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From rare earth doped zirconia to 1 kW solid oxide fuel cell system

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Abstract

Toho Gas R&D efforts and achievements regarding solid oxide fuel cells (SOFCs) from rare earth doped zirconia to 1 kW SOFC system are described in this article. In this research, the use of Sc_2O_3–ZrO_2 (scandia stabilized zirconia, ScSZ), which has the highest oxide ion conductivity among the zirconia systems, was investigated as an electrolyte for SOFCs. To improve the performance and mechanical reliability of SOFC cells and stacks, tetragonal phase ScSZ ceramic sheets were developed as electrolytes for SOFCs. They have flexibility, high-strength and twice the conductivity of a Y_2O_3–ZrO_2 (Yttria stabilized zirconia, YSZ) system. Because of this electrolyte, the SOFC operating temperature was reduced to 1073 K. Also, to validate the performance of this cell, the first Japanese-made 1 kW SOFC system was fabricated, and its carefully designed internal manifold type 68-cell stack produced an output of over 1 kW (DC) at 1073 K under thermal self-sustaining conditions.

Keywords: Solid oxide fuel cell; Zirconium oxide; Scandium oxide

1. Introduction

Rare earth doped zirconia is popularly used for solid-state ionic materials such as electrolytes for solid oxide fuel cells (SOFCs). The SOFC is a promising technology for environmental solutions because of its high electric conversion efficiency and wide choice of potential fuels. In addition, the SOFC electrodes do not need a platinum-based catalyst because the SOFC unit cells are made entirely of ceramics, which include many types of rare earth elements. In an electrolyte-supported SOFC, the ohmic resistance of the electrolyte is the most significant portion of internal resistance. Therefore, a highly conductive electrolyte is expected to improve the power density and reduce the operating temperature of SOFCs. Recent R&D has tended to focus on reduced-temperature SOFCs because of their cost advantages (since they use low-cost metallic interconnect plates) and their thermal cycle abilities. From the standpoint of reducing the operating temperature from 1273 K to around 1073 K, alternative electrolyte materials with higher conductivities (i.e., doped CeO_2 and $LaGaO_3$) and thin film electrolyte cells have been examined and reported [1–6]. However, these cells do not have enough mechanical reliability because of their low mechanical strength and lack of stability in the microstructure of the Ni-based substrates after the reduced and oxidized atmospheres (RedOx) cycle. Sc_2O_3–ZrO_2 (scandia stabilized zirconia, hereinafter describe as ScSZ), which has a higher ionic conductivity than Y_2O_3–ZrO_2 (Yttria stabilized zirconia, hereinafter describe as YSZ), is one of the candidate materials for reduced-temperature SOFCs [7–13]. In this article, basic properties of ScSZ electrolytes in SOFC applications, such as ionic conductivity, mechanical properties, and electrochemical performance of the cells, are described. Since tetragonal phase ScSZ, which has extremely high mechanical strength and fracture toughness, is particularly effective in improving the reliability of SOFCs [14–16], tetragonal phase ScSZ electrolyte sheets and electrolyte-supported type cells were designed for reduced-temperature operation at around 1073 K. Also, in order to validate the performance of these tetragonal-ScSZ cells in stack conditions and system

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conditions, a 1 kW class SOFC system was fabricated and examined.

2. Design, fabrication and experiment

2.1. Fabrication of materials and single cells

The specifications of component materials of the SOFC cells used for the 1 kW system are shown in Table 1. The stabilized ZrO_2 (ScSZ, YSZ) powders were made by the co-precipitation method by Daiichi Kigenso Kagaku Kogyo Co., Ltd. (DKKK). The perovskite cathode powders (LSCF, LSM) were supplied by Seimi Chemical Co., etc. Where, the LSCF is $La_{1-x}Sr_xCo_{1-y}Fe_yO_3$ and LSM is $La_{1-x}Sr_xMnO_3$. The powders for the Ni–ScSZ cermet anode were prepared by mechanical ball milling of NiO powders and ScSZ powders with predetermined weight ratios [17,19]. In that process, the NiO in the anode was reduced to Ni by hydrogen fuel in situ SOFC operating conditions. Sintered ScSZ specimens for conductivity measurement and strength measurement were prepared by cold isostatic pressing and sintering. The mechanical strength of the sintered samples was measured by a three-point bending test using rectangular bar specimens 3 mm (B) × 4 mm (W) × 40 mm (L) at room temperature. The span was 30 mm and the cross-head speed was 0.5 mm/min. The electrolyte sheets were made by forming a ScSZ slurry in sheets using the doctor blade tape-casting method. Then, a binder was carefully pyrolyzed and fired at around 1673 K. Dense 4ScSZ (4 mol % Sc_2O_3–96 mol % ZrO_2) ceramic sheets were manufactured according to cell design values by Nippon-Shokubai Co., and they were inspected for dimensional accuracy and for the existence of defects (visible pores or cracks). Porous Ni–ScSZ cermet anodes and LSCF ($La_{1-x}Sr_xCo_yFe_{1-y}O_3$, x = 0.2–0.4, y = 0.5–0.8) cathodes were prepared on the electrolyte substrates with the screen printing method (where the electrode thickness was checked by measuring the weight changes before and after screen printing) followed by the firing at 1423–1673 K.

2.2. Electrochemical measurement

Electrical conductivity was measured using the AC impedance method with platinum paste electrodes. The frequency range was 0.1 Hz to 100 kHz Basic cell performance was evaluated with small cells with an electrode area of 0.2 cm^2. Pure hydrogen humidified at room temperature was used as fuel gas and air as oxidant gas. The flow rate of both gases was 0.1 L/min. Electrode characteristics were evaluated by the AC impedance method using a reference electrode on each side of the electrolyte.

Fig. 1. Planar type 1 kW SOFC stack with 68 cells prepared for 1 kW SOFC system.

2.3. Design and fabrication of SOFC stack and system

The planar type SOFC stack was designed with 68 cells and ferritic stainless steel (ZMG232, Hitachi metals Co.) interconnecting plates with gas manifolds. Figure, dimensions and electrode area of single cells are disk with center holes, outer diameter of 12 cm, and active electrode area of 67 cm^2, respectively. The fuel gas/air flow was a radial co-flow obtained with coaxial gas manifolds. Excess fuel and air were mixed and burned in the outer edge of the stack. Fig. 1 shows the compact planar cell stack, which has a volume of 2 l/1 kW. As shown in Fig. 2, the SOFC system was designed as a proof-of-concept system for future small-scale cogeneration systems with simplified construction, and it was fabricated by Sumitomo Precision Products Co. (SPP). The design specifications are shown in Table 2. A compact catalytic partial oxidation (CPOx) reformer was used for the fuel reforming of natural gas.

Table 1
Specifications and fabrication process of component materials for SOFC cells used for 1 kW stack and system test

Component	Materials	Thickness (μm)	Fabrication process
Electrolyte	4ScSZ (4 mol %Sc_2O_3–96 mol % ZrO_2)	100	Co-preciptation powder, tape casting/sintering
Anode	Ni–10Sc1CeSZ (composite of Ni and 10 mol %Sc_2O_3–1 mol %CeO_2–89 mol % ZrO_2)	50	Mechanical mixed powder, screen printing/sintering
Cathode	LSCF ($La_{1-x}Sr_xCo_{1-y}$ Fe_yO_3, x = 0.2–0.4, y = 0.5–0.8)	30	Supplied powder, screen printing/sintering

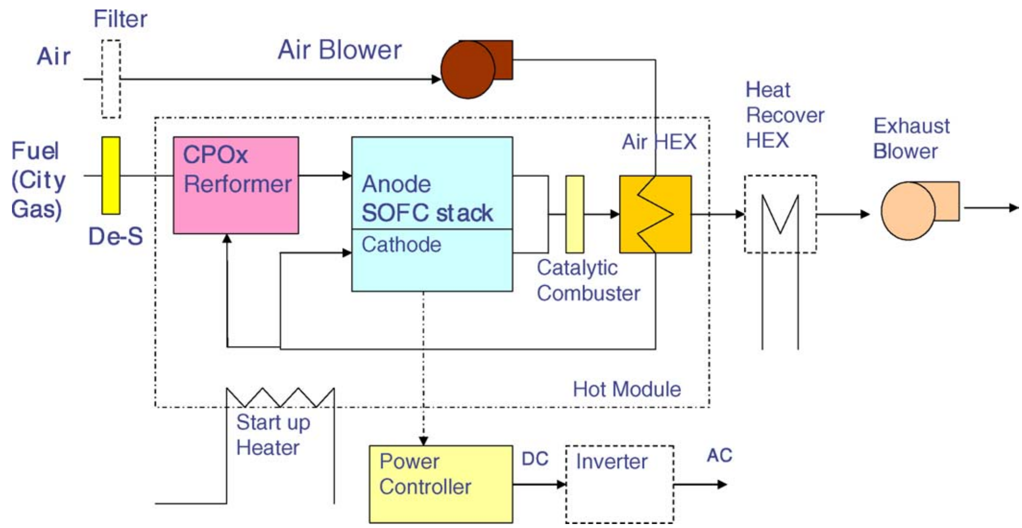

Fig. 2. Schematic diagram of 1 kW SOFC system for proof-of-concept test.

3. Results and discussion

3.1. Properties of Sc_2O_3–ZrO_2 (ScSZ) electrolytes for SOFC applications

Generally, the crystal phase of rare earth doped zirconia changes with the dopant ratio and temperature. The observed crystal phase in the Sc_2O_3–ZrO_2 system also changed with the Sc_2O_3 content. The Sc_2O_3–ZrO_2 samples containing less than 4 mol % Sc_2O_3 were a mixture of monoclinic (m-) and tetragonal (t-) phases, and they were not suitable for SOFC applications because a large amount of m-phase decreases conductivity and mechanical strength. Full t-phase ceramics are obtained by the addition of 4 mol % Sc_2O_3, and that dopant amount is a little larger than that of the Y_2O_3–ZrO_2 system. Well-controlled t-phase Sc_2O_3–ZrO_2 ceramics have high mechanical strength, fracture toughness, and flexibility, as shown in Fig. 3. The maximum mechanical strength of 1000 MPa was obtained around the 3–4 mol % Sc_2O_3 as shown in Fig. 4. From 4 to 7 mol % Sc_2O_3, a mixture of t-phase and cubic (c) phase was observed, and the c-phase increased with the dopant ratio. In this region, conductivity increased with the dopant ratio as shown in Fig. 5(a). In the region above 11 mol % Sc_2O_3, a rombohedral (r-) phase was observed at room temperature, and significant conductivity

Table 2
Design and specifications of 1 kW SOFC system

Item	Specifications
Output	1 kW DC
Operating temperature of SOFC stack	1073 K
Fuel	Natural Gas 4 L/min (or hydrogen 12 L/min)
Air	150 L/min
SOFC stack	12 cm diameter 4ScSZ cell 68 cells
Reformer	Catalytic partial oxidation
Dimensions	800 (*W*) × 600 (*D*) × 1400 (*H*)

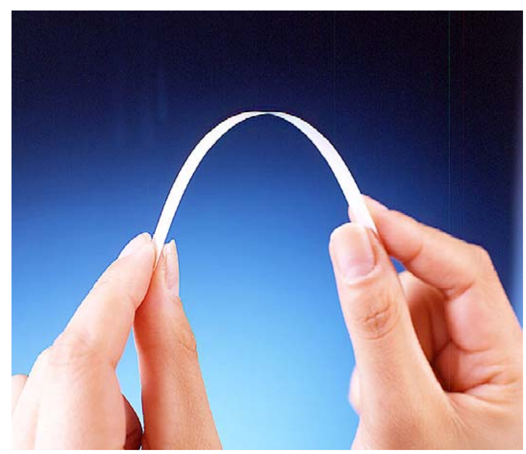

Fig. 3. Flexible scandia stabilized zirconia (ScSZ) ceramics sheet with a composition of 4 mol %Sc_2O_3–96 mol %ZrO_2.

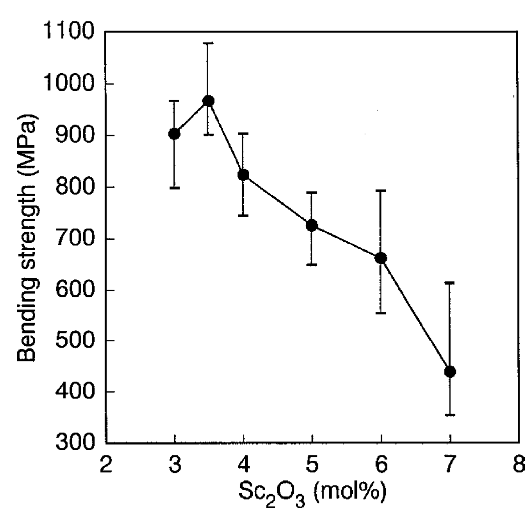

Fig. 4. Relation between dopant ratio and room-temperature bending strength of Sc_2O_3 in Sc_2O_3–ZrO_2 (scandia stabilized zirconia, ScSZ) ceramics.

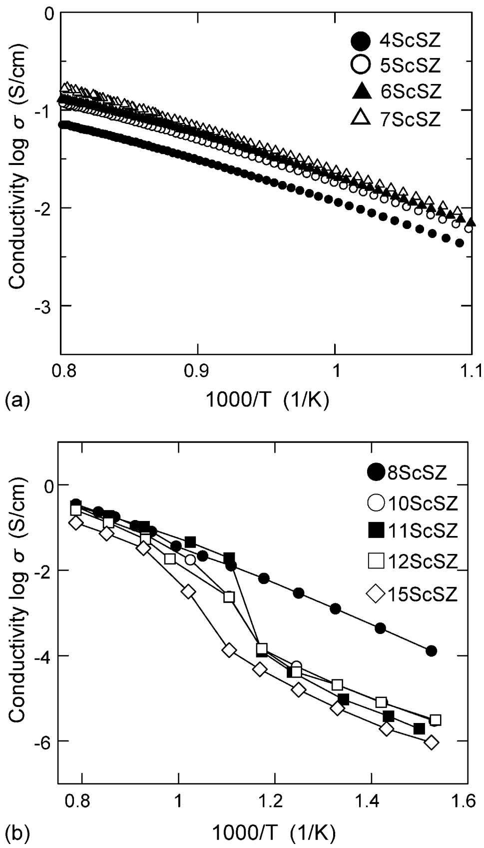

Fig. 5. Temprature dependence of conductivity for several dopant ratios of ScSZ: (a) for 4 mol % (4ScSZ) to 7 mol % Sc_2O_3 (7ScSZ) specimens (mainly tetragonal phase and cubic phase) and (b) for 8 mol % (8ScSZ) to 15 mol % Sc_2O_3 (15ScSZ) specimens (mainly cubic phase and rombohedral phase).

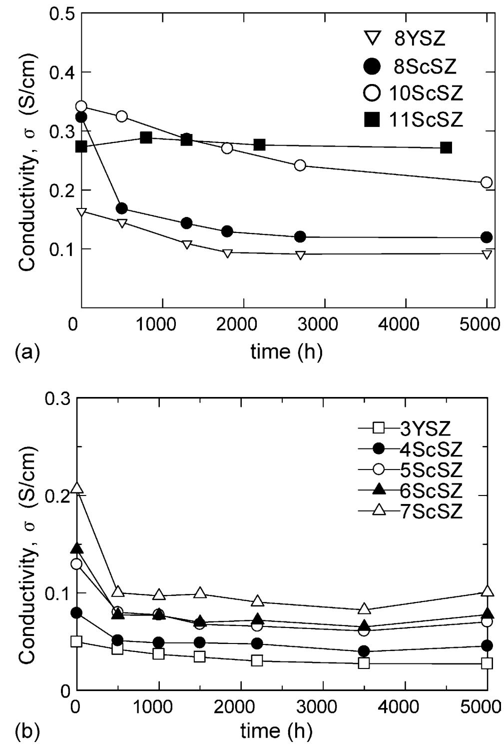

Fig. 6. Changes in electrical conductivity of Sc_2O_3–ZrO_2 (scandia stabilized zirconia, ScSZ) and Y_2O_3–ZrO_2 (yttria stabilized zirconia, YSZ) as a function of annealing period at 1273 K: (a) for cubic phase and rombohedral phase specimens and (b) for tetragonal phase and cubic phase specimens.

changes were observed at the phase transition temperature around 900 K, as shown in Fig. 5(b). Also, phase transformations are not suitable for SOFC applications because of their sudden thermal expansion and mechanical failure, so many researchers have tried to inhibit this cubic-rombohedral phase transformation by addition of second dopants. As second dopants, Al_2O_3, La_2O_3, Y_2O_3, Yb_2O_3, CeO_2 Gd_2O_3, etc. have been reported [9,10,18–20].

To achieve long-life SOFC cells, the aging effect on the conductivity change of the zirconia system should be investigated. Fig. 6(a) shows the changes in electrical conductivity of 8YSZ, 8ScSZ, 10ScSZ and 11ScSZ as a function of the annealing period at 1273 K. In the initial several hundred hours of the annealing period, 8ScSZ showed a significant conductivity decrease, and 8YSZ showed a conductivity decrease up to 2000 h. The electrical conductivity of these materials annealed for 5000 h, recovered compared to those of the as-sintered samples by re-sintering at 1873 K. On the other hand, 11 mol % samples of 11ScSZ showed no significant decrease in conductivity due to annealing at 1273 K, indicating that they would be suitable materials for use in SOFCs. It is well known that electrical conductivity has an aging phenomenon on stabilized zirconia, and many aging mechanisms, such as phase stability, ordering of the crystal lattice, and segregation of the impurities at the grain boundaries, have been proposed. Nomura et al. conducted a Raman scattering study for annealed ScSZ samples, reporting that decreased conductivity in the ScSZ system due to annealing is caused by cubic to tetragonal phase changes [21]. This result suggests that the full c-phase ScSZ has the largest degradation ratio and full t-phase ScSZ has no degradation. Fig. 6(b) shows the aging characteristics of 4ScSZ (full t-phase)–7ScSZ (mixture of t-phase and c-phase). As expected, 7ScSZ showed the largest degradation ratio in conductivity and 4ScSZ showed the smallest. However, 4ScSZ still showed a little aging effect, which suggested the presence of another aging mechanism for this composition. Muller et al. also reported on the long-term stability of 8YSZ (c-phase), 3YSZ (t-phase) and 4ScSZ (t-phase) [22]. They found that sintering conditions affect the degradation ratio, and that constant heating rate/rate controlled sintering (RCS) was effective in improving the stability of 8YSZ but not that of 3YSZ and 4ScSZ. These results also suggest that another aging mechanism should be considered for tetragonal phase zirconia electrolytes.

3.2. *Electrochemical performance of Sc_2O_3–ZrO_2 (ScSZ) in SOFC applications*

The electrical conductivity of ScSZ (c-phase and t-phase) is almost twice that of Yttria stabilized zirconia, which is

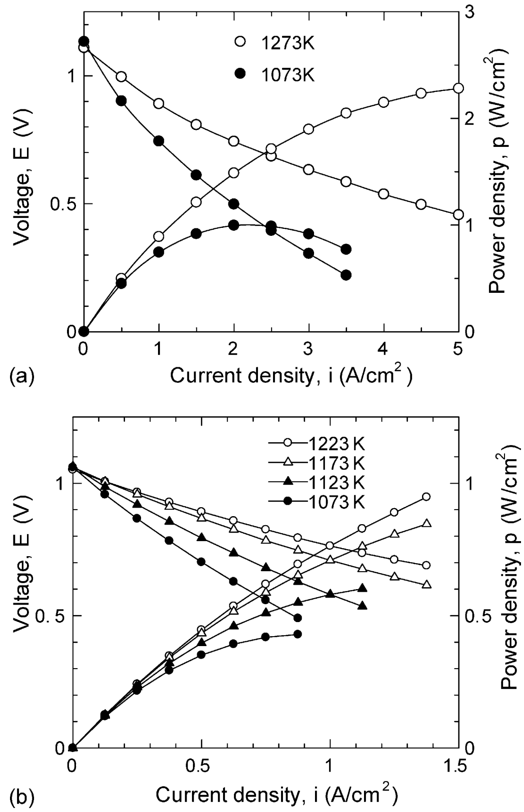

Fig. 7. *I–V* and *I–P* characteristics of electrolyte-supported cells with Sc_2O_3–ZrO_2 (scandia stabilized zirconia, ScSZ) electrolytes at several operating temperatures: (a) for cubic phase ScSZ cell and (b) for tetragonal phase ScSZ cell.

conventionally used in SOFCs. Therefore, power density improvement and the possibility of reducing the operating temperature of SOFCs have been reported [9,11]. Fig. 7 shows the *I–V* characteristics of electrolyte-supported type cells using c-ScSZ [Fig. 7(a)] and t-ScSZ [Fig. 7(b)]. The thickness of the electrolytes was 250 and 140 μm, respectively. As shown in Fig. 7(a), an extremely high power densities of 2.3 W/cm^2 at 1273 K and 1.0 W/cm^2 at the reduced operating temperature 1073 K were achieved with the c-ScSZ cell. These performance figures were almost twice those of the 8YSZ cells. Also, as shown in Fig. 7(b), power densities of 0.95 W/cm^2 at 1223 K and 0.4 W/cm^2 at 1073 K were obtained with the t-ScSZ (4ScSZ) cell. These performance results indicate that ScSZ electrolyte-supported cells can be used in actual SOFC applications even at 1073 K.

On the other hand, thin-film electrolyte SOFCs with YSZ electrolytes have been developed in order to reduce the operating temperature [5,6]. Generally, if an alternative electrolyte is used, cell performance does not seem to improve with a thin-film electrolyte SOFC because the electrolyte resistance is a small part of the whole cell resistance. However, recent research has reported that the conductivity of the electrolyte influences the SOFC electrode characteristics [23]. Therefore, ScSZ can also be used in thin-film electrolyte SOFCs. We confirmed that the performance of an anode-supported type thin-film electrolyte SOFC – as well as that of an electrolyte-supported type SOFC – was improved by using a ScSZ electrolyte, compared with a YSZ electrolyte [24]. Fig. 8 shows the performance of our latest anode-supported thin-film electrolyte cell, which uses a 4ScSZ electrolyte with a thickness of 20 μm and two types of cathode materials [25,26]. The maximum power density of 2.4 W/cm^2 at 1073 K that was obtained with an LSCF cathode was over 4 times as high as that of an electrolyte-supported ScSZ cell. This extremely high performance is very attractive for SOFC applications, but important problems to be solved are the mechanical durability and reduced and oxidized cycle stability of the anode substrates.

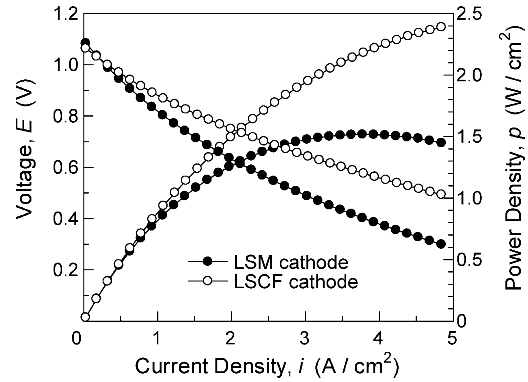

Fig. 8. *I–V* and *I–P* characteristics of anode-supported type thin-film electrolyte cell using Sc_2O_3–ZrO_2 (scandia stabilized zirconia, ScSZ) electrolyte with different cathode materials.

The application of ScSZ in a composite electrode was also examined. It has been reported that the overpotential of a Ni–ScSZ anode was lower than that of a Ni–YSZ anode in methane reforming conditions, and that the Ni–ScSZ anode showed a stable performance at very low steam/carbon (S/C) ratios [27,28]. Fig. 9 shows the changes in output voltage of cells with a Ni–ScSZ anode and with a Ni–YSZ anode in methane fuel at 1273 K. The S/C ratio of this experiment was 0.03 (almost dry), whereas the general internal reforming

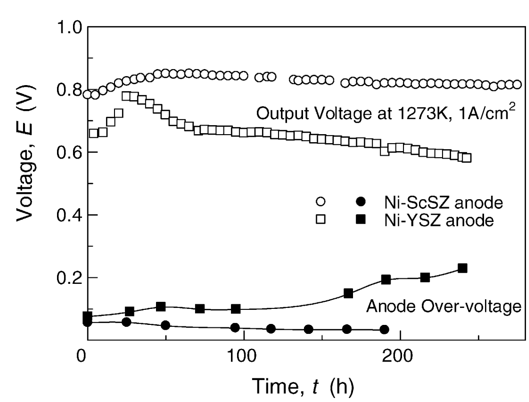

Fig. 9. Changes in output voltage of cells with Ni–YSZ anode and Ni–ScSZ anode at current density of 1 A/cm^2 under almost dry methane fuel conditions.

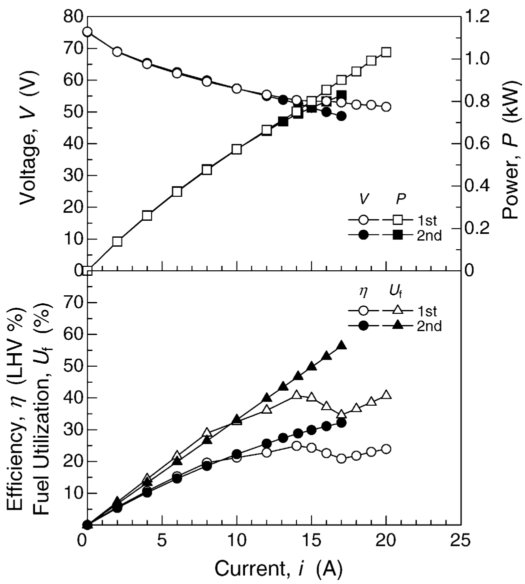

Fig. 10. Operating results of 1 kW SOFC system with hydrogen fuel. Operating temperature is 1073 K and all operating conditions are controlled by thermal self-sustaining.

condition was S/C > 2.0 in order to avoid the poisoning of the anode by deposited carbon.

Remarkable degradation was observed in the cell with the Ni–YSZ anode, whereas stable performance was shown in the cell with the Ni–ScSZ anode. After the anodes were exposed to methane fuel, carbon deposits with different morphologies and crystallization ratios were found on all the anode surfaces [17]. These results suggest that the ScSZ can be used not only in SOFC electrolytes and electrodes but also in catalytic applications such as hydrogen production by direct decomposition of methane.

3.3. *Proof-of-concept 1 kW SOFC system*

The 1 kW SOFC system was installed at Toho Gas in September 2003. Considering reliability and mass-producibility, electrolyte-supported cells with 4ScSZ electrolytes were used. Mass production processes for this cell were examined by Nippon-Shokubai Co. Ltd., Japan, and several hundred thin 4ScSZ disk cells with a diameter of 12 cm and a low weight of 6 g were successfully manufactured. Also, low-cost, thin ferritic stainless steel plates were machined to bi-polar interconnect plates. Preliminary stack tests were conducted using a single cell stack (single cell with interconnect plates on both sides), and a 1 kW stack performance was estimated. As a result, an efficiency of 40% (based on low heat value, LHV) was obtained with a single cell stack and with a hydrogen fuel rate of 200 cm^3/min, current of 23 A, and voltage of 0.7 V. Fig. 10 shows the first results of the 1 kW system operation with hydrogen fuel. An output power of 1032 W was obtained at the first trial under thermal self-sustaining conditions, and the electrical efficiency was 24% (LHV) and fuel utilization was 40%. Unfortunately, system operation with natural gas fuel failed because of uncontrollable system software. In the second trial performed under higher fuel utilization conditions, an electrical efficiency of 32% was obtained at 818 W output. Although the primary single cell stack showed enough efficiency and output, these results were lower than the design values of the 1 kW system. Consideration of the detailed operating data showed a non-uniform voltage distribution in the 1 kW stack. Several cells showed lower voltages, which limited the performance of the whole cell stack. It was found that the uniformity of the stack temperature distribution and of the gas flow rate to each cell, as well as the electrical contact between the cells and interconnecting plates, should be improved. In the future, the system operation with natural gas fuel will be demonstrated and system durability tests will be conducted.

4. Conclusions

Toho Gas R&D efforts and achievements regarding solid oxide fuel cells (SOFC) that use Sc_2O_3–ZrO_2 (scandia stabilized zirconia) electrolytes are summarized as follows.

(1) Cubic-ScSZ and tetragonal-ScSZ have a higher electrical conductivity than that of the conventionally used Yttria stabilized zirconia, and they are effective in improving the performance and reducing the operating temperature of SOFCs.
(2) The aging effect due to changing conductivity was examined, and it was found that >10 mol % Sc_2O_3 was stable for cubic-ScSZ and that a smaller dopant ratio was better for tetragonal-ScSZ.
(3) An adequate composition of high strength ScSZ was 4 mol % Sc_2O_3, and 12 cm diameter disk cells were successfully developed.
(4) ScSZ electrolyte-supported cells could reduce the SOFC operating temperature to around 1073 K even under actual system operating conditions.
(5) A proof-of-concept test was performed for the 1 kW SOFC system, and an electrical output of 1 kW was obtained in the test under thermal self-sustaining conditions.

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