Novel Composite Electrolytes of Zr$_{0.92}$Y$_{0.08}$O$_2$-$\alpha$(8YSZ)-Low Melting Point Glass Powder for Intermediate Temperature Solid Oxide Fuel Cells

Hongtao Wang *, Ruifeng Du, Ruijuan Shi and Junlong Liu

School of Chemical and Material Engineering, Fuyang Normal College, Anhui Provincial Key Laboratory for Degradation and Monitoring of Pollution of the Environment, Fuyang 236037, China; ruifengdu1@163.com (R.D.); rjshi@fync.edu.cn (R.S.); jlliu@fync.edu.cn (J.L.)

* Correspondence: hongtaoking3@163.com; Tel.: +86-558-2596-249; Fax: +86-558-2596-703

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Abstract: In this study, Zr$_{0.92}$Y$_{0.08}$O$_2$-$\alpha$(8YSZ) powders were synthesized by the sol-gel method. The chemical physics changes and phase formation temperature of 8YSZ crystal were determined by thermogravimetry analysis and differential scanning calorimetry (TGA-DSC). 8YSZ-low melting point glass powder (8YSZ-glass) composite electrolytes with various weight ratios were prepared and calcined at different temperatures. The X-ray diffraction (XRD) patterns of the composite electrolytes were tested. The effects of synthesis temperature, weight ratio, test temperature, and oxygen partial pressure on the conductivities of 8YSZ-glass composite electrolytes, were also investigated at 400–800 $^\circ$C. The result of the log $\sigma$ ~ log($p$O$_2$) plot indicates that the 8YSZ-20% glass (700 $^\circ$C) is almost a pure ionic conductor. The oxygen concentration discharge cell illustrates that the 8YSZ-20% glass (700 $^\circ$C) composite electrolyte is a good oxygen ion conductor.

Keywords: defects; electrolyte; fuel cell; ceramics; sol-gel preparation

1. Introduction

ZrO$_2$-based electrolytes doped, with rare earth metallic cations, are excellent oxide ionic conductors, and they are widely used in oxygen sensors and solid oxide fuel cells (SOFCs) [1–4] due to their good mechanical strength and high ionic transport numbers. During the past decades, continuous research concentrating on Y$_2$O$_3$-stabilized ZrO$_2$ (YSZ) has been done [5,6]. For example, Caruso et al. investigated the influence of different parameters on the morphology and microstructure of YSZ powders synthesized by the sol-gel method [5].

However, SOFCs using YSZ as electrolyte membrane usually run at high temperatures (800–1000 $^\circ$C). Therefore, researchers have focused on two ways to lower the operating temperature of YSZ. One strategy is to use YSZ films, and the other way is to construct composite electrolytes, which may have the combined advantages of each component [7–11]. A few research reports have shown that the thin film fuel cells using YSZ as membrane electrolytes generated maximum power output densities of 200–400 mW·cm$^{-2}$ at 800 $^\circ$C [12–14]. Singh et al. reported that the YSZ-SDC (samarium doped ceria) composite electrolyte with a weight ratio 8.5:1.5 has a higher electrical conductivity than single material YSZ at 400–700 $^\circ$C [9]. It is well known that silicate, borate, mica, and other glass systems are commonly used as sealing materials in fuel cell systems [15–18]. It may be expected that using silicate low melting point glass powder, as a sintering aid, as well as composite electrolytes with improved gas tightness, durability, and better component matching, could be synthesized.
In this study, novel composite electrolytes of $\text{Zr}_{0.92}\text{Y}_{0.08}\text{O}_{2-x}$ (8YSZ)-low melting point glass powder were synthesized. The morphology, structure, and intermediate temperature electrochemical properties of the composite electrolytes were investigated by a variety of methods.

2. Experimental

We initially synthesized $\text{Zr}_{0.92}\text{Y}_{0.08}\text{O}_{2-x}$ (8YSZ) electrolyte via a sol-gel method using citric acid as a chelating agent as reported previously [19]. All the reagents used were analytical-grade. Firstly, $\text{Y}_2\text{O}_3$ was dissolved in nitric acid and $\text{Zr(NO}_3)_4\cdot 5\text{H}_2\text{O}$ was dispersed into distilled water. The solution was then mixed with citric acid and $\text{NH}_4\text{OH}$ and evaporated at 90 $^\circ\text{C}$ to get a gel. After gelation and ashing treatment, the obtained ash was calcined at 700 $^\circ\text{C}$, 1200 $^\circ\text{C}$ and 1550 $^\circ\text{C}$ for 6 h, respectively, to get $\text{Zr}_{0.92}\text{Y}_{0.08}\text{O}_{2-x}$ (8YSZ) powder. The low melting point glass powder was used as a sintering aid to form composite. The composition of the low melting point glass powder is $\text{Na}_2\text{O}-\text{CaO}-\text{SiO}_2-\text{ZnO}$ (Taizhou Xinhai Special Materials Factory, 300 mesh, m.p. is 550 $^\circ\text{C}$). 8YSZ and low melting point glass powder were mixed with a weight ratio of four to one and heated at 700 $^\circ\text{C}$, 1200 $^\circ\text{C}$ and 1550 $^\circ\text{C}$ for 2 h, correspondingly. The composites with weight ratio of 8YSZ: low melting point glass powder = 9:1 and 7:3 were also synthesized at 700 $^\circ\text{C}$. These results are summarized in Table 1.

| Sample Synthesis Temperature Abbreviation |
|-----------------------------------------|-----------------------------------|-----------------|
| $Zr_{0.92}\text{Y}_{0.08}\text{O}_{2-x}$ 10 wt% low melting point glass 700 $^\circ\text{C}$ | 8YSZ-10% glass 700 $^\circ\text{C}$ |
| $Zr_{0.92}\text{Y}_{0.08}\text{O}_{2-x}$ 20 wt% low melting point glass 700 $^\circ\text{C}$ | 8YSZ-20% glass 700 $^\circ\text{C}$ |
| $Zr_{0.92}\text{Y}_{0.08}\text{O}_{2-x}$ 30 wt% low melting point glass 700 $^\circ\text{C}$ | 8YSZ-30% glass 700 $^\circ\text{C}$ |
| $Zr_{0.92}\text{Y}_{0.08}\text{O}_{2-x}$ 20 wt% low melting point glass 1200 $^\circ\text{C}$ | 8YSZ-20% glass 1200 $^\circ\text{C}$ |
| $Zr_{0.92}\text{Y}_{0.08}\text{O}_{2-x}$ 20 wt% low melting point glass 1550 $^\circ\text{C}$ | 8YSZ-20% glass 1550 $^\circ\text{C}$ |

The chemical physics changes and phase formation temperature of 8YSZ crystal were determined by thermogravimetry analysis and differential scanning calorimetry (TGA-DSC) (TGA-DSC, Universal V 3.7A, TA Instruments, New Castle, DE, USA). The X–ray diffraction (XRD) (XRD, X‘pert Pro MPD, Panama Xinhai Special Materials Factory, 300 mesh, m.p. is 550 $^\circ\text{C}$). 8YSZ and low melting point glass powder were mixed with a weight ratio of four to one and heated at 700 $^\circ\text{C}$, 1200 $^\circ\text{C}$ and 1550 $^\circ\text{C}$ for 2 h, correspondingly. The composites with weight ratio of 8YSZ: low melting point glass powder = 9:1 and 7:3 were also synthesized at 700 $^\circ\text{C}$. These results are summarized in Table 1.

The TGA and DSC curves of the $Zr_{0.92}\text{Y}_{0.08}\text{O}_{2-x}$ (8YSZ) gel heated at 15 $^\circ\text{C}\cdot\text{min}^{-1}$ in nitrogen atmosphere up to 1000 $^\circ\text{C}$ are shown in Figure 1. It can be seen that the TGA curve shows a weight loss about 7% from 35 $^\circ\text{C}$ to 130 $^\circ\text{C}$ corresponding to two weak endothermic peaks in DSC curve, which is attributed to the residual water in the 8YSZ gel [24,25]. About seventy percent of weight loss of 8YSZ gel occurred up to c.a. 500 $^\circ\text{C}$. The first calcined temperature was fixed at 700 $^\circ\text{C}$ because there is almost no weight loss at 520 $^\circ\text{C}$ and above [26,27].

The XRD patterns of 8YSZ and 8YSZ-glass obtained with different weight ratio and calcined at different synthesis temperature are shown in Figure 2. Figure 2a shows the XRD patterns of the 8YSZ-glass with different weight ratio calcined at 700 $^\circ\text{C}$, i.e., 0%, 10%, 20% and 30%. All the samples possess coexisting tetragonal and monoclinic phases, where tetragonal is the major phase. The XRD angles at 30.14$^\circ$, 34.72$^\circ$ and 35.04$^\circ$ belong to the (101), (002), and (110) crystal planes of t-Zr$_{0.9}$Y$_{0.1}$O$_{1.95}$.
(JCPDS 82-1241), respectively. From Figure 2b, when the synthesis temperature reaches 1200 °C and 1550 °C, the XRD patterns of 8YSZ are merely tetragonal structures. However, there are still a few obvious diffraction peaks of monoclinic structure in 8YSZ-20% glass calcined at 1200 °C and 1550 °C, respectively. Mori et al. observed that the Ti⁴⁺-doped 8YSZ electrolyte transform from a pure cubic structure to two-phase compound containing small amount of tetragonal phase with increasing Ti content [28]. And a monoclinic-to-tetragonal phase transformation was found in 9 mol% MgO doped ZrO₂ above 1300 °C [29]. Therefore, it is probably the high synthesis temperature and 20% weight ratio of low melting point glass changes from amorphous to crystalline at high temperature.

Figure 1. TGA-DSC test of the Zr₀.₉₂Y₀.₀₈O₂₋₀.₆(8YSZ) gel heated at 15 °C·min⁻¹.

Figure 2. Cont.
The conductivities vs. different synthesis temperature and weight ratio were tested at 400–800 °C in nitrogen atmosphere as shown in Figure 3. It is clear that the conductivities of composite electrolytes increase with the increase in glass concentration. And the highest conductivities are obtained for the 8YSZ-20% glass (700 °C), 8YSZ-20% glass (1200 °C), and 8YSZ-20% glass (1550 °C) to be $5.7 \times 10^{-2}$ S·cm$^{-1}$, $4.1 \times 10^{-3}$ S·cm$^{-1}$, and $2.3 \times 10^{-2}$ S·cm$^{-1}$ at 800 °C, respectively. A recent investigation by Lee et al. [29] reported that a single cubic phase of 8YSZ showed higher conductivity than 9 mol% MgO doped ZrO$_2$ which has a mixed phase. Similarly, the conductivities of the 8YSZ-20% glass (700 °C) (Figure 3a) and 8YSZ-20% glass (1550 °C) (Figure 3b) are higher than that of 8YSZ-20% glass (1200 °C) (Figure 3b) which has evidently tetragonal and monoclinic biphasic structure in Figure 2b. The conductivities of the 8YSZ-20% glass (700 °C) are lower than that of 8YSZ-30% glass (700 °C) composite electrolyte as shown in Figure 3a. However, the 8YSZ-30% glass (700 °C) composite electrolyte is unstable because it will cause segregation and reduce the mechanical hardness in the molten state when the glass powder is too high in percentage.

![Figure 2.](image-url)

**Figure 2.** (a) XRD patterns of the 8YSZ, 8YSZ-10% glass, 8YSZ-20% glass and 8YSZ-30% glass calcined at 700 °C for 6 h; (b) XRD patterns of the 8YSZ and 8YSZ-20% glass calcined at 1200 °C and 1550 °C, respectively.

![Figure 3.](image-url)

**Figure 3.** The conductivities vs. (a) different weight ratio of the 8YSZ-10% glass, 8YSZ-20% glass and 8YSZ-30% glass after calcined at 700 °C; (b) different synthesis temperature of the 8YSZ-20% glass (1200 °C, 1550 °C) in nitrogen atmosphere at 400–800 °C.
Figure 4 shows the variation of conductivity of 8YSZ-30% glass (700 °C) composite electrolyte with time in nitrogen atmosphere at 800 °C. The conductivity reaches a steady state in the first hour. However, with increasing time, the conductivity of 8YSZ-30% glass (700 °C) composite electrolyte gradually decreased. This suggests that it cannot be used for long period at 800 °C.

![Graph showing conductivity variation](image)

**Figure 4.** The variation of conductivity of 8YSZ-30% glass (700 °C) with time in nitrogen atmosphere at 800 °C.

The external (a) and cross-sectional (b) surface SEM images of the 8YSZ-20% glass (700 °C) composite electrolyte are displayed in Figure 5. The 8YSZ agglomerated with low melting point glass powder, few pores are observed and the microstructure is homogeneous after heating at 700 °C, which is attributed to high fluidity of molten glass. Figure 5 shows that the two components are evenly dispersed and intimately connected and do not react with each other due to their high chemical stability [3,5,9,11].

![SEM images](image)

**Figure 5.** The external (a) and cross-sectional (b) surface SEM images of the 8YSZ-20% glass (700 °C) composite electrolyte.

In order to investigate ionic conduction of the 8YSZ-20% glass (700 °C), the relationship between the oxygen partial pressure (pO₂) and conductivities was studied. As shown in Figure 6, there is almost

![Graph showing pO2 vs conductivity](image)
a straight line within the whole $pO_2$ range. The result indicates that the 8YSZ-20% glass (700 °C) is almost a pure ionic conductor [20–23]. In the $pO_2$ range of $10^{-20}$–$10^{-15}$ atm, the curve is slightly upwarped, indicating that there is a trace electron conduction in the 8YSZ-20% glass (700 °C) in reducing atmosphere.

![Figure 6](image_url)

**Figure 6.** The conductivities of the 8YSZ-20% glass (700 °C) composite electrolyte as a function of $pO_2$ at 750 °C is almost a pure ionic conductor.

It is well known that ZrO$_2$-based electrolyte is a good oxygen ion conductor. To study the oxide ionic conduction of the 8YSZ-20% glass (700 °C) composite electrolyte, an oxygen concentration discharge cell was tested at 800 °C as shown in Figure 7. The calculational electromotive forces (EMF$_{cal}$) could be obtained from EMF$_{cal} = \frac{RT}{nF} \ln[pO_2 \, (A)/pO_2 \, (B)]$ when $t_O = 1$. The air ($pO_2 \, (B)$) and pure O$_2$ ($pO_2 \, (A)$) are introduced into the anode and cathode, correspondingly. From Figure 7, the open circuit voltage is 35.6 mV, which is close to the calculated EMF (36.1 mV). Moreover, a stable discharge line could be seen in Figure 7. All the results illustrate that the 8YSZ-20% glass (700 °C) composite electrolyte is a good oxygen ion conductor.

![Figure 7](image_url)

**Figure 7.** The oxygen concentration discharge cell: air, Pd-Ag|8YSZ-20% glass (700 °C)|Pd-Ag, O$_2$ at 800 °C.

The H$_2$/O$_2$ fuel cell electrochemical performance was tested at 800 °C for the 8YSZ-20% glass (700 °C) as shown in Figure 8. It can be seen that the 8YSZ-20% glass (700 °C) reveals a high open circuit voltage (1.09 V) which means the composite electrolyte is dense [3]. The maximum power
density of the 8YSZ-20% glass (700 °C) is 72.7 mW·cm⁻² (thickness = 1.1 mm) at 800 °C. The result is lower than that previous reported cathode supported thin film fuel cell with 200–400 mW·cm⁻² at 800–850 °C [12–14,30], which can be attributed to the electrolyte thickness and the electrode/electrolyte interface. Further work is in progress to optimize the composition of composite electrolytes and develop the stable and high performance solid oxide fuel cell [31,32].

Figure 8. H₂/O₂ fuel cell of the 8YSZ-20% glass (700 °C) at 800 °C.

4. Conclusions

In this study, low melting point glass powder was chosen as a sintering aid to prepare novel Zr₀.₉₂Y₀.₀₈O₂₋ₐ(8YSZ)-low melting point glass composite electrolytes. The results of XRD indicate that the major phase in composite electrolytes is tetragonal and no diffraction peaks of low melting point glass are found. The influences of amount of additive, synthesis temperature, test temperature, and oxygen partial pressure on the electrical conductivities of the composite electrolytes were investigated at 400–800 °C. The results of the XRD and conductivities show that the 8YSZ-20% glass (700 °C) is a suitable choice. The oxygen concentration discharge cell illustrates that the 8YSZ-20% glass (700 °C) composite electrolyte is a good oxygen ion conductor. The maximum power density of the 8YSZ-20% glass (700 °C) is 72.7 mW·cm⁻² (thickness = 1.1 mm) at 800 °C.

Author Contributions: H.W. and R.D. conceived and designed the experiments; R.S. and J.L. performed the experiments; H.W. and R.D. analyzed the data; R.S. and J.L. contributed the used materials and analysis tools; H.W. and R.S. wrote the paper.

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