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Effect of voltage and temperature on NO removal with power generation in SOFC with V_2O_5-added LSCF-GDC cathode

Ta-Jen Huang*, Chien-Liang Chou

Department of Chemical Engineering, National Tsing Hua University, 101 Kuang Fu Road, Sec. 2, Hsinchu 300, Taiwan, ROC

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ABSTRACT

A solid oxide fuel cell (SOFC) unit, operating at 800–875 °C, is constructed with Ni-(Ce,Gd)O_{2-x} (GDC) as the anode, yttria-stabilized zirconia as the electrolyte, and V_2O_5-added (LaSr)(CoFe)O_3-GDC as the cathode. Both the current–voltage measurement and the fixed-voltage test were performed on electrochemical NO reduction, i.e. DeNO$_x$, with power generation. As the voltage decreases, the NO conversion increases but only slightly. DeNO$_x$ by SOFC is operational with the O_2 concentration of 2–5% and temperature of 800–875 °C. The NO conversion increases with decreasing O_2 and NO concentrations but with increasing temperature. This process of DeNO$_x$ by SOFC should be suitable for NO removal from the exhaust of lean burn engines.

1. Introduction

The removal of nitric oxide (NO) and nitrogen dioxide (NO_2), i.e. DeNO$_x$, where NO$_x$ denotes NO plus NO_2, from industrial flue gases is an important task to protect our environment. The commercial DeNO$_x$ process is the selective catalytic reduction (SCR), in which both NO and NO_2 react with a reducing agent, usually ammonia, to produce nitrogen and water [1]; however, the residual ammonia can pose a problem and needs additional treatment. Therefore, the electrochemical NO reduction without any reducing agent has been studied extensively [2–7]. However, this process of electrochemical NO reduction is performed with an applied current—that is, DeNO$_x$ with power consumption—whose current efficiency is generally only a few percent [7]. Therefore, DeNO$_x$ by solid oxide fuel cell (SOFC) with power generation can be an environmental-friendly and energy-efficient alternative.

Simultaneous NO reduction and power generation has been shown to be feasible, by the current–voltage measurements, in SOFCs with V_2O_5 or Cu-added (LaSr)(CoFe)O_3-(Ce,Gd)O_{2-x} cathodes [8]—that is, NO can be used as an oxidant to generate electricity in SOFCs. Additionally, DeNO$_x$ by SOFC (DeNO$_x$ with power generation) has been shown to be able to reduce both NO and NO_2 to zero, by the fixed-voltage tests, in SOFCs with Cu-added (LaSr)(CoFe)O_3-(Ce,Gd)O_{2-x} cathodes [9]. However, the effect of operating voltage and temperature has not yet been studied for DeNO$_x$ by SOFC. For electrochemical NO reduction with an applied current, the applied voltage has an effect on NO reduction; additionally, the selectivity for NO reduction is strongly dependent on the temperature, being highest at low temperature [7]. Therefore, the study on the effect of voltage and temperature should be interesting for DeNO$_x$ by SOFC.

Recently, the problem of CO_2 emission has become very critical. The lean burn engines are promising to decrease the fuel consumption for automotive use and thus decrease the CO_2 emission. However, for NO abatement from the exhaust of lean burn engines, an efficient NO reduction at high oxygen concentrations is needed. The process of DeNO$_x$ by SOFC can be useful in this aspect. For the application of DeNO$_x$ by SOFC for the lean burn engines, the understanding on the effects of various operating conditions, such as voltage, temperature, O_2 and NO concentrations, should be useful due to the highly varying conditions of the engine operations.

V_2O_5 is the catalytic component of the commercial SCR catalyst [10]. In this work, this catalytic component was added into the cathode materials of $La_{0.58}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (LSCF)-gadolinia-doped ceria (GDC) composite, which exhibits good cathode performance [11]. LSCF is the well-known material for the cathode in intermediate temperature SOFCs, and adding GDC into LSCF to make the LSCF-GDC composite as the cathode can increase the oxygen reduction activity of the cathode [11].

In this work, DeNO$_x$ was carried out in an electrolyte-supported SOFC unit cell operating at 800–875 °C. The cathode is made of LSCF-GDC composite, with adding 2 wt% V_2O_5. Since NO_2 can be fully converted to NO in an SOFC [9], only NO reduction was studied in this work. The effects of voltage, temperature as well as O_2 and NO concentrations were studied on NO removal with power

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* Corresponding author. Tel.: +886 3 5716260; fax: +886 3 5715408.
E-mail address: tjhuang@che.nthu.edu.tw (T.-J. Huang).

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generation. The results show that the effect of voltage is very small. However, an increase in temperature leads to a considerable increase in the NO conversion; notably, an increase in temperature results in a decrease in the selectivity for NO reduction during electrochemical NO reduction with an applied current [7].

2. Experimental

2.1. Preparation of LSCF and GDC and V_2O_5 addition

LSCF was prepared by the glycine-nitrate process with a composition of $La_{0.58}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$. Appropriate amounts of reagent-grade (Showa, Japan) metal nitrates $La(NO_3)_3 \cdot 6H_2O$, $Sr(NO_3)_2$, $Co(NO_3)_2 \cdot 6H_2O$ and $Fe(NO_3)_3 \cdot 9H_2O$ were dissolved in de-ionized water. Glycine (Sigma, USA) was also dissolved in de-ionized water. Then, these two solutions were mixed together with a glycine to NO_3 ratio of 1:0.8. The mixture was then heated under stirring at 110 °C until combustion occurred. The obtained product was ground to powders. The LSCF powders were calcined by heating to 500 °C and held for 2 h, then to 900 °C and held for 4 h, and then slowly cooled down to room temperature. In this work, the heating was always done in air at a rate of 5 °C/min.

GDC was prepared by the co-precipitation method with a composition of $(GdO_{1.5})_{0.1}(CeO_2)_{0.9}$. The details of the method have been described elsewhere [12]. The GDC powders were calcined by heating to 900 °C and held for 4 h before cooling down.

The LSCF-GDC composite was prepared by mixing the above-prepared LSCF and GDC powders at LSCF:GDC = 100:50 in weight and thus designated as LSCF-50GDC. The mixture was ground for 24 h, then calcined by heating to 500 °C and held for 2 h, and then to 900 °C, held for 10 h before cooling down.

The addition of V_2O_5 to LSCF-50GDC powder was done by mixing V_2O_5 powder (Showa, Japan) with LSCF-50GDC powder in de-ionized water. After drying, the powder was calcined by heating to 500 °C and held for 2 h, and then to 800 °C and held for 2 h before cooling down. The V_2O_5 loading was 2 wt% with respect to LSCF and thus this cathode material is designated as LSCF-50GDC-2V_2O_5.

2.2. Preparation of SOFC unit cell

The commercial yttria-stabilized zirconia (YSZ) tape (Jiuhow, Taiwan) was employed to make an electrolyte-supported SOFC cell. A disk of 1.8 cm in diameter was cut from the YSZ tape. One side of the disk was spin-coated with the paste of Ni-GDC as the anode, being composed of 60 wt% Ni in terms of GDC, which has been shown to have an optimum anode performance [13]. The details of the preparation of the Ni-GDC paste have been described elsewhere [14]. The other side of the disk was spin-coated with a thin interlayer of LSCF-50GDC-2Cu to enhance adhesion and then with LSCF-50GDC-2V_2O_5 as the cathode. The preparation of LSCF-50GDC-2Cu has been described elsewhere [8]. The thus-prepared SOFC unit cell has an anode-layer thickness of 25 μm, an electrolyte-layer thickness of 156 μm, an interlayer thickness of 5 μm, a cathode-layer thickness of 20 μm, and a cathode area of 1.1 cm^2.

2.3. Current–voltage measurement

The measurement of current–voltage curve was performed at 800 °C with pure hydrogen flowing on the anode side and various gaseous mixtures, all balanced by argon, on the cathode side. The rate of gaseous flow was 150 Nml/min if not cited otherwise. The flow rates of anode and cathode gases were always the same. The flow rates of various components in the gaseous mixtures were measured by mass flow meters, respectively, before entering into a mixer. The overall flow rate was measured by a gas bubble meter at the outlet of the experimental setup. The voltage was varied by an adjustable resister, and both the voltage and the current were measured by a Multimeter (TES 2730).

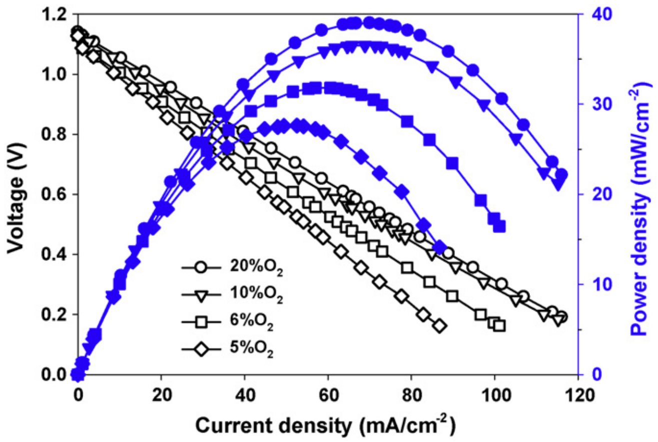

Fig. 1. Variation of voltage–current and power–current profiles with O_2 concentration. Open symbol: voltage; filled symbol: power density.

2.4. Fixed-voltage test

The fixed-voltage tests were performed at 800 °C with the operating voltage kept constant at 0.2, 0.5 and 0.8 V, respectively, for the study on the effect of voltage. For the study on the effect of temperature, the tests were carried out at 800–875 °C, respectively, and a constant voltage of 0.5 V. The anode gas was pure hydrogen. The inlet cathode gas was a mixture of O_2 and/or NO, balanced in argon, as designated in the figure and table legends.

The tests were conducted with introducing a designated gas mixture to the cathode side of the SOFC unit cell. After a steady state was obtained for over 60 min, the cathode side was purged with argon and a new designated gas mixture was introduced or the temperature was increased to a higher one. Throughout the test, the electrical current, the voltage, and the outlet gas compositions were always measured. The NO content in the outlet cathode gas was measured by an NO analyzer (NGA 2000, Emerson, Germany). The uncertainty or error on NO measurements was smaller than ±10 ppm.

3. Results and discussion

3.1. Electrochemical NO reduction with current–voltage variation

Fig. 1 shows the variation of voltage–current and power–current profiles with O_2 concentration of 5–20%. The open circuit voltage is close to the theoretical one; thus, there should be no leakage in the SOFC unit cells of this work. When the O_2 concentration decreases from 20% to 5%, the generated voltage decreases; the extent of this decrease becomes larger as the current density increases. This may be attributed to a mass transfer effect [15]. Notably, the mass transfer resistance increases with increasing reaction rate until the overall reaction rate becomes mass transfer controlled [16]. In this work, the electrochemical reaction rate is represented by the current density. Notably, also, an increase of the current density is an increase of the rate of electrochemical O_2 reduction over the cathode and that of subsequent migration of the O species from the cathode to the anode, where the O species is consumed by the reaction of fuel oxidation. A decrease of the O_2 concentration results in a decrease of the rate of electrochemical O_2 reduction. The overall effect of the decrease of O_2 concentration on the SOFC performance is shown by a decrease of the maximum power density.

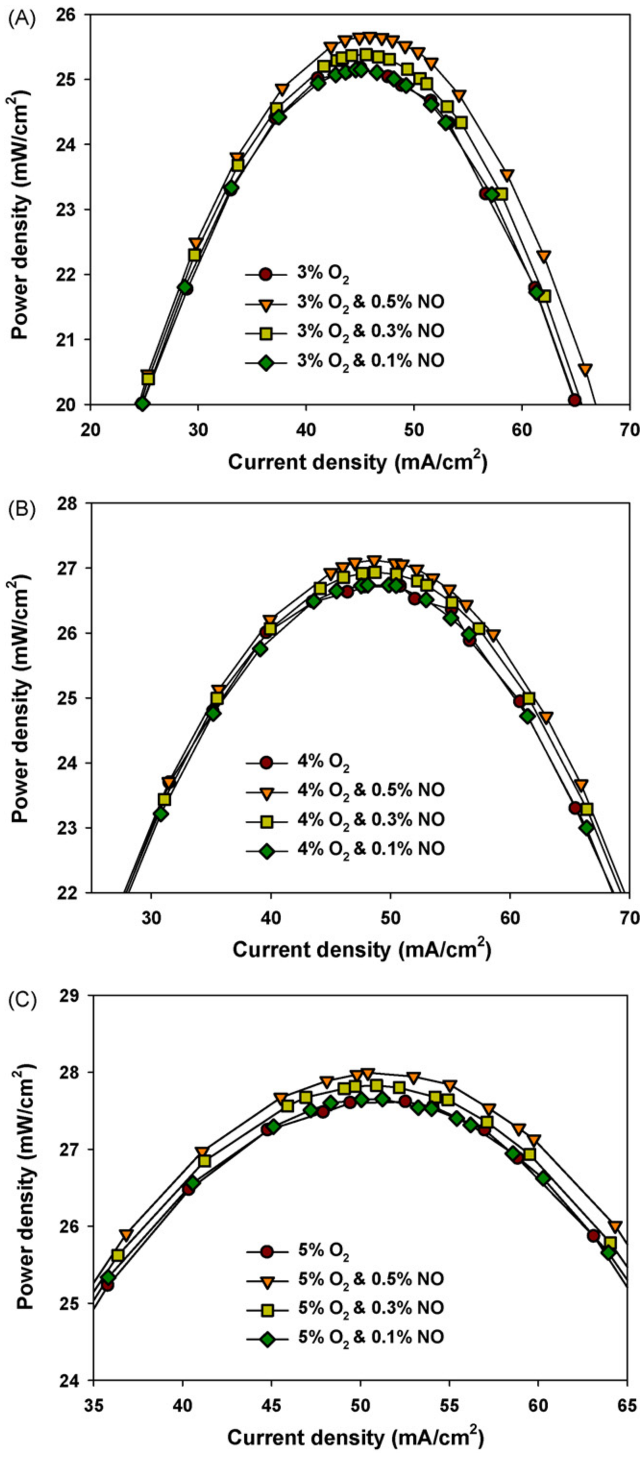

Fig. 2. Variation of power–current profile with O_2 concentration of (A) 3%, (B) 4% and (C) 5% plus various concentrations of NO.

Fig. 2 shows the variation of power–current profile with the O_2 concentration of 3–5% and with the NO concentration varying from 0.1% to 0.5%. As the O_2 concentration decreases from 5% to 3%, the power density decreases. However, the power density always increases with adding NO—that is, an amount of power density is added due to the addition of NO into the gaseous mixture with O_2 as the cathode gas. Additionally, the amount of added power density becomes larger with higher NO concentration at the same O_2 concentration. Thus, the added power density should indicate the occurrence of electrochemical NO reduction. The added amount of maximum power density (MPD) is listed in Table 1; notably, this added amount equals (MPD with O_2 + NO-MPD with O_2) at the same O_2 concentration. This added power density decreases with increasing O_2 concentration.

Table 1
Variation of the added amount of maximum power density with O_2 concentration of 3–5% plus various concentrations of NO.

Cathode gas	Added amount of maximum power density (mW/cm^2)
3% O_2 + 0.1% NO	0.02
3% O_2 + 0.3% NO	0.23
3% O_2 + 0.5% NO	0.51
4% O_2 + 0.1% NO	0.02
4% O_2 + 0.3% NO	0.22
4% O_2 + 0.5% NO	0.40
5% O_2 + 0.1% NO	0.01
5% O_2 + 0.3% NO	0.21
5% O_2 + 0.5% NO	0.37

Table 2 shows that, with the cathode gas being a gaseous mixture of 2–6% NO in Ar, there is a maximum power density—that is, there is a generation of an electrical current. This indicates the occurrence of NO dissociation to produce the adsorbed N and O species:

$$NO \rightarrow N + O \tag{1}$$

Followed by the charge transfer reaction to form the oxygen ion:

$$O + 2e^- \rightarrow O^{2-} \tag{2}$$

Notably, an electrical current is generated when the oxygen ion migrates from the cathode to the anode. Notably, also, the oxygen ion can be formed when there is a supply of the O species, indiscriminating the source of the O species. This directly verifies the occurrence of electrochemical NO reduction:

$$NO + 2e^- \rightarrow N + O^{2-} \tag{3}$$

Additionally, the maximum power density increases with increasing NO concentration; this is the same trend as that indicated by the above-observed increase of the added power density with increasing NO concentration in the presence of oxygen.

Fig. 2 also shows that the major driving force for electrochemical NO reduction, i.e. DeNO$_x$ by SOFC, is the O_2 content. This may be attributed to the fact that the migration of the O species from the cathode to the anode is one step in the overall process of electrochemical NO reduction. When the rate of electrochemical O_2 reduction increases with increasing O_2 concentration, the concentration of the O species on the cathode side increases and thus the migration rate of the O species from the cathode to the anode increases; this may help the migration of the O species generated from electrochemical NO reduction from the cathode to the anode and thus increase the rate of NO dissociation.

3.2. *Effect of operating voltage*

Table 3 shows the steady state values of current density, power density, NO conversion and NO consumption varying with the operating voltage, the O_2 and NO concentrations during the fixed-voltage tests. Notably, these values became constant, i.e. steady state, very quickly after the start of each fixed-voltage test. As the

Table 2
Variation of maximum power density with 100 Nml/min of 2–6% NO in Ar as the cathode gas at 800 °C.

Cathode gas	Maximum power density (mW/cm^2)
2% NO	0.577
4% NO	0.767
6% NO	1.053

Table 3
Variation of current density, NO conversion and NO consumption with voltage, O_2 and NO concentrations during fixed-voltage tests at 800 °C.

Cathode gas	Voltage (V)	Current density (mA/cm^2)	Power density (mW/cm^2)	NO conversion (%)	NO consumption (ppm)
5% O_2 + 0.5% NO	0.8	27.5	22.0	3.9	195
	0.5	55	27.5	4.15	207.5
	0.2	88	17.6	4.25	212.5
5% O_2 + 0.3% NO	0.8	27.2	21.8	6.32	189.6
	0.5	55	27.5	6.46	193.8
	0.2	82.8	16.6	6.66	199.8
5% O_2 + 0.1% NO	0.8	26.87	21.5	13.94	139.4
	0.5	54	27	14.04	140.4
	0.2	82.7	16.5	14.16	141.6
4% O_2 + 0.5% NO	0.8	26.94	21.6	5.1	255
	0.5	53.52	26.8	5.13	256.5
	0.2	79.48	15.9	5.22	261
4% O_2 + 0.3% NO	0.8	26.93	21.5	7.84	235.2
	0.5	53.0	26.5	8.06	241.8
	0.2	79.14	15.8	8.18	245.4
4% O_2 + 0.1% NO	0.8	26.67	21.3	19.13	191.3
	0.5	53.0	26.5	20	200
	0.2	78.71	15.7	21.46	214.6
3% O_2 + 0.5% NO	0.8	25.4	20.3	6.21	310.5
	0.5	50.4	25.2	6.46	323
	0.2	74	14.8	6.56	328
3% O_2 + 0.3% NO	0.8	25.4	20.3	10.11	303.3
	0.5	49.4	24.7	10.35	310.5
	0.2	74	14.8	10.42	312.6
3% O_2 + 0.1% NO	0.8	24.83	19.9	23.82	238.2
	0.5	49.27	24.6	23.87	238.7
	0.2	73.66	14.7	24.87	248.7
2% O_2 + 0.5% NO	0.8	24.2	19.4	7.93	396.5
	0.5	46.1	23.1	8.1	405
	0.2	65.8	13.2	8.13	406.5
2% O_2 + 0.3% NO	0.8	23.95	19.2	13.04	391.2
	0.5	46.1	23.1	13.27	398
	0.2	65.14	13.0	13.33	400
2% O_2 + 0.1% NO	0.8	23.16	18.5	29.8	298
	0.5	44.63	22.3	30.18	301.8
	0.2	64.4	12.9	31.14	311.4

operating voltage decreases from 0.8 to 0.2 V, the NO conversion always increases but only slightly, noting that the current density increases largely with decreasing voltage. However, the power density is at its maximum at an intermediate voltage of around 0.5 V. Therefore, the increase of the NO conversion may be attributed to the increased current density—that is, the amount of NO consumption increases with increasing current density. This increase of the NO conversion may also be attributed to a synergistic effect between NO and O_2 reduction as shown by Huang and Chou [8], who have reported that, over Cu-added LSCF-GDC cathode, adding 2% NO into O_2 resulted in a maximum power density which is always larger than the sum of the maximum power density with O_2 and that with 2% NO; additionally, this synergistic effect increases with increasing O_2 concentration from 3% to 5%.

The above results show that the power density is at maximum with an intermediate operating voltage. Thus, a lower operating voltage is beneficial for the NO conversion but an intermediate operating voltage is needed to achieve a maximum power generation. Since the effect of the operating voltage on the NO conversion is very small, an intermediate operating voltage may be the choice for DeNO$_x$ by SOFC (DeNO$_x$ with power generation).

Table 3 also shows that, as the O_2 concentration decreases from 5% to 2%, the NO conversion also increases, noting that the current density decreases with decreasing O_2 concentration. With decreasing O_2 concentration at the same NO concentration, the trend of increase in the NO conversion during the fixed-voltage test is the same as that in the added power density during the current–voltage measurement. This is attributed to a decreased extent of the competition between NO and O_2 adsorption onto the active sites over the cathode. This also indicates that NO and O_2 adsorption can occur over the same active site and thus a competition for the active site may occur.

The NO conversion also increases always as the NO concentration decreases from 0.5% to 0.1% at the same voltage and O_2 concentration, noting that the current density decreases very slightly or even is the same under these conditions. Notably, the increase of the NO conversion with the same current density would be at the expense of electrochemical O_2 reduction. The increase of the NO conversion with decreasing NO concentration is attributed to the fact that the amount of the active sites available for NO reduction should be about the same if the effect of competition between NO and O_2 does not vary much under these conditions. Thus, a lower NO concentration means that a larger portion of the NO species can be adsorbed and converted; this leads to a larger NO conversion. This is beneficial for the treatment of trace amount of NO in the exhausts.

For NO$_x$ removal from the exhaust of lean burn engines, the operating conditions may vary frequently. For the application of the SCR process, the amount of the reducing agent may not be able to match the varying NO concentration; if excess amount of the reducing agent is used, the residual of the reducing agent, especially ammonia, may cause a problem. However, the varying operating

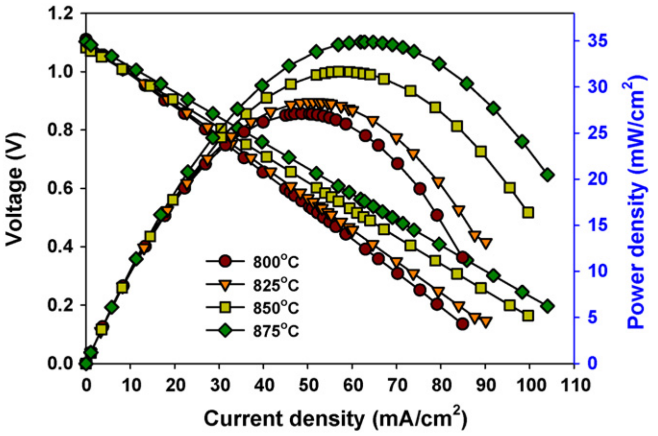

Fig. 3. Variation of voltage–current and power–current profiles with temperature. The cathode gas was 4% O_2 and 0.5% NO in Ar.

conditions should be not a problem in the application of DeNO$_x$ by SOFC; this is due to the fact that the major driving force for DeNO$_x$ by SOFC is the O_2 content; additionally, the variation in the operating voltage has only very small effect on the NO conversion, as shown in Table 3. For a lean burn engine, the NO$_x$ concentration in the exhaust is around 300–1000 ppm [17]; notably, with Cu-added LSCF-GDC as the SOFC cathode, this NO content can be easily reduced to zero [9]. This makes the process of DeNO$_x$ by SOFC suitable for NO removal from the exhaust of lean burn engines.

3.3. *Effect of temperature*

Fig. 3 shows the variation of voltage–current and power–current profiles of 4% O_2 and 0.5% NO with temperature. The maximum power density increases with increasing temperature. Fig. 4 shows the variation of NO consumption with temperature during the fixed-voltage tests with 4% O_2 and 0.5% NO. The NO consumption increases as the operating temperature increases. With increasing temperature, the trend of increase in the NO consumption is the same as that in the maximum power density. This indicates that the increase in the maximum power density from the current–voltage measurement is associated with that in the NO consumption from the fixed-voltage test. Therefore, the result from the current–voltage measurement is useful on studying the effect of the operating temperature. However, for the steady state behavior of NO reaction, the fixed-voltage test should be used.

Table 4 shows the variation of the steady state values of the NO consumption and the NO conversion with temperature and NO concentration at 4% O_2. As the NO concentration decreases from 0.5% to 0.1%, the NO consumption decreases only slightly. Notably, the amount of the active sites for electrochemical NO reduction should be about the same. The slight decrease of the NO consumption is due to the lower NO concentration which should result in a smaller reaction rate. Consequently, the NO conversion increases dramatically with decreasing NO concentration from 0.5% to 0.1%.

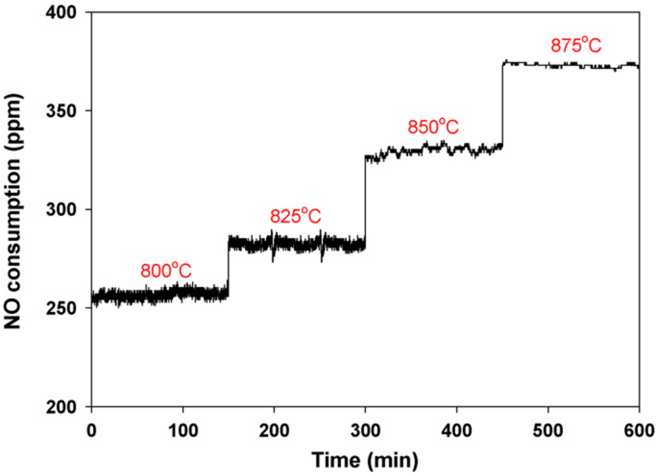

Fig. 4. Variation of NO consumption with temperature during fixed-voltage tests at 0.5 V with cathode gas of 4% O_2 and 0.5% NO in Ar.

Table 4 also shows that the extent of increase in the NO conversion with decreasing NO concentration increases with increasing temperature. However, when the NO concentration increases from 0.3% to 0.5%, this extent is smaller than that of NO concentration increased from 0.1% to 0.3%. This is attributed to the fact that the amount of the active sites for NO reduction should be about the same as discussed in the above. When the operating temperature increases, the oxygen ion conductivity of the materials increases and thus the rate of migration of the O species from NO dissociation increases; this can increase the NO conversion. Notably, the rate of NO dissociation can increase with increasing NO concentration. However, the limit on the amount of the active sites for NO adsorption should put a limit on the extent of increase in the rate of NO dissociation. Therefore, the increasing trend on the rate of electrochemical NO reduction should decrease with increasing NO concentration.

The increase of NO conversion with increasing temperature is beneficial for NO removal from the exhaust of lean burn engines, where the operating condition may be highly varying. During the variation of operating condition which demands a varying NO conversion, the variation of the operating temperature in the process of DeNO$_x$ by SOFC, such as by electrical heating, should be easier than that of the amount of reducing agent in the SCR process. This

Table 4
Variation of NO consumption and NO conversion with temperature and NO concentration at a fixed-voltage of 0.5 V.

Temperature (°C)	Cathode gas	NO consumption (ppm)	NO conversion (%)
800	4% O_2 + 0.5% NO	256.5	5.13
	4% O_2 + 0.3% NO	241.8	8.06
	4% O_2 + 0.1% NO	200	20
825	4% O_2 + 0.5% NO	282.2	5.64
	4% O_2 + 0.3% NO	272.8	9.09
	4% O_2 + 0.1% NO	232	23.2
850	4% O_2 + 0.5% NO	328.8	6.58
	4% O_2 + 0.3% NO	325	10.83
	4% O_2 + 0.1% NO	300.7	30.07
875	4% O_2 + 0.5% NO	373	7.46
	4% O_2 + 0.3% NO	370.2	12.34
	4% O_2 + 0.1% NO	359	35.9

also makes the process of $DeNO_x$ by SOFC suitable for NO removal from the exhaust of lean burn engines.

The increase of NO conversion with increasing temperature is somewhat contrast to that of electrochemical NO reduction with applied voltage, where an increase in temperature results in a decrease in the NO selectivity [7]. This is attributed to the fact that the major driving force for $DeNO_x$ by SOFC ($DeNO_x$ with power generation) is the O_2 content while that for electrochemical NO reduction with an applied current ($DeNO_x$ with power consumption) is the applied voltage; consequently, a higher temperature would increase the generation of the electrical current during $DeNO_x$ by SOFC but its consumption during electrochemical NO reduction with an applied current. Therefore, the process of $DeNO_x$ by SOFC is very energy-efficient; the NO conversion contributes to power generation; notably, the anode fuel is consumed only when the oxygen ion is transported to the anode to carry out the electrochemical oxidation to generate an electrical current.

4. Conclusions

(1) As the operating voltage decreases, the NO conversion always increases but only slightly.
(2) An intermediate operating voltage is needed to achieve a maximum power generation.
(3) The major driving force for $DeNO_x$ by SOFC ($DeNO_x$ with power generation) is the O_2 content.
(4) The NO conversion increases with decreasing O_2 and NO concentrations but with increasing temperature.
(5) The extent of increase in the NO conversion with decreasing NO concentration increases with increasing temperature.

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