Advances in Solid Oxide Fuel Cells II

A Collection of Papers Presented at the 30th International Conference on Advanced Ceramics and Composites
January 22–27, 2006, Cocoa Beach, Florida

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Narottam P. Bansal

General Editors
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Contents

Preface xi

Introduction xiii

Overview and Current Status

Development of Two Types of Tubular SOFCs at TOTO 3
Akira Kawakami, Satoshi Matsuoka, Naoki Watanabe, Takeshi Saito, Akira Ueno, Tatsumi Ishihara, Natsuko Sakai, and Harumi Yokokawa

Cell and Stack Development

Development of Solid Oxide Fuel Cell Stack Using Lanthanum Gallate-Based Oxide as an Electrolyte 17

Anode Supported LSCM-LSGM-LSM Solid Oxide Fuel Cell 27
Alidad Mohammadi, Nigel M. Sammes, Jakub Pusz, and Alevtina L. Smirnova

Characterization/Testing

Influence of Anode Thickness on the Electrochemical Performance of Single Chamber Solid Oxide Fuel Cells 37
B. E. Buergler, Y. Santschi, M. Felberbaum, and L. J. Gauckler

Investigation of Performance Degradation of SOFC Using Chromium-Containing Alloy Interconnects 47
D. R. Beeaff, A. Dinesen, and P. V. Hendriksen

Degradation Mechanism of Metal Supported Atmospheric Plasma Sprayed Solid Oxide Fuel Cells 55
D. Hathiramani, R. Vassen, J. Mertens, D. Sebold, V. A. C. Haanappel, and D. Stover
Effect of Transition Metal Ions on the Conductivity and Stability of Stabilized Zirconia  
D. Lybye and M. Mogensen

Thermophysical Properties of YSZ and Ni-YSZ as a Function of Temperature and Porosity  
M. Radovic, E. Lara-Curzio, R. M. Trejo, H. Wang, and W. D. Porter

Physical Properties in the Bi2O3-Fe2O3 System Containing Y2O3 and CaO Dopants  
Hsin-Chai Huang, Yu-Chen Chang, and Tzer-Shin Sheu

Electrical Properties of Ce0.8Gd0.2O1.9 Ceramics Prepared by an Aqueous Process  
Toshiaki Yamaguchi, Yasufumi Suzuki, Wataru Sakamoto, and Shin-ichi Hirano

Structural Study and Conductivity of BaZr0.90Ga0.10O2.95  
Istaq Ahmed, Elisabet Ahlberg, Sten Eriksson, Christopher Knee, Maths Karlsson, Aleksandar Matic, and Lars Borjesson

Hydrogen Flux in Terbium Doped Strontium Cerate Membrane  
Mohamed M. Elbaccouch and Ali T-Raissi

A Mechanical-Electrochemical Theory of Defects in Ionic Solids  
Narasimhan Swaminathan and Jianmin Qu

Electrodes

Nanostructured Ceramic Suspensions for Electrodes and the Brazilian SOFC Network “Rede PaCOS”  
R. C. Cordeiro, G. S. Trindade, R. N. S. H. Magalhães, G. C. Silva, P. R. Villalobos, M. C. R. S. Varela, and P. E. V. de Miranda

Modeling of MIEC Cathodes: The Effect of Sheet Resistance  
David S. Mebane, Erik Koep, and Meilin Liu

Cathode Thermal Delamination Study for a Planar Solid Oxide Fuel Cell with Functional Graded Properties: Experimental Investigation and Numerical Results  
Gang Ju, Kenneth Reifsnider, and Jeong-Ho Kim

Electrochemical Characteristics of Ni/Gd-Doped Ceria and Ni/Sm-Doped Ceria Anodes for SOFC Using Dry Methane Fuel  
Caroline Levy, Shinichi Hasegawa, Shiko Nakamura, Manabu Ihara, and Keiji Yamahara
Control of Microstructure of NiO-SDC Composite Particles for Development of High Performance SOFC Anodes
Koichi Kawahara, Seiichi Suda, Seiji Takahashi, Mitsunobu Kawano, Hiroyuki Yoshida, and Toru Inagaki

Electrochemical Characterization and Identification of Reaction Sites in Oxide Anodes

Interconnects and Protective Coatings

Corrosion Performance of Ferritic Steel for SOFC Interconnect Applications
M. Ziomek-Moroz, G. R. Holcomb, B. S. Covino, Jr., S. J. Bullard, P. D. Jablonski, and D. E. Alman

High Temperature Corrosion Behavior of Oxidation Resistant Alloys Under SOFC Interconnect Dual Exposures
Zhenguo Yang, Greg W. Coffey, Joseph P. Rice, Prabhakar Singh, Jeffry W. Stevenson, and Guan-Guang Xia

Electro-Deposited Protective Coatings for Planar Solid Oxide Fuel Cell Interconnects
Christopher Johnson, Chad Schaeffer, Heidi Barron, and Randall Gemmen

Properties of (Mn,Co)$_3$O$_4$ Spinel Protection Layers for SOFC Interconnects
Zhenguo Yang, Xiao-Hong Li, Gary D. Maupin, Prabhakar Singh, Steve P. Simner, Jeffry W. Stevenson, Guan-Guang Xia, and Xiaodong Zhou

Fuel Cell Interconnecting Coatings Produced by Different Thermal Spray Techniques
E. Garcia and T. W. Coyle

Surface Modification of Alloys for Improved Oxidation Resistance in SOFC Applications
David E. Alman, Paul D. Jablonski, and Steven C. Kung

Seals

Composite Seal Development and Evaluation
Matthew M. Seabaugh, Kathy Sabolisky, Gene B. Arkenberg, and Jerry L. Jayjohn

Investigation of SOFC-Gaskets Containing Compressive Mica Layers Under Dual Atmosphere Conditions
F. Wiener, M. Bram, H.-P. Buchkremer, and D. Sebold
Performance of Self-Healing Seals for Solid Oxide Fuel Cells (SOFC) 287
Raj N. Singh and Shailendra S. Parihar

Properties of Glass-Ceramic for Solid Oxide Fuel Cells 297
S. T. Reis, R. K. Brow, T. Zhang, and P. Jasinski

Mechanical Behavior of Solid Oxide Fuel Cell (SOFC) Seal Glass-Boron Nitride Nanotubes Composite 305
Sung R. Choi, Narottam P. Bansal, Janet B. Hurst, and Anita Garg

Mechanical Behaviour of Glassy Composite Seals for IT-SOFC Application 315
K. A. Nielsen, M. Solvang, S. B. L. Nielsen, and D. Beeaff

Mechanical Property Characterizations and Performance Modeling of SOFC Seals 325
Brian J. Koeppel, John S. Vetrano, Ba Nghiep Nguyen, Xin Sun, and Moe A. Khaleel

Mechanical Properties

Fracture Test of Thin Sheet Electrolytes 339
Jurgen Malzbender, Rolf W. Steinbrech, and Lorenz Singheiser

Failure Modes of Thin Supported Membranes 347
P. V. Hendriksen, J. R. Høgsberg, A. M. Kjeldsen, B. F. Sørensen, and H. G. Pedersen

Comparison of Mechanical Properties of NiO/YSZ by Different Methods 361
Dustin R. Beeaff, S. Ramousse, and Peter V. Hendriksen

Fracture Toughness and Slow Crack Growth Behavior of Ni-YSZ and YSZ as a Function of Porosity and Temperature 373
M. Radovic, E. Lara-Curzio, and G. Nelson

Effect of Thermal Cycling and Thermal Aging on the Mechanical Properties of, and Residual Stresses in, Ni-YSZ/YSZ Bi-Layers 383

Three-Dimensional Numerical Simulation Tools for Fracture Analysis in Planar Solid Oxide Fuel Cells (SOFCs) 393
Janine Johnson and Jianmin Qu

viii . Advances in Solid Oxide Fuel Cells II
Modeling

Electrochemistry and On-Cell Reformation Modeling for Solid Oxide Fuel Cell Stacks

Modeling of Heat/Mass Transport and Electrochemistry of a Solid Oxide Fuel Cell
Yan Ji, J. N. Chung, and Kun Yuan

Author Index
Preface

The third international symposium “Solid Oxide Fuel Cells: Materials and Technology” was held during the 30th International Conference on Advanced Ceramics and Composites in Cocoa Beach, FL, January 22–27, 2006. This symposium provided an international forum for scientists, engineers, and technologists to discuss and exchange state-of-the-art ideas, information, and technology on various aspects of solid oxide fuel cells. A total of 125 papers, including three plenary lectures and eleven invited talks, were presented in the form of oral and poster presentations indicating strong interest in the scientifically and technologically important field of solid oxide fuel cells. Authors from four continents and 14 countries (Brazil, Canada, Denmark, France, Germany, India, Iran, Italy, Japan, Sweden, Switzerland, Taiwan, Ukraine, and U.S.A.) participated. The speakers represented universities, industries, and government research laboratories.

These proceedings contain contributions on various aspects of solid oxide fuel cells that were discussed at the symposium. Forty one papers describing the current status of solid oxide fuel cells technology and the latest developments in the areas of fabrication, characterization, testing, performance analysis, long term stability, anodes, cathodes, electrolytes, interconnects and protective coatings, sealing materials and design, interface reactions, mechanical properties, cell and stack design, protonic conductors, modeling, etc. are included in this volume. Each manuscript was peer-reviewed using the American Ceramic Society review process.

The editor wishes to extend his gratitude and appreciation to all the authors for their contributions and cooperation, to all the participants and session chairs for their time and efforts, and to all the reviewers for their useful comments and suggestions. Financial support from the American Ceramic Society is gratefully acknowledged. Thanks are due to the staff of the meetings and publications departments of the American Ceramic Society for their invaluable assistance. Advice, help and cooperation of the members of the symposium’s international organizing committee (Tatsumi Ishihara, Tatsuya Kawada, Nguyen Minh, Mogens Mogensen,
Nigel Sammes, Prabhakar Singh, Robert Steinberger-Wilkens, and Jeffry Steven-
son) at various stages were instrumental in making this symposium a great success.

It is our earnest hope that this volume will serve as a valuable reference for the
engineers, scientists, researchers and others interested in the materials, science and
technology of solid oxide fuel cells.

NAROTTAM P. BANSAL
Introduction

This book is one of seven issues that comprise Volume 27 of the Ceramic Engineering & Science Proceedings (CESP). This volume contains manuscripts that were presented at the 30th International Conference on Advanced Ceramic and Composites (ICACC) held in Cocoa Beach, Florida January 22–27, 2006. This meeting, which has become the premier international forum for the dissemination of information pertaining to the processing, properties and behavior of structural and multifunctional ceramics and composites, emerging ceramic technologies and applications of engineering ceramics, was organized by the Engineering Ceramics Division (ECD) of The American Ceramic Society (ACerS) in collaboration with ACerS Nuclear and Environmental Technology Division (NETD).

The 30th ICACC attracted more than 900 scientists and engineers from 27 countries and was organized into the following seven symposia:

- Mechanical Properties and Performance of Engineering Ceramics and Composites
- Advanced Ceramic Coatings for Structural, Environmental and Functional Applications
- 3rd International Symposium for Solid Oxide Fuel Cells
- Ceramics in Nuclear and Alternative Energy Applications
- Bioceramics and Biocomposites
- Topics in Ceramic Armor
- Synthesis and Processing of Nanostructured Materials
The organization of the Cocoa Beach meeting and the publication of these proceedings were possible thanks to the tireless dedication of many ECD and NETD volunteers and the professional staff of The American Ceramic Society.

ANDREW A. WERESZCZAK
EDGAR LARA-CURZIO
General Editors

Oak Ridge, TN (July 2006)
Overview and Current Status
DEVELOPMENT OF TWO TYPES OF TUBULAR SOFCS AT TOTO

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ABSTRACT

The current status of two types of SOFC R & D at TOTO is summarized. We have developed 10kW class tubular SOFC modules for stationary power generation using Japanese town gas (13A) as fuel. A small module which consisted of 5 stacks (a stack consisted of 2 x 6 cells) generated 1.5kW at 0.2A/cm² and achieved 55%-LHV efficiency at an average temperature of 900°C. A thermally self-sustaining module consisting of 20 stacks achieved 6.5kW at 0.2A/cm² and 50%-LHV.

We have also developed micro tubular SOFCs for portable application, which operate at relatively lower temperatures. The single cell generated 0.85, 0.70, and 0.24W/cm² at 700°C, 600°C, and 500°C, respectively. We built and evaluated a stack consisting of 14 micro tubular cells, and it successfully demonstrated 43W, 37W and 28W at a temperature of 700°C, 600°C, and 500°C, respectively.

INTRODUCTION

TOTO is the top sanitary ware manufacturer in Japan and highly experienced in traditional and advanced ceramic products. Solid Oxide Fuel Cells (SOFCs) are mainly composed of ceramics, and our fabrication technology has been utilized to produce high performance SOFCs at a low cost. TOTO started the research and development of tubular type SOFCs in 1989. From 2001 to 2004, we successfully completed a 10kW class thermally self-sustaining module test in a New Energy and Industrial Technology Development Organization (NEDO) project. Since 2004, TOTO started a new collaboration with Kyushu Electric Power Co., Inc. and Hitachi, Ltd. in a new NEDO project. We are developing a co-generation system by integration with the TOTO stack. On the other hand, TOTO also started the development of micro SOFCs using micro tubular cells (diameter is less than 5mm) from 2002 under another NEDO project. In this paper, we summarized the current status of two types of SOFCs R & D at TOTO, i.e. tubular SOFC for stationary power generation and micro tubular SOFC for portable power application.

TOTO TUBULAR SOFC
Cell development

The schematic viewgraphs of the TOTO tubular cell are shown in Figure 1. A perovskite cathode tube is formed by extrusion molding. A zirconia electrolyte and a nickel/zirconia cermet...
Development of Two Types of Tubular SOFCs at TOTO

Anode are coated onto the tube by the TOTO uet process. A vertical interconnector is coated along the tube in a strip. The diameter of the cell is 16.5 mm and the active length is 660 mm. Fuel gas is supplied to the outside of the cell, and air is supplied to the inside by a thinner air supply tube. Recently, the materials used for cells are changed as shown in Table 1. The new material configuration resulted in the improvement of cell performance as shown in Figure 2, especially in lower operating temperatures. Our latest cell worked best at temperatures over 850°C.

![Cross Section](image1)

Figure 1. Schematic view of TOTO tubular cell.

<table>
<thead>
<tr>
<th>Components</th>
<th>Previous Type Cell</th>
<th>New Type Cell</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cathode Tube</td>
<td>(La,Sr)MnO₂</td>
<td>(La,Sr)MnO₂</td>
</tr>
<tr>
<td>Electrolyte</td>
<td>YSZ</td>
<td>SCSZ</td>
</tr>
<tr>
<td>Anode</td>
<td>Ni/YSZ</td>
<td>Ni/YSZ</td>
</tr>
<tr>
<td>Interconnector</td>
<td>(La,Ca)CrO₃</td>
<td>(La,Ca)CrO₃</td>
</tr>
</tbody>
</table>

![Graph](image2)

Figure 2. Cell performances as a function of operating temperature.
Development of Two Types of Tubular SOFCs at TOTO

Stack development

Twelve tubes are bundled in a 2x6 stack with nickel materials connecting an interconnector of one cell and the anode of the next (Figure 3). A stack was installed in a metal casing and heated by an electric furnace. Simulated fuel of partially steam reformed town gas was supplied to the stack. The town gas was assumed to be 50% steam reformed under SIC (steam carbon ratio) = 3.0. A stack generated 0.34 kW at a current density of 0.2 A/cm² at a temperature of 940°C. The maximum efficiency for DC output of the stack was 57%. Lower Heating Value (LHV) calculated on the basis of equivalent town gas (Figure 4).

Small module (quarter size module)

The thermally self-sustaining operation indicates that the modules generate power without any external heat supply. A quarter-size small size modules, which consisted of 5 stacks were made and tested for basic evaluations to realize the thermally self-sustaining operation. The small module and its metal casing were covered with a ceramic insulator. In this test, the desulfurized town gas was partially steam reformed through a reactor. The reformer was installed outside of the module with an electric furnace as shown in Figure 5. The conversion of steam

---

Figure 3. Appearance of TOTO stack.

Figure 4 Performance of 2x6 stack.

Figure 5. Flow diagram of small SOFC module.
Development of Two Types of Tubular SOFCs at TOTO

reforming can be controlled independently with reformer temperature. However the higher hydrocarbons such as ethane (C2H6), propane (C3H8) and butane (C4H10) were completely converted. The residual CH4 was internally reformed on the anode, and the endothermic effect was utilized to maintain the homogeneous temperature distribution of the module. The outlet fuel and air were mixed and burned above the module, and this combustion heat was used for air preheating. Those improvements in temperature distribution and fuel gas distribution strongly affected on the module performance. We succeeded to operate a module with 1.6kW at 0.2A/cm² at an average temperature of 900°C which corresponds to the efficiency of 40%-LHV for 3000 hours. The maximum efficiency was 55%-% LHV, which obtained for different module.

Thermally self-sustaining module

A ten kW class module consisting of four quarter-size modules is fabricated for thermally self-sustaining operation (Figure 6). An integrated heat exchanging steam reformer was mounted above the module. It consisted of an evaporator, pre-heater and reformer. However, the stability of the evaporator was not clearly demonstrated, therefore steam was supplied by another evaporator with an electric furnace. The gas flow was simple, and it does not include any gas recycles as shown in Figure 7. The module, the after-burning zone, and the reformer were covered with a ceramic insulator. A commercial steam reforming catalyst was embedded in the reforming section. The desulfurized town gas was supplied to the modules after being partially steam reformed. In the test, the heat of exhaust gases was used for steam reforming through the reformer. The module was heated by a partially oxidation burner and air heaters from room temperature.

The voltages of each stack and of the 2-cells in a quarter module, were monitored, and the variation of the voltages was quite small. The module generated 6.5kW at 0.19A/cm² and 46%-LHV (Table 2 Test 1). The improved module generated 6.5kW at 0.2A/cm² and 50%-LHV (Table 2 Test 2). The thermally self-sustainability of both operating condition were confirmed. The test was shifted to evaluation of long-term stability under the condition of Test 1. No degradation was observed in the module or the integrated heat exchanging steam reformer during the operating time of 1000 hours (Figure 8).

![Stack](Image)

Figure 6. Stack layout and appearance of thermally self-sustaining SOFC module.
Development of Two Types of Tubular SOFCs at TOTO

Figure 7. Flow diagram of thermally self-sustaining SOFC module system.

Table 2. Performance of thermally self-sustaining SOFC module.

<table>
<thead>
<tr>
<th>Items</th>
<th>Test 1</th>
<th>Test 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Power (kW)</td>
<td>6.4</td>
<td>6.5</td>
</tr>
<tr>
<td>$U_f$ (Yo)</td>
<td>70</td>
<td>75</td>
</tr>
<tr>
<td>Current Density ($A/cm^2$)</td>
<td>0.19</td>
<td>0.20</td>
</tr>
<tr>
<td>Ave. Cell Voltage (V)</td>
<td>0.69</td>
<td>0.70</td>
</tr>
<tr>
<td>Efficiency (%-LHV)</td>
<td>46</td>
<td>50</td>
</tr>
</tbody>
</table>

Figure 8. Long term stability of thermally self-sustaining SOFC module.
Development of Two Types of Tubular SOFCs at TOTO

<table>
<thead>
<tr>
<th>Component</th>
<th>Material</th>
<th>Fabrication</th>
<th>Firing</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anode Tube</td>
<td>NiO/YSZ</td>
<td>Extrude Molding</td>
<td></td>
</tr>
<tr>
<td>Anode Interlayer</td>
<td>NiO/GDC 10</td>
<td></td>
<td>Co-Firing</td>
</tr>
<tr>
<td>Electrolyte</td>
<td>LDC40(Ga2O3)-LSGM (Double Layered)</td>
<td>Slurry coating</td>
<td></td>
</tr>
<tr>
<td>Cathode</td>
<td>LSCF</td>
<td></td>
<td>Firing</td>
</tr>
</tbody>
</table>
Figure 9 is a picture of a micro tubular single cell. The diameter of cell is 5mm, and the active length is 50mm. The single cell was jointed to the current collector cap with silver braze metal and its performance was tested in a furnace. Figure 10 shows the evaluation method for single cell performance. A fuel gas was supplied inside the cell, and air was supplied to the outside of the cell. The current voltage and impedance of the single cells were measured using a potentiostat and a frequency response analyzer in the 500 to 700°C temperature range.

Figure 9. TOTO micro tubular cell.

Figure 10. Evaluation method for single cell performance.

Figure 11 show the typical I-V curves of a micro tubular single cell using dry H₂ in N₂ as fuel. H₂ flow was fixed at 0.12L/min. The open circuit voltage (OCV) was close to the theoretical value. It indicated that the electrolyte has a good gas tightness and the chemical reaction between LSGM and Ni are effectively avoided by the LDC40 layer. The maximum

Figure 11. I-V characteristic of micro tubular single cell.
Development of Two Types of Tubular SOFCs at TOTO

power densities were 0.85, 0.70, and 0.24 W/cm² at 700°C, 600°C, and 500°C, respectively. Figure 12 shows the impedance spectra of a micro tubular cell measured under 0.125 A/cm² at various temperatures. It has been generally assumed that the intercept with the real-axis at the highest frequency represents the ohmic resistance, and the width of low frequency arc represents the electrode resistance. The electrode resistance increased significantly with decreasing operation temperature, and ohmic resistance at 500°C was very high. The most likely cause of the high resistance is the low ionic conductivity of LDC40. Therefore, it is expected that the cell performance can be improved by optimizing the anode electrode and the thickness of LDC40 layer.

Figure 13 shows the fuel utilization effects on micro tubular cell performance measured under 0.125 A/cm² at 700°C and 600°C. The observed cell voltage was close to the theoretical value calculated by the Nernst equation. It indicates that the micro tubular cell can be operated at a high efficiency.

The cell performances using H₂ in N₂ (1:1) gas mixture or simulated reformate gas were compared in Figure 14. The composition of simulated reformate was 32%H₂, 13%CO, 5%CO₂, and 50%N₂ based on the preliminary experiment of the catalytic partial oxidation (CPOX) reforming of LPG. As shown in the figure, the difference in cell performances was small at lower current densities. However, the performance using reformate gas was lower at higher current densities at temperatures of 600°C and 700°C. The differences became significant with increasing operating temperatures. To identify the differences, the impedance spectra were measured under a current density of 0.8 A/cm² at 700°C (Figure 15). The electrode resistance on simulated reformate was higher than that on H₂ in N₂, and it was thought that this difference was caused by the CO transport resistance from the anode in the high current density area. Therefore, we are now trying to improve the anode performance.

Figure 16 shows the cell performance using DME + air mixture as fuel at 550°C. The DME flow rate was fixed at 85 mL/min and the excess air ratios (air-fuel ratio/ theoretical air-fuel ratio) were 0.1, 0.2, 0.3, and 0.4 respectively. Direct use of fuel without a reformer in SOFCs will simplify the system greatly, and this is important for SOFCs, especially in portable and transportation applications. DME is an attractive fuel because it is highly active and easily
Development of Two Types of Tubular SOFCs at TOTO

Figure 14. I-V curve tested on H₂ in N₂ and simulated reformate gas.

Figure 15. Impedance tested on H₂ in N₂ and simulated reformate gas.

Figure 16. I-V curve tested on DME+air mixture as fuel at 550°C.

Figure 17. DME conversion rate and exhaust gas composition tested on DME+air mixture.

liquefied and stored. As shown in the figure, the use of DME + air as fuel resulted in higher performance than that of H₂ in N₂ and no carbon deposition was observed during operation. Figure 17 shows the DME conversion rate and the exhaust gas composition analyzed by a gas chromatography. (The water content was not measured.) The DME conversion rate and CO₂ content increased with excess air ratio. Therefore, the increased performance achieved by using DME + air mixture is probably due to the raising cell surface temperatures caused by the decomposition and the combustion of DME. (The furnace temperature was kept at 550°C). These results demonstrated the high possibility of micro tubular cells being used for direct fueled operations.
Development of Two Types of Tubular SOFCs at TOTO

Micro tubular stack development

In order to evaluate the performance of cells in a bundle, we built the stack consisting of 14 micro tubular cells as shown in Figure 18. This stack was evaluated in a furnace using hydrogen as a fuel and successfully demonstrated 43W, 37W and 28W power generation at a temperature of 700°C, 600°C, and 500°C, respectively (Figure 19). Table 4 summarizes the results we have obtained from the stack evaluation. The maximum stack power densities were 478W/L and 239W/kg at 700°C. These results demonstrated that micro tubular SOFCs have a high potential for portable and transportation applications.

Figure 18. Appearance of micro tubular SOFC stack.

Figure 19. Performance of micro tubular SOFC stack.
Table 4. Performance of micro tubular SOFC stack at 700°C.

<table>
<thead>
<tr>
<th>Item</th>
<th>Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>Maximum power (W)</td>
<td>43</td>
</tr>
<tr>
<td>Stack volume (L)</td>
<td>0.09</td>
</tr>
<tr>
<td>Stack weight (kg)</td>
<td>0.18</td>
</tr>
<tr>
<td>Stack power density (W/L)</td>
<td>478</td>
</tr>
<tr>
<td>Stack power density (W/kg)</td>
<td>239</td>
</tr>
</tbody>
</table>

SUMMARY
TOTO tubular SOFC
The 2x6 stack, small module and thermally self-sustaining module of the TOTO tubular SOFC were designed and made. They were evaluated using town gas or simulated fuel and showed excellent performance. We continually improve the cell performance and the durability to advance the module performance. TOTO started the small-scale production of SOFC and trial delivery in 2004. The SOFC can be supplied as a stack for the development of stationary power generation systems. In 2004, TOTO started a new collaboration with Kyushu Electric Power Co., Inc. and Hitachi, Ltd. in a new NEDO project. We are developing a co-generation system by integration with the TOTO stack.

TOTO micro tubular SOFC
The anode-supported micro tubular cells with thin lanthanum gallate with strontium and magnesium doping were developed. The single cells and the cell stack were tested using various fuels, i.e., hydrogen, simulated reformate gas of LPG, direct fueling of DME, and they showed excellent performance at lower temperatures from 500-700°C. These results demonstrated that micro tubular SOFCs have a high potential for portable and transportation applications. Further development on durability, quick start up, and compactness of the stack is being undertaken.

ACKNOWLEDGMENT
The development was supported by NEDO in Japan.

REFERENCES