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Simplified processing of anode-supported thin film planar solid oxide fuel cells

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Abstract

The concept of anode-supported planar solid oxide fuel cells (SOFCs) involves different fabrication procedures and several sintering steps. This contribution is an attempt towards minimizing the number of sintering steps. By adopting an improved technique a pre-firing step was eliminated. Cells obtained following this co-firing technique showed relatively higher power density ($\sim 1 \text{ A cm}^{-2} \text{ at } 0.7 \text{ V}$) at an operating temperature of 800 °C compared to the cells prepared by the conventional technique. In the present investigation, cells as large as 10 cm × 10 cm were prepared and characterized following the new co-firing approach. Detailed microstructural, leak-rate testing (gastightness of the sintered electrolyte film) and electrochemical characterizations were carried out. © 2004 Elsevier Ltd. All rights reserved.

Keywords: Suspension; YSZ; ZrO₂; Fuel cells; Co-firing; Sintering

1. Introduction

The major focus in current solid oxide fuel cell (SOFC) research is the reduction of costs to an affordable range. Various cost-reduction steps have been considered for SOFCs including the use of low-purity raw materials, mass production of cell components, use of inexpensive and simplified fabrication processes, operation at intermediate temperature, etc.^{1–6} The Solid State Energy Conversion Alliance (SECA) program by the US Department of Energy (DOE) is perhaps one of the best collective efforts towards reaching the ultimate cost target.⁷ Of all the available SOFC designs the anode-supported solid oxide fuel cell (SOFC) concept provides a great opportunity for commercializing a low-cost product by introducing a metal (steel) as the interconnect material instead of traditional ceramic interconnectors. In addition, reduction of electrolyte thickness down to the range of $5-10 \,\mu\text{m}$ is a remarkable advantage, particularly in re-

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ducing the operating temperature of SOFCs in the range of 750–800 °C. Other advantages of such anode-supported structures have been discussed in the literature.^{8–10}

While dealing with such anode-supported SOFC structures, a large number of processing steps are often encountered which unnecessarily increase the complexity, fabrication time and cost. The present contribution is an attempt to develop high-performance cells by eliminating a key processing step.

2. Experimental

2.1. Fabrication of cells by conventional techniques

In this investigation conventional cell component materials were used for fabricating anode-supported thin film SOFC structures.^{5,8} Two simple fabrication techniques vacuum slip casting (VSC[®]) and wet powder spraying (WPS[®])—were used to develop uniform thin particulate films (5–12 μ m) of electrolyte based on yttria stabilized zirconia (YSZ) and anode functional layer (AFL, a mixture of NiO and YSZ). A schematic of these two key fabrication techniques is shown in Fig. 1. The common features of these two techniques are: (a) simple slurry chemistry, (b)

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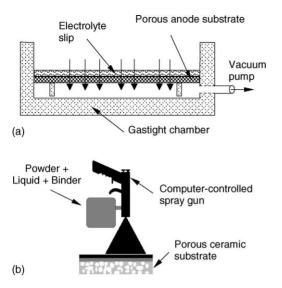


Fig. 1. Schematic of particulate deposition technique (a) vacuum slip casting (VSC) and (b) wet powder spraying (WPS).

ability to form thin films down to $5 \,\mu$ m, (c) low cost, and (d) potential for industrial production.

Complete cell fabrication at Forschungszentrum Jülich involves several processing steps followed by intermedi-ate firing steps.^{2,5,8,11} Porous NiO/YSZ cermet substrates were prepared by warm pressing and sintering. The porosity of the substrate was normally maintained within the range of 35–40%. Thin films of AFL (\sim 5 µm), electrolyte $(\sim 5 \,\mu m)$, cathode functional layer (CFL) $(\sim 15 \,\mu m)$, and cathode (\sim 40 µm) were deposited sequentially on these substrates with intermediate firing steps and thus a single SOFC was formed. A schematic of such an anode-supported structure is shown in Fig. 2. The anode substrate in this case acts as the support for all deposited films. After an AFL film had been deposited on the anode substrate, it underwent a pre-firing step at 1000 °C. A 1400 °C sintering step followed after a YSZ electrolyte film had been deposited on top of the fired AFL film. This firing step gave a gastight electrolyte film. On top of this dense electrolyte film, two cathode layers were sequentially deposited by WPS. One of the thick films was the CFL, which is a mixture of cathode material La_{0.65}Sr_{0.3}MnO₃ (LSM) and YSZ. The other film on top of the CFL is a cathode film (LSM) for current distribution. Sintering at 1100 °C was the final production process step.

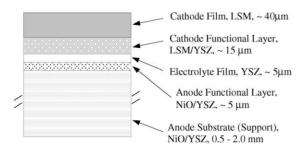


Fig. 2. Schematic of an anode-supported thin film structure.

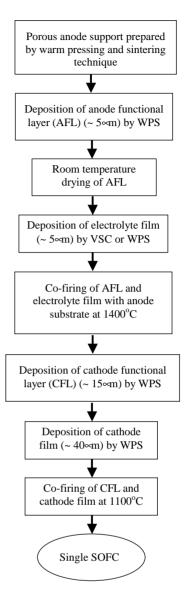


Fig. 3. Flow chart showing processing and firing steps for manufacturing of SOFCs by co-firing technique.

2.2. Fabrication of cells by co-firing technique

The present work aimed at the fabrication of an SOFC structure by co-deposition of AFL (NiO/YSZ) and electrolyte (YSZ) film in one step, followed by single-step firing at 1400 °C. The deposition and firing of two cathode thick films performed during cell fabrication in the same manner as described above. The fabrication steps involved in the present route (co-firing) are shown in Fig. 3. Notably, in this co-firing approach there was no intermediate firing step at 1000 °C, which was essential in the conventional approach at Forschungszentrum Jülich.

2.3. Preparation of electrolyte films

A combination of different fabrication approaches and corresponding firing schedules is listed in Table 1. While

 Table 1

 Sample preparation details with identification code

Preparation details	Sample preparation code
AFL (by VSC) + air drying + $1000 \degree C/1 h$ + electrolyte (by VSC) + $1400 \degree C/5 h$	SP-I
AFL (by WPS) + air drying + $1000 \degree C/1 h$ + electrolyte (by VSC) + $1400 \degree C/5 h$	SP-II
AFL (by VSC) + air drying + $1000 \circ C/1 h$ + electrolyte (by WPS) + $1400 \circ C/5 h$	SP-III
AFL (by VSC) + electrolyte (by VSC) + $1400 \degree C/5 h$	SP-IV
AFL (by VSC) + air drying + electrolyte (by VSC) + $1400 \circ C/5 h$	SP-V
AFL (by WPS) + air drying + electrolyte (by VSC) + $1400 \degree C/5 h$	SP-VI
AFL (by VSC) + air drying + electrolyte (by WPS) + $1400 \degree C/5 h$	SP-VII
AFL (by WPS) + air drying + electrolyte (by WPS) + $1400 \degree C/5 h$	SP-VIII

SP-I describes the conventional preparation procedure and SP-II and SP-III are variations of this procedure with an intermediate pre-firing step at 1000 °C, SP-IV to SP-VIII represent the co-firing procedure without any such intermediate firing schedule. Anode substrates having dimensions of $50 \text{ mm} \times 50 \text{ mm} \times 1.5 \text{ mm}$ and $100 \text{ mm} \times 100 \text{ mm} \times 1.5 \text{ mm}$ were used in this investigation. Among the large number of samples used in this investigation, a few representative samples with specific sample preparation codes are listed in Table 2. These samples were prepared following the processing techniques listed in Table 1. In the first set, the SP-I technique was applied to prepare samples used as references for anode-supported cells. Similarly, from the second set, three co-firing techniques, SP-V, SP-VI, and SP-VIII, were chosen.

All these four techniques—SP-I, SP-V, SP-VI, and SP-VIII-involved the deposition of particulate thick films. Therefore, the suspension chemistry played a crucial role in producing good quality films. Ethyl-alcohol-based AFL (NiO/YSZ) and electrolyte (YSZ) suspensions were prepared by extensive 12 h ball milling of 8YSZ (Tosoh), NiO (J.T. Baker) and organic binder. The particle size distribution and d_{50} value of the suspensions were routinely monitored using a Shimadzu particle size analyzer SA-CP3. The suspension preparation details are reported elsewhere.^{5,11} Special precautions were taken to prevent the escape of NiO aerosol into the environment while spraying NiO/YSZ (AFL) suspensions onto the substrate.

Table 2 Details of the samples used in the present investigation

Sample ID	Dimensions	Sample preparation code
A-1 and A-2	$50\mathrm{mm} imes50\mathrm{mm} imes1.5\mathrm{mm}$	SP-I
B-1 and B-2	$50 \mathrm{mm} \times 50 \mathrm{mm} \times 1.5;\mathrm{mm}$	SP-V
C-1 to C-4	$50\mathrm{mm} imes50\mathrm{mm} imes1.5\mathrm{mm}$	SP-VI
D-1 to D-4	$50\mathrm{mm} imes50\mathrm{mm} imes1.5\mathrm{mm}$	SP-VIII
E-1	$100 \text{ mm} \times 100 \text{ mm} \times 1.5 \text{ mm}$	SP-I
F-1	$100 \text{ mm} \times 100 \text{ mm} \times 1.5 \text{ mm}$	SP-VI
G-1	$100 \text{ mm} \times 100 \text{ mm} \times 1.5 \text{ mm}$	SP-VIII

2.4. Quantitative leak testing

After sintering the electrolyte film (for both sets of cells), the gastightness of these ceramic films on the porous substrates was measured using a He leak-test technique.^{11,12} This characterization step was very important in determining the quality of an individual cell. For conventionally fabricated cells the threshold value of such a leak-rate is $<2 \times 10^{-5}$ mbar l cm⁻² s⁻¹—the lower the threshold value the higher the gas tightness.

2.5. Microstructural studies

In this investigation, an extensive microstructural study was carried out using both high-resolution optical microscopy and scanning electron microscopy (SEM, LEO 440). For this purpose, polished cross-sections of the anode-supported thin film structures were used.

2.6. Electrochemical test

Electrochemical tests were performed on a few selected samples prepared by SP-I, SP-V, SP-VI, and SP-VIII. Once the gastightness of the electrolyte had been measured in the leak-test experiment, two cathode layers were deposited onto the electrolyte film by WPS. Finally, a sintering step was performed at 1100 °C. The thickness of the CFL and cathode film was maintained at ~ 15 and $\sim 40 \,\mu$ m, respectively (Fig. 3). While samples with identical dimensions of $50 \text{ mm} \times 50 \text{ mm} \times 1.5 \text{ mm}$ were used for the electrochemical test, the cathode film was deposited on an area of $40 \text{ mm} \times 40 \text{ mm}$ (effective cell area). The electrochemical test was performed in a test rig made of alumina. Moist hydrogen was used as the fuel and air as the oxidant. The flow rates of H₂, H₂O, and air were maintained at 1000, 30, and 1000 ml/min, respectively. Gold foil was used as sealing. The anode was reduced in situ during the measurement, and remained under H₂ atmosphere. The cell performance was characterized at four different temperatures: 750, 800, 850, and 900 °C. The current applied was within the range of 0-12 A with an interval of 1 A. The current and voltage data were collected continuously by a dedicated computer.

3. Results and discussion

3.1. Development of anode functional layer (AFL) for co-firing technique

As already mentioned, the first step in developing an SOFC structure was depositing a film of NiO/YSZ (AFL) on top of the pre-fired anode substrate (NiO/YSZ). In the conventional method, AFL was deposited only by VSC, while in the present co-firing approach, either VSC or WPS was used. Therefore, at the beginning it was necessary to standardize the processing parameters that control the WPS. The thickness of the film was built up by successive deposition of particulate layers from the NiO/YSZ suspension. Once the layers had been deposited, they were allowed to dry in air. The dried film surface was thoroughly observed under the optical microscope and no crack or surface defect was detected. A maximum temperature of 1400 °C was used to sinter the dried AFL film. Optical and scanning electron microscopy (SEM) were employed to check the uniformity and overall quality of the sintered film as well as to estimate the film thickness. The different stages of AFL development on the porous substrates are shown in Fig. 4. Deposition of a single particulate layer of NiO/YSZ was not sufficient; therefore several layers were deposited sequentially. A minimum of seven layers were required to build up a layer thickness of around 6 µm. The sintered layers of NiO/YSZ were fairly uniform as was observed in the SEM cross-sections (Fig. 4). Although the coatings looked dense, the overall porosity normally reached about 20% after reduction of the NiO to Ni in hydrogen.

3.2. Development of zirconia electrolyte film

Once the AFL layer had been formed, the next step was to deposit electrolyte films on the top of either the unfired or pre-fired AFL. It was noted that the YSZ films were prepared both by WPS and VSC followed by sintering at 1400 °C. Fig. 5 depicts SEM cross-sectional images taken from different samples prepared both by conventional (SP-I) and co-firing techniques (SP-V, SP-VI, and SP-VIII). Fig. 5a represents a typical microstructure for samples prepared by SP-I. The microstructures obtained by using the above-mentioned three co-firing options are shown in Fig. 5b and d. All the microstructures as depicted in Fig. 5 resembled each other irrespective of fabrication approaches. The quality of the film as well as variation in electrolyte and AFL thicknesses were noted. This variation was primarily due to the amount of slurry (in the case of VSC) or number of layers (in the case of WPS) needed during deposition.

3.3. Gastightness testing of sintered electrolyte film

The microstructural investigations do not provide direct information regarding the gastightness of the electrolyte. Therefore He leak-test experiments were performed on samples sintered at 1400 °C. Fig. 6 shows the leak-rate values amongst the different preparation approaches adopted in this investigation. Among the three co-firing approaches the leak-rate values for samples B-1 and B-2 prepared using the SP-V approach were 9.47×10^{-5} mbar $1 \text{ cm}^{-2} \text{ s}^{-1}$ and 8.05×10^{-5} mbar 1 cm⁻² s⁻¹—the values were well above the leak-rate threshold as indicated by the horizontal line in Fig. 6. It was furthermore noted that the leak-rates were auite satisfactory for the other two co-firing approaches, i.e. SP-VI and SP-VIII. Even in the case of the co-fired technique, it was possible to prepare gastight zirconia electrolyte films with leak-rate values of $\sim 10^{-6}$. The leak-rate regime as obtained from SP-VI and SP-VIII was very close to the samples prepared by SP-I. It was also verified that not only the $5 \text{ cm} \times 5 \text{ cm}$ co-fired samples, but also co-fired samples with larger areas $(10 \text{ cm} \times 10 \text{ cm})$ were comparable to the conventional technique. As expected, the densification (gastightness) is independent of cell dimensions. However, the variation in gastightness as observed particularly for SP-V may suggest a strong influence on the fabrication process. In this case, when YSZ electrolyte deposition was performed, the particulate arrangements within the electrolyte film could disturb and affect the green compaction. As a result, the sintered film did not provide complete gastightness for the SP-VIII approach (Fig. 6).

3.4. Electrochemical study

The electrochemical tests were performed for a few selected samples and measured at four different temperatures: 750, 800, 850, and 900 °C. Fig. 7 shows the current (I) versus voltage (V) characteristic at 750 $^{\circ}$ C for samples prepared by conventional (SP-I) and co-firing techniques (SP-V, SP-VI, and SP-VIII). Fig. 8 depicts the power density of the samples as a function of the testing temperature keeping the voltage constant at 0.7 V. Although the leak-rate values for samples prepared by SP-VIII (co-firing) were below the threshold limit of 2×10^{-5} mbar l cm⁻² s⁻¹ the measured current density values at 750 and 800 $^{\circ}$ C were only 0.2 and 0.35 A cm⁻². On the other hand, the corresponding values for another co-firing approach, SP-VI, were remarkably high-0.6 and 1 A cm⁻² at 750 and 800 °C, respectively. Furthermore, the current density values for the SP-I approach at the above two temperatures were 0.45 and 0.7 A cm⁻², respectively. Measurements were also performed at 850 and 900 °C. However, the focus of interest for the present investigation was to develop cells to operate at somewhat lower temperatures (preferably between 750 and 800 °C). Fig. 8 summarizes the cell performance for the four samples used in this investigation. Interestingly, it was noted that the lowest current density was obtained from sample B-1 prepared by SP-V measured at 900 °C. The result was in good agreement with the poor leak-rate values as shown in Fig. 6. However, a deviation from the same trend was observed for other measurement temperatures between 750 and 850 °C. One reason for such a deviation might be the relatively poor quality

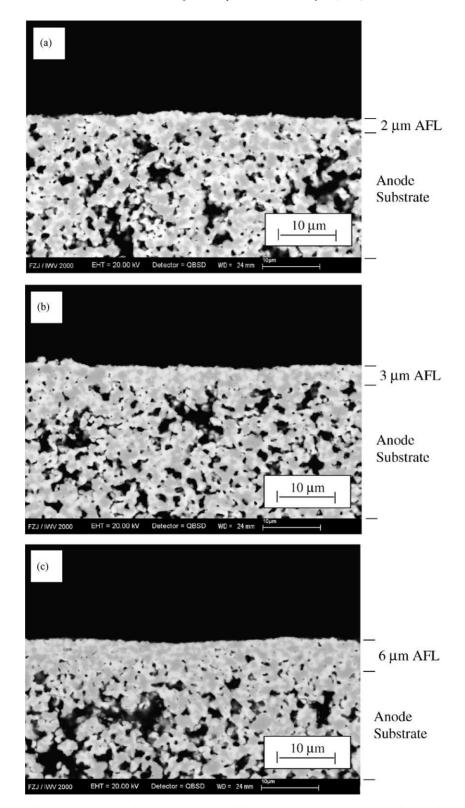


Fig. 4. SEM images of the polished cross-sections of sintered samples ($1400 \circ C/5 h$) showing the development of anode functional layer (AFL) on the porous anode substrates. The images shown above were obtained after deposition of (a) three, (b) five, and (c) seven layers of NiO/YSZ particulates from AFL suspensions by WPS.

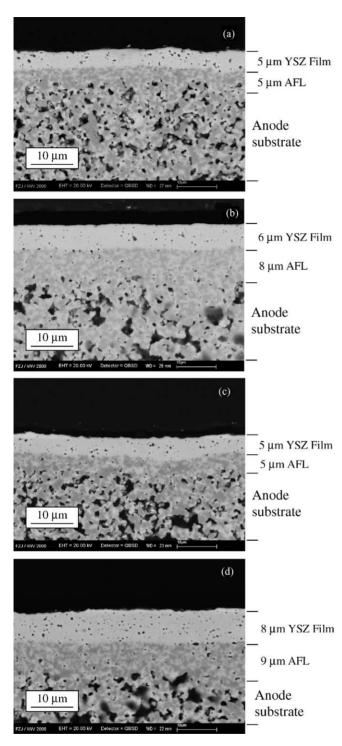


Fig. 5. Cross-sectional views of SEM micrographs of polished samples prepared by conventional and various co-firing techniques: (a) SP-I, (b) SP-V, (c) SP-VI, and (d) SP-VIII.

electrolyte film (leak-rate for sample B-1: 9.47×10^{-5} mbar l mbar l cm⁻² s⁻¹) obtained when prepared by SP-V (Table 1). Note that in this case VSC was performed to deposit particulate films of both AFL and electrolyte.

The next set of data as obtained from another co-firing approach SP-VIII (sample D-1) was provided moderate cur-

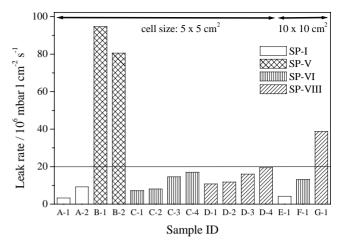


Fig. 6. Comparison of leak-rate values for the samples used in this investigation. The horizontal line indicates the threshold value of 2×10^{-6} mbar $1 \text{ cm}^{-2} \text{ s}^{-1}$ (see text).

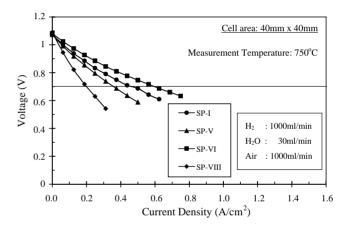


Fig. 7. Current–voltage characteristics as a function of processing technique for operating temperature 750 $^\circ\text{C}.$

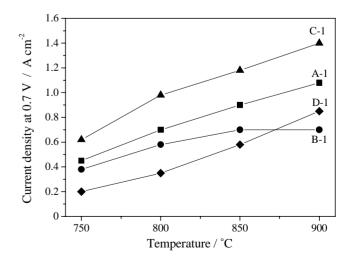


Fig. 8. Comparison of cell performances as a function of processing technique (cf. Tables 1 and 2).

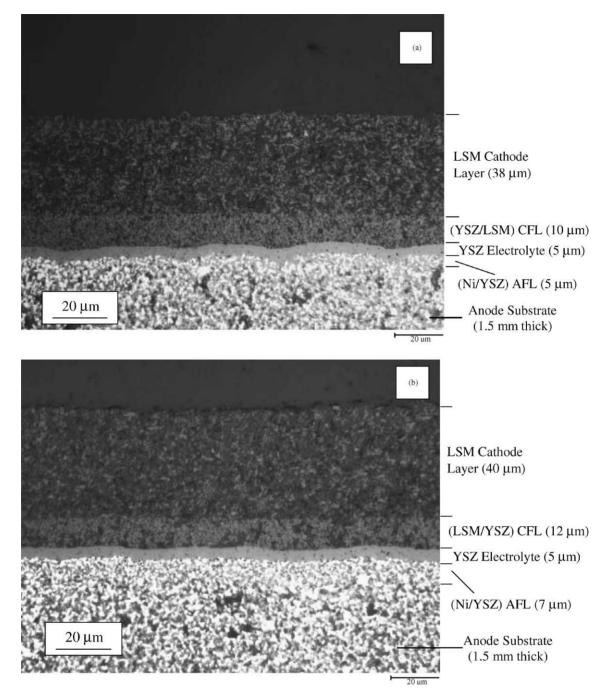


Fig. 9. Optical micrographs of the cross-sectional views of the samples: (a) A-1, prepared using conventional technique, SP-I and (b) C-1, prepared using co-firing technique, SP-VI. Cathode functional layer and cathode layer were simultaneously deposited on the sintered YSZ film and then finally fired at 1100 °C for 3 h. All samples were reduced during the electrochemical test. The micrographs were taken of the polished cross-section of such reduced cells.

rent density values compared to approach SP-VI (sample C-1). While the co-firing technique indicated a possibility of an improved technique for fabricating cells by bypassing a firing step, the performance data of sample A-1 (conventional technique, SP-I) used as the reference unambiguously demonstrated the potential of one co-firing approach, in particular SP-VI (Table 1). This approach offered a fairly high current density (0.6 A cm⁻² at 0.7 V) even at the lowest operating temperature of 750 °C (Fig. 7). Not only at

750 °C, but also at other measurement temperatures the current densities were superior to other co-firing as well as conventional techniques (sample C-1 in Fig. 8). It should be noted here that the SP-VI approach consisted of a combination of two particulate deposition processes—deposition of AFL by WPS followed by electrolyte film deposition by VSC without any intermediate firing. Therefore, one of the reasons that such a high current density was obtained in this co-fired case (SP-VI) might be the combination of the two

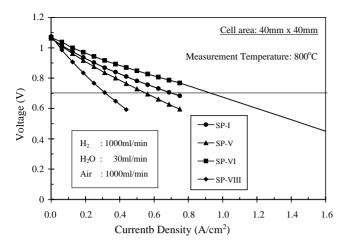


Fig. 10. Current–voltage characteristics as a function of processing technique for operating temperature 800 °C.

above-mentioned deposition techniques in the green condition producing a favorable particulate compaction. Hence, due to the combination of these processes, some changes in the microscopic level might occur at the interface resulting in a good quality product.⁵

3.5. Microstructures of complete cells

Though the electrochemical study shows SP-VI as one of the promising co-firing techniques, the microstructures of complete cells did not reflect this fact. Fig. 9 depicts a microstructural comparison between the two complete cells—sample A-1 (conventional technique, SP-I) and C-1 (co-firing technique, SP-VI). No remarkable microstructural differences were observed.

Fig. 10 shows a typical SEM image of the polished cross-section and surface morphology (without polishing) of the most promising sample prepared by the co-firing approach (SP-VI). A fully dense well-adhering zirconia film was observed on the top of the porous NiO/YSZ anode substrate.

4. Conclusions

In this investigation of how to manufacture an anodesupported thin film structure, the particulate layers (3–12 μ m) of NiO/YSZ (anode functional layer) and YSZ (electrolyte) were deposited on a porous and flat anode (NiO/YSZ) substrate using the VSC and WPS techniques. Both deposition techniques are simple, inexpensive and suitable for mass production. The suspension chemistry and processing parameters for WPS were optimized. Approximately 6–7 deposition runs (by WPS) were required to build ~5 μ m thick AFL (NiO/YSZ), while 5–6 deposition runs were needed to achieve a ~5 μ m thick electrolyte (YSZ) film. In this investigation, a pre-firing step at 1000 °C was eliminated. After electrolyte deposition the composite was co-fired at 1400 °C. A remarkably high current density of 1 A cm⁻² was found at 800 °C (measured at 0.7 V) for the co-fired samples where the NiO/YSZ film was deposited by WPS and the electrolyte (YSZ) by VSC. Samples prepared using this co-firing technique were even better than the samples produced by the conventional technique. These samples offered a leak-rate value within the range of 10^{-6} mbar l cm⁻² s⁻¹ obtained by a He leak-test. One of the reasons for such a high current density as that obtained in the case of co-fired samples might be the combination of these two deposition techniques (AFL by WPS and electrolyte by VSC) which produced a relatively better particulate compaction during fabrication of the SOFC structure. Cell areas as large as $10 \text{ cm} \times 10 \text{ cm}$ were also manufactured by the present simplified co-firing approach and a similar quality of product was obtained.

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