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Title

Study of the solid-state reaction at the interface between lanthanoid-doped ceria and yttria-stabilized zirconia for solid-oxide fuel cell applications

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Abstract

To develop more highly-efficient SOFCs, we have investigated the thickness of highly resistive layer which consist of solid solutions of $\text{CeO}_2\text{-ZrO}_2$ system generally observed between YSZ electrolyte and cathode-interlayer made of doped CeO_2 . In terms of the effect of the dopant in the CeO_2 -based interlayer materials on the thickness of the solid solution, the use of YDC or LDC for the interlayer results in a thinner solid solution compared to that obtained when a GDC interlayer was used. When adapted into SOFCs, I-V tests at 800°C indicated that the cell with a YDC interlayer exhibited substantially better performance than the cell with a GDC interlayer.

Keywords

Solid oxide fuel cell, efficiency, diffusion, interlayer.

Main text

1. Introduction

Fuel cells convert fuels to electricity with greater efficiency than conventional gas engines or gas turbines because they directly convert the chemical energy of fuels into electrical energy. Among all fuel cell types, solid oxide fuel cells (SOFCs) are expected to achieve the highest power generation efficiency. Although the efficiency of commercialized SOFC systems for residential applications is 46.5%LHV [1], results of 60%LHV in validation experiments have been reported [2, 3], indicating that SOFC systems can potentially achieve greater generating efficiency. Thus, the development and practical implementation of high-efficiency SOFCs is an effective approach to counteract the limited quantity of fossil fuels and promote a sustainable society. Various approaches [4-8] for enhancing the power generation efficiency of SOFCs have been proposed, such as developing new materials, increasing fuel utilization, and improving high-efficiency auxiliary machines and power conditioners. Among these

approaches, enhancing efficiency through the development of materials is important because it is highly flexible and does not depend on the system composition.

For the purpose of constructing high-efficiency SOFCs, highly conductive and highly electrode-reaction-active materials are needed. However, the manufacturing processes of SOFCs involve heat treatments at temperatures that exceed SOFC operating temperatures. More highly active materials tend to result in the easier formation of reaction products between the highly active materials and the materials they contact.

For example, high-resistance SrZrO_3 has been reported to form between YSZ, a very common electrolyte material for SOFCs, and LSC, an active material used as a conventional cathode for SOFCs [9]. A well-known solution to this problem is to insert an interlayer consisting of CeO_2 -based materials between the cathode and the electrolyte [10-13]. However, highly resistive solid solutions have also been reported to sometimes form between a CeO_2 -based material layer and a YSZ electrolyte layer [14-16], resulting in deterioration of the power generation efficiency of the SOFC [17, 18]. With respect to this challenge, two approaches for achieving SOFCs with high

efficiency have been proposed. One approach is to select a highly conductive interlayer material; the other is to reduce the thickness of the highly resistive layers.

Some studies on the conductivities of CeO_2 -based materials have been presented, including their electronic conductivity under different oxygen partial pressure conditions [19, 20], and dependence of their ion conductivity on cationic species [21, 22]. With respect to the solid solubility between the CeO_2 -based material and YSZ, the diffusion rate of Ce into Zr has been reported to be more rapid than that of Zr into Ce [23]. However, few reports have been presented on the thickness of the formed highly resistive layers.

In this study, we examined the effect of dopant species in CeO_2 on the thickness of the highly resistive solid solution between a YSZ electrolyte and a CeO_2 -based material interlayer. We used lanthanoid-doped CeO_2 (LnDC), in which Ln is La, Y, or Gd, as the interlayer and 8YSZ as the electrolyte material. First, YSZ discs were fabricated and the interlayer materials were printed on the discs and sintered at 1300, 1400, and 1500°C for 10 h while the diffusion behaviour of Ce was analysed. Next, SOFCs with

each material interlayer were fabricated and the cell performances were characterized.

2. Experimental

2.1. Sample preparation

The samples tested were manufactured from commercial ceramic powders using standard ceramic processing techniques. The sample compositions are shown in Table 1.

2.2. Preparation of samples for diffusion tests

As an electrolyte material, YSZ (Tosoh Corporation: TZ-8Y) and 15 mass% polyvinyl butyral (Sekisui Chemical Corporation) were well mixed and processed into a paste. The paste was shaped into films of 0.3 mm thickness, from which 28 mm diameter discs were stamped. After this process, the stamped discs were sintered at 1500°C for 2 h. As an interlayer material, LnDC (Ln = La, Y, Gd) materials (KCM Corporation Co., Ltd.) were processed into a paste (Kyoto Elex Co., Ltd.), which was used to screen print a coating with a diameter of 16 mm and a thickness of 10 µm onto

the electrolyte discs. The discs with the interlayer material were sintered at 1300, 1400, and 1500°C for 10 h. After the calcination step, the discs were embedded into resin materials and the cross-sections of the samples were observed using SEM/EDX (SEM, JEOL Ltd., JSM-7001F) at 15.0 kV acceleration voltage to analyse the diffusion behaviour of each element of the LnDC and YSZ materials at the interface.

2.3. Sample preparation for power generation tests

As an anode support substrate material, NiO (Kanto Chemical Co., Inc.):YSZ was prepared at a mass ratio of 60:40. The anode support substrate supplies fuel and needs to possess high porosity. Thus, a pore-forming carbon (Showa Denko K.K.) was added to the mixture at a concentration of 10 mass%; the resulting mixture was well mixed and crushed using a ball mill for 24 h and then pressed uniaxially into circular pellets with a 24 mm diameter at a pressure of 6.0 MPa for 120 s. For the electrolyte and the interlayer materials, the same pastes used to prepare the diffusion test samples were used. The electrolyte and interlayer materials were screen-printed as 20 μm diameter and 10 μm diameter coatings, respectively, onto the anode substrates. The substrates

were subsequently calcined at 1000°C for 2 h and then at 1350°C for 2 h. After these processes, the cathode material LSCF (Praxair) was also screen-printed and sintered at 950°C for 2 h. The cell's cathode area was 0.64 cm², and a Pt mesh was placed on the cathode surface for power collection. Fig. 1 shows a schematic of the setup used to measure the performance of the button SOFCs. In the power generation performance tests, the electric furnace was heated to 900°C over a period of 4.5 h in air. Then, H₂ was supplied to the anode side at 100 cc/min, air was supplied to the cathode side at 150 cc/min, and the I-V performance was measured.

3. Results and discussion

3.1. Results

3.1.1. Diffusion tests

Fig. 2 shows SEM/EDX images of a cross-section of the YSZ/GDC sample calcined at 1500°C for 10 h. The images show that the peak intensity of Ce decreased from the GDC interlayer side to the YSZ electrolyte side. In the same manner, the peak

intensity of Zr decreased from the YSZ electrolyte side to the GDC interlayer side.

This result agrees with previously discussed findings [15].

Figs. 3 and 4 show SEM/EDX images of the cross-sections of the YSZ/YDC and YSZ/LDC samples, respectively. The SEM/EDX images of the YSZ/YDC sample were nearly the same as those of the YSZ/GDC sample; this trend also corresponds to previously reported results [24]. The SEM/EDX images show a slightly higher peak intensity of the dopant in the YSZ side. Table 2 shows the dopant atomic concentrations of the points in Fig. 5. Fig. 5 shows the EDX mappings of Ce and dopant in YSZ/GDC and YSZ/YDC. These results show a prominent dopant enrichment in the YSZ region adjacent to the interface. Arachi et al [25] reported that the conductivities of $\text{ZrO}_2\text{-Y}_2\text{O}_3$ system is higher than those of $\text{ZrO}_2\text{-Gd}_2\text{O}_3$ system. This could cause performance advantages of the cell with YDC interlayer, but the conductivity decline by the $(\text{Ce, Zr})\text{O}_2$ solid solution formations is bigger than the decline by the dopant condensation formations. Therefore, the dopant condensation formations have low impact on the cell performance. The cause of the local

enrichment of the dopants remains to be identified. The SEM/EDX images and spectra of YSZ/LDC show a slightly higher peak intensity of the dopant, La, in the YSZ side compared to that observed in the spectra of the other 2 samples. The condensation of La was assumed to result from the formation of $\text{La}_2\text{Zr}_2\text{O}_7$ between the interlayer and the electrolyte [9, 26]. The conductivity of $\text{La}_2\text{Zr}_2\text{O}_7$ in the air at 750°C is 4.66×10^{-4} S/cm [27] which is much lower than the conductivity of YSZ (1.8×10^{-2} S/cm) [28] and GDC (7.9×10^{-2} S/cm) [28]. So the use of LDC for the interlayer is not suitable.

Eguchi et al. reported that Ce diffuses faster than Zr in the YSZ/YDC interdiffusion couple; they also observed the Kirkendall effect [29], whereas we focused on the diffusion behaviour of Ce. To compare the dependency of the diffusion behaviour of Ce on the dopant species of the CeO_2 -based materials, we extracted the intensity data of Ce from the spectra of YSZ/LnDC (La = La, Gd, Y) calcined at 1500°C ; the results are shown in Fig. 6. Each line was normalized by the average Ce intensity which was calculated using the Ce intensity from the surface of observation point to the $1\text{ }\mu\text{m}$

inside toward the interface between interlayer and electrolyte. The zero position in each line was set to the start point of the line analysis. The graph shows that the diffusion length between the highest level of Ce intensity and the lowest level of Ce intensity in YSZ/GDC is more than the diffusion lengths between the highest concentration levels and the lowest concentration levels of Ce in the other 2 samples.

Next, the diffusion coefficients of Ce in YSZ for each YSZ/LnDC sample were calculated using the solution of the following diffusion equation (1):

$$\frac{\partial c}{\partial t} = D \frac{\partial^2 c}{\partial x^2} \quad (1)$$

Because the thickness of the YSZ is much greater than the diffusion length of Ce, the boundary condition of the diffusion equation can be written as follows (2):

$$\left(\frac{\partial c}{\partial t} \right)_{x=0} = 0, t > 0 \quad (2)$$

The solution of equation (1) with boundary condition (2) is written as follows (3):

$$c(x, t) = \frac{M}{\sqrt{\pi D t}} \exp\left(-\frac{x^2}{4 D t}\right) \quad (3)$$

where D is the diffusion coefficient, M is the amount of solute, and t is time. The equation was fitted to the measured data, and the diffusion coefficients were calculated.

Fig. 7 shows the fitting results for the intensity of Ce in YSZ/YDC samples calcined at temperatures ranging from 1300 to 1500°C. These data were normalized, and the background intensity was removed, resulting in a good fit to equation (3). Fig. 8 shows the temperature dependence of the diffusion coefficient for the diffusion of Ce into YSZ in YSZ/GDC and YSZ/YDC samples. Fig. 9 shows the calculated diffusion behaviour of Ce at the YSZ/GDC and YSZ/YDC interfaces, assuming a calcination at 1350°C for 2 h. Broken line corresponds to 1/e in the vertical axis. The values of 0.33 and 0.49 described on the horizontal axis indicate the distances from the interface at the intensity decreased to 1/e of the initial values in YSZ/YDC and YSZ/GDC, respectively. Thus, the distances where the peak intensity of Ce decreased to 1/e of the initial value are shown to be 0.33 μm in YSZ/YDC and 0.49 μm in GDC/YDC. Therefore, these results indicate that the thickness of highly resistive (Ce,Zr)O₂ solid solution, as a result of Ce diffusion, depends on the interlayer materials. Consequently, the performance of SOFCs can be improved through the use of YDC rather than GDC as an interlayer material.

3.1.2. Power generation test results

Fig. 10 shows the power generation test results for the SOFCs with GDC and YDC interlayers at 800°C. Humidified hydrogen that was passed through a 30°C bubbler was supplied to the cells as fuel. The fuel and air were supplied at the same rates during the power generation tests. The I-V performance of cells with a YDC interlayer was better than that of cells with a GDC interlayer. Table 3 shows the ohmic and polarization resistances of the cells with GDC and YDC interlayer at 800°C and 0.5 A/cm² with the current interrupt method. This result shows that the cell with YDC interlayer has lower ohmic and polarization resistances than the cell with GDC interlayer. As Uchida et al. reported [30] that there is a relationship between ohmic and polarization resistances and the polarization resistance is supposed to reduce with ohmic resistance decreasing.

3.2. Discussion

3.2.1. Differences in Ce diffusion behaviour with different dopants

As shown in Fig. 6, the diffusion length of Ce in YSZ/GDC was longer than that in

YSZ/YDC and YSZ/LDC; i.e., the mobility of Ce in GDC is greater than that in YDC and LDC. The empirical equation for the lattice parameter changes of CeO₂ with different cation dopants is expressed as follows (4) [31, 32]:

$$d_{Ce} = 0.5413 + \sum_k (0.0220\Delta r_k + 0.00015\Delta z_k)m_k \quad (4)$$

where d_{Ce} (in nm) is the lattice constant of CeO₂ with dopant at room temperature.

The term Δr_k is the difference in ionic radius ($r_k - r_h$) of the k^{th} dopant, r_k , and the host cation, r_h , in eight-fold coordination, as calculated from Shannon's compilation. The

term Δz_k is the valence difference of ($z_k - z_h$), and m_k is the mole percent of the k^{th} dopant

in the form of MO_x. By equation (4), the ionic radius which doesn't change the lattice

parameter of CeO₂ can be calculated when the second term of equation (4) is equal to

zero. The dopant radius which doesn't change the lattice constant is defined as the

critical ionic radius. By equation (4), a triad cation is added to CeO₂; thus, the critical

ionic radius, r_C (in nm), is given as follows (5):

$$r_C = r_{Ce} + \frac{0.00015}{0.022} \quad (5)$$

In this case, the host cation is Ce⁴⁺ in 8-fold coordination (0.097 nm [33]), and the

critical ionic radius is calculated as 0.1038 nm. The ionic radius of the triad dopant Gd (0.1053 nm [33]) is closer to the critical ionic radius than are the other two cations of Y (0.1019 nm [33]) and La (0.1160 nm [33]). Though each crystal structure of GDC, YDC, and LDC is thought to maintain their cubic structure, their lattice constant depends on dopant species. The lattice constant changes are thought to affect local strains, which leads to the low mobility of Ce in LnDC. In GDC, the dopant radius is closer to the critical ionic radius and the less effect of local strains is thought to be introduced into GDC, which leads to the high mobility of Ce in GDC.

In terms of strains, Nicholas et al. [34] showed the relationship between strain energy and the dopant's size and charge by using the Vegard's Slope, X , described by Eq. (6) followed by (7) and (8),

$$X = 0.0220\Delta r_i + 0.00015\Delta z_i \quad (6)$$

$$W = 6Ga_0(\Delta a)^2 \quad (7)$$

$$\Delta a = Xm_i \quad (8)$$

where Δr_i is the difference in the ionic radius between the dopant and Ce^{4+} , Δz_i is the

difference in valence number between the dopant and Ce^{4+} . W is the strain energy, G is the shear modulus, a_0 is the initial lattice parameter, Δa is the change in lattice parameter, and m_i is the concentration of dopant. Eqs. (6) to (8) indicates that the strain energy has dependence on the absolute value of X that is the difference of ionic radius and balance number between the dopant ion and the host ion. In comparison with each Vegard's Slope, X_k ($k=\text{Gd, Y, La}$), the strain energy of GDC calculated from Vegard's Slope showed the smallest value among the three types of LnDC ($\text{Ln}=\text{Gd, Y, La}$). That suggests that the less effect of local strains is introduced into GDC than the other 2 samples of YDC and LDC. Deguchi et al. also reported that YDC and LDC include numerous large local strains, which are thought to obstruct Ce diffusion towards the interface of YSZ and YDC or the interface of YSZ and LDC [35, 36].

Thus, local strains caused by the difference between the critical ionic radius and the dopant radius as well as the strain energy estimated from the difference of the ionic radii and/or the valence numbers were found to be consistent with the dependence of the Ce-diffusion rates on the dopants in LnDC.

4. Conclusions

To enhance SOFC efficiency, we examined the thickness of the highly resistive solid solution of $(\text{Ce,Zr})\text{O}_2$ formed at the interface between the YSZ electrolyte and the CeO_2 -based interlayer.

Regarding the effects of dopant in the CeO_2 -based materials used for the interlayer on the thickness of the solid solution, we examined the diffusion behaviour of YSZ/LnDC ($\text{Ln} = \text{La}, \text{Gd}, \text{Y}$). We successfully determined that the use of YDC and LDC as interlayers can reduce the thickness of the solid solution compared to the use of a GDC interlayer. This result indicates that the use of YDC as an interlayer material can improve the performance of SOFCs compared to the use of GDC.

The cell with YDC as an interlayer was also confirmed to exhibit significantly better cell performance compared to the cell with GDC as an interlayer.

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Captions

Fig. 1 Schematic of the setup used to measure the performance of SOFC button cells.

Fig. 2 SEM/EDX images of the cross-section of YSZ/GDC.

Fig. 3 SEM/EDX images of the cross-section of YSZ/YDC.

Fig. 4 SEM/EDX images of the cross-section of YSZ/LDC.

Fig. 5 EDX mappings of Ce and dopant in YSZ/GDC and YSZ/YDC.

Fig. 6 Comparison of Ce diffusion behaviour in YSZ/LnDC (La = La, Gd, Y) samples

calcined at 1500°C.

Fig. 7 Fitting results of the peak intensity of Ce in YSZ/YDC samples calcined at 1300, 1400, and 1500°C.

Fig. 8 Temperature dependence of the diffusion coefficient for the diffusion of Ce into YSZ in YSZ/GDC and YSZ/YDC samples.

Fig. 9 Calculated diffusion behaviour of Ce at the YSZ/GDC and YSZ/YDC interfaces, assuming calcination at 1350°C for 2 h. Broken line corresponds to 1/e in the vertical axis. The values of 0.33 and 0.49 described on the horizontal axis indicate the distance from the interface at which the intensity decreased to 1/e of the initial values in YSZ/YDC and YSZ/GDC, respectively.

Fig. 10 Power generation test results of the SOFCs with GDC and YDC interlayers at 800°C.

Table 1. Sample details.

Table 2. The dopant atomic concentrations

Table 3. The ohmic and polarization resistances of the cells

Table 1. Sample details.

Species	Company	Average diameter D ₅₀ [μm]	Specific surface area [m ² g ⁻¹]
(Ce _{0.8} , La _{0.2})O _{1.9}	KCM Corporation Co., Ltd.	0.50	8.14
(Ce _{0.8} , Gd _{0.2})O _{1.9}	↑	0.49	4.24
(Ce _{0.8} , Y _{0.2})O _{1.9}	↑	0.49	8.17

Table 2. The dopant atomic concentrations.

Sample	Point	Atomic concentration [%]	
		Gd	Y
YSZ /GDC	1	5.46	---
YSZ /GDC	2	6.64	---
YSZ /GDC	3	5.28	---
YSZ /YDC	1	---	8.61
YSZ /YDC	2	---	11.70
YSZ /YDC	3	---	7.40

Table 3. The ohmic and polarization resistances of the cells.

Interlayer	Ohmic resistance [mV]	Polarization resistance [mV]
YDC	122	73
GDC	168	129

Figure(s)
Fig.1

Fig.2

Fig.3

Fig.4

Fig.5

Fig.6

Fig.7

Fig.8

Fig.9

Fig.10