**Determination of Oxygen Surface Exchange Constant of
 $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ coated with $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ by Isotope Exchange
Technique**

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ABSTRACT

The surface oxygen exchange coefficient (k^*) of $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ (LNF) coated with porous $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (GDC) was determined by the isotope exchange technique. The LNF coated with GDC was subjected to isotope exchange treatment where $^{16}\text{O}_2$ was abruptly changed into $^{18}\text{O}_2$ at range of temperature 873 – 1073 K under 10^{-1} and 10^{-2} bar of the oxygen partial pressure ($p(\text{O}_2)$). The line-scanning mode of secondary ion mass spectrometry was used to determine the surface oxygen exchange of the LNF coated with GDC. The k^* of LNF coated with GDC enhanced compared to the bare LNF due to the formation of triple phase boundary between LNF, GDC, and gas phase of oxygen. The role of GDC is enhancing the incorporation process of the oxygen dissociated at the LNF surface. The adsorption and dissociation of oxygen takes places at LNF surface and incorporated into GDC porous.

Keywords: Surface oxygen exchange kinetics, Isotope exchange, Secondary ion mass spectrometry

1. Introduction

Mixed ionic and electronic conductor (MIEC) type materials have been studied for a few decades due to the ability to reduce oxygen at the surface [1,2]. The MIEC gives a chance to improve the performance of the solid oxide fuel cell (SOFC). Several MIECs such as $(\text{La,Sr})\text{CoO}_{3-\delta}$ (LSC) and $(\text{La,Sr})(\text{Co,Fe})\text{O}_{3-\delta}$ (LSCF) have been utilized as the state the art cathode of SOFC. On the other hand, $\text{La}(\text{Ni,Fe})\text{O}_{3-\delta}$ electrode is relatively new cathode material which has been studied in the last decade and shows promising performance [3-5].

$\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ (LNF) shows superior electronic conductivity and ionic conductivity. Nishi et al. mentioned that LNF shows a comparable value on the ionic conductivity to LSCF [6]. In addition, they suggested that the oxygen surface exchange kinetics (k^*) of LNF is relatively high among MIEC electrode. In order to enhance the electrochemical performance, the MIEC is often mixed with ionic conducting oxide such as Ytria-stabilized Zirconia (YSZ) or doped ceria based electrolyte [7-9]. The performance of the composite electrode is enhanced drastically compared to single phase electrode.

In our preceding work, composite electrode of LNF and $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (GDC) has been studied and showed drastic enhancement on the electrochemical performance [10]. Such a large enhancement on MIEC which has relatively high ionic conductivity is difficult to be understood by a simple explanation of the extended contact area between electronic and ionic conducting oxide. Another factor may exist for the enhancement by the GDC existence on the mixed composite electrode.

The study by Hong et al. on the surface reaction of LSCF coated with samaria-doped ceria (SDC) gives another way to understand the reaction mechanism of oxygen reduction process in a composite electrode [11]. They found that the k^* of LSCF enhances drastically with the existence of SDC particles on the surface [11].

This result suggests that the enhanced was not the ionic conduction but the surface reaction rate in a porous composite electrode.

In this study, an LNF bulk sample, which is coated by GDC has been investigated by isotope exchange technique to determine the k^* at controlled temperature and $p(\text{O}_2)$. Isotope exchange technique is one of powerful techniques to determine the surface exchange kinetics [12,13]. The analysis by secondary ion mass spectrometry determines the diffusion profile of $^{18}\text{O}/^{16}\text{O}$ inside the bulk sample. By combine the isotope exchange and secondary ion mass spectrometry, the k^* of LNF coated with GDC has been determined and compared to bare LNF. In this study, the enhancement of the surface exchange kinetics will be discussed in order to determine the reaction pathways of the oxygen reduction process.

2. Experimental

Sample Preparation

A $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ (LNF) powder was prepared by Pechini method. The powder was calcined at 1073 K for 6 hours to obtain a single phase, which was confirmed by XRD (M18X CE, Mac Science). The LNF powder was pulverized and then pressed into a pellet at 200 MPa. The pressed sample was sintered at 1523 K for 6 hours and cut into a rectangular shape of 2 mm x 2 mm x 5 mm. The sample was polished by diamond suspension to get a fine surface prior to GDC deposition.

A $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (GDC) powder was provided by a commercial powder (Shin-Etsu Rare Earth, Shin-Etsu Chemical). The GDC was mixed with ethyl cellulose (Wako Pure Chemical Industries, Ltd.) and organic solvent, TMS-1 (Tanaka Kikinzoku Co.), to get slurry of GDC. The GDC was deposited onto LNF bulk surface by screen printing method. The samples of LNF coated with GDC were sintered at 1273 K. After sintering, the samples were pre-annealed for the 3 hours

prior to the isotope exchange treatment. The average particle size of GDC particle which coated to LNF surface was determined by scanning electron microscopy (SEM, JEOL JSM-700 1F). The average particle size of GDC is around 1 μm as shown in the Fig. 1.

Isotope Exchange and Secondary Ion Mass Spectrometry

The sample was placed into the alumina chamber, which was placed into glass for isotope exchange treatment. The thermocouple (Pt/Pt-3%Rh) were attached to alumina chamber to monitor the temperature. The sample was heated up to the constant temperature at $^{16}\text{O}_2$ for 30 minutes before the gas was abruptly changed to the isotope enriched one ($^{18}\text{O}_2$, 99% purity). The exchange times were varied depending on the temperature and $p(\text{O}_2)$ condition. For 873 K under 10^{-2} bar, the exchange time was around 30 min after subjected into $^{16}\text{O}_2$. In the other case, at 1073 K under 10^{-2} bar, the exchange time was around 10 min after $^{16}\text{O}_2$ abruptly changed into $^{18}\text{O}_2$. After that, the sample was quenched to the room temperature in 60 second.

The quenched samples after the isotope exchange were used for SIMS measurement. For LNF coated with GDC sample, the GDC porous layer was removed from LNF prior to SIMS measurement. The sample was put into an acetone in order to remove the GDC porous layer from the surface of the LNF bulk. The sample was cut to get the cross section surface and then polished to get a fine surface. The secondary ion mass spectrometer (SIMS, CAMECA IMS-7F) was utilized to determine the diffusion profile of the samples. The Cs^+ (15 keV) were used as a primary ion beam of the SIMS. The line scanning mode was used where the points of measurement were taken from the edge of the sample up to 500 μm with a primary

ion beam of 0.5 nA. The negative ion signal of $^{16}\text{O}^-$ and $^{18}\text{O}^-$ were collected from the central part (20 μm in diameter) of the sputtered area.

3. Results and Discussion

Enhancement of surface oxygen exchange kinetics (k^)*

The oxygen isotope ratio ($[^{18}\text{O}]/([^{18}\text{O}]+[^{16}\text{O}])$) profile is shown in the Fig. 2 and 3. The oxygen ratio from the samples was higher than natural abundance of isotope oxygen (0.002) which indicates that the isotope exchange treatments were successful. At the low temperature (873 K) and low oxygen partial pressure (10^{-2} bar), the exchange time was 30 min during isotope exchange treatment. The relatively long exchange time has been chosen since the isotope oxygen needs time to dissolve into the samples.

As shown in Fig. 2 and 3, large difference in the isotope ratio was observed between the LNF samples with and without GDC. The high concentration of ^{18}O in the LNF with GDC indicates an enhancement on the surface reaction of LNF by the existence of GDC particles. The oxygen coefficient and, D^* , and surface exchange coefficient, k^* , were calculated by following equation [14]:

$$C(^{18}\text{O})(x, t) = C_0 + (C_g - C_0) \left[\operatorname{erfc} \left(\frac{x}{2\sqrt{D^*t}} \right) - \exp \left(\frac{k^*x + k^*t}{D^*} \right) \operatorname{erfc} \left(\frac{x}{2\sqrt{D^*t}} + \frac{k^*\sqrt{D^*t}}{D^*} \right) \right] \quad (1)$$

where $C(^{18}\text{O})(x, t)$, C_0 , C_g and, t are the ^{18}O isotope ratio at depth x which determined by the experimental, the natural abundance of ^{18}O (0.2%), the ^{18}O isotope ratio in the gas phase (99%) and the annealing time, respectively.

The enhancement of isotope exchange rate was in the factor of 10 independently of the temperature and $p(\text{O}_2)$ as shown in the table 1. Fig. 4 shows the surface exchange coefficients of LNF coated with GDC and bare LNF for each temperature

and oxygen partial pressure. The activation energy of k^* of the bare LNF (216 kJ/mol) was different to the reference (74 kJ/mol) [6]. It may be caused by the reference sample which was suffered by the secondary phase (NiO were found at the surface of the sample). Although the secondary phase was observed, they suggested that the oxygen D^* of the LNF weren't affected. However, the surface exchange kinetics might be affected by NiO on the surface of LNF sample.

There is no information how the NiO appears in their sample. If NiO was formed in their sample due to the segregation therefore the k^* could be low. In other case, if in their sample the LNF has more NiO due to nonstoichiometric composition during the sample preparation, therefore it is possible to have higher k^* compared to the single phase LNF. As a reference, a work has been reported on $\text{La}_{0.8}\text{Sr}_{0.2}\text{CoO}_{3-\delta}$ film electrode which added more Sr in the surface of the film electrode [15]. As a result the oxygen reduction activity was enhanced drastically compared to single phase of $\text{La}_{0.8}\text{Sr}_{0.2}\text{CoO}_{3-\delta}$ film electrode [15]. Thus, it gives another insight that the enhancement of k^* is possible due to the secondary phase at the surface of the electrode.

The Effect of GDC existence on the diffusivity (D^)*

The D^* of the LNF coated by GDC and bare LNF are shown in the Fig. 5 and table 2. There are no clear dependence of the D^* due to the GDC existence and there is no clear dependence of the D^* to the oxygen partial pressure as well. The GDC existence is only affected the k^* since the GDC only enhances the oxygen reduction process at the very surface of the LNF bulk. The D^* which obtained in this study is close to the data from the reference [6]. It indicates that the secondary phase from the reference doesn't affect the D^* as shown in the Fig. 5. The isotope diffusion

coefficient can be correlated with ionic conductivity, σ_i , by the Nernst-Einstein equation as [12]:

$$D^* = fD_o = (fR T\sigma_i) / (4c_oF^2) \quad (2)$$

Where D_o is the self-diffusion coefficient of the oxygen, R is the gas constant, T is the temperature and F is the Faraday constant. The correlation factor, f , for the LNF which is perovskite-type structure is 0.69 [16]. The correlation factor, f , assumes that the equal probability of jump of the vacancy without any interaction between cation-vacancy or vacancy – vacancy interaction. The oxide ion conductivity of LNF and LNF coated with GDC is shown in the Fig. 6. There is no clear difference in the ionic conductivities of LNFs with/without the GDC existence.

Mechanism of oxygen reduction process

Measurement by secondary ion mass spectroscopy proves that GDC existence on the LNF bulk sample enhances the k^* . If the surface exchange of the GDC is faster than the LNF, therefore the reaction reduction process of oxygen will be at GDC. However, the k^* of GDC (bulk sample) is estimated around 2×10^{-9} cm/s [17]. In this study, the k^* of GDC is lower than LNF bulk sample and it is difficult to think reaction took place at the GDC. Therefore, the reaction might proceed at or near the triple phase boundary of the GDC particle, LNF surface, and the gas phase. It should be noticed that, the D^* of the LNF sample was lower than that in GDC bulk sample. The LNF is mixed ionic and electronic conductor, however the concentration of oxygen vacancy isn't so high compared other electrode.

MIEC of LNF has a high electronic conductivity as reported in the references [3,5]. Therefore, during the oxygen reduction process the electrons are sufficient and the charge transfer process may be fast. Because of relatively low ionic conductivity,

the incorporation process can assumed as limiting step in oxygen reduction process. If the oxygen reduction process separated into three steps therefore:

where O_2 , V_{O_s} , V_{O} , and O_0^x are oxygen molecules at gas phase, oxygen vacancy at the surface, oxygen vacancy at the bulk, and oxygen in the lattice, respectively.

The adsorption and dissociation process will be faster at LNF surface. It would be reasonable if the incorporation process at the surface will be at GDC particles on the LNF surface. The reaction will take place at separated place between LNF and GDC. The GDC porous layer has role to enhance the incorporation during oxygen reduction reaction process.

Another possible explanation of the enhanced surface reaction rate is the modification of chemical composition of the LNF surface by the existence of GDC. . In our study on LNF film coated by GDC porous, we measured the compositional change by auger electron spectroscopy (AES) [18]. The high Ce concentration was observed at the interface of the LNF film-GDC porous layer. The concentration of Ce as high as La concentration, which it has some possibility of Ce inclusion into LNF structure. However, the result needs to be proven by another experiment after that the mechanism of Ce inclusion into LNF structure need to be clarified. Further study is needed to understand the details of the oxygen reduction reaction process.

Conclusion

The isotope exchange treatment and secondary ion mass spectroscopy was utilized to determine the surface oxygen exchange kinetics (k^*) of $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ (LNF) coated

with $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (GDC). The k^* of LNF coated with GDC enhanced compared to the bare LNF due to the formation of triple phase boundary between LNF, GDC, and gas phase of oxygen. The GDC only affects the k^* , while the diffusivity (D^*) is no clear dependence to the GDC existence. The role of GDC at LNF surface involved in the oxygen reduction can be to serve the incorporation site for the adsorbed and dissociated oxygen at the LNF surface.

Acknowledgments

This work was supported by JST, Japan, as part of “Phase Interface Science for Highly Efficient Energy Utilization” project in strategic basic research program, CREST.

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Table 1. The surface oxygen exchange, k^* / cm s⁻¹)

Sample	1073 K		973 K		873 K	
	10 ⁻¹ bar	10 ⁻² bar	10 ⁻¹ bar	10 ⁻² bar	10 ⁻¹ bar	10 ⁻² bar
LNF	5.11x10 ⁻⁰⁶	4.03x10 ⁻⁰⁷	5.67x10 ⁻⁰⁷	7x10 ⁻⁰⁸	2x10 ⁻⁰⁸	3.49x10 ⁻⁰⁹
LNF-GDC	1.99x10 ⁻⁰⁵	2.19x10 ⁻⁰⁶	2.64x10 ⁻⁰⁶	3.1x10 ⁻⁰⁷	7x10 ⁻⁰⁸	1.15x10 ⁻⁰⁸

Table 2. The oxide ion diffusion, D^* / cm² s⁻¹)

Sample	1073 K		973 K		873 K	
	10 ⁻¹ bar	10 ⁻² bar	10 ⁻¹ bar	10 ⁻² bar	10 ⁻¹ bar	10 ⁻² bar
LNF	7.06x10 ⁻⁰⁹	1.07x10 ⁻⁰⁸	1.52x10 ⁻⁰⁹	1.57x10 ⁻⁰⁹	1.4x10 ⁻¹⁰	9.05x10 ⁻¹⁰
LNF-GDC	1.07x10 ⁻⁰⁸	3.27x10 ⁻⁰⁸	1.57x10 ⁻⁰⁹	2.1x10 ⁻⁰⁹	9.05x10 ⁻⁰⁹	1.12x10 ⁻⁰⁹

Figure Caption

Figure 1 SEM image of $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (GDC) coated on the surface of $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$.

Figure 2 Profile of oxygen ratio of (a) bare $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ and (b) $\text{LaNi}_{0.4}\text{Fe}_{0.6}\text{O}_{3-\delta}$ coated with $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (GDC) at 1073 K under 10^{-1} bar of $p(\text{O}_2)$. There is slightly enhancement of oxygen concentration on the $\text{LaNi}_{0.4}\text{Fe}_{0.6}\text{O}_{3-\delta}$ coated with $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (GDC). The solid line indicates the curve fitting result.

Figure 3 Profile of oxygen ratio of (a) bare $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ and (b) $\text{LaNi}_{0.6}\text{Fe}_{0.6}\text{O}_{3-\delta}$ coated with $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (GDC) at 973 K under 10^{-1} bar of $p(\text{O}_2)$. Large enhancement can be seen due to the GDC existence. The solid line indicates the curve fitting result.

Figure 4 The surface oxygen exchange kinetics (k^*) of bare $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ and $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ coated with $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (GDC) at various temperature and $p(\text{O}_2)$. The enhancement of k^* can be seen due to the GDC existence on the LNF surface.

Figure 5 The diffusivity (D^*) of the bare $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ and $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ coated with $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (GDC) at various temperature and $p(\text{O}_2)$. There is no clear dependence of the D^* due to the GDC existence and $p(\text{O}_2)$.

Figure 6 Ionic conductivity of bare $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ and $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ coated with $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (GDC) at various temperature and $p(\text{O}_2)$.

Figure 1. SEM image of $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (GDC) coated on the surface of $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$.

(a)

(b)

Figure 2 Profile of oxygen ratio of (a) bare $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ and (b) $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ coated with $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (GDC) at 1073 K under 10^{-1} bar of $p(\text{O}_2)$. There is slightly enhancement of oxygen concentration on the $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ coated with $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (GDC). The solid line indicates the curve fitting result.

(a)

(b)

Figure 3 Profile of oxygen ratio of (a) bare $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ and (b) $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ coated with $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (GDC) at 973 K under 10^{-1} bar of $p(\text{O}_2)$. Large enhancement can be seen due to the GDC existence. The solid line indicates the curve fitting result.

Figure 4 The surface oxygen exchange kinetics (k^*) of bare $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ and $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ coated with $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (GDC) at various temperature and $p(\text{O}_2)$. The enhancement of k^* can be seen due to the GDC existence on the LNF surface.

Figure 5 The diffusivity (D^*) of the bare $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ and $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ coated with $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (GDC) at various temperature and $p(\text{O}_2)$. There is no clear dependence of the D^* due to the GDC existence and $p(\text{O}_2)$.

Figure 6 Ionic conductivity of bare $\text{LaNi}_{0.6}\text{Fe}_{0.44}\text{O}_{3-\delta}$ and $\text{LaNi}_{0.6}\text{Fe}_{0.4}\text{O}_{3-\delta}$ coated with $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (GDC) at various temperature and $p(\text{O}_2)$.