Performance degradation and analysis of 10-cell anode-supported SOFC stack with external manifold structure

Dong Yan^a, Lingjiang Liang^a, Jiajun Yang^a, Tao Zhang^{*b}, Jian Pu^a, Bo Chi^a, Jian Li^a

^aSchool of Materials Science and Engineering, State Key Laboratory of Material Processing and Die & Mould Technology, Huazhong University of Science & Technology, 430074 Wuhan, Hubei, China

^bSchool of Naval Architecture & Ocean Engineering, Huazhong University of Science & Technology, 430074 Wuhan, Hubei, China

Abstract:

This study describes the development of solid oxide fuel cell (SOFC) stack with external manifold structure. The stack used anode supported cells with size of 11cm×11cm which are fabricated by tape casting, screen-printing and co-sintering. Computer simulation was employed to evaluate the gas distribution in the external manifold stack. The 10-cell stack was operated at 750°C with hydrogen as fuel and air as oxidant and generated output power of 360 W, corresponding to power density of 440mW/cm². The degradation performance was investigated for over 750h under the current density of 370 mA/cm². Posttest analysis of the SOFC stack was operated to investigate the factors related to the performance degradation such as microstructure of electrodes, sealing, contact resistance and oxidation of metal interconnect. The study presented here showed the stack with external manifold can be considered as a strong candidate for SOFC stack design.

Keywords: Solid oxide fuel cell; stack; Performance durability; Microstructure analysis; oxidation

1. Introduction

Solid oxide fuel cell is gaining more attention in recent years. It is considered as a promising power generation technology due to its high efficiency, low emission and high fuel flexibility[1-3]. However, the durability and cost are still the major challenges towards commercialization in long term operation[4-6]. Depending on considerable progress in SOFC materials system, the operating temperature of the stack has been reduced from 1000°C to the range of 600~800°C, thus, cheaper metals like stainless steel can be used as interconnect which makes SOFCs technology more attractive[7-9].

* zhangt7666@hust.edu.cn

Planar type anode-supported cell is a promising one which has been confirmed to have high power density and reliability[10]. In our previous work, the fabrication process and performance of single cell have been investigated. The cell with active area of 81 cm^2 can obtain an OCV(open circuit voltage) of 1.2 V and power density of 770 mW/cm^2 at the current density of 950 mA/cm^2 and its performance degradation is lower than 1.56%/1000h[11]. However, when cells are assembled into a stack to achieve higher power outputs, the stack performance will be unavoidable affected by several inherent issues, especially like surface contact resistance, gas distribution, sealing, oxidation of metallic interconnect, which have been confirmed by researches[12-14].

Although considerable progresses have already been achieved in improving the initial stack performance by enhancing cell and interface contact[15], the stack stability remains a critical concern for SOFC development. The degradation mechanism of stack has been investigated in the recent decades. The general degradation features include deactivation of catalysts, oxidation of metallic component, element diffusion and chemical reaction, microstructure changes from electrode, sealing failure, etc. which is difficult to examine and identify the degradation origin[16-19]. As described in the literature[20], two parameters, area-specific resistance (ASR) and degradation rate (DR) are proposed to evaluate the degradation behavior. The increase of ASR was widely believed to be the result of degradation.

Different types of stack have been invented like internal-manifold type[21,22], external-manifold type[23, 24], envelope type[25] disc type[26], etc. Within these types, the external manifolds are simple in structure and cost less to build. We have already built and tested a 3-cell stack and described the gas flow and temperature distribution in the stack with the help of computer simulation in our previous work [24].

Based on previous researches [11, 24, 27, 28], the gas distribution in the stack is evaluated with computational fluid dynamic method. A 10-cell stack was built and evaluated during a long-time operation. The effect of cell microstructure, cathode contact materials, sealing and oxidation of metallic interconnect on performance degradation of stack is discussed in detail as well as intensive post-test analysis.

2. Stack configuration and simulation

Fig.1 shows the schematic diagram of the external-manifold SOFC stack with cross-flow configuration. The serially connected cells in the stack are fed with gas in parallel. If fuel starvation developed in some of the cells, the whole stack performance will be affected. High current density may reverse the electric potential of the starving cells, oxide the anode and lead to structural failure. Gas distribution was investigated by computer simulation before the stack was assembled. A 3D module of the manifold in full scale was built as shown in Fig.2 (a). The gas flow rate of each cell was set to be 0.8 SLM and the cells were numbered from top to bottom. For an open manifold without outlet back pressure, the gas distribution is uneven and the middle cell has the lowest mass flow rate as shown in Fig.2 (b). This result represents the situation of absence of stack core, without the current collectors in the stack providing resistance for the gas flow. By increasing the back pressure to 600 Pa and 1200 Pa, the mass flow rates between each cell improved their uniformity as shown in Fig.2 (c) and (d). The flow rate fluctuation reduced from $\pm 18\%$ for 0 Pa to $\pm 0.09\%$ and $\pm 0.07\%$ for 600 Pa and 1200 Pa respectively. Judging from the calculation results, the manifold flow chamber is capable to provide a uniform flow distribution for the 10-cell stack core.

3. Experimental

3.1 Stack construction

The Ni foam was used as current collector and gas distributor for the anode. In order to eliminate the mismatch of coefficient of thermal expansion between interconnect and manifold, stainless steel SUS 430 was used to fabricate all the metallic components. Detail of the integrated interconnect has been described elsewhere[24]. As previously reported[27], the anode supported cells were fabricated by tape casting, screen printing, and single step co-fire process. The Cells have dimensions and active area of $11 \times 11 \text{ cm}^2$ and $9 \times 9 \text{ cm}^2$ respectively. To enhance the ability of current collection, a layer of $\text{LaCo}_{0.6}\text{Ni}_{0.4}\text{O}_{3-\delta}$ (LCN) was applied between the cathode and current collector. The electronic conductivity of LCN is above 1000 S/cm at 800°C, higher than conventional cathode contact materials LSM and LNF[29]. The powder was pre-calcined at 1100°C for 3 hours to prevent grain coarsening during the operation at 750 °C. In this study, the cell was sealed by flexible alumina-based strip-shape tapes developed from our previous work[13, 30]. The thickness of sealing tape was optimized to meet a precise match with cell and corrugation plate that can

achieve good contact and sealing at the same time.

3.2 Stack operation and post analysis

The 10-cell stack was heated from room temperature to the operating temperature of 750°C in the furnace. The inlet gases were heated while passing through coiled steel tubes inside the furnace. The cathode was fed with dry air and the anode was fed with nitrogen and 4 vol. % of hydrogen mixture gas so that the NiO in the anode can be gradually reduced. Upon reaching 750°C, the gas of anode side was changed to pure hydrogen. The voltage and power density of stack as a function of current density were measured under current passage ranging from 0 to 60 A. The stack with more than 700 h operating time was tested under current density of 370 mA/cm² for evaluating the performance stability at long time operation. During the operation, flammable gas detectors were deployed in the testing room to detect hydrogen leaking, exhaust fans were also used to ensure the ventilation. When the test ended, the stack was cooled down to the room temperature under hydrogen atmosphere of anode side.

The stack was then disassembled and examined. The cell microstructure, morphology of the LCN powder and oxide scale of interconnect were characterized by scanning electron microscopy (SEM, FEI quanta 200, Phillips) and energy dissipation spectroscopy (EDS, AMETEK). The phase structure of anode and LCN were identified by X-ray diffraction (XRD, X'Pert PRO diffractometer).

4. Results and discussion

4.1 Electrical performance and degradation of stack

Fig.3 shows the stack voltage and power density as functions of current density in 10-cell stack. The open circuit voltage (OCV) of 11.79 V and power density of 440 mW/cm² (power of 360 W) were achieved at the current density of 740 mA/cm² (current of 60 A). It can be inferred that the sealant did not met severe failure like cracks or delamination which could lead to massive gas leaking and internal combustion, according to the high OCV, and the adjacent components in the stack contacted well with each other and resulted a low electrical resistance in testing. The area specific resistance (ASR) of stack was 0.44 Ω·cm² calculated from the linear portion of voltage-current curve, which is twice the value of the single cell ASR of only 0.202 Ω·cm² [11]. This difference between cell and stack depends on stack structure, components connect and materials combinations, and some problems like gas distribution, interface contact and sealing can often occur on stack other than cell,

leading to unwanted performance degradation. Dey et al.[12] suggest that the loss of actual contact area (2.5% on the anode and 6.7% on cathode) for cell electrode plays critical roles towards the performance of the scaled up cell. Obviously, the initial performance difference can be mainly ascribed to the contact resistance between cell and the adjacent components when cell structure is almost the same.

The long term stability was characterized by the voltage degradation when the stack was discharging at a constant current. The degradation was mainly caused by electrochemical and ohmic resistance increasing. A working voltage model has been developed to discuss the degradation mechanism of stack according to the equation[20]:

$$V(i, t) = (E_N - \eta_L) - (iR_i + \eta_{aa} + \eta_{ac} + \eta_{ca} + \eta_{cc}) \quad (1)$$

Where V is the stack voltage, i is the current density, t is the time, E_N is the Nernst potential, R_i is the total resistance of all cell components, η_L is the loss caused by leakage, η_{aa} and η_{ac} are the activation over potentials of anode and cathode, η_{ca} and η_{cc} are the concentration over potentials, respectively. Additionally, the definitions of ASR and average DR in terms of the Eq. (1) are:

$$ASR(i_s, t) = \frac{E(t) - V(i_s, t)}{i_s} \quad (2)$$

And,

$$\overline{DR}(t) = 100 \frac{V(i_s, t_0) - V(i_s, t)}{V(i_s, t_0) \times (t - t_0)} \quad (3)$$

Where E can be either the ideal Nernst potential or OCV, i_s is the constant operating system current density, and t_0 can be described as the initial starting time. Fig.4 shows the voltage degradation of 10-cell stack with current density of 370 mA/cm^2 , taken operation time at 700h. The stack voltage increased from 8.02 V to 8.55 V at the initial stage of the polarization, and then decreased slowly to 7.46 V. According to the Eqs. (2) and (3), the ASR and average DR were calculated and shown as a function of time in the Fig.5. The average DR of the stack for 700 h was 17.6%/1000h, which displays a clearly linear degradation. The ASR increased from $0.88 \text{ } \Omega \cdot \text{cm}^2$ to $1.17 \text{ } \Omega \cdot \text{cm}^2$ per cell. Generally, the performance degradation of stack originated from ASR increase may be ascribed to several reasons including microstructure coarsening of cell, oxidation of metallic interconnect, mechanical delamination between electrode/interconnect interface, and leakage driven degradation.

4.2 Degradation analysis

4.2.1 Cell structure

After the test, the stack was disassembled and cells were examined. None of the cells was broken and delaminated. The CTE mismatch among sealant, interconnect and the cell or the manifold did not develop thermal stress high enough to break the cell.

The cross sectional microstructure of cell is shown in Fig.6. The thin dense electrolyte is almost free of pore or cracks, and well bonded to the porous cathode and functional anode. Further, the porous electrode provides the gas diffusion path to the triple phase boundary near the electrolyte.

Fig.7 shows the XRD patterns of anode sides before and after reduction with electrolyte carefully removed by polishing to expose the anode functional layer.. Peaks are denoted with JCPDF file 00-001-1258 for Ni, 00-001-1239 for NiO and 00-037-1307 for YSZ. Result shows that NiO had been fully reduced to metallic Ni and no other phase formed, the materials of cell remained stable during the test.

4.2.2 Contact

Fig.8 gives the X-ray diffraction patterns of cathode contact material $\text{LaCo}_{0.6}\text{Ni}_{0.4}\text{O}_{3-\delta}$ layer before and after test and compare those with the JCPDF file 00-054-0834. The peaks match well and no difference can be observed. It can be confirmed that the LCN has a chemical compatibility both with cathode and interconnector in the cathode atmosphere under operational temperature and remains stable during the operation, which is in agreement with previously reported results[29]. The morphologies and particle sizes of LCN powder before (a) and after (b) test are shown in Fig.9. Both LCN powders had similar mean grain size of about $1.5\mu\text{m}$, while the particle after test had narrower size distribution and morphology showed sintering neck between neighboring particles. It is noted that the neck-contacted LCN particles could remain the geometric dimension and microstructure during the operation basically, although the grain coarsening and formation of sintering neck might bring the shrinkage of the LCN layer increasing the contact resistance. Low temperature sintering could happen in contact materials according to the report of McCarthy et al.[31]. The pre-calcine treatment at higher temperature could effectively reduce the particle surface energy and thus stable the microstructure and prevent particle growth.

In order to evaluate the degradation of LCN contact layer, the ASR of LCN was measured under a simulation environment with four-probe method and the results

were shown in Fig.10 accompanied by the schematic diagram, a layer of LCN slurry was sandwiched between two SUS430 steel plate and was heat to 750 °C. During the simulation test, the ASR increased from 70 mΩ·cm² to 130 mΩ·cm² in first 50h and slowly went up to 160 mΩ·cm² in 200h. Based on the fitting of the curve, the total ASR increment was 100 mΩ·cm² at 700h. Subtracting the increment of 10 mΩ·cm² caused by steel oxidation calculated according to the previous research[32], the ASR increment caused by LCN degradation was 90 mΩ·cm², contributing to 31% of total degradation (290 mΩ·cm²).

4.2.3 Sealant

Fig.11(a) shows the photograph of cathode side. Part of the black contact layer was carried away by the current collector and cathode surface was exposed. At the edge of the contact area, some LCN powder's color turned into dark brown. Samples were taken from these region and characterized by XRD, Result shown in Fig.11 (b) suggested that the LCN powder was decomposed into La₂O₃, LaCrO₃, and LaNiO₃, which have higher resistance than LCN. This change will increase the ASR, reduce the active area and increase the current density in the active area. Consider the position where decomposition happened, we suspect that there was a sealing failure in these area. When H₂ invaded into the cathode side from the edge, burning elevated the local temperature and also changed the oxygen partial pressure to a condition that LCN couldn't retain stable and decomposed. On the anode side, no burning mark of air leakage was found and the sealant remained relatively intact shown in Fig.12. Meanwhile, the Ni foam was firmly adhered with cell and interconnect indicating a good contact. Although excellent performance was achieved at simulated out-of-stack leak test and single cell test[33], the sealant might be broken down at severer SOFC stack operation condition including high temperature gradient, rigorous reducing and oxidizing environment and uneven stress distribution and past experimental results revealed that the gas leakage through interface of sealant and cell/interconnect takes the dominant part.

4.2.4 Interconnect oxidation

Fig.13 shows the cross-sectional microstructures of interconnect (anode side) and corrugation (cathode side), and EDS compositional profile of line scan across the oxide scale. The scale was less than 5 μm thick and well bonded on the metallic substrate. The EDS result shown in Fig.13 reveals a double layer structure, with higher Mn content in the outer layer, higher Cr content in the inner layer and absence

of Fe. According to the reported literature[8, 34], the scale is composed of MnCr_2O_4 on top of Cr_2O_3 . The absence of Fe in the scale means the dense oxidant layer acted as a protective layer and prevented the oxidation of Fe.

Theoretically, the ASR can be calculated by the expression as a combination of the electrical resistivity (ρ) and the thickness (t) of the oxide scale:

$$\text{ASR} = \rho_{\text{Cr}_2\text{O}_3} \Delta t_{\text{Cr}_2\text{O}_3} + \rho_{\text{MnCr}_2\text{O}_4} \Delta t_{\text{MnCr}_2\text{O}_4} + \rho_{\text{Fe}_2\text{O}_3} \Delta t_{\text{Fe}_2\text{O}_3} \quad (4)$$

Where $\rho_{\text{Cr}_2\text{O}_3}$, $\rho_{\text{Fe}_2\text{O}_3}$ and $\rho_{\text{MnCr}_2\text{O}_4}$ are the constant with values of $78 \Omega \cdot \text{cm}$ [35], $9.2 \Omega \cdot \text{cm}$ and $0.5 \Omega \cdot \text{cm}$ [36]. Thus, the total estimated ASR value of the oxidant was $82 \text{ m}\Omega \cdot \text{cm}^2$ and the equivalent cell voltage decrement caused by oxidation was 0.03 V , making up 29% of the stack degradation. The oxidation of metallic interconnect is inevitable, which is deteriorate to the performance of stack after long oxidation in SOFC operation environment.

Thus, it can be tentatively concluded that the oxidation of interconnect, the degradation of cathode contact material LCN, seal failure and other issues made great contribution to the performance degradation shown in Fig.14.

5. Conclusions

We have developed an external manifold SOFC stack using a simple interconnect with corrugation plate. Flow simulation by computer finds out that the gas flow in the manifold can maintain a relative uniformity and the difference between each cell was less than $\pm 0.1\%$ for the 10 cells with the proper flow resistance. The stack composed of 10 anode-supported SOFCs and provided an OCV of 11.79 V and output of 440 mW/cm^2 (power of 360 W). The long term performance of the stack for 700h was conducted and the degradation behavior was discussed. The average degradation rate was $17.6\%/1000\text{h}$ at the current density of 370 mA/cm^2 . In the post test examination, we have investigated several issues related to degradation including contact decaying, oxidation of interconnects and sealing. Although the contact material was pre-calcined to prevent low temperature sintering, there was still a minor microstructure change, and the ASR of the LCN contact material increased and contributes almost 1/3 of the degradation. Stainless steel interconnect oxidation provided another 1/3 of the total degradation as the uncoated steel operated under complex atmospheres with a relative high current density. Sealant retained intact during the test, but there is still a leaking at some part of the stack, this yield the decomposition of LCN contact material and also provided performance degradation. These are believed to contribute most of the

stack degradation and should be improved in the future work.

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Appendix

SOFC	solid oxide fuel cell
OCV	open circuit voltage
ASR	area specific resistance
DR	degradation rate
SLM	standard liter per minute
CTE	coefficient of thermal expansion
E_N	Nernst potential
R_i	total resistance of all cell components
η_L	voltage loss caused by leakage
η_{aa}	activation over potentials of anode
η_{ac}	activation over potentials of cathode
η_{ca}	concentration over potentials of anode
η_{cc}	concentration over potentials of cathode
i_s	constant operating system current density
t_0	initial starting time
ρ	electrical resistivity
t	thickness of oxidation scale

Figure Captions:

Fig. 1 — Schematic view (a) and assembled stack view (b) of the external-manifold stack with cross-flow configuration.

Fig. 2 — Mass flow rate distribution in the manifold: (a) the schematic view of the manifold; (b) the gas flows without resistance; (c) the gas flows with resistance of 600 Pa; (d) the gas flows with resistance of 1200 Pa

Fig. 3 — Cell voltage and power density as a function of current density for 10-cell stack ($9 \times 9 \text{ cm}^2$ active area per cell) at 750°C

Fig. 4 — Degradation of 10-cell stack under the current density of 370 mA/cm^2 at 750°C .

Fig. 5 — Calculated ASR(t) per cell and average degradation rate of 10-cell stack under 370 mA/cm^2 constant load at 750°C .

Fig. 6 — SEM micrograph of the cross section of single cell used in 10-cell stack, (a) the thickness of electrolyte is about $10 \text{ }\mu\text{m}$ and cathode is about $5 \text{ }\mu\text{m}$, (b) shows the microstructure details of the porous anode supporter, sintered particles have an average diameters of about $1 \text{ }\mu\text{m}$.

Fig. 7 — X-ray diffraction patterns of NiO/YSZ anode layer before and after reduction.

Fig. 8 — X-ray diffraction patterns of LCN before and after test.

Fig. 9 — SEM morphologies and particle sizes before (a) and after (b) test of LCN powder.

Fig. 10 — ASR analysis of LCN contact material employing four-electrode method.

Fig. 11 — Photograph of cathode side of cell (a) and X-ray diffraction patterns of LCN (b).

Fig. 12 — Photograph of anode side with cell and interconnect.

Fig. 13 — Cross-sectional SEM morphologies with the EDS compositional analysis of anode (a, c) side of interconnect and cathode (b, d) side of corrugation.

Fig. 14 — The estimation of degradation composition.

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