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Journal of the European Ceramic Society 24 (2004) 1329–1335

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Novel fast oxide ion conductor and application for the electrolyte of solid oxide fuel cell

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Abstract

Effects of Co doping to Ga sites of a $LaGaO_3$ based oxide on the oxide ion conductivity was investigated. Oxide ion conductivity increased by doping with Co and it was found that usage of a $LaGaO_3$-based perovskite type oxide, doped with Sr for the A site and Co and Mg for the B site ($La_{0.8}Sr_{0.2}Ga_{0.8}Mg_{0.115}Co_{0.085}O_3$ denoted as LSGMC), for the electrolyte of the fuel cell gave a notably large power density at an intermediate temperature of 873 K on a cell using H_2 and O_2 as fuel and oxidant, respectively. The power density increased as the thickness of the electrolyte was decreased. The maximum power density was attained at values of 1.4 and 0.5 W/cm^2 at 1073 and 873 K, respectively, when 0.18 mm thick LSGMC was used for the electrolyte. Electrical conductivity in the LSGMC was also estimated using polarization methods. Electrical conductivity was also increased by doping with Co, resulting in an increased amount of chemically leaked oxygen. Consequently, the theoretical calculation demonstrated that the highest energy conversion efficiency would be achieved when the thickness of the LSGMC electrolyte was 100 μm.

Keywords: Electrical conductivity; Fuel cells; Ionic conductivity; Perovskite

1. Introduction

Fuel cell technology is considered as the method for the next generation energy production as it exhibits almost no emission of air pollutant and a high efficiency of energy conversion, namely, a low CO_2 emission. At present, a polymer electrolyte membrane fuel cell (PEMFC) is attracting much interest as a power source for the electric vehicle. However, this has a serious drawback regarding fuel, i.e. highly purified hydrogen is essential. In contrast, a solid oxide fuel cell, denoted as SOFC, is another type of interesting fuel cell, from the power generator point of view, because of the high energy conversion efficiency and the flexibility regarding fuel: natural gas can be directly used as fuel.[1,2] Therefore, the total system for SOFC including fuel processing becomes quite simple. At present, Y_2O_3 stabilized ZrO_2(YSZ) is widely used as an oxide ion conductor. However, requirements for high operating temperatures and thin films are major drawbacks for the electrochemical devices utilizing YSZ because of its limited oxide ion conductivity.[3] The reduction of the operating temperature is essential for the development of SOFCs in order to improve their reliability as power generators and to enable the use of cheap refractory materials. To decrease the operating temperature, the development of an ion conductor with a high electrical conductivity is paramount. As a result, the development of an oxide ion conductor with high electrical conductivity and stability against reduction is a highly demanded subject.[1] In particular, the fast and stable oxide ion conductor, over a wide P_{O2} range is strongly required for the electrolyte of SOFC in order to achieve a high efficiency of energy conversion.

In our previous study, it was found that $LaGaO_3$ doped with Sr and Mg (denoted as LSGM) exhibited an oxide ion conductivity as high as that of Gd-doped CeO_2.[4] The high oxide ion conductivity of LSGM is recognized by various other research groups[5–10] and it was also demonstrated that a high power density was obtained by using LSGM as electrolyte material in SOFCs.[11–15] On the other hand, it is generally believed

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doi:10.1016/S0955-2219(03)00508-9

that doping with transition metal cations such as Co, Ni or Fe enhance electron or hole conduction. Therefore, the use of these cations as dopants does not seem to be desirable insofar as the oxide ion transport properties are concerned. However, it was found in our previous study that oxide ion conductivity increased by doping LSGM with small amounts of Co without decreasing the transport number of the oxide ion.[11] In the previous study, application of Co doped LSGM for the electrolyte of SOFC was studied and it was found that cells using LSGMC as an electrolyte exhibited a notably high power density.[12,13] In this study, application of a Co doped $LaGaO_3$ based oxide for intermediate temperature SOFCs was investigated in detail under operating conditions. Durability of the cell was also investigated in order to examine the stability of Co doped LSGM as an electrolyte.

2. Experimental

All specimens used in this study were prepared by a conventional solid state reaction technique employing powders of La_2O_3, Ga_2O_3, MgO, $SrCO_3$, and CoO. Details of the preparation method can be found elsewhere.[4] The final sintering was achieved at 1773 K for 6 h in air. The disk obtained was ground to 0.5 mm in thickness using a diamond grinding machine. The compositions of the prepared specimens of $La_{0.8}Sr_{0.2}Ga_{0.8}Mg_{0.2\text{-}X}Co_XO_{3\text{-}\delta}$, in particular, $La_{0.8}Sr_{0.2}Ga_{0.8}Mg_{0.115}Co_{0.085}O_{3\text{-}\delta}$, were generally used in this study.

A single planar SOFC with 0.2 mm^2 effective electrode area was fabricated in this study. Commercial NiO (Wako, reagent grade) and prepared $Sm_{0.5}Sr_{0.5}CoO_3$, which were made into an oxide paste by mixing with *n*-butylic acethic acid, were painted with a blush for the anode and cathode, respectively. For long-term stability measurements, Ni-$Ce_{0.8}Gd_{0.2}O_2$ (GDC) cermet (10 vol% NiO mixed with GDC) was used for the anode. A platinum mesh (80 mesh) was the push-on electrode with a mullite tube for the current corrector. Platinum wire was attached next to the cathode with platinum paste (Tanaka TR7902) for the reference electrode. After painting the electrode, the cell was fired at 1173 K for 30 min to remove the organic solvent. Before power density measurement, NiO in the cermet anode was reduced to Ni in a H_2 stream at 1273 K for 1 h. Pure O_2 or dry air and hydrogen humidified with 3 vol% H_2O were fed to the cathode and the anode side, respectively. It is noted that the theoretical electromotive force under the conditions used was 1.10 V. The power generation characteristics were measured by the four-probe method. A galvanostat (Hokuto, HA-301) was used to provide the electrical load. Internal resistance was measured with a current interrupting method. A current pulse was generated with a current pulse generator (Hokuto, HC-111) and the corresponding potential was measured with a spectrum analyzer (Takeda TR-9201).

A polarization method was used for the estimating the minor carrier of electronic hole and electron conduction in a Co-doped $LaGaO_3$-based oxide. Fig. 1 shows the schematic view of the experimental set-up used for the polarization measurement. Platinum paste and molten Pyrex glass were used for the electrode and the gas shealing, respectively. The reference electrode was attached close to the anode side and the applied potential was controlled with a potentiostat/galvanostat (Hokuto HA-501). Air was used for the reference atmosphere.

3. Results and discussion

3.1. Effects of Co doping on the oxide ion conductivity

Fig. 2 shows the electrical conductivity in a N_2 atmosphere ($P_{O_2} = 10^{-5}$ atm) and the estimated transport number of oxide ion by using a H_2–O_2 gas concentration as a function of the amount of Co doping for the Ga site in $LaGaO_3$. It is seen that the electrical conductivity monotonically increased with increasing amounts of Co. However, the estimated ionic transport number decreased with increasing amounts of Co. In particular, a decrease in the ionic transport number is significant when the amount of Co is higher than 10 mol% for Ga site. This decrease in the ionic transport number can be explained by the increased hole conduction by doping the Co. Since it is considered that the improved hole conduction can be assigned to the hopping conduction of hole between Co^{2+} and Co^{3+}, the hole conduction became significant when the amount of Co became large. The electrical conductivity shown in Fig. 2 is the total conductivity consisting of the hole and oxide ion conduction. In order to determine the effect of Co on the oxide ion conductivity, the electrical

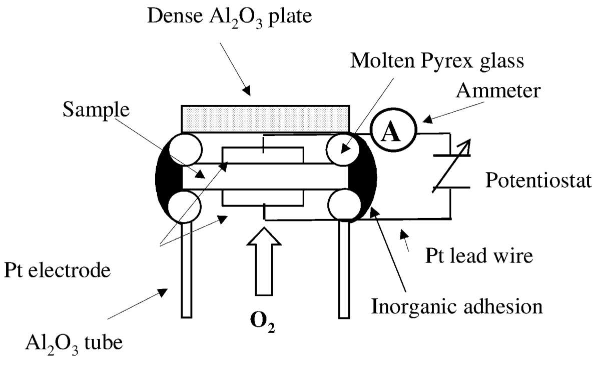

Fig. 1. Schematic view of the experimental set-up for polarization method used.

conductivity by the electronic hole should be removed from the total conduction. This can be achieved theoretically by multiplying the total conductivity by the estimated transport number using an oxygen concentration cell. As a first approximation, the oxide ion conductivity was estimated by multiplying the total conductivity by the estimated transport number estimated by the oxygen concentration cell. The oxide ion conductivity was estimated with the transport number and total conductivity and superimposed to Fig. 2. It was seen that the estimated oxide ion conductivity increases with increasing amounts of doped Co and attains a maximum around 10 mol%. On the other hand, the apparent activation energy for electrical conductivity monotonically decreases with increasing Co content and reaches to a value of 0.45 eV for 10 mol% Co doped: this value is almost half of that for $Zr_{0.84}Y_{0.16}O_2$. Although the highest oxide ion conductivity is obtained at $X=0.1$, the transport number of the oxide ion decreases to a value lower than 0.9. Therefore, it was concluded that the most promising composition for the electrolyte of SOFC is $La_{0.8}Sr_{0.2}Ga_{0.8}Mg_{0.115}Co_{0.085}O_3$ (denoted as LSGMC).

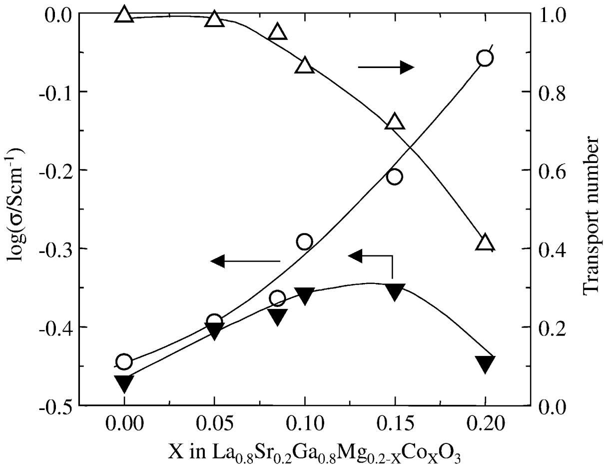

Fig. 2. Electrical conductivity at 1223 K and the estimated transport number of the oxide ion at 1223 K by using H_2–O_2 gas concentration as a function of Co amount doping for the Ga site in $LaGaO_3$. (○) Conductivity at 1223 K; (□) Transport number at 1223 K; (▼) Estimated oxide ion conductivity.

Fig. 3 shows a comparison of the oxide ion conductivity of LSGMC (Co content 8.5% for B site) with that of the conventional fluorite oxides. It is seen that the oxide ion conductivity of LSGMC is higher than that of CeO_2 doped with Gd_2O_3 and slightly lower than that of Bi_2O_3 doped with Yttria. However, it is well known that Bi_2O_3 based oxides exhibit pure oxide ion conductivity in a limited P_{O_2} range, Considering the wide P_{O_2} range for the electrolyte domain (oxide ion transport number, $t_i > 0.9$), LSGMC is highly attractive as an electrolyte material for SOFCs operating at intermediate temperatures.

3.2. Power generating property of the cell using Co doped LSGM

Fig. 4 shows the open circuit potential as well as the maximum power density at 1073 K as a function of the Co content in the $LaGaO_3$ based oxide electrolyte. It is clear that the open circuit potential (OCV) decreased monotonically with increasing Co amounts. In particular, the decrease in OCV was significant when the Co content was higher than 10 mol% in Ga site. This was caused by the appearance of hole conduction by doping with Co, as discussed, and the dependence of the OCV on the amount of doped Co agrees well with that of the transport number of oxide, as shown in Fig. 2. On the other hand, the power density increased with increasing

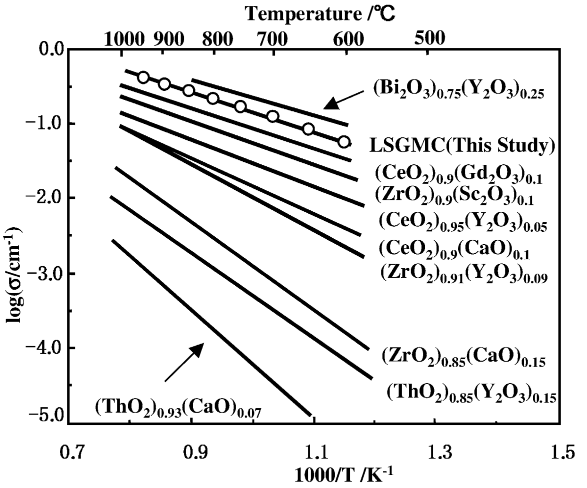

Fig. 3. Arrhenius plots of the electrical conductivities of $La_{0.8}Sr_{0.2}Ga_{0.8}Mg_{0.115}Co_{0.085}O_3$ and of conventional fluorite structured oxide ion conductors.

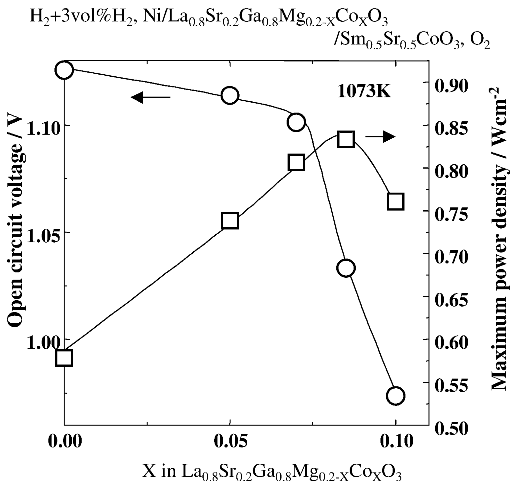

Fig. 4. Open circuit potential as well as the maximum power density at 1073 K as a function of Co content in the $LaGaO_3$ based oxide electrolyte. (□) Maximum power density; (○) Open circuit potential.

Co content and it attained a maximum value at 8.5 mol% Co doped for the Ga site. The improvement in power density was explained by improved oxide ion conductivity by doping Co. When the amount of doping Co was in excess, the hole conduction was significant and the cell became short-circuited resulting in a decrease in power density.

Fig. 5 shows details of the potential drop of the cell at 0.3 A/cm^2, 1073 K, as a function of the Co content. It is clear that not only electrical resistance but also overpotential of the cathode and anode decreased with increasing Co content. Therefore, the improved power density was obtained not only by the improved oxide ion conductivity in the electrolyte but also by the improved electrode reaction. The decrease in potential drop by internal electric resistance (IR) loss was simply explained by the improved oxide ion conductivity by doping Co. Since the electrical resistance monotonically decreased with increasing Co content, the IR loss also exhibited a similar dependence on the Co content. On the other hand, a detailed mechanism for a decrease in the overpotential of the cathode and anode was not clear up to now. However, several reports suggested that some part of the electrode reaction is strongly dependent on the electrolyte performance and the electrode overpotential decreased with increasing oxide ion conductivity.[14,15] Therefore, increased oxide ion conductivity by doping Co could play an effective role in improving the electrode reaction. This may be the case for the improved cathodic overpotential. As discussed above, the addition of small amounts of Co does not simply increases the oxide ion conductivity but also increase the hole conductivity. Therefore, the better anodic performance exhibited by doping Co for LSGM seems most likely to be assigned to an appearance of hole conduction, which may accelerate the charge transfer step. Anyway, both IR loss and the overpotential of the cathode and anode were at a minimum at a Co content of 8.5 mol%. Consequently, the largest power density was attained at this composition (Fig. 4).

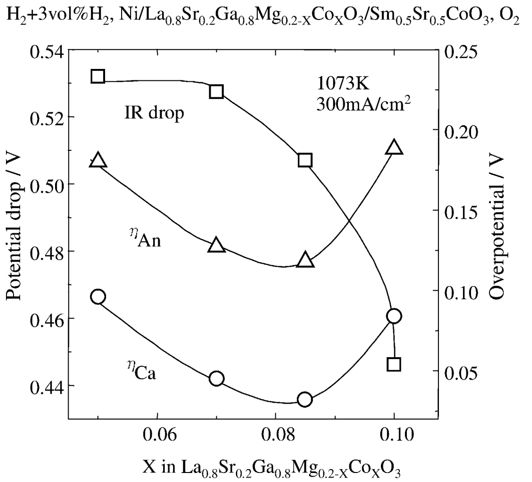

Fig. 5. Details of the potential drop of the cell at 0.3 A/cm^2, 1073 K as a function of Co content. Here, η_{Ca}, η_{An} and IR means potential drop by cathodic overpotential, anodic overpotential, and electrical resistance respectively.

Fig. 6 shows the temperature dependence of the maximum power density together with the open circuit potential. In the same figure the temperature dependence of the maximum power density of the cell using $La_{0.9}Sr_{0.1}Ga_{0.8}Mg_{0.2}O_3$ (LSGM) under the same conditions is also shown for comparison. It is obvious that the high power density was obtained in both LSGMC and LSGM cells. Compared with the power density of the LSGM cell, the LSGMC cell exhibited a higher power density at each temperature. In particular, high power density values were observed even at temperatures lower than 973 K when LSGMC was used as the electrolyte material. This is explained by the smaller activation energy for oxide ionic conduction in LSGMC than LSGM.[11] Therefore, LSGMC is highly attractive as an electrolyte of fuel cells operating at intermediate temperatures.

Fig. 7 shows the *I–V* curve at 1073 K and details of the potential drop of the LSGMC cell which was estimated by the current interrupting method. It is obvious that the main reason for the potential drop was electrical resistance which was in good agreement with the results of the impedance measurement. This is because the electrolyte for the cell is 0.5 mm thick. The effects of electrolyte thickness were further studied.

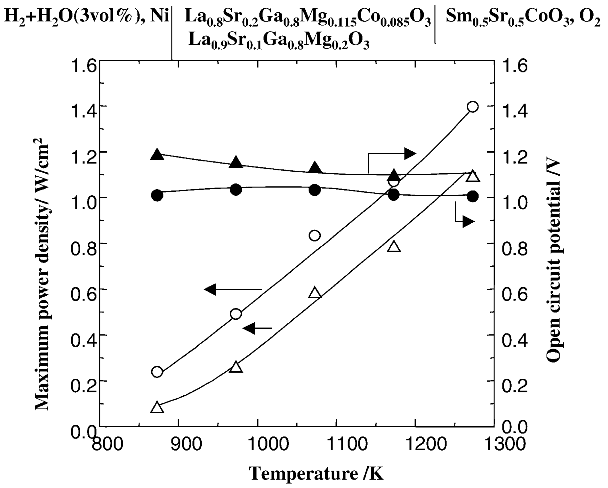

Fig. 6. Temperature dependence of electrical power generation characteristics of SOFC for which $La_{0.8}Sr_{0.2}Ga_{0.8}Mg_{0.115}Co_{0.085}O_3$ and $La_{0.9}Sr_{0.1}Ga_{0.8}Mg_{0.2}O_3$ were applied. Electrolyte thickness is 0.5 mm and $Sm_{0.5}Sr_{0.5}CoO_3$ and Ni were used for the cathode and anode respectively.

(▲) Open circuit potential
(□) Maximum power density } for LSGM electrolyte

(●) Open circuit potential
(△) Maximum power density } for LSGMC electrolyte

3.3. Power generating characteristics of the cell using thinner LSGMC plates

Fig. 8 shows the power density of the H_2–O_2 cell at 1073 and 873 K as a function of electrolyte thickness. As expected, the power density of the cell monotonically increased with decreasing the thickness of the LSGMC electrolyte. On the other hand, the open circuit potential exhibited a tendency to decrease with decreasing the thickness of the electrolyte. This was explained by the increased amount of leaked oxygen with decreasing thickness, since LSGMC exhibits a small hole conduction. At an electrolyte thickness of 0.18 mm, in particular, the open circuit potential decreased to 0.94 V at 873 K. Therefore, from the conversion efficiency point of view, it is expected that the suitable thickness of the electrolyte will be existed for the application of SOFCs. However, an extremely large power density was attained with a thinner electrolyte. In particular, maximum power density values (1.53 and 0.50 W/cm^2 at 1073 and 873 K, respectively) were attained with a 0.18 mm thick electrolyte as shown in Fig. 8. This is simply explained by the decrease in electrical resistance of the electrolyte, which is the main internal resistance of the cell. Therefore, further improvements in the power density are expected by further decreasing the electrolyte thickness. However, as discussed above, the decrease in the open circuit potential also becomes significant with decreasing the thickness of LSGMC. This is caused by chemically leaked oxygen and an amount of fuel will be combusted without forming electric power, resulting in the decreased energy conversion efficiency. To estimate the role of electronic conduction on theoretical energy conversion efficiency, the measurement of electron and hole conduction is of importance.

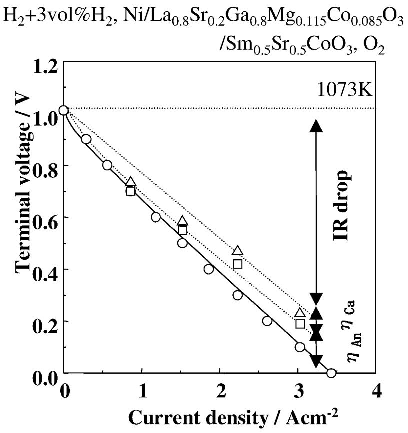

Fig. 7. *I–V* curve at 1073 K and details of the internal resistance of the LSGMC cell.

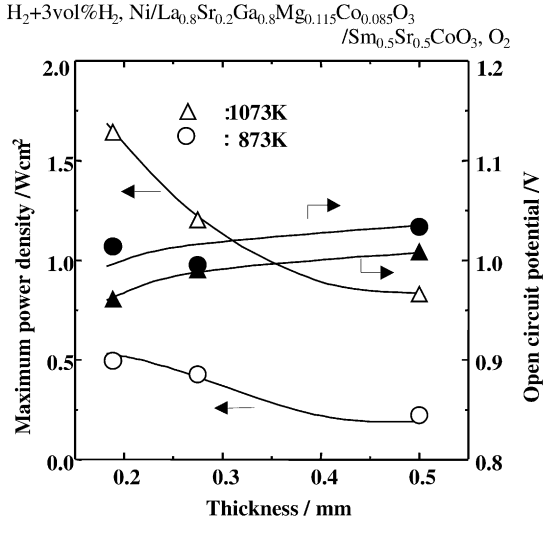

Fig. 8. Maximum power density and open circuit potential of the H_2–O_2 cell with LSGMC electrolyte as a function of the thickness of the electrolyte.

(▲) Open circuit potential
(○) Maximum power density } at 873 K

(●) Open circuit potential
(△) Maximum power density } at 1073 K

3.4. Measurement of electron and hole conduction in LSGMC oxides

Fig. 9 shows the plots of polarization curves, i.e. the residual electronic current against applied potential in LSGMC at 1073 K. Here, the preliminary polarization measurement was done by using LSGMC with a smaller Co content than the optimized one (i.e. $La_{0.8}Sr_{0.2}Ga_{0.8}Mg_{0.15}Co_{0.05}O_3$), since the polarization in mixed conducting ceramics is rather difficult. According to the Hebb–Wagner theory, the electron and the hole conductivity can be estimated by the slope of the polarization curve. It was seen that the typical "S" shaped curve corresponding to a p–n transformation was observed on the I_e vs. V plot for LSGMC. Differentiation of the I_e vs. V relationship gives the P_{O_2} dependence

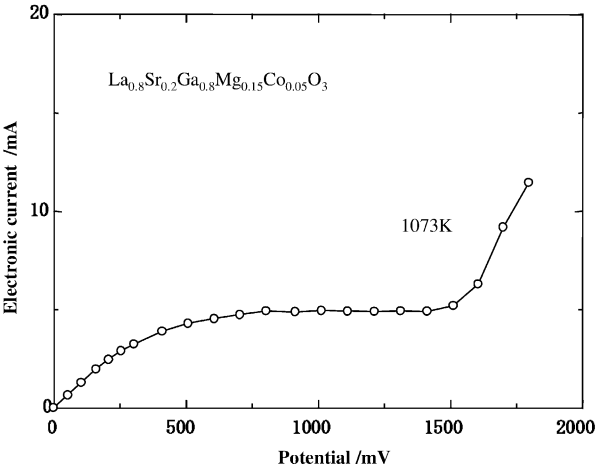

Fig. 9. Polarization curve at 1073 K for $La_{0.8}Sr_{0.2}Ga_{0.8}Mg_{0.15}Co_{0.05}O_3$.

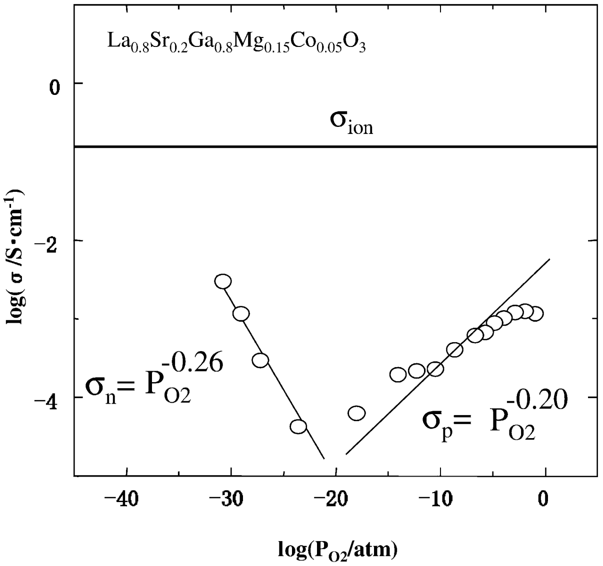

Fig. 10. P_{O_2} dependence of the electron, hole and oxide ion conductivity at 1073 K for $La_{0.8}Sr_{0.2}Ga_{0.8}Mg_{0.15}Co_{0.05}O_3$.

of the electronic conduction according to the following equation:

$$\sigma(P_{O_2}) = L/S(\Delta V/\Delta I_e)$$

where L and S are the thickness of the specimen and the effective electrode area, respectively.

Fig. 10 shows the P_{O_2} dependence of the electronic, hole and oxide ion conductivity. Oxide ion conductivity can be obtained by subtracting the electron or hole conduction from the total conductivity. It is seen that the P_{O_2} dependencies of hole and electron conductivities are close to the power of 1/4 and −1/4, respectively, although the P_{O_2} dependence of hole conduction is slightly smaller than 1/4 power of P_{O_2}. This result is in good agreement with that of LSGM reported by other groups.[16,17] Therefore, the main defect in LSGMC seems to be $V_{\ddot{O}}$ or $h^{\bullet}$ at high oxygen partial pressure and V''_M and e' at low oxygen partial pressure.[16] Here V''_M means cation vacancies at Ga or La site. In the case of LSGM ($La_{0.9}Sr_{0.1}Ga_{0.8}Mg_{0.2}O_3$), the origin of formation of electron has not been identified, however, in the case of LSGMC, the mixed valence of Co at the Ga site may be the main origin of the electronic hole and electron conduction.

Theoretical efficiency of the fuel cell using LSGMC as electrolyte at 1073 K was estimated according to the reports by Kawada[18] and Choudhury[19] and the results are reported in Fig. 11 as a function of the thickness of the electrolyte, 0.3 A/cm^2 being the current density. In the case of $La_{0.9}Sr_{0.1}Ga_{0.8}Mg_{0.2}O_3$, it was reported that the maximum energy conversion efficiency of almost 1.0 is achieved at 1 μm thickness. On the other hand, the maximum electrolyte efficiency was around 0.9 at 300 μm. Therefore, the improved hole conduction by doping the Co causes a decrease in the maximum theoretical conversion efficiency and also decreases the transport number of the oxide ion suggesting that a thicker electrolyte is required for achieving a high energy conversion efficiency. Therefore, a thinner electrolyte film may give a high power density, however, it is not desirable insofar as energy conversion efficiency issues are concerned. Consequently, the optimum thickness of the LSGMC electrolyte for the fuel cell seems to be around 100 μm from the view point of the power density as well as a reasonable energy conversion efficiency. Further detailed study on the measurement of electron or hole conduction is now under investigation and the results will be reported elsewhere.

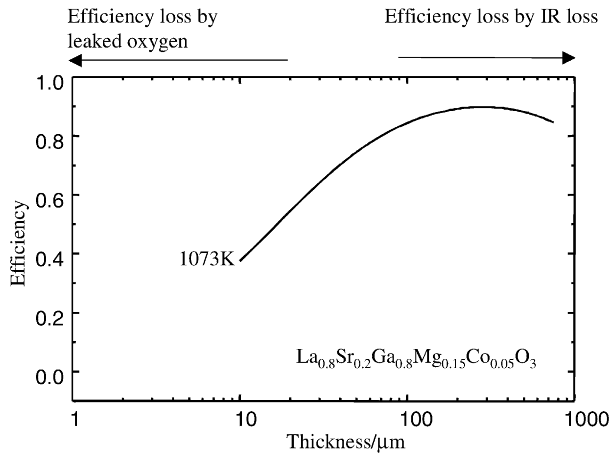

Fig. 11. Theoretical electrolyte efficiency of $La_{0.8}Sr_{0.2}Ga_{0.8}Mg_{0.15}Co_{0.05}O_3$ at 1073 K, 0.3 A/cm^2, as a function of thickness.

4. Conclusions

This study revealed that the use of LSGMC as electrolyte material gives a notably large power density at temperatures as low as 873 K in the solid oxide fuel cell using H_2 and O_2 as fuel and oxidant, respectively. In spite of the 0.186 mm thick electrolyte, the maximum power density was at 1.53 and 0.50 mW/cm^2, respectively. Consequently, this study suggested the possibility that the operating temperature of SOFCs could decrease down to 873 K. It is considered that the energy conversion efficiency of SOFCs will decrease with decreasing operating temperature. However, the application of a high oxide ion conductor is effective in decreasing the operating temperature without decreasing the efficiency. In addition, development of SOFCs power generating systems could further be accelerated by decreasing the operating temperature, since stacking of the cell becomes easier. In particular, operating around 900 K has great potential for the development of SOFC. Namely, reasonably priced metals, such as stainless steel, can be used for constructing the cell stacking and a requirement for an excessively thin electrolyte film can be removed. Considering the temperature of 900 K is close to the lower limit for CH_4 steam reforming, which is considered as the fuel processing reaction. If an SOFC system operable at this temperature range can be developed, the intermediate temperature SOFC would be a highly attractive power generator with a simple structure. From this point of view, a SOFC using LSGMC as electrolyte is highly interesting.

Acknowledgements

The authors acknowledge the financial support from the Grant-in-Aide for Science Promotion (No.11102006) and The Development of Innovative Technology for New Millennium from Ministry of Education, Culture, Sports, Science, and Technology (No.12307).

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