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Low temperature solid oxide fuel cells with pulsed laser deposited bi-layer electrolyte

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deposition

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ABSTRACT

Solid oxide fuel cells using a pulsed laser deposited bi-layer electrolyte have been successfully fabricated and have shown very good performance at low operating temperatures. The cell reaches power densities of 0.5 W cm^{-2} at 550°C and 0.9 W cm^{-2} at 600°C , with open circuit voltage (OCV) values larger than 1.04 V. The bi-layer electrolyte contains a $6 \sim 7 \mu\text{m}$ thick samarium-doped ceria (SDC) layer deposited over a $\sim 1 \mu\text{m}$ thick scandium-stabilized zirconia (ScSZ) layer. The electrical leaking between the anode and cathode through the SDC electrolyte, which due to the reduction of Ce^{4+} to Ce^{3+} in reducing environment when using a single layer SDC electrolyte, has been eliminated by adopting the bi-layer electrolyte concept. Both ScSZ and SDC layers in the bi-layer electrolyte prepared by the pulsed laser deposition (PLD) technique are the highly conductive cubic phases. Poor conductive $(\text{Zr, Ce})\text{O}_2$ -based solid solutions or β -phase ScSZ were not found in the bi-layer electrolyte prepared by the PLD due to low processing temperatures of the technique. Excellent reliability and flexibility of the PLD technique makes it a very promising technique for the fabrication of thin electrolyte layer for SOFCs operating at reduced temperatures.

INTRODUCTION

High operating temperature is one of the main barriers for wide scale adoption of solid oxide fuel cells (SOFC) technology [1]. Therefore, most of the research work is focused on the development of low temperature SOFCs operating at around 500-600°C [2-4]. To achieve the objective of low operating temperatures, a number of approaches from the viewpoint of new materials, novel processes, and unique architectures have to be re-examined.

The practical operating temperature of SOFCs is mainly determined by the conductivity and thickness of the electrolyte. Samarium-doped ceria (SDC) is a promising material for its uses as the electrolyte in reduced temperature SOFCs and has been investigated widely over the past two decades [3, 4]. SDC exhibits relatively high conductivity of 0.1 S/cm at 700 °C [5], 2-3 times higher than that of yttrium-stabilized zirconia (YSZ). Its thermal expansion coefficient ($12.5 \times 10^{-6} \text{ K}^{-1}$) is also more compatible with that of the Ni-cermet anode and commercial ferritic stainless steel interconnect, than with that of YSZ. Although the material exhibits better chemical and structural compatibility with electrodes as well as higher ionic conductivity than YSZ, the reduction of Ce^{4+} to Ce^{3+} induces n-type electronic conduction which tends to decrease the power output of solid oxide fuel cells due to an internal electrical shorting. The problem can be eliminated by using a barrier of thin scandium-stabilized zirconia (ScSZ) layer deposited under the SDC layer to improve the stability of the SDC layer under the reducing atmosphere. The ionic conductivity of ScSZ is the highest among all the ZrO_2 -based electrolytes [6],

however, it is not considered as the candidate electrolyte for the reduced temperature SOFCs since its conductivity decreases rapidly with temperature below 800°C [7]. When the bi-layer SDC/ScSZ electrolyte was used, ScSZ layer acted mainly as the barrier and its layer thickness can be in the range of a few tens to a few hundreds of nanometers, therefore its relative low conductivity will not contribute significantly to the overall ohmic loss of the bi-layer SDC/ScSZ electrolyte for the SOFCs. For the fabrication of the bi-layer SDC/ScSZ electrolyte on anodic-supported SOFCs, wet ceramic processes like screen printing and tape castings have been used. These processes normally require processing temperatures over 1300°C. At such high co-fired temperatures, doped ceria (i.e., SDC) reacts easily with stabilized zirconia (e.g., ScSZ, YSZ) to form (Zr, Ce)O₂-based solid solutions which have a very low ionic conductivity [8, 9]. Ohmic loss due to the low-conductive interfacial layer will result in poor performance of SOFCs [10]. In addition, highly conductive cubic phase ScSZ, stabilized at high temperatures, will transfer to a less conductive β -phase when ScSZ is cooled down below 800°C. To avoid those issues, low temperature techniques (under 800°C) such as pulsed laser deposition (PLD) are attractive. PLD is an emerging physical vapor deposition technique that offers better control over the film properties, such as microstructures, chemical composition, density and interfacial properties. Grain size from a few nanometers to a few hundred nanometers can be easily prepared by selecting appropriate processing parameters. Recently, an enhancement of ionic conduction was found in the nanocrystalline ScSZ, whose specific grain boundary conductivity is 1-2 orders of magnitude higher than that of the microcrystalline specimen at 700-850°C [11]. PLD technique usually can operate at

low processing temperatures, which can suppress grain growth to achieve nanocrystalline materials.

Lower temperature processes like PLD is particularly suitable for the fabrication of miniaturized SOFCs based on multilayer thin films. This method not only can deposit a dense electrolyte thin layer, but also porous anode and cathode layers, therefore it possesses the potential for automation. In addition, this technique holds promise for improved quality control and integrated testing to enhance the reliability, performance and durability of the resulting product while dramatically reduces the costs.

The focus of this research is on the development of the PLD process to fabricate SDC/ScSZ bi-layer electrolyte with controlled microstructures, density and interfacial properties. Si(100) wafers and NiO-SDC modified NiO-YSZ cermet discs are used as the substrates in this study. The Si substrates were used to develop the optimal PLD processing parameters for the deposition of a dense and highly conductive bi-layer SDC/ScSZ electrolyte. The bi-layer electrolyte was then deposited on porous anode substrates under optimized processing conditions and their structures and morphologies were characterized by XRD and SEM. A unit cell that utilized the PLD prepared bi-layer electrolyte and wet-processing prepared NiO-SDC anode and $\text{Sm}_{0.5}\text{Sr}_{0.5}\text{CoO}_3$ -SDC composite cathode was tested for its electrochemical performances.

EXPERIMENTAL

Starting Materials and cell substrates

Table 1 lists the starting materials used in this study. All the materials for wet ceramic processing and PLD processing are commercially obtained. Figure 1 shows a schematic drawing of the cell structure and its fabrication methods.

The cell substrate was the NiO-YSZ cermet consisting of 57 wt% NiO and 43 wt%YSZ, prepared by the tape casting method. NiO-SDC modified layer, used as the anode, was applied by screen-printing on the cell substrate. The composition of the NiO-SDC anode was 47 wt.% SDC and 53 wt.% NiO. The Ni volume percent (41 vol.%) in the modified layer is the same as that in the NiO-YSZ cermet substrate. The modified cell substrate of 20mm diameter (by punching) was fired at 1380°C for 2hrs. The heat-treated cell substrate has 17.5 mm in diameter and 0.8 mm in thickness. The NiO-SDC anode layer is approximately 15 μm thick with the average surface roughness of approximately 0.66 μm measured by the Wako (WYKO??) instrument. The average density of the cell substrate after the anode layer deposition is 5.16 g cm^{-3} measured by Archimedes' method. It indicates a 19.4 % porosity in the NiO-YSZ cermet substrate based on the cermet substrate composition of 53 wt.% NiO. The substrate porosity was increased to approximately 37 % after reducing NiO to Ni with the hydrogen gas.

Pulsed Laser Deposition (PLD) of electrolytes

The bi-layer SDC/ScSZ electrolyte films were deposited by ablating a 90 mm diameter rotating SDC target (SDC, 99.9%, from Praxair) and a ScSZ target (prepared by slip casting and sintering at 1500°C for 5 hrs), subsequently, in an advanced deposition chamber (PVD. Inc., PLD-3000) by means of a pulsed KrF excimer laser ($\lambda = 248 \text{ nm}$,

Lambda Physik, LPX-210i), at a repetition rate of 50 Hz. The laser beam was focused down to a spot size of $\sim 4 \text{ mm}^2$ on the target surface. The on-target laser beam fluence was adjusted to about 3 to 5 J/cm^2 . Silicon wafers [(100) orientation, p-type, $\rho = 10\text{-}30 \ \Omega \text{ cm}$, from Polishing Corporation of America] and the NiO-SDC modified NiO-YSZ anode substrates.

Before introducing a Si wafer into the deposition chamber, it was cleaned by acetone, and isopropyl alcohol, and then etched in 2.5 % HF acid for five minutes to remove the native oxide. No further cleaning was necessary for the NiO-SDC modified NiO-YSZ substrates. After loading the substrate, the system was pumped down to a base pressure below 2×10^{-7} Torr using a turbo-molecular pump. The substrate was then heated, under vacuum, using a programmable non-contact radioactive heater. The substrate to be coated was facing the target, with a stand-off distance of 12 cm. To achieve uniform deposition over the entire substrate surface, the laser beam was rastered over the radius of the rotating target. Oxygen gas pressure was adjusted to be 0 to 5.32 Pa (40 mTorr) during deposition. Detailed information about the deposition processes has been given in our previous communication [12].

The film structure was examined by using a X-ray diffraction equipment (XRD, Philips, X-Pert MRD) with monochromatized Cu K_{α} in the θ_0 - 2θ thin film configuration, where θ_0 was fixed at 0.5° for single layer films and 0.5 to 3° for bi-layer films. The cross-sections of the laser deposited SDC/SCSZ bi-layer films were investigated by Hitachi's FE-SEM S-4500 using the new super ExB filter technology. FE-SEM images were

taken at a magnification of 100,000 with a 5 kV beam voltage. Based on the deposition rates, the thickness of the bi-layer electrolyte on cell substrates was approximately 1 μm ScSZ plus 6 ~ 7 μm SDC.

Cell characterization

The surface of the PLD deposited bi-layer electrolyte appears shining and transparent. The electrolyte shows very low helium gas cross leakage rate, 0.05ml (min cm)⁻¹ at 13.8kPa (about 2psi), indicating that the electrolyte layer was almost fully dense. A SSC#3 cathode layer consisting of 75 wt.% SScO and 25 wt.% SDC with surface area of 0.551cm² was applied to the centre of the bi-layer electrolyte by stencil printing, and dried at 100°C for 1h. The thickness of the cathode layer is approximately 40-50 μm after *in-situ* sintering at 800°C for 2 hrs.

Figure 2 schematically shows the electrochemical testing setup for the button cell. A split- tube furnace (Thermcraft incorporated) was used to heat up the testing cell. The testing cell was mounted at the end of an alumina tube and sealed with glass. The temperature of the testing cell was controlled with a thermo couple that was located nearby the cell at the anode side. A thin layer of SSC#3 paste was applied to the Pt mesh at the cathode side to create a good electrical contact between the cathode and the current collector (Pt mesh) in the testing apparatus. A spring with a constant loaded force, of approximately 1.0 kgf according to the deformation of loading springs, was applied to the surface of the cathode located inside the air supply alumina tube. The cell was heated up to 800 °C at a ramp rate of 300 °C h⁻¹, and held at 800 °C for 2hrs for *in-situ* sintering of

the cathode layer. The temperature was then decreased down to 700°C for the reduction of the anode and for the measurements of cell electrochemical performance. Ambient air was introduced at a flow rate of 100ml min⁻¹ to the cathode side. Hydrogen gas with the same flow rate was first passed through a bubbler-type humidifier at room temperature. The humidified hydrogen gas (corresponding to 3% H₂O content) was then introduced to the anode side during the cell test. Electrochemical measurements were performed at the temperatures ranging from 700 to 450°C. Cell performance was measured with a Multi-channel Potentiostat/Galvanostat (Solartron 1480 8 channel multi-stat) with the Corrview software. The current-voltage-power (I-V-P) curves were obtained using the linear sweep voltametry at a sweep rate of 4 mV s⁻¹ from OCV to 0.3V.

RESULTS AND DISCUSSIONS

On Si(100) Substrate

PLD technique was used to deposit thin films of SDC and ScSZ on Si(100) substrates at the substrate temperature range of 200 ~ 600°C and reactive oxygen gas atmosphere of 1 mTorr ~ 40 mTorr. By varying the deposition temperatures and oxygen gas pressures, SDC and ScSZ films with various crystallite sizes, density and interfacial properties were grown successfully. XRD results indicate that both SDC and ScSZ films were crystallized to cubic phases (PDF card # 75-0158 for SDC and card # 75-0158 for ScSZ [13]) at relatively low deposition temperatures (~ 200°C for SDC and ~ 400°C for ScSZ, respectively). The density of the SDC films increases with the increase in the deposition temperature while for ScSZ it decreases with the increase in the deposition temperature. The densities of 200°C and 400°C SDC films are only 63% and 70%, respectively, of the

density of the 600°C SDC films. In contrast, the 200°C ScSZ film is denser than that of the 400 °C and 600°C ScSZ films. The densities of 400 °C and 600°C film are 82% and 77%, respectively, of the density of 200 °C film.

Figure 3 shows the XRD spectra of SDC/ScSZ bi-layer films deposited on silicon wafers at 200 °C and 600 °C in 40 mTorr O₂ process gas. The XRD spectra consist of diffraction peaks originating from both cubic SDC and ScSZ phases. Peak assignment for the SDC top layer and the ScSZ underneath layer were also shown in Figure 3. No additional peaks due to (Zr, Ce)O₂-based solid solutions or β-phase ScSZ were found in the diffraction pattern, which indicates that during the deposition process, no chemical reaction occurred at the interface between SDC and ScSZ. This means that PLD technique is an excellent technique for growing both highly conductive cubic ScSZ and SDC phases at very low processing temperatures. Laser deposition process can indeed stabilize the ScSZ cubic phase at low temperatures that prohibits the phase transition from more conductive cubic phase to less conductive β-phase which occurs during the cooling step for most of the high temperature wet-ceramic processes. SDC layer shows strong (111) preferential orientation on the top of the cubic phase ScSZ at both temperatures. The XRD peaks for the bi-layer film deposited at 200 °C are quite broad and not well-defined, indicating that the grain size of the low temperature deposited bi-layer film is small and its crystallinity is poor. As the deposition temperature increases, the full-width at half maximum of the XRD peaks decreases indicating that the grain size and crystallinity of films increases with the increase in deposition temperatures.

Figure 4 shows the cross-section FE-SEM graphs of ScSZ/SDC bi-layer films deposited at a temperature of 200°C and 600°C in 5.32Pa of oxygen. At 200°C, both ScSZ and SDC layers contains small grains. The SDC layer is very porous, while ScSZ layer is relatively dense. At 600°C, both ScSZ and SDC layers contains columnar grains oriented perpendicularly to the substrate and are very dense. The grain diameter for SDC layer is about 60-70 nm while for the ScSZ film it is around 30-40 nm. The surface of the 600 °C film is very smooth and does not show any cracks and pin-hole. The FE-SEM results are consistent with the previously described XRD results.

All the above results clearly indicate that by simply selecting the process parameters of the PLD technique, bi-layer SDC/ScSZ films with various microstructures, density and interfacial properties can be deposited. It is obvious that a dense and gas impermeable layer is required for the electrolyte layer in SOFCs, therefore appropriate process parameters should be selected for producing a dense SDC/ScSZ bi-layer electrolyte for SOFCs. When considering the application of the PLD technique for the deposition of anode and/or cathode layers in SOFCs where very porous structures are desired, different process parameters should be used to produce a microstructure similar to that shown in Figure 4 (a). The added flexibility of the PLD process indeed makes it a very promising technique for the fabrication of all solid-state devices based on multilayer thin films, such as miniaturized SOFCs.

On NiO-SDC Modified NiO-YSZ Substrates

PLD process was also used to deposit the ScSZ/SDC bi-layer electrolyte on NiO-SDC modified NiO-YSZ anode substrates. The anode substrates were prepared and

characterized for its surface roughness and helium gas permeability before the PLD processing. The surface roughness of the cell substrates was about $0.66\mu\text{m}$ after the NiO-SDC modification. SEM micrograph in Figure 5a shows that the unmodified face is quite rough and uneven, roughness is about $1.5\mu\text{m}$. While the modified surface consists of crack-free, smooth layer with small size and uniform crystallites (Figure 5b). The cross-sectional view in Figure 5c clearly shows that NiO-SDC modified layer consists of much smaller grains ($1 \sim 2 \mu\text{m}$) compare to the NiO-YSZ layer ($10 \sim 20\mu\text{m}$).

Thin SDC films of approximately $2 \mu\text{m}$ thick were initially deposited on the anode substrates at the substrate temperatures $500\sim 600 \text{ }^\circ\text{C}$ and oxygen pressure of $5 \sim 40 \text{ mTorr}$. The Low Angle XRD results confirmed that SDC films on the anode substrates were indeed single-phase fluorite type structure SDC. The $2 \mu\text{m}$ SDC film coated anode substrates have decreased He permeability up to five times. However, the cross-leak of the samples is too high to conduct cell performance tests. FE-SEM examination of the SDC coated anode revealed the failure of $2 \mu\text{m}$ SDC layer to cover the $1\sim 2 \mu\text{m}$ diameter pinholes, existed on the anode surface. Much thicker SDC layer was needed in order to achieve the required gas tightness, which will increase the electrical resistance of the electrolyte layer significantly.

In order to effectively utilize the ScSZ/SDC bi-layer electrolyte concept for reduced temperature SOFCs, the ScSZ bottom layer should be as thin as possible and also be gas impermeable. The ScSZ cannot directly be deposited on anode substrates prepared in this manner since it requires a relatively thick ScSZ layer to achieve the required gas

tightness. Therefore, an additional NiO-SDC layer of about 4 μm thickness was initially deposited by the PLD on the anode surface. This layer reduces the gas leakage of the anode substrate significantly and provides smoother surface for the deposition of the ScSZ layer due to the nanostructural nature of the PLD films. After the deposition of the additional NiO-SDC layer, ScSZ of above 1 μm thick was deposited by the PLD at substrate temperatures of 200~ 400 $^{\circ}\text{C}$ and in 5 ~ 20 mTorr of oxygen pressure. Following the ScSZ layer, a relatively thick SDC layer about 6 ~ 7 μm was deposited at substrate temperatures of 400~ 600 $^{\circ}\text{C}$ and in 5 ~ 20 mTorr. The surface and cross section of the bi-layer electrolyte was examined by the FE-SEM. No cracks or pin-holes were found in the SEM micrographs. The obtained He leakage of this bi-layer electrolyte coated anode substrates is less than 0.05 ml (min cm^{-2}) at 13.8kPa He. The above results clearly indicate that PLD technique is a most suitable method to deposit dense and gas impermeable layers not only on smooth surfaces like silicon wafers but also on porous ceramic substrates like NiO-SDC modified NiO-YSZ cermet.

A composite cathode was then applied onto the surface of the bi-layer electrolyte by the stencil printing followed by drying and *in-situ* sintering. The unit cell thus prepared was characterized for its electrochemical performance. Figure 6 shows the cell performance with the SSC cathode in-situ sintered at 800 $^{\circ}\text{C}$ for 2hrs. It can be seen that the cell shows very high performance in the tested temperature range of 450-700 $^{\circ}\text{C}$. The peak power density reaches 1.6 W cm^{-2} at 700 $^{\circ}\text{C}$, and approximately 0.5 W cm^{-2} even at 550 $^{\circ}\text{C}$. What is most significant is that the cell shows a very high OCV value, 1.04V, in comparison to the single 20 μm -SDC electrolyte cell which only shows only 0.87 V at 600 $^{\circ}\text{C}$ [9]. A

high OCV value indicates that there is very low electronic conductivity and physical defects (cracks, pin-holes etc.) in the ScSZ/SDC bi-layer electrolyte. High cell performance also indicates the interfacial reaction between ScSZ and SDC are successfully eliminated due to low deposition temperatures.

This is our first report on the performance of reduce temperature SOFCs using the pulsed laser deposited bi-layer electrolyte. In order to get a better understanding of the effect of the microstructures, chemical composition and interfacial properties of the PLD electrolyte on the performance of the SOFCs, more detailed studies are being pursued and the results will be reported in our future communications. Besides, according to our studies for the composite cathode [14], the cell performance could be improved further by optimizing the cathode preparation conditions. Our future work will also address the cell stability and performance improvement.

CONCLUSION

PLD technique has been used in this study to deposit the SDC\ScSZ bi-layer electrolyte on Si(100) substrates and on NiO-SDC modified NiO-YSZ cell substrates. By varying the deposition process parameter conditions, SDC/ScSZ bi-layer electrolyte films with various crystallite sizes, density and interfacial properties were successfully grown. The PLD technique was able to deposit the highly conductive cubic phases for both ScSZ and SDC layers. Interfacial reaction between the ScSz and SDC layers that forms low conductive (Zr, Ce)O₂-based solid solutions were eliminated due to the low processing temperatures of the PLD technique. A unit cell that uses the PLD ScSZ/SDC bi-layer

electrolyte cell shows very high performance and high open circuit voltage. Comparing to other wet ceramic processes which require high processing temperatures, PLD method is a very promising low temperature process for the deposition of high quality thin electrolyte layer for the low temperature SOFCs.

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Table 1. Chemical composition, properties, suppliers of starting materials

Name	Composition	Properties	Supplier
SSCo	$\text{Sm}_{0.5}\text{Sr}_{0.5}\text{CoO}_3$	D50: 0.80 μm Surface area: 5.16 $\text{m}^2 \text{g}^{-1}$	Praxair
SDC	$(\text{SmO}_{1.5})_{0.2}(\text{CeO}_2)_{0.8}$	D50: 0.40 μm Surface area: 7.08 $\text{m}^2 \text{g}^{-1}$	Praxair
ScSZ	$(\text{ScO}_{1.5})_{0.20}$ $(\text{CeO}_2)_{0.01}(\text{ZrO}_2)_{0.79}$	D50: 0.46 μm Surface area: 7.0 $\text{m}^2 \text{g}^{-1}$	Daiichi chemicals
YSZ	$(\text{YO}_{1.5})_{0.16}(\text{ZrO}_2)_{0.84}$	D50: 0.46 μm Surface area: 7.0 $\text{m}^2 \text{g}^{-1}$	Tosoh
NiO-f	NiO type F	D50: 1.0 μm Surface area: 4.0 $\text{m}^2 \text{g}^{-1}$	Novamet
NiO-s	NiO-standard	D50: 16.0 μm Surface area: < 1 $\text{m}^2 \text{g}^{-1}$	Novamet
Silicon wafer	Si (100)	(100) orientation, p-type, 10 ~ 30 Ω	Polishing Corporation of America

FIGURE CAPTIONS

- Figure 1 : A schematic drawing of the cell structure and its fabrication method
- Figure 2 : A schematic setup for testing the button cell
- Figure 3 : XRD spectra of SDC/ScSZ bi-layer films deposited on silicon wafers at (a) 200 °C and (b) 600 °C in 40 mTorr O₂
- Figure 4 : FE-SEM graphs of ScSZ/SDC bi-layer films deposited silicon wafer at a substrate temperature of (a) 200, and (b) 600⁰C in 40 mTorr of O₂.
- Figure 5 : SEM Micrograph of surface view of (a) unmodified NiO-YSZ anode, (b) NiO- SDC modified NiO-SDC and cross-sectional view of (c) NiO- SDC modified NiO-SDC .
- Figure 6 : Current - Voltage power curves of the bi-layer electrolyte cell at different operating temperatures.

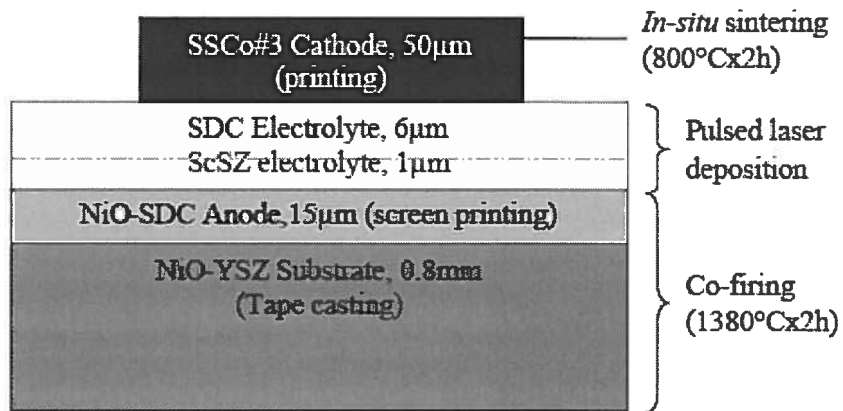


Figure 1

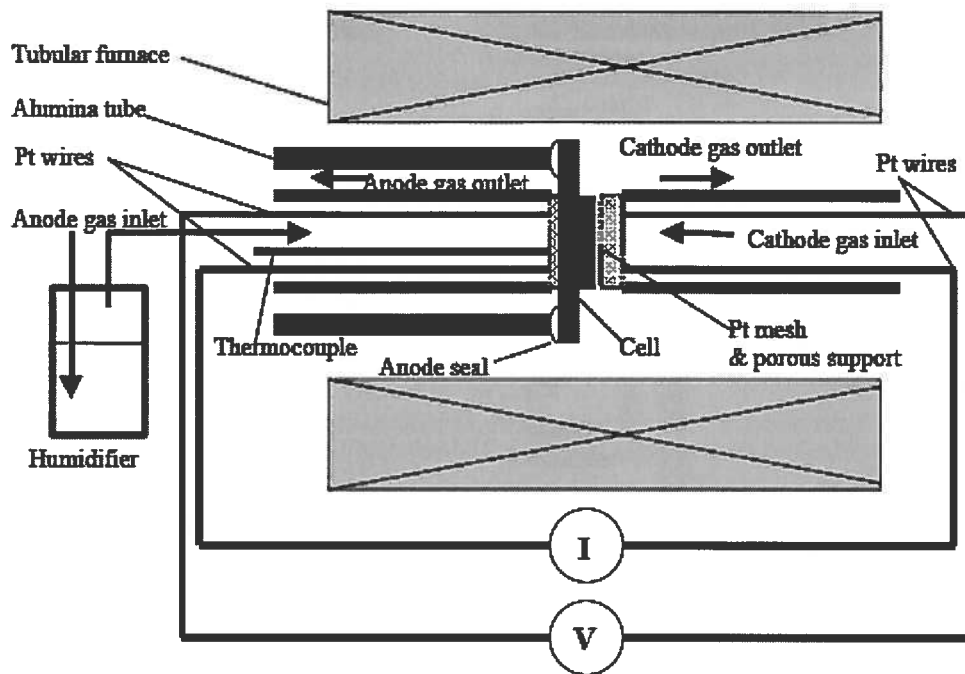


Figure 2

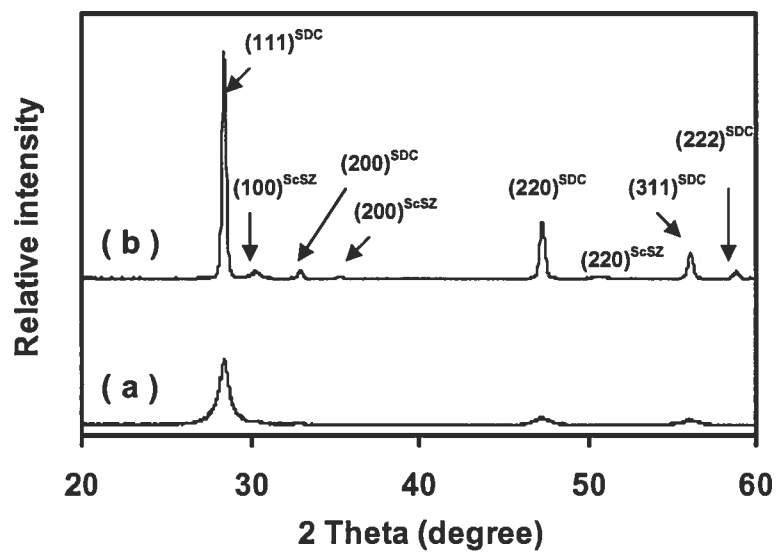


Figure 3

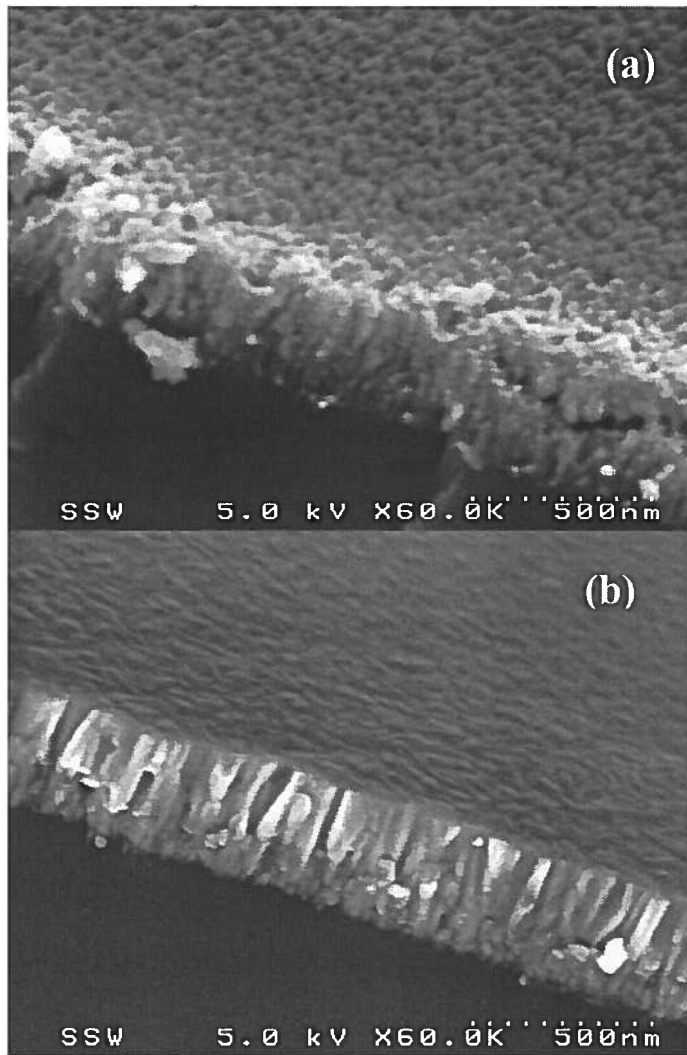


Figure 4

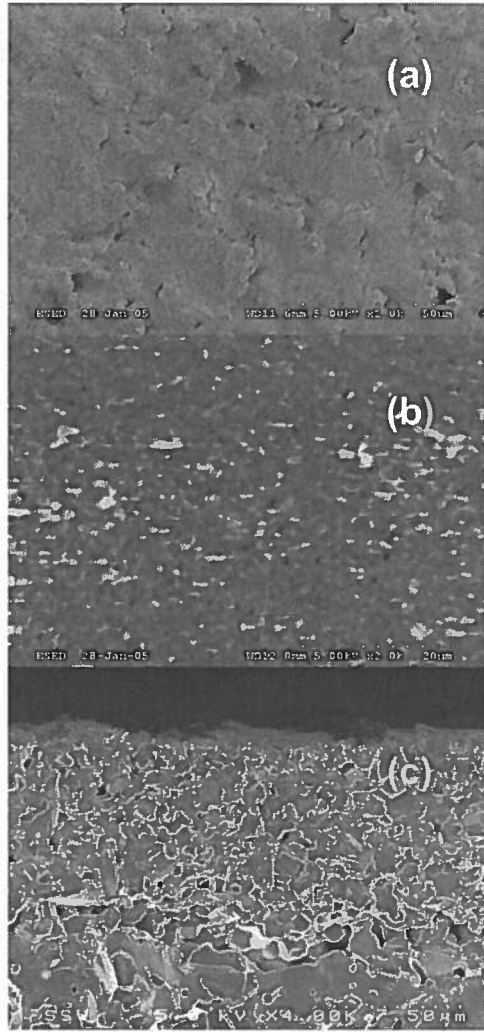


Figure 5

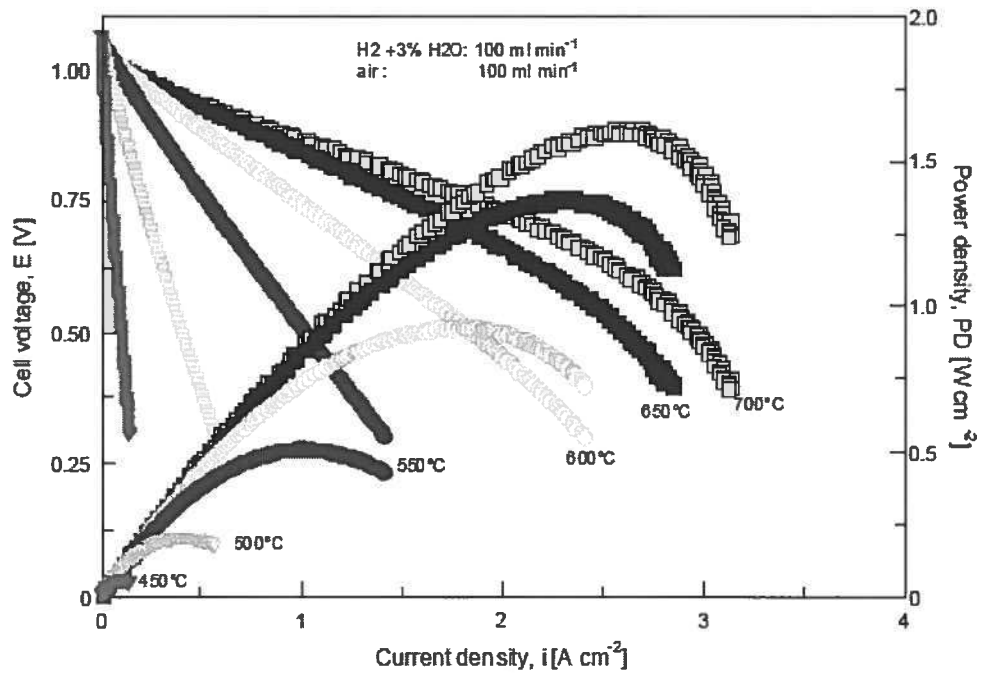


Figure 6