte pellets from synthesis 8mol%YSZ powders revealed activation energy 0.774(eV) less than 3YSZ electrolytes with ohmic resistance to be about 1.10($\Omega\cdot\text{cm}^2$). The lower activation energy is the higher conductivity and vice versa [30]. The electrical properties reduced as a result of the appearance of monoclinic phases [31].

From all frequency and testing temperatures of 8YSZ electrolytes, a clear semicircular arc with the Nyquist plot without distortion can be observed.

It is seen from Figure 14 that ionic conductivity with 8 YSZ electrolytes is higher than 3YSZ electrolyte shown in Figure 12.

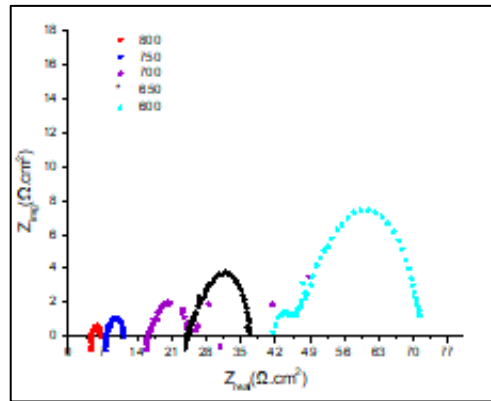


Figure 11: Impedance plots of 3YSZ electrolyte pellet measured at (800,750, 700, 650, 600) °C

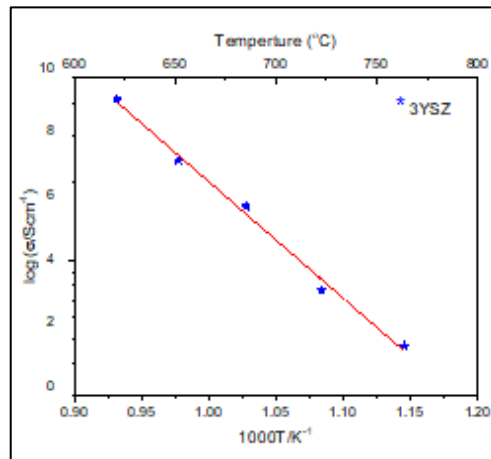


Figure 12: Arrhenius plots of 3YSZ electrolyte pellet measured at (800) °C

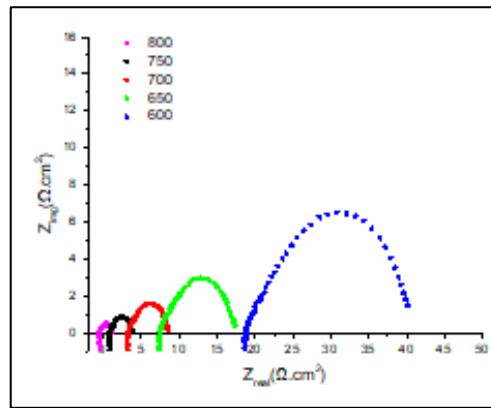


Figure 13: Impedance plots of 8YSZ electrolyte pellet measured at (800,750, 700, 650, 600) °C

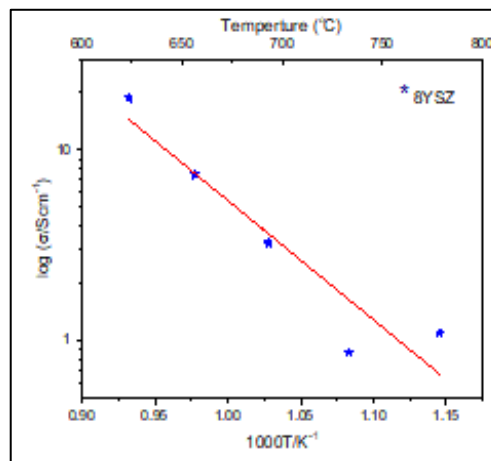


Figure 14: Arrhenius plots of 3YSZ electrolyte pellet measured at (800) °C

The results of electrical characterization for 3YSZ and 8YSZ electrolytes are listed in Table 3. From the impedance spectroscopic, the conductivity with Arrhenius plots can be calculated according to Eq. (1) below:

$$\ln(\sigma T) = \ln \sigma_0 + \frac{-Ea}{RT} \tag{1}$$

Where σ : ionic conductivity, Ea : activation energy, R : gas constant [32].

Table3: Electrical characterization of sintered 3YSZ and 8YSZ

Specimen code	Temperature ° C				
	3YSZ	600	650	700	750
Ohmic Resistance (Ω.cm²)	41.5	24.	161	7.6	4.6
	2	2		7	8
Ionic conductivity (S/cm)	0.02	0.0	0.0	0.1	0.2
		4	6	3	1
Activation energy(ev)	0.901				
8YSZ	Temperature ° C				
	600	650	700	750	800
Ohmic Resistance (Ω.cm²)	18.7	7.4	3.2	0.8	1.1
	7	5	5	7	0
Ionic Conductivity (S/cm)	0.05	0.1	0.3	1.1	0.9
		3	1	5	1
Activation energy(ev)	0.774				

4. Conclusion

By comparing the results of the electrical conductivity between 3mol%YSZ and 8mol%YSZ, it was found that there are increases in the electrical conductivity with increasing temperatures Also, 8YSZ and 3YSZ electrolytes obtained gain size 13568 nm and 978 nm in respectively. Thus, the sintered

8YSZ gave 0.906 (S/cm) at 800°C on the other hand, the sintered 3YSZ electrolyte gave a lower ionic conductivity about 0.214 (S/cm) at 800°C. This is due to the appearance of the secondary phases of tetragonal Zirconia (T+M) with monoclinic Zirconia oxide and monoclinic Zirconia oxide (M) with 3YSZ electrolyte. The cubic stabilized Zirconia phase obtained in XRD results with 8YSZ electrolytes had a higher ionic conductivity at higher temperatures. The (RD)% was higher in 8YSZ electrolyte than in 3YSZ electrolyte related to the higher cubic percentage. Thus, the denser 8YSZ electrolyte showed activation energy 0.90 (ev) which less than other literature with ohmic resistance 4.677 ($\Omega \cdot \text{cm}^2$). From the impedance results, it revealed a higher ionic conductivity 0.91(S/ cm^2) and lower ohmic resistance 1.10 ($\Omega \cdot \text{cm}^2$) than sintered 3YSZ electrolyte at 800°C. This can be due to the lower conductivity of 3YSZ concerning 8YSZ at high temperatures.

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