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# Determination of partial conductivities and electrolytic domain of yttrium-doped zirconia prepared from Indonesian zircon sand

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In this work, yttrium stabilized zirconia (YSZ) was synthesized using zirconium oxychloride (ZOC) which was extracted from high-grade zircon sand mine in Indonesia. The ZOC was extracted from the zircon sand by caustic fusion method which was used to prepare 8 mole% yttrium doped zirconia (YSZ8) powder with average particle diameter of 422 nm by co-precipitation method. The powder was then granulated to ~15  $\mu$ m granules by spray drying process and the granules were used to fabricate dense sintered body. The sintering temperature of 1600 °C was determined by monitoring the relative shrinkage rate with temperature.<ed>cedeck the technical meaning> The prepared materials were analyzed by X-ray diffraction (XRD), particle size distribution and Inductively coupled plasma mass spectrometry (ICP-MS), and scanning electron microscopy (SEM). The XRD indicated that the sintered YSZ8 has a cubic structure. Total electrical conductivity of the sintered YSZ specimen was calculated in 700-900 °C and  $0.21 \le (pO_2/atm) \le 10^{-19}$  range by DC 4-probe method. The materials showed a maximum conductivity of 7.78 × 10<sup>-2</sup> S.cm<sup>-1</sup> at 900 °C with an activation energy of  $0.83 \pm 0.02$  eV and the conductivity was lower than commercial 8YSZ at various temperature. The Hebb-Wagner polarization experiments were performed to measure partial electronic conductivity. The activation energies for conduction of holes and electrons were 1.48 ± 0.02 and 4.98 ± 0.94 eV, respectively. The electrolyte domain for YSZ8 was determined.

Key words: Zircon sand, Yttrium stabilized zirconia, Hebb-Wagner ion-blocking method, Partial electronic conductivity, Electrolytic domain.

## Introduction

Zirconia ceramics has been used extensively in a number of applications due to its high fracture toughness, high hardness and wear resistance, good frictional behavior, high temperature capability up to 2,400°C, nonmagnetic, low thermal conductivity and coefficient of thermal expansion similar to iron, modulus of elasticity similar to steel. It is also employed in thermal barrier coatings, high density grinding media, medical prostheses and components of automobile and machines [1-3]. In addition, it's electrically insulating at normal temperature but high oxygen ion conductivity at elevated temperatures makes it a widely used material in electrochemical devices such as sensors, fuel cells, electrolyzers, and separation membranes [4-9]. According to the US Geological Survey, diversified and commercial use of zirconia, contributed to a global mine production og 1440 mrtric tons in 2014.in 2014 its global mine production was 1440 metric tons [10].

Zirconia crystals, which form three different patterns-

monoclinic, tetragonal, and cubic- with temperature, can be stabilized in tetragonal phase by mixing ZrO<sub>2</sub> with small amounts of other metallic oxides, such as MgO, CaO, or Y<sub>2</sub>O<sub>3</sub> [11]. yttrium stabilized zirconia (YSZ) is one of the extensively studied and commercially produced zirconia based material. It has been observed that, doping small amounts (< 8 mole%) of Y<sub>2</sub>O<sub>3</sub>, at room-temperature, the crystal structure of zirconia transforms from monoclinic phase to tetragonal phase, and when the doping amount is  $\geq 8$  mole% it further transforms to cubic phase [12]. The YSZ with cubic structure has been reported to have high oxygen ion conductivity, with 8 mole% yttria doped zirconia (YSZ8) showing maximum ionic conductivity which is a key factor to its application as electrolyte in solid oxide fuel cells [11-14]. The zirconium silicate mineral, zircon (ZrSiO<sub>4</sub>), is the dominant economic source of zirconium, which is found in deposits located in Australia, South Africa, India, as well as in Indonesia [10]. It has been observed that the calcined powders of YSZ8 prepared from the ZrO<sub>2</sub>, extracted from zircon sand and originating from the tin mines show a mixed phase composition at the room temperature, with the major phase being cubic along with the traces of monoclinic and tetragonal phases [15]. For the application of the YSZ8 as fuel cell electrolyte, it is

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Determination of partial conductivities and electrolytic domain of yttrium-doped zirconia prepared from Indonesian zircon sand

required to have high ionic but minimum electronic conductivities. YSZ8 has been an excellent ionic conductor and shows very low electronic conductivity. Therefore, researchers have paid little attention on the quantification of its electronic conductivity and the contribution of the hole and electrons in the electronic conduction in YSZ8. However, the YSZ8 prepared from the ZrO<sub>2</sub> extracted from zircon sand and originating from the tin mines have impurities of other oxides such as Na<sub>2</sub>O, SiO<sub>2</sub>, CaO, Fe<sub>2</sub>O<sub>3</sub>, and TiO<sub>2</sub> [15], which might have significant effects on the ionic and electronic conductivity of such YSZ8. Any change in the electronic conduction of the YSZ8 based on zircon sand would limit the fuel cell efficiency by reducing its driving force. Therefore, it is necessary to quantify the amount of electronic conductivity as well as the contribution of the hole and electrons in the electronic conduction in YSZ8. In the present study, DC 4 probe conductivity measurements were performed by measuring the total electrical conductivity in different thermodynamic conditions by varying the temperature and oxygen partial pressure (pO<sub>2</sub>). Using the ion-blocking Hebb-Wagner polarization method, we measured the electronic conductivity and then quantified the partial electron and hole conductivity. Finally, the electrolyte domain was determined from the top limit, bottom limit, and boundary (p = n) of the conductivity vs. oxygen partial pressure curve.

### **Experimental**

Zircon sand was supplied from Indonesia Caustic fusion method, as shown in flow chart in Fig. 1, was employed to extract ZrOCl<sub>2.8</sub>H<sub>2</sub>O (ZOC) from the zircon sand. Briefly, zircon sand was mixed with NaOH in 7 : 1 molar ratio and then heated at 750 °C for 30 min. The reaction product was washed with copious amount of water to leach out Silica in the form of soluble Na<sub>2</sub>SiO<sub>3</sub>. Then the acid leaching process was performed by dissolving the precipitate in HCl solution to remove Na<sub>2</sub>O in the form of soluble NaCl. The precipitate was repeatedly washed with water and then reacted with 5 M HCl solution at 95 °C for 2 hrs to produce zirconium oxychloride. In this step, the remaining silica was precipitated out as H<sub>2</sub>SiO<sub>3</sub>. The solution was then filtered and the filtrate was crystalized to ZrOCl<sub>2</sub>. 8H<sub>2</sub>O by evaporation.

YSZ8 was prepared from the extracted ZOC crystals by a co-precipitation method involving the reaction between ZOC and commercial  $Y_2O_3$ , as shown in flow chart in Fig. 2. The Inductively coupled plasma mass spectrometry (ICP-MS) analysis was conducted for the analysis of impurities and data is given in Table 1. The particle size distribution and specific surface area of the calcined powder was measured by BET experiment. The particle size distribution of the YSZ8 as-calcined powder is given in Fig. 3. The specific surface area and the average particle diameter was  $28.4 \text{ m}^2/\text{g}$  and



Fig. 1. Flow chart for extraction of ZOC from zircon sand.



Fig. 2. Flow chart for co-precipitation process for preparation of YSZ8 powders from ZOC.

**Table 1.** Data from ICP of the calcined YSZ8 powder fromzircon sand.

Na	Mg	Si	Са	Ti	Fe	Zn	$Y_2O_3$
(ppm)	(wt %)						
11.6	7.1	-	50.0	3.9	67.0	19.0	13.0

422 nm, respectively.

As-calcined YSZ8 powder was used to produce YSZ8 granules through the process of granulation based on spray drawing and developed by the Jeonnam Technopark foundation, as shown in flow chart in Fig. 4. First, 15 vol% of 8YSZ powder was briefly milled in a



Fig. 3. Particle size distribution of calcined YSZ8 powders.



Fig. 4. Flow chart for granulation of YSZ8 powder by spray drying.

roller mill for 20 hrs in isopropyl alcohol (IPA, Dae-Jung, Korea) medium with 0.5 wt% of dispersing agent (DISPERBYK-2013). Then 2 wt% of binder (polyvinyl butyral, M.W. 40000), 2 wt% of plasticizer (polyethylene glycol M.W. 400) and 1 wt% of plasticizer (polyethylene glycol, M.W. 3700) was added to the milled mixture which was further milled for another 4 hrs. continued for another 4 hrs. The milled slurry was aged for about 1 hr and then spray dried using a sprayer at a rate of 10 kg/h with inlet temperature 70 °C and the outlet temperature 60 °C. The granulated powder was analyzed by X-ray diffraction (XRD, D/MAX Ultima III, Rigaku, Japan) and scanning electron microscopy (SEM, SS-550, Shimadzu, Japan).

The granulated powders were molded into discs and bar shapes by uniaxial die pressing and the discs were later cold isostatically pressed (CIP) under 150 MPa. The CIPed specimens were heat-treated at different temperatures for 10 hrs in air atmosphere and the relative shrinkage ratewas monitored to determine the optimum sintering temperature. For comparison, a molded specimen of the commercial YSZ8 powder (Tosoh zirconia, TZ-8Y) was also fabricated and heattreated under similar conditions. The sintered YSZ8 disc specimens were polished into a 1.5 mm thick disc for Hebb-Wagner polarization experiment whereas the sintered bar specimens were polished into  $1.8 \times 1.8 \times$ 11 mm<sup>3</sup> samples for DC 4 probe electrical conductivity measurements. The DC 4 probe electrical conductivity measurements were performed by combining a digital multimeter (Keithley 2700) and a current source (Keithley 6220) at various temperatures,  $700 \leq (T/^{\circ}C) \leq 900$ , and partial pressures of oxygen,  $0.21 \leq (pO_2/atm) \leq 10^{-19}$ . The partial conductivity of YSZ8 was measured by preparing a Hebb-Wagner polarization cell with an ion-blocking electrode (Pt foil) on one side and a reversible electrode (Pt mesh) on the other side. For detailed information about the Hebb-Wagner polarization experiment refer to our previous work [16].

# **Results and Discussion**

### Physical characterization

Fig. 5(a) shows XRD pattern of the YSZ8 granules obtained from the spray drying process. Compared to the commercial YSZ8 powder, the granulated powder shows mixed phase of low crystallinity. Although the cubic zirconia is the major crystalline phase, there are additional diffraction peaks which can be assigned to either tetragonal and monoclinic zirconia phases or to some common impurities observed in the YSZ8 prepared from zircon sand [15, 17]. Fig. 5(b) shows the SEM image of the YSZ8 granules. The morphology of the granules resembles an apple-shaped spheres and the average granule diameter was 15  $\mu$ m.

Fig. 6(a) shows the relative shrinkage of the molded YSZ8 specimens during heat-treatment at different temperatures. From the figure, the temperature for maximum shrinkage rate was higher for the specimen prepared from the granulated powder. In addition, it's only at 1600 °C that the shrinkage rate becomes negligible hence this was taken as the sintering temperature for the YSZ8 specimen. Fig. 6(b) shows XRD pattern of the YSZ8 specimens sintered at 1600 °C for 10 hrs. Unlike the granulated YSZ8 powder, XRD pattern of the

Determination of partial conductivities and electrolytic domain of yttrium-doped zirconia prepared from Indonesian zircon sand





Fig. 5. (a) XRD patterns and (b) SEM image of YSZ8 granules.

sintered body is equivalent to that prepared from the commercial powder and the diffraction peaks can be assigned to the cubic zirconia phase. Fig. 6(c) shows SEM image of the polished cross-section of the sintered YSZ8 specimen. Almost no pores are visible in the sintered body indicating good sintering of the YSZ8 specimen during the heat-treatment. The density of the sintered body was measured using Archimedes' method and it was found to be 98.6% of the theoretical density.

# **Determination of electrical conductivity**

Fig. 7 shows variation of total electrical conductivity of the YSZ8 in a temperature range of 700-900 °C. As expected, the conductivity increased with increasing temperature and with an activation energy of  $0.83\pm0.02$ eV it reached  $7.73 \times 10^{-3}$  S.cm<sup>-1</sup> at 900 °C. Nevertheless, the electrical conductivity of YSZ8 was lower than that observed in Tosoh YSZ8. The Arhenius behavior of the zircon YSZ8 can be represented by Eq. (1) whereas the same for Tosoh YSZ8 can be represented by Eq. (2).

$$\sigma_{ion} = 3.03 \times 10^2 \exp(-0.83 \pm 0.02 eV/kT) \tag{1}$$

$$\sigma_{ion} = 2.24 \pm 0.10 \times 10^2 \exp(-0.83 \pm 0.01 eV/kT)$$
(2)

As seen in the equations, the actvation energies for electrical conduction in the YSZ8 zircon sand and Tosoh YSZ8 were comparable. However, the comparison of the pre-exponential factors showed that it was higher for zircon YSZ8 compared to the Tosoh YSZ8. The preexponential factor is a term which depends on a number of factors, such as the concentration of mobile charge



Fig. 6. (a) Relative shrinkage rate of molded YSZ8 specimen during heat-treatment, (b) XRD patterns and (c) SEM image of YSZ8 sintered specimen.



Fig. 7. Variation of total electrical conductivity of YSZ8 specimen with temperature.



Fig. 8. Defect diagram of YSZ8 showing the variation of the concentrations of various charge carriers vs.  $pO_2$ .

carriers, hopping rate, and entropy. Higher zircon sand YSZ8 values is indicates higher vacancy concentration. [18]. The trace impurities of ions such as  $Fe^{3+}$ ,  $Ca^{2+}$ , Na<sup>+</sup>, Zn<sup>2+</sup>, etc., that were present in the zircon sand YSZ8 granules, might dissolve into the zirconia bulk during the high temperature sintering. Such soluble impurities act as aliovalent dopants which increase oxygen vacancy concentration.

Determination of partial conductivities from Hebb-Wagner polarization

The defect reaction on the doping of  $Y_2O_3$  to  $ZrO_2$  can be expressed as Eq. (3), where various symbols have their usual meaning in standard Kroger-Vink notation.

$$Y_2 O_3 \rightarrow 2Y_{Zr} + 3O_0^X + V_0^{\bullet}$$
(3)

The external equilibia upon oxygen release or inorporation can be represented by Eqs. (4) and (5) where  $K_{re}$  is equilibrium constant for reduction reaction,  $K_{ox}$  is equilibrium constant for oxydation reaction, n is electron concentration and p is hole concentration in YSZ8. In-Ho Kim, Bhupendra Singh, Ji-Won Lim and Sun-Ju Song

$$K_{Re} = n^2 \left[ V_o^{\bullet} \right] P_{O_2}^{1/2} \tag{4}$$

$$K_{Ox} = p^2 [O_i^{ll}] P_{O_2}^{-1/2}$$
(5)

1

1

Assuming the prevalence of anti-Frenkel defects in the stoichiometric YSZ8, the internal defect equilibria can be represented by Eqs. (6) and (7)

$$K_I = n \cdot p \tag{6}$$

$$K_{AF} = [Q_i^{ll}] [V_o^{\bullet}]$$
(7)

Furthermore, the electrical neutrality condition in the defect reactions can be represented by Eq. (8)

$$n+2[Q_i^{ll}]+[O_{Zr}^{l}]=p+2[V_o^{\bullet}]$$
(8)

The defect diagram of YSZ8 was constructed using the Eqs. (1)-(8) and is shown in Fig. 8. The 8 mol%-doping of  $Y_2O_3$  in  $ZrO_2$  can be considered as a high-doped case, and its characteristics in the electrolytic domain can be represented by  $2[V_0^{-}]=[Y'_{Zr}]$ . Therefore, partial conductivities by electron and hole can be expressed in terms of oxygen partial pressure as:

$$\sigma_n \propto P^{-1/4} \tag{10}$$

$$\sigma_p \propto P^{+1/4}_{O_2} \tag{11}$$

Furthermore, the contribution of a charge carrier in electrical conductivity in terms of carrier concentration ([]), charge (q), and mobility (u) can also be represented by Eq. (12)

$$\sigma_i = []_i \cdot q \cdot u_i \tag{12}$$

Ion-blocking cell experiments have been widely employed in the determination partial conductivities [16, 19-21]. The details about the Hebb-Wegner ionblocking experiments can be referenced to our previous work [16]. In ion-blocking experiments, the current density at steady-state is due to only electrons. The slope of the current-voltage curve can provides a way to estimate the partial electronic conductivity using Eq. (13) where L, A, and U represents length, area, and voltage at both ends of electrodes in the sample, respectively.

$$\sigma_e(\mu_{O_2} = \mu^{ll}_{O_2}) = -\frac{L}{A} \left( \frac{dj_e}{dU} \right)$$
(13)

The electronic conductivity has contributions from both electrons and holes, i.e.,  $\sigma_e = \sigma_n + \sigma_p$ . As seen from the defect diagram of YSZ8 in Fig. 8, the concentrations of electron and hole are proportional to the partial pressure of oxygen to the powers of -1/4 and 1/4, respectively, therefore the electronic conductivity can be represented by Eq. (14)



Fig. 9. (a) Current-voltage characteristic of ion-blocking cell measurement at 900 °C in air; (b) partial conductivities in YSZ8.

$$\sigma_{e} = \sigma_{n}^{*} \left( \frac{pO_{2}}{pO_{2}} \right)^{-1/4} + \sigma_{p}^{*} \left( \frac{pO_{2}}{pO_{2}^{*}} \right)^{1/4}$$
(14)

Considering the chemical potential of oxygen molecule and the partial pressure of oxygen, the partial conductivities of electron ( $\sigma_e$ ) and hole ( $\sigma_p$ ), at steadystate the current-voltage characteristics can be represented by Eq. (15) where A, L, I<sub>e</sub>, E, and F are area, length, current, voltage, and Faraday constant, respectively.

$$I_{e} = \frac{ART}{FL} + \left[\sigma_{p}^{*}\left\{exp\left(\frac{EF}{RT}\right)\right\} - 1\right] + \sigma_{p}^{*}\left[\left\{1 - exp\left(-\frac{EF}{RT}\right)\right\}\right]$$
(15)

In ion-blocking cell experiments, the voltage measurement at the steady-state under a specific partial pressure of oxygen at applied constant current provides data sets, as shown for example in Fig. 9(a) at 900 °C in air. Based on these data after proper fitting, we calculated the partial conductivity of electron and hole in the 8YSZ system, which can be represented by Eqs. (16) and (17)

$$\sigma_n = 1.02 \times 10^{11} \exp(-4.98 \pm 0.94 eV/kT) (pO_2)^{-1/4}$$
 (16)

$$\sigma_p = 1.41 \times 10 \exp(-1.48 \pm 0.02 eV/kT)(pO_2)^{1/4}$$
 (17)

The contribution of ionic conductivity can be extracted by subtracting the electronic contribution from the total electrical conductivity calculated earlier by DC 4 probe method. The contribution of ionic conductivity can be represented by Eq. (18)

$$\sigma_{ion} = 3.03 \times 10^2 \exp(-0.83 \pm 0.02 eV/kT)$$
(18)

The variation of partial conductivities with  $pO_2$  is shown in Fig. 9(b). The points represent values obtained from the experiments whereas solid lines represent the extrapolated ones. YSZ8 is a widely used electrolyte material in SOFCs and based on the high ionic conductivity of the YSZ8 from zircon sand observed in the present study it appeared to be suitable enough to be used as SOFC electrolyte. As seen in Fig. 9(b), under normal  $pO_2$  conditions, the contribution of partial ionic conductivity is much higher than that of electronic conductivity. therefore, under these conditions the YSZ8 from zircon sand can easily be used as SOFC electrolyte. However, in extreamly low pO2 conditions, the contributions from the partial electronic conductivities become significant and can not be ignored. Therefore, it is imperative to determine the electrolytic domain for the YSZ8 from zircon sand. The condition for a material with mixed ionic-electronic conductivity to act as an electrolyte is that the ionic transference number (tion) should be  $\geq 0.99$ . The dependence of contributions of partial conductivities by oxygen-ions, electrons and holes on oxygen partial pressure can be expressed by Eqs. (18-20).

$$\sigma_i = A_i \exp\left(-\frac{E_i}{kT}\right) \tag{18}$$

$$\sigma_n = A_n \exp\left(-\frac{E_i}{kT}\right) p O_2^{-\frac{1}{4}}$$
(19)

$$\sigma_p = A_p \exp\left(-\frac{E_p}{kT}\right) p O_2^{\frac{1}{4}}$$
(20)

From the upper and lower limits of oxygen partial pressure ( $P_p$  and  $P_n$ ), where the contribution from hole and electron, respectively, are maximum and oxygen partial pressure ( $P_{p=n}$ ) where contributions from electron and hole become equal, in the regions where the transport number of oxygen ion is above 0.5 (i.e. ti = 0.5), we have



Fig. 10. Representation of calculated electrolytic domain boundaries in YSZ8.

$$P_{p} = \left(\frac{A_{i}}{A_{p}}\right)^{4} \exp\left[-\frac{4(E_{i}-E_{p})}{kT}\right]$$
(21)

and 
$$P_p = \left(\frac{A_i}{A_p}\right)^4 \exp\left[-\frac{4(E_i - E_p)}{kT}\right]$$
 (22)

Similarly, in regions where ti=0.99, we have

$$P_p = \left(\frac{A_i}{99A_p}\right)^4 \exp\left[-\frac{4(E_i - E_p)}{kT}\right]$$
(23)

and 
$$P_n = \left(\frac{A_i}{99A_n}\right)^4 \exp\left[-\frac{4(E_i - E_p)}{kT}\right]$$
 (24)

From the values of activation energies and pre-exponential factors, the mixed conducting and electrolytic region was calculated, as shown in Fig. 10. The doted lines indicate the border between electrolytic region and mixed conducting region, whereas the solid lines show the condition where ti = 0.5. Using data given in Fig. 10, the electrolytic domain for YSZ8 from zircon sand can be decided; for example, at 700 °C it extends across  $10^{27}$ - $10^{-155}$  atm range whereas at 1000 °C it will be in  $10^{20}$ - $10^{-109}$  atm range, indicating that YSZ8 from zircon sand is very much suitable as fuel cell electrolyte in normal operating conditions of high temperature SOFCs.

# Conclusions

8 mole% yttrium stabilized zirconia (YSZ8) was synthesized from high-grade zircon sand mine in Indonesia. First,  $ZrOCl_2$  was extracted from the zircon sand by caustic fusion method and was used to prepare YSZ8 powder with an average particle diameter of 422 nm. The powder was then granulated to ~15 mm granules by spray drying process and the granules were

#### In-Ho Kim, Bhupendra Singh, Ji-Won Lim and Sun-Ju Song

used to fabricate a dense sintered body at 1600 °C. The XRD showed that the sintered YSZ8 has a cubic structure. Total electrical conductivity of the sintered YSZ specimen was calculated in 700-900 °C and  $0.21 \le (pO_2/atm) \le 10^{-19}$  range by DC 4-probe method. The material showed a maximum conductivity of  $7.73 \times 10^{-3} \text{ S.cm}^{-1}$  at 900 °C with an activation energy of  $-0.83 \pm 0.02$  eV but the conductivity was lower than commercial 8YSZ at various temperatures. The Hebb-Wagner polarization experiments were performed to measure partial electronic conductivity. The conductivities for electron and hole are negligibly low  $(2.23 \times 10^{-9})$  and  $1.39 \times 10^{-5}$  S.cm<sup>-1</sup> respectively) at 1000 °C in air, compared to  $1.53 \times 10^{-1}$  S.cm<sup>-1</sup> for ionic conductivity. Finally, the electrolytic domain for the prepared 8YSZ was calulated.

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Determination of partial conductivities and electrolytic domain of yttrium-doped zirconia prepared from Indonesian zircon sand

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