



Short communication

Electrochemical performances of solid oxide fuel cells based on Y-substituted SrTiO₃ ceramic anode materials

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ABSTRACT

Yttrium-substituted SrTiO₃ has been considered as anode material of solid oxide fuel cells (SOFCs) substituting of the state-of-the-art Ni cermet anodes. Sr_{0.895}Y_{0.07}TiO_{3-δ} (SYT) shows good electrical conductivity, compatible thermal expansion with yttria-stabilized ZrO₂ (YSZ) electrolyte and reliable stability during reduction and oxidation (redox) cycles. Single cells based on SYT anode substrates were fabricated in the dimension of 50 mm × 50 mm. The cell performances were over 1.0 A cm⁻² at 0.7 V and 800 °C, which already reached the practical application level. Although Ti diffusion from SYT substrates to YSZ electrolytes was observed, it did not show apparent disadvantage to the cell performance. The cells survived 200 redox cycles without obvious OCV decrease and macroscopic damage, but performance decreased due to the electronic properties of the SYT material. The influence of water partial pressure on cell performance and coking tolerance of the cells are also discussed in this study.

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1. Introduction

Compared to conventional combustion systems, solid oxide fuel cells (SOFCs) are more competitive because they have higher fuel efficiency and less emission. Towards the commercialization of SOFC, Ni/YSZ cermet, the state-of-the-art anode material for SOFCs, shows significant drawbacks because of the problems like carbon deposition [1–4] and poor mechanical stability during reduction and oxidation cycles (redox) [5–10]. Hence, redox-stable and coking-free anodes based on full ceramic materials have attracted increasing interest in the recent years [11–14], and some single cells in laboratory-level already have been reported (see Table 1), exhibiting good potentials for further developments.

However, there are still significant deficiencies for these reported cells. For single cells in practical-using level, the maximum power out put should be over 1.0 W cm⁻². The reported electrical performances of the cells are still not satisfactory enough. In addition, in some of the cells noble-metal catalysts like Ru and Pd were used, which is commercially unacceptable. Regarding the mentioned cells in Table 1, only one was exposed to a redox test of 7 cycles [18]. However, with respect to commercial purposes, at least 100 redox cycles should be tested to determine the robustness of the cells. Even more important is the dependence of the redox strain limit of a single cell on the dimension of the cell [9,10]. All

the former reported cells are small pellet-sized, compared to single cells in commercial size, they suffer much smaller strain during redox cycles. In order to better evaluate the redox stability of anode materials, larger cells should be fabricated.

Compared to other redox-stable anode materials, donor-substituted SrTiO₃ materials have better chance to replace the state-of-the-art Ni/YSZ cermet because of their high electrical conductivity (after heat treatment in reducing atmosphere), matching thermal expansion to that of YSZ and good dimensional stability upon redox cycling [13,14,19]. In the former reports of our research group [20], 50 mm × 50 mm-sized half-cells based on Sr_{0.895}Y_{0.07}TiO₃ (SYT) anode substrates were prepared, some properties of the cells were investigated. In this work, the electrochemical properties of the cells were characterized in detail.

2. Experimental

2.1. Synthesis

Titanium (IV) isopropoxide (97%), Sr(NO₃)₂ (99%) and Y(NO₃)₃·6H₂O (99.9%) were used as starting materials for the preparation of Sr_{0.895}Y_{0.07}TiO₃ (SYT) powders. The titanium (IV) isopropoxide was dropped into distilled water that was being stirred. The precipitate was filtered and washed, and then dissolved in HNO₃ solution. Y(NO₃)₃·6H₂O was directly dissolved in distilled water. The Ti⁴⁺ and Y³⁺ concentration in solution was determined by thermal gravimetry. Sr(NO₃)₂ was weighed directly. Corresponding amounts of the nitrates were then com-

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Table 1Reported single cells based on redox-stable anode materials. All cells tests were performed with H₂ as fuel and air as oxidant.

Cell type	Dimension	Anode	Performance (maximum power density)	Redox testing	Reference
Electrolyte supported	20 mm diameter, 0.6 mm thickness	(La _{0.75} Sr _{0.25}) _{0.9} Cr _{0.5} Mn _{0.5} O ₃	0.30 W cm ⁻² at 850 °C	No test	[15]
Cathode supported	~20 mm diameter	Ceria and Ru-infiltrated Sr _{0.88} Y _{0.8} TiO ₃ /YSZ	0.51 W cm ⁻² at 800 °C	No test	[16]
Cathode supported	~1 cm ² , 0.4 mm thickness	Ceria and Pd-infiltrated La _{0.3} Sr _{0.7} TiO ₃ /YSZ	0.43 W cm ⁻² at 800 °C	No test	[17]
Anode supported	~15 mm diameter, 0.8 mm thickness	La _{0.2} Sr _{0.8} TiO ₃ support, NiO-Ce _{0.8} Sm _{0.2} O ₂ /NiO-YSZ anode	0.85 W cm ⁻² at 800 °C	7 cycles	[18]

bined to obtain one solution. After homogenization, the solution was spray-pyrolyzed in a commercial spray dryer (Nubilos, Konstanz, Germany). After spray pyrolysis the raw powder was heated to 900 °C in air for 5 h. La_{0.6}Sr_{0.4}CoO_{3-δ} (LSC) powder was prepared accordingly. (Sr_{0.89}Y_{0.07})_{0.91}TiO_{2.91} (SYT2) powder was prepared by Pechini's method [21] with the same starting materials as SYT. The powder was then mixed with commercial 8YSZ powder (Zr_{0.852}Y_{0.148}O_{1.926}, Tosoh, Japan) in a volume ratio of 2:1 (1.7:1 in weight ratio) by ball milling for 72 h (SYT2-YSZ). The screen-printing pastes of LSC and SYT2-YSZ were prepared by mixing the corresponding powders with a binder consisting of ethyl cellulose in terpineol.

2.2. Fabrication

The SYT powders were coat-mixed and warm pressed [22] into plates with dimensions of 70 mm × 70 mm × 2 mm. After debinding and pre-sintering at 1250 °C in air, the plates were screen-printed with SYT2-YSZ paste and sintered at 1000 °C. Then the YSZ electrolyte was vacuum slip-cast onto the anode and sintered in Ar/4% H₂ at 1350 °C for 3 h. The size of the plates after sintering was 50 mm × 50 mm × 1.5 mm with a YSZ layer of about 49 mm × 49 mm in area and ~15 μm in thickness. The porosity of the sintered anode substrate was about 30%. The anode and anode substrate were impregnated with Ni nitrate solution applying a vacuum pump to reduce the remaining air inside the pores of the substrate. After subsequent heating of the half-cells up to 500 °C, about 3 wt% NiO was immersed in the porous structures. A Gd_{0.2}Ce_{0.8}O_{1.9} (CGO; ~1 μm) and a YSZ (~0.5 μm, only to certain cells) interlayer between electrolyte and LSC cathode were applied by magnetron sputtering with a commercial physical vapor deposition (PVD) cluster system (CS 400 ES, von Ardenne Anlagentechnik, Germany). LSC cathodes were also fabricated by screen printing. The cathodes were sintered directly at 900 °C during performance testing.

2.3. Characterization

X-ray diffraction using a Siemens D5000 diffractometer with Cu Kα radiation showed that the SYT powders were pure perovskite after calcination. The microstructure of the cells after performance testing was investigated by scanning electron microscopy (SEM, Zeiss Ultra55).

2.4. Electrochemistry

The cells tested under H₂/H₂O as fuels have effective cathode areas of 10 mm × 10 mm, with 2 LSC reference electrodes in the size of 4 mm × 3 mm. The electrochemical performance was tested with

a single cell test bench [23]. The cells were integrated into an Al₂O₃ housing and sealed by a gold frame. The sealed area was about 45 mm × 15 mm. Gold meshes as current collectors were pressed onto the 1 cm² cathode and the matching position of the anode side by an Al₂O₃ flowfield (1 mm × 1 mm gas channels, contact rib width of 1 mm). The gas flows to the anode and the cathode (counter-flow) were controlled by digital mass flow controllers (Bronkhorst F-201CV). The flow rates were related to standard conditions (25 °C, 1 atm). The current–voltage curves of the cells were measured at temperatures ranging from 850 to 600 °C using dry H₂ as fuel and air as oxidant. However, a steam content of 3–4% in the fuel gas was produced by undesired fuel utilization from gas leakages. The impedance spectra of the cells were measured at open-circuit voltage during performance tests using a Solartron 1260A frequency response analyzer in the frequency range of 10⁶ Hz to 0.2 Hz. The redox behavior of the cells was investigated after performance testing. The anode gas was first changed from H₂ to N₂ and maintained for 1 min at a N₂ flow rate of 0.3 l_n min⁻¹, then air (0.15 l_n min⁻¹) was fed to the anode for certain time. After 1 min of purging with 0.3 l_n min⁻¹ N₂ once again, 0.25 l_n min⁻¹ H₂ was delivered to the anode chamber for certain time to reduce the anode again.

The cells tested under CH₄/H₂O as fuels had effective cathode areas of 32 mm × 32 mm. The cells were sealed with Au frames. Pt current collectors were used for both anode and cathode. The performance of the cells was tested under anode gas flow of 130 and 500 ml min⁻¹ of CH₄ and N₂, respectively, with steam/carbon (S/C) ratios of 2, 1.5, 1 and 0.5. As oxidant a gas flow of 1600 ml min⁻¹ N₂ and 400 ml min⁻¹ O₂ was used.

3. Results and discussion

Two kinds of single cells were fabricated based on Sr_{0.895}Y_{0.07}TiO₃ (SYT) anode substrates and (Sr_{0.89}Y_{0.07})_{0.91}TiO_{2.91}/YSZ (2:1 in volume, SYT2-YSZ) anode layers. The reason for using A-site deficient SYT2 as anode material is the higher conductivity after mixing with YSZ [20]. Both types of cells have planar design with the dimension of 50 mm × 50 mm. The details of cell architecture used in this work are shown in Table 2.

Previously Ti diffusion was found in the YSZ electrolyte after co-sintering with SYT support [20]. It is known that Ti additions to YSZ increase the electronic conduction [11,24]. Therefore an additional PVD layer of YSZ was coated on top of the sintered YSZ layer of cell A to ensure at least a thin Ti-free YSZ layer as shown in Fig. 1. The layer is about 0.5 μm in thickness, dense and adheres well to both CGO protecting layer and sintered YSZ electrolyte. Since the PVD layers were prepared at about 800 °C, solid state reaction between CGO and YSZ is unlikely to take place. Ti diffusion into the PVD-YSZ layer can also be neglected. In comparison, cell B has only a co-

Table 2

Characteristics of cells used in this study.

Cell type	Anode support	Anode	Electrolyte	Diffusion barrier layer	Cathode
A	SYT (1.2 mm) infiltrated with NiO	SYT2/YSZ (~3 μm) infiltrated with NiO	YSZ (~14 μm + ~0.5 μm by PVD)	CGO (~1 μm)	LSC (~50 μm)
B	SYT (1.2 mm) infiltrated with NiO	SYT2/YSZ (~3 μm) infiltrated with NiO	YSZ (~14 μm)	CGO (~1 μm)	LSC (~50 μm)

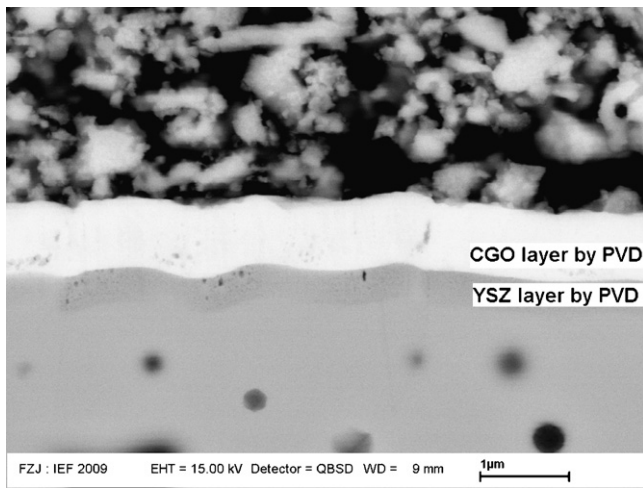


Fig. 1. Cross-sectional view of YSZ/PVD-YSZ/PVD-CGO/LSC interfaces of cell type A.

sintered YSZ layer. The electrochemical performances of the two types of cells are shown in Fig. 2.

The OCV of the cell A is 1.05 V at 800 °C and 1.11 V at 600 °C, very close to the theoretical value indicating the electrolyte layer is sufficiently dense and has negligible electronic conduction. The current density of the cell is 0.94 A cm⁻² at 0.7 V and 800 °C. This leads to power densities for practical use and gives good prospect for commercial application. However, the current density decreases significantly with decreasing temperature. At 600 °C the current density is only 0.08 A cm⁻² at 0.7 V. Further optimization of the cell design is necessary to improve area-specific resistance and electrochemical performance.

The only difference of cell B in comparison to cell A is the omitted PVD-YSZ layer on top of the co-sintered YSZ layer. The other components of the two kinds of cells are exactly the same and prepared in the same batch. A lower OCV was anticipated for cell B because there was no Ti-free YSZ layer to block the possible electronic conduction. However, surprisingly the OCV of cell B is even slightly higher than that of cell A. The values are 1.09 and 1.14 V at 800 and 600 °C, respectively. One possible reason is that the amount of Ti in the YSZ layer is still not sufficient to create pronounced electronic conduction. The performance of cell B is 1.22 A cm⁻² at 0.7 V and 800 °C and the calculated maximum power density is more than 1 W cm⁻². It is the best electrochemical performance of an SOFC based on a ceramic anode reported until now. This performance is also better than that of cell A, but the higher current

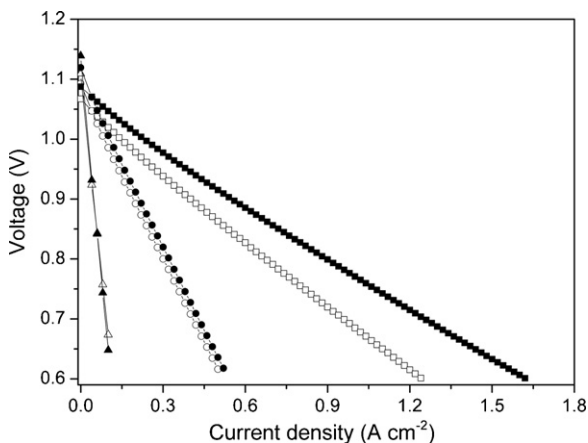


Fig. 2. Cell voltages as a function of current density for cell A (hollow symbols) and cell B (closed symbols) at 800 °C (squares), 700 °C (circles) and 600 °C (triangles).

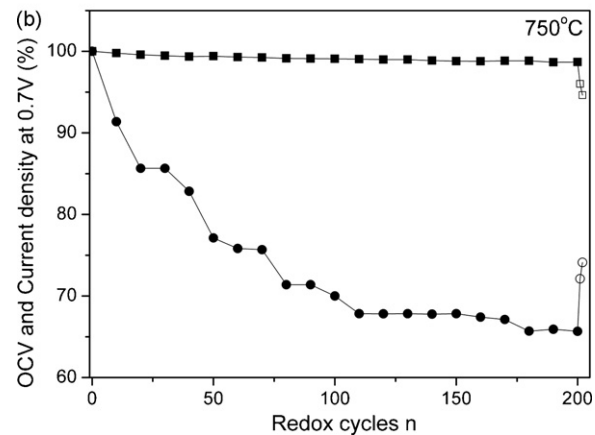
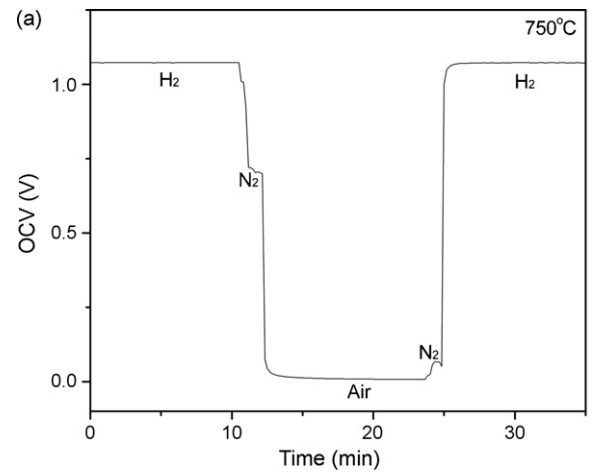


Fig. 3. (a) Test protocol for one redox cycle indicating the sequence of gas changes for the first 200 redox cycles. (b) Percentage of OCV (squares) and current density at 0.7 V (circles) of cell A to the data before redox cycles in dependence of the number of redox cycles at 750 °C. Closed symbols indicate exposing time in air and H₂ for 10 min each as showed in (a), open symbols indicate the exposing time for 5 h each.

densities cannot be explained by the small additional bulk and contact resistances of the extra PVD-YSZ layer. It is more likely that the contacting of the two anodes is somewhat different due to the small bending of the cells. Similar to cell A, the performance of cell B also rapidly decreases with temperature resulting in 0.08 A cm⁻² at 600 °C and 0.7 V.

Redox tests were performed on cell A at 750 °C. The procedure of changing the anode gas for each redox cycle is shown in Fig. 3a. Fig. 3b shows the changes of OCV and current density at 0.7 V in dependence of the numbers of redox cycles. Impressively, after 200 redox cycles, the OCV only decreased by 1.3% indicating the high robustness and stability of the cell. This is the first time that an anode supported cell (ASC) – and emphasizing here the size of 50 mm × 50 mm – sustained 200 redox cycles. In contrast, SOFCs based on Ni/YSZ cermet usually collapsed or had apparent losses of OCV after just one redox cycle [5–10]. However, the performance of the cell decreased by 35% after 200 redox cycles. The reason is most likely the gradual loss of electrical conductivity after each redox cycle either due to an irreversible decrease of charge carriers or the slow kinetics between the reduced and oxidized state of SYT. It was reported that the conductivity of Y-substituted SrTiO₃ sintered in reducing atmosphere significantly depends on the oxygen partial pressure during testing [14,20,25]. The values are nearly 100 S cm⁻¹ after sintering in Ar/H₂ and only about 0.01 S cm⁻¹ in air, both measured at 800 °C. If the atmosphere is changed from air back to Ar/H₂, the conductivity can restore, but it needs minutes to hours depend-

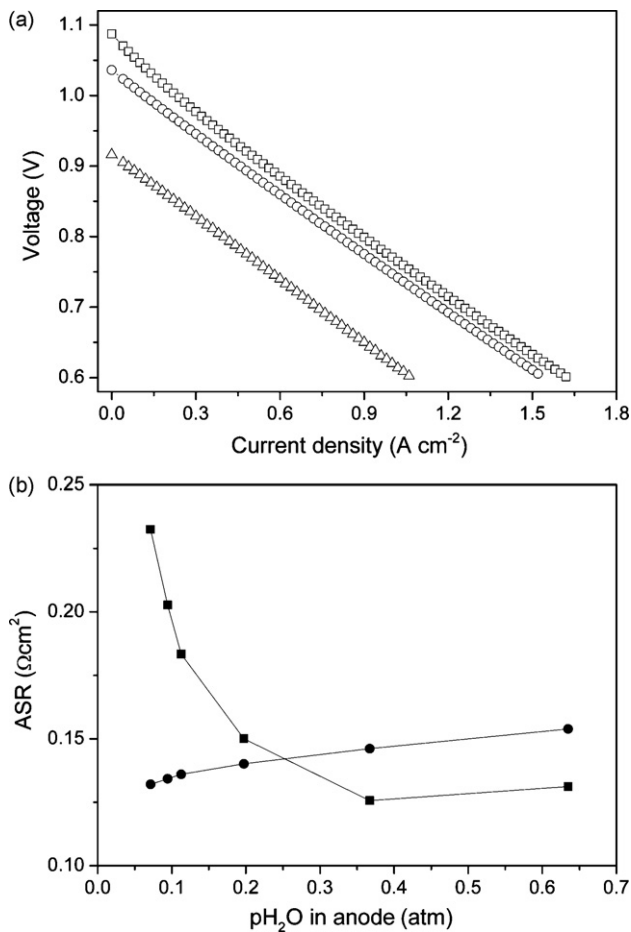


Fig. 4. (a) Cell voltages as a function of current density for cell B at 800 °C using 3% H₂O/H₂ (squares), 8% H₂O/H₂ (circles) and 60% H₂O/H₂ (triangles) as fuels. (b) Ohmic resistance (R_0 , circles) and total polarization resistance (R_{pol} , squares) of cell B as a function of water partial pressure at 800 °C determined from impedance spectra.

ing on the density of the anode substrate. In this study, it is very likely that the time for reduction during each redox cycle is not long enough to restore the conductivity of the anode after the oxidizing period, which results in a continuous performance decrease with the redox cycling. The amount of performance loss is not a severe problem because during practical application of SOFCs the event of re-oxidation is regarded as a kind of ‘accident’ during operation and there is enough time to recover the anode status after that failure mode. It is also showed in Fig. 3b that after the 200 redox cycles, when the exposing time in air and H₂ was changed from 10 min to 5 h, the OCV of the cell had apparent decrease, and meanwhile the power output of the cell was also increasing, indicating there was already cracks on the electrolyte of the cell. Although the redox stability of SYT based ASC is much higher than Ni/YSZ cermet based cells, the cell will also break down if the redox conditions are too severe. But fortunately, the redox condition as severe as this is also unlikely to happen during practical application of SOFCs.

Fig. 4a shows the performance of cell B at 800 °C with 3%, 8% and 63% H₂O in H₂ as fuel gases. It is reasonable that the OCV and current density at 0.7 V are decreasing with increasing water partial pressure ($p(\text{H}_2\text{O})$), because the oxygen partial pressure in moistured H₂ is orders of magnitude higher than in dry H₂. But it is uncommon that the area-specific resistance (ASR) of the cell calculated from the i - V curve also shows a tendency of increase with increase of $p(\text{H}_2\text{O})$, which is 0.276, 0.274 and 0.299 Ω cm² for 3%, 8% and 63% H₂O/H₂, respectively. For typical Ni/YSZ cermet-based cells, since the conductivity of Ni is only related to temperature,

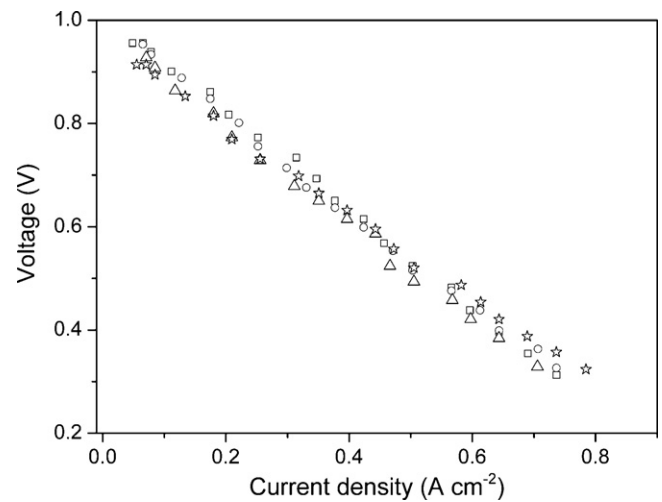


Fig. 5. Cell voltages as a function of current density of cell B using H₂O/CH₄ mixtures as fuel gases. The squares, circles, triangles and stars represent S/C ratios of 0.5, 1, 1.5 and 2, respectively.

the ohmic resistance of the cell does not change with $p(\text{H}_2\text{O})$ as long as the oxygen partial pressure is not high enough to oxidize Ni to NiO. And the polarization resistance of the cell generally decreases with increasing $p(\text{H}_2\text{O})$ because the steam in the fuel gas atmosphere has a catalytic impact on the anode performance [26–30]. Therefore, the ASR of Ni/YSZ cermet-based cells always decreases with increasing $p(\text{H}_2\text{O})$. Impedance spectra of cell B were recorded at different $p(\text{H}_2\text{O})$ at open-circuit conditions to explain the unusual ASR increase of SYT-based cells (see Fig. 4b). Unlike Ni/YSZ cermet-based cells, the ohmic resistance of cell B increases with the increasing of $p(\text{H}_2\text{O})$, because the conductivity of SYT is very sensitive to the oxygen partial pressure [14,20,25]. In addition, the conductivity of the SYT anode substrate is normally between 10 and 100 S cm⁻¹, much smaller than the value for Ni/YSZ cermets (100–1000 S cm⁻¹). Therefore the ohmic resistance of SYT-based cells is comparable to the polarization resistance and even higher in high $p(\text{H}_2\text{O})$ atmospheres (see Fig. 4b). This is the main reason for the ASR increase of SYT-based cells with increasing $p(\text{H}_2\text{O})$. Similar to Ni/YSZ-based cells, the measured polarization resistance also decreases with increasing $p(\text{H}_2\text{O})$ indicating that steam can also positively affect the anodic reaction of SYT-based cells. However, at high $p(\text{H}_2\text{O})$ (>50%) an increase in polarization resistance is observed. The explanation of this phenomenon needs further more detailed experiments.

It is generally agreed that for Ni/YSZ cermets, an S/C ratio of at least 2 is necessary to prevent coking [31–33], although the efficiency of the cells is decreased because of the higher oxygen partial pressure in the fuel gas. High coking tolerance of SYT-based cells can be anticipated due to the low amount of Ni in the anode (3 wt%). Fig. 5 shows the electrochemical performance of cell B applying S/C ratios from 0.5 to 2 as fuel gas. The measured voltages are quite similar and no apparent performance loss was found for low S/C ratios indicating that the catalytic activity is not affected by coking. However, long-term stability tests are needed to further prove the coking tolerance and determine the limitations in fuel gas composition.

4. Conclusions

Single cells based on SYT anode supports were prepared and tested in this study. The performance of the cells can be as high as 1.22 A cm⁻² at 0.7 V and 800 °C, which implies good prospect for commercial application. The Ti diffusion from SYT to YSZ has no

apparent influence on the OCV of the cells indicating negligible electronic conduction is caused by Ti in the YSZ layer. The cells can also sustain 200 redox cycles between H₂ and air without significant decrease in OCV. Compared to Ni/YSZ cermet-based cells, p(H₂O) in anode gases slightly decreases the performance of SYT-based cells, because the ohmic resistance of SYT is sensitive to oxygen partial pressure. Preliminary experiment shows the coking tolerance of SYT-based cells, but further experiments are needed to prove this.

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