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An ion-plasma technique for formation of anode-supported thin electrolyte films for IT-SOFC applications

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ABSTRACT

This paper describes a preparation method and structural and electrochemical properties of a thin bilayer anode-electrolyte structure for a solid oxide fuel cell operating at intermediate temperatures (IT-SOFC). Thin anode-supported yttria-stabilized zirconia electrolyte films were prepared by reactive magnetron sputtering of a Zr–Y target in an Ar–O₂ atmosphere. Porous anode surfaces of IT-SOFCs were modified by a pulsed low-energy high-current electron beam prior to film deposition; the influence of this pretreatment on the performance of both the deposited films and a single cell was investigated. The optimal conditions of the pulsed electron beam pretreatment were obtained. For the electrolyte thickness about 2.5 μm and the value of gas permeability of the anode/electrolyte structure $1.01 \times 10^{-7} \text{ mol m}^{-2} \text{ s}^{-1} \text{ Pa}^{-1}$, the maximum power density achieved for a single cell at 800 °C and 650 °C was found to be 620 and 220 mW cm⁻² in air, respectively.

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1. Introduction

Solid oxide fuel cells (SOFCs) are regarded as a most promising source for electrical power generation. The development of solid oxide fuel cells, especially those operating at intermediate temperatures (IT-SOFC), which are capable of delivering high power densities at reduced temperatures, is a line of research intensively pursued worldwide. The operation of SOFCs at lower temperatures (650–800 °C) offers a number of advantages, to mention just a few: a lower corrosion rate of the cell components and a possibility of using inexpensive metal components for fabrication of interconnecting and structural elements. Yttria-stabilized zirconia (YSZ) with high ionic conductivity, low electronic conductivity and high

chemical stability under oxidizing and reducing atmosphere, are the most common materials used as electrolytes in solid oxide fuel cells, and the most typical anode material in SOFCs is a Ni–YSZ cermet [1,2].

To reduce the operating temperature two approaches are generally used: application of materials with appreciably higher conductivity (such as samaria or gadolinia-doped ceria) [3,4] or reduction in the SOFC overall thickness, particularly that of the electrolyte [5,6]. Thus, the electrolyte layer is one of the key components to reach the aims of reducing the operating temperature and increasing cell efficiency.

Many different processes have been applied to fabricate YSZ electrolyte in the form of coating, including pulsed laser deposition [7], tape casting, sol-gel and colloidal spray

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deposition [8,9], liquide-state deposition and screen-printing methods, and so on [10,11]. But most of them allow no forming of electrolyte layers with the thickness of a several micrometers or fabrication methods are expensive.

For planar structure SOFCs with anode support, thin electrolyte films have to be deposited onto the porous electrode substrate. In this case, the porous structure of the anode surface layer is an important parameter for the thickness of the electrolyte film. To fabricate gastight thin films on porous substrates, preliminary surface modification of the porous anode substrates to form modified and interface layers on their surfaces are applied [12,13].

Yen-Yu Chen et al. [14] described prepare process ultra-thin YSZ electrolyte film (with an average thickness of 0.5 μm) on a porous anode substrate by a liquid-state deposition method. Moreover, to overcome some processing defects such as cracks, pores or delamination, a multi-coating method, which repairs the crack step-by-step after cycling coating was applied. The results of cell testing of the YSZ-coated sample showed the best power density of 477 mW cm^{-2} at 600 $^{\circ}\text{C}$ and 684 mW cm^{-2} at 800 $^{\circ}\text{C}$. Despite the excellent results obtained, the production process used here involves several distinct stages and is quite lengthy.

In the present study, the formation of thin-film anode-electrolyte structure was carried out by vacuum plasma methods. We have studied not only the deposition of thin YSZ films, but also preliminary surface modification of the porous anode substrates. Thin YSZ films were deposited onto porous anode substrates (Ni-YSZ cermet) by reactive magnetron sputtering, which is a suitable deposition technique for these purposes [15,16]. However, the originality of this work was using the method of pulsed electron beam (e-beam) treatment to decrease the pore size in the anode substrate surface layer and also to form an intermediate layer between the porous anode support and the gastight electrolyte. The ability to use the e-beam treatment for these purposes has not been investigated previously, but this treatment method successfully used for processing other materials [17,18].

Combination of the e-beam treatment together with magnetron sputtering allows realize the preparation process of the anode-electrolyte structure of SOFC (including the preliminary anode surface modification with following YSZ electrolyte deposition) in a single vacuum process. As a result, the duration as well as the cost of manufacturing process is reduced. In this work, we studied influence of e-beam treatment parameters on microstructure of both the modified anode surface and thin-film electrolyte. In addition, we investigated the electrochemical performance of the cells thus formed.

2. Experimental

2.1. Preparation of porous anode substrates

The substrates used for the electron beam treatment and sputtering of thin YSZ films were reduced porous anodes (cermet anodes composed of 60 vol. % Ni and 40 vol. % $\text{Zr}_{0.9}\text{Y}_{0.1}\text{O}_{1.95}$ (Ni-YSZ)) of the SOFCs. The initial workpieces for the Ni-YSZ anodes were green sheets measuring 180 and

15 μm in thickness prepared by tape casting (from ESL ElectroScience). To form a single anode pellet, three green disks each 180 μm in thickness and one green disk 15 μm in thickness were laminated onto each other and sintered in air at 1450 $^{\circ}\text{C}$ for 2 h. The green anode was 24 mm in diameter. The resulting porous anode pellet had a bilayer structure (Fig. 1) consisting of a porous anode support (about 0.5 mm thick) and a less porous functional coating (15 μm thick layer). Upon reduction of NiO to Ni at 800 $^{\circ}\text{C}$ for 2 h in H_2 atmosphere, the pore size of the functional coating was found to be within 0.5–1.3 μm , and the pore size of the anode support was in the range from 1.5 to 3 μm . The sintered anode diameter was 21 mm. The gas permeability of this porous bilayer substrate was measured to be $\sim 6.25 \times 10^{-5} \text{ mol m}^{-2} \text{ s}^{-1} \text{ Pa}^{-1}$.

2.2. Pulsed electron beam treatment

Modification of the porous anode surface was performed with an e-beam source [19] generating a low-energy high-current electron beam with an electron energy of 10–12 eV, a beam current of 15 A, a beam diameter of 70–80 mm and a pulse duration of 2–3.5 μs . The conditions of the pulsed electron beam treatment were as follows: the energy densities (E_e) were 0.8, 1.5, 2.5, 3.5 and 4.5 J cm^{-2} , the pulse count (N) was from 1 to 9, and fast quenching from the melt was 10^{10} K s^{-1} . The operating pressure in the vacuum chamber was 3.8×10^{-4} Torr. Some of the samples were modified under heating to 600–700 $^{\circ}\text{C}$ (so-called background heat). Electron beam treatment was applied in two different ways. The first consisted in an e-beam treatment of initial porous anode samples. The second consisted in an e-beam treatment of a porous anode support together with a thin YSZ sublayer (0.5–2 μm) previously deposited onto it by magnetron sputtering.

2.3. Deposition of YSZ electrolytes by magnetron sputtering

Thin films of $\text{Y}_2\text{O}_3\text{-ZrO}_2$ (YSZ) were deposited onto porous anode substrates (Ni-YSZ) before and after their electron beam modification by magnetron sputtering of a composite

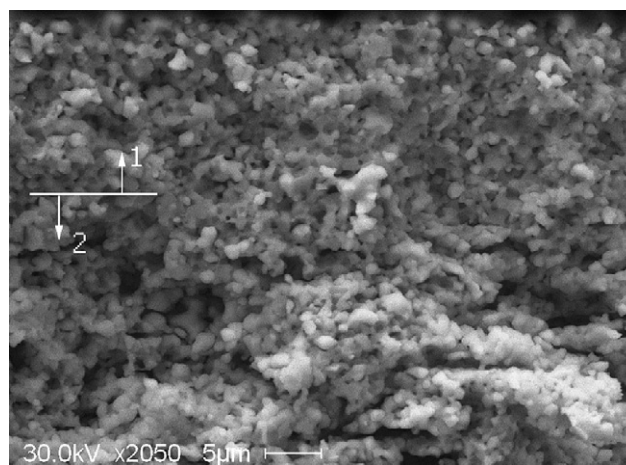


Fig. 1 – Cross section of the reduced bilayer anode support: (1) functional layer and (2) anode support.

Zr_{0.86}Y_{0.14} target in the reactive pulsed mode with a frequency of 50 kHz. Sputtering was carried out in an oxygen-argon atmosphere at the working pressure between 0.2 and 0.3 Pa. A constant current in the range 3.2–3.6 A was applied to the target at a voltage of 420–460 V. With a power of 1.5 kW on the target a deposition rate of 2.3 μm h⁻¹ was achieved. The distance between the magnetron target and substrate was about 80 mm. Prior to film deposition, the substrates were heated to 550–600 °C.

2.4. Fuel cell assembly and investigation

The microstructure of different multilayer assemblies and films, porous anodes after their electron beam treatment and electrode/electrolyte interface and the thickness of the thin electrolyte were characterized by scanning electron microscopy (Philips SEM 515). The SEM analysis was also used to examine the sample surface and cross section. The thickness of the YSZ film deposited onto the porous Ni–YSZ substrates was determined by an interferometer and compared to that estimated by SEM. For the gas tightness measurement of the anode substrates before and after e-beam treatment and that of YSZ films deposited on these substrates the homemade N₂ leakage system was used.

The electrochemical properties of thin YSZ electrolytes were studied by DC voltammetry on fully assembled fuel cells. For cell testing we prepared a few fully assembled cells having the following structure: air (or oxygen), Pt mesh, La_{0.80}Sr_{0.20}MnO₃ (acting as cathode)/ZrO₂:Y₂O₃ (1–3 μm thick and acting as electrolyte)/Ni–ZrO₂:Y₂O₃ (acting as support and anode), Pt, H₂.

The La_{0.80}Sr_{0.20}MnO₃ (LSM) cathode coating was prepared by screen-printing with a commercial LSM ink (Fuelcellmaterials.com) whereupon it was dried at 125 °C for 2 h. As current collector were used Pt mesh of 12 and 20 mm in diameter for both air and fuel side, respectively. On the cathode side Pt mesh fixed to electrode surface by LSM ink in order to improve surface contact between cathode and current collector. After following drying (at 125 °C for 1 h) Pt mesh adhered well to the electrode. On the anode side, Pt mesh attach directly to the air electrode without application of a contact paste. Lead connections were made using a 0.5 mm Pt wires. Two platinum wires were connected to each platinum mesh and are served as respective voltage and current probes. Activation of the cathode layer was carried out at the working temperatures (600–800 °C) during the first experiment.

The cell seal was realized by glass gaskets. Air/oxygen (35 ml min⁻¹) on the cathode side acted as an oxidant and hydrogen (110 ml min⁻¹) on the anode side - as a fuel.

Fig. 2 shows a built test station Probostat™ (High Temperature Electrochemical Test Fixture from NorECS) for study of the single fuel cell performance.

The measurements were carried out in a temperature range of 550–800 °C. The heating cell rate was as low as 300 °C h⁻¹ up to the point of reaching the operating condition. In the course of cell testing, the open circuit voltage (OCV) and current–voltage (*I*–*V*) characteristics were obtained. Finally, the power density values were calculated as a function of current density.

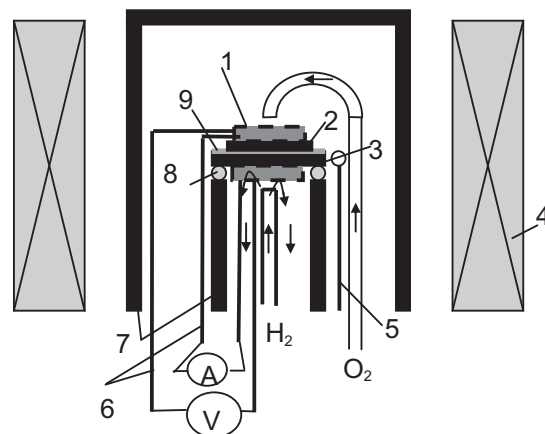


Fig. 2 – Schematic SOFC assembly for investigation of electrochemical performance: (1) Pt mesh, (2) cathode, (3) anode, (4) working space of furnace, (5) thermocouple, (6) Pt wires, (7) alumina tubes (an internal alumina tube is used as a support for mounting a single fuel cell, and an external tube is used as a cap), (8) anodic sealing, (9) thin electrolyte.

3. Result and discussion

3.1. Modification of Ni–YSZ anode substrate microstructure

A SEM image of the changed microstructure in the surface layer of the porous anode substrate is shown in Fig. 3. It is clear from the figure that the electron beam treatment gives rise to melting of the substrate surface layer down to a depth of about 1–1.5 μm, with the depth of the modified layer being practically independent of the e-beam energy density. It is also evident that at $E_s \leq 2.5 \text{ J cm}^{-2}$ and $N \leq 3$ the melted surface remains sufficiently rough (Fig. 3(a) and (c)). Later on, this would promote formation of an extra three-phase boundary between the porous anode substrate and the thin gastight YSZ electrolyte.

An increase in the e-beam energy density ($E_s > 2.5 \text{ J cm}^{-2}$) and the number of pulses ($N > 3$) resulted in nearly complete smoothing of the surface layer of the anode substrates (Fig. 3 (b) and (d)). Under this electron beam treatment conditions a number of defects such as microcracks, from a few hundred nanometers up to several microns wide, and microcraters about 25 μm in diameter were observed to form on the surface. It should be noted that microcracks (indicated by arrows in Fig. 3(b)) extend down the full depth of the modified layer. Crack formation results from the thermal and phase stresses in the surface layer quenched from a melted state, and the formation of craters might have been due to local overheating of the substrate material in the regions of the second phase with a lower thermal conductivity.

However, heating of the anode substrate to the temperature 600–700 °C during the e-beam treatment (background heating) allowed us to significantly reduce both the size and the number of microcracks on the modified anode surfaces. This effect is attributed to the lower temperature gradients generating under pulsed heating.

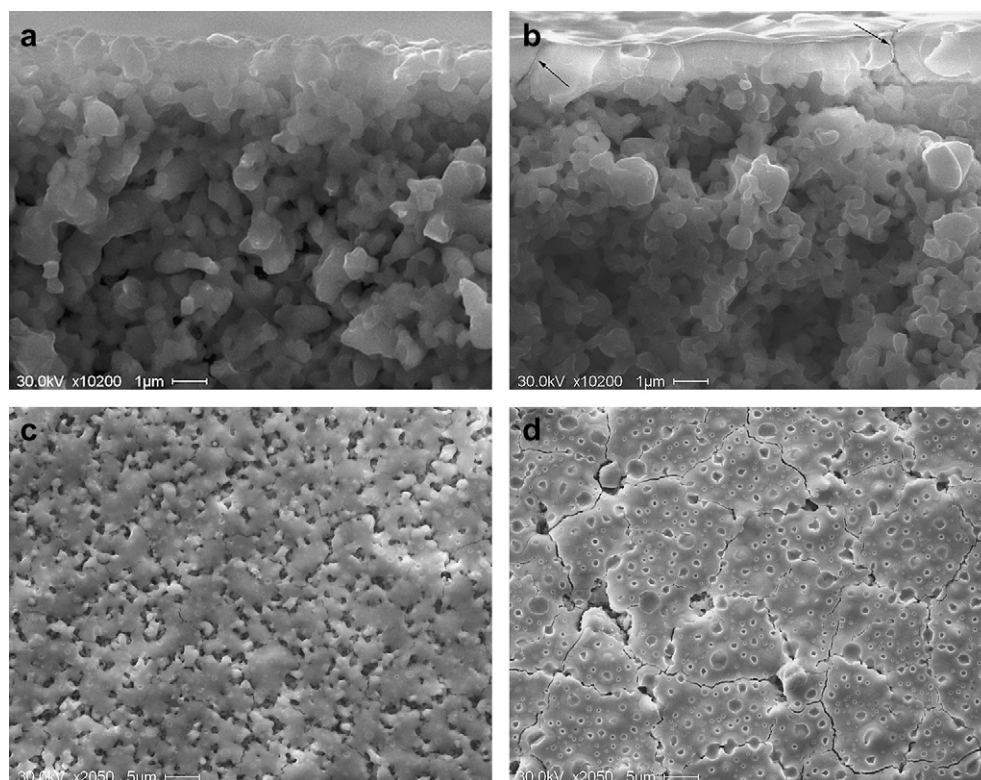


Fig. 3 – SEM images from the anode support: (a) fracture surface and (c) plan view of anode support modified at $E_s = 0.8 \text{ J cm}^{-2}$, $N = 3$; (b) fracture surface and (d) plan view of anode support modified at $E_s = 4.5 \text{ J cm}^{-2}$, $N = 3$, respectively.

Fig. 4 shows a SEM image of the cross section of an anode substrate with a thin YSZ sublayer about $1 \mu\text{m}$ thick after their combined electron beam treatment.

As a result, a dense $1\text{--}1.5 \mu\text{m}$ layer of Ni alloy with $\text{ZrO}_2\text{--Y}_2\text{O}_3$ is formed on the anode substrate surface, with a network of microcracks of less than $1 \mu\text{m}$ in width observed on it (see Fig. 5).

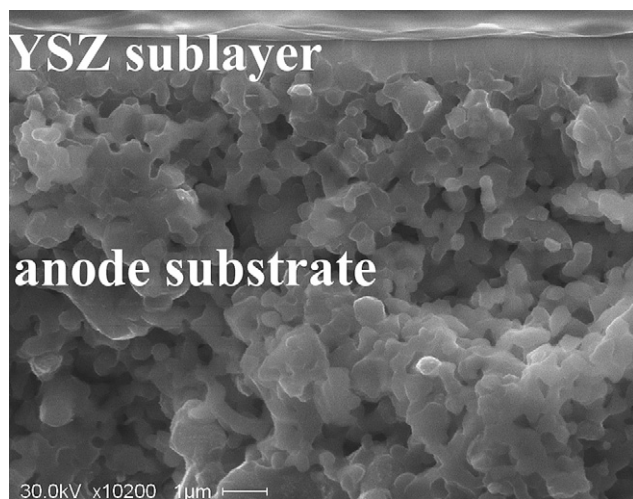


Fig. 4 – SEM image from the cross section of the anode substrate processed by a pulsed electron beam at $E_s = 0.8 \text{ J cm}^{-2}$ and $N = 3$ together with a thin $1 \mu\text{m}$ YSZ coating (so-called sublayer).

It is clear (Fig. 5(a)) that the cracks do not extend down the full depth of the modified YSZ sublayer and remain on the surface. The initial heating (i.e. background heating) of the porous anode substrate coated with a thin YSZ sublayer to $600\text{--}700 \text{ }^\circ\text{C}$ prior to their pulsed electron beam treatment significantly reduces crack formation on the surface (Fig. 5(b)) in the same way as described above.

3.2. Characterization of YSZ electrolyte

The effect of the pulsed electron beam pretreatment on the electrolyte structure is clearly seen in Fig. 6. Microstructure of the YSZ thin film deposited by reactive magnetron sputtering onto the initial anode substrate (without preprocessing) is columnar as is evident from Fig. 6(a). Despite the columnar structure, the film is sufficiently dense and the adhesion between the anode support and the electrolyte is excellent. The electrolyte film deposited on the pre-treated anode substrate has a similar structure. The best results in terms of the structure of the electrolyte films were obtained in the case of a thin YSZ film deposited onto an YSZ coated anode substrate modified by an electron beam (Fig. 6(b)). Due to this preprocessing, the electrolyte films subsequently grow not from the grain tops of the porous substrate but from an almost smooth surface, and hence a dense and quite homogeneous structure is formed. The YSZ electrolyte films are pore-free and well adhered to the Ni–YSZ supports under all deposition conditions.

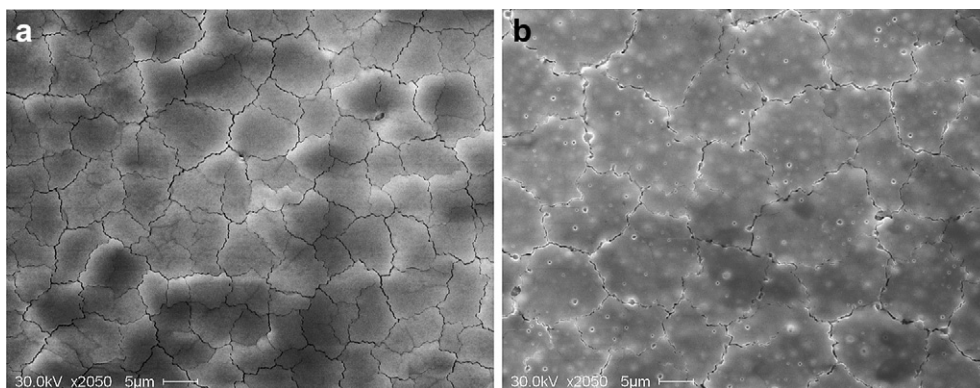


Fig. 5 – SEM images from the anode support surfaces e-beam treated together with a thin 1 μm YSZ sublayer at $E_s = 0.8 \text{ J cm}^{-2}$ and $N = 3$: (a) without background heating; (b) with background heating up to 700 $^{\circ}\text{C}$.

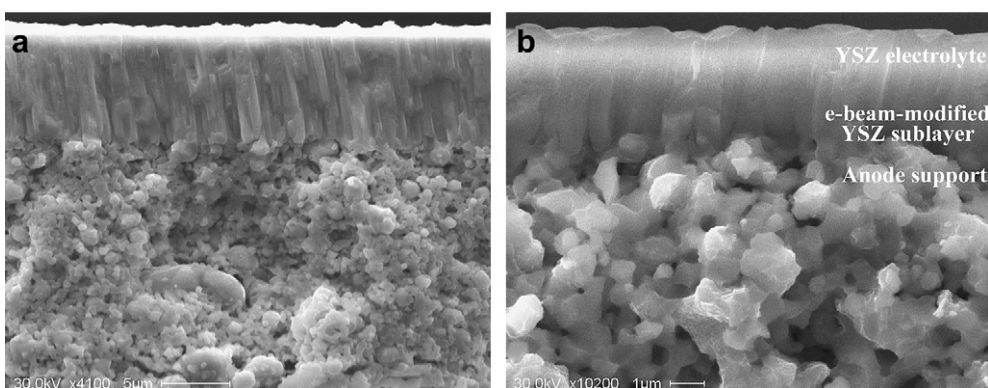


Fig. 6 – SEM micrographs from fracture surface of anode-supported electrolyte: (a) on an initial anode substrate (without preprocessing); (b) after electron beam treatment of the YSZ coated anode support.

3.3. Gas permeability characterization

Table 1 presents the e-beam treatment conditions, thin electrolyte parameters and the results of gas permeability measurements for thirteen samples under study.

The gas permeability measurements of the substrates after their modification by electron beam have shown that this treatment decreases the permeability values of porous anode supports by a factor of 10 and higher (Samples 3–8 in Table 1). Additionally, we found the gas permeability values for the complete anode/electrolyte structures. It is clear from Table 1 that the gas permeability levels of Samples 2 and 10 are within the same order of magnitude (2.2×10^{-7} and $2.6 \times 10^{-7} \text{ mol m}^{-2} \text{ s}^{-1} \text{ Pa}^{-1}$, respectively). Note, however, that Sample 10 exhibits this gas permeability for the electrolyte film thickness twice smaller than that of Sample 2. The low gas tightness of Sample 9 of $7.4 \times 10^{-7} \text{ mol m}^{-2} \text{ s}^{-1} \text{ Pa}^{-1}$ is attributed to the presence of a network of microcracks, which was successfully reduced by applying the background heating. These results show that the use of the pulsed electron beam treatment for porous anode modification promotes formation of fully gastight electrolyte films with a much smaller thickness (no more than 3 μm); this processing however should be carried out under optimal conditions. The most gastight anode/electrolyte structure was achieved for the electrolyte film sputtered

onto an YSZ coated porous anode substrate treated by an electron beam (Sample 13 in Table 1). The gas permeability value for Sample 13 was found to be $1.01 \times 10^{-7} \text{ mol m}^{-2} \text{ s}^{-1} \text{ Pa}^{-1}$ for the YSZ electrolyte thickness of 2.5 μm .

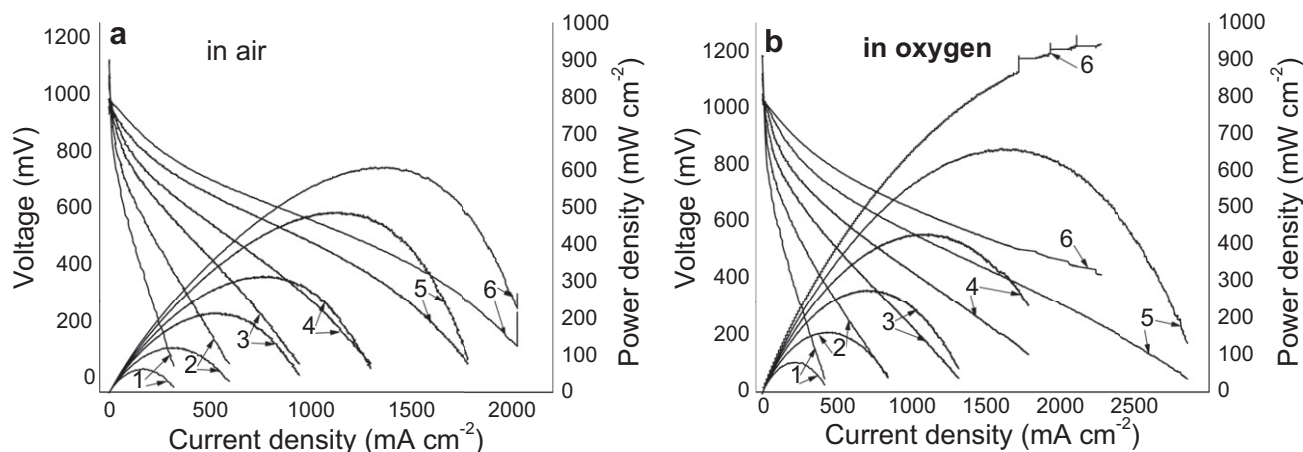
Based on the results obtained, we selected the optimum e-beam parameters. These are: E_s - as low as 2.5 J cm^{-2} and N - no more than 3. Moreover, in order to achieve the most gastight anode/electrolyte structure it is preferable to apply a pulsed electron beam to the porous anode substrate together with the thin YSZ sublayer deposited onto it.

3.4. Single cell performance

Fig. 7 shows the voltage/power density versus the current density trace for a cell based on Sample 13 (Table 1). The open circuit voltage (OCV) obtained for the single cell tested at 800 $^{\circ}\text{C}$ is 1.03 V. This value is close to the theoretical and indicates that the thin YSZ film deposited onto the YSZ coated and modified anode substrate blocks gas leakage through the electrolyte. During testing, the cell current–voltage characteristics were close to linear; the electrical characteristics were stable throughout the measurement period. When the temperature was increased, the power density was observed to grow rapidly. The maximum power densities were 940 and 620 mW cm^{-2} at 800 $^{\circ}\text{C}$ for oxygen and air used as oxidants,

Table 1 – Pulsed electron beam modification parameters of anode substrates and values of gas permeability of the modified anodes and the modified anode/thin YSZ film structures.

Samples	Thickness of sublayer (μm)	Electron beam treatment parameters			Electrolyte thickness (μm)	Gas permeability ($\text{mol m}^{-2} \text{s}^{-1} \text{Pa}^{-1}$)
		Energy density, E_s (J cm^{-2})	Pulse count, N	Value of background heat, T ($^{\circ}\text{C}$)		
1	–	–	–	–	–	6.25×10^{-5}
2	–	–	–	–	8.9	2.2×10^{-7}
3	–	0.8	3	20	–	1.33×10^{-5}
4	–	1.5	3	20	–	1.04×10^{-5}
5	–	2.5	3	20	–	0.41×10^{-5}
6	–	2.5	3	700	–	0.31×10^{-5}
7	–	3.5	3	20	–	0.44×10^{-5}
8	–	4.5	3	20	–	0.7×10^{-5}
9	–	0.8	2	20	2.5	7.4×10^{-7}
10	–	0.8	2	700	2.5	2.6×10^{-7}
11	0.5	2.5	3	20	–	47.6×10^{-7}
12	0.5	2.5	3	700	–	29.7×10^{-7}
13	1	0.8	2	700	2.5	1.01×10^{-7}

**Fig. 7 – Current–voltage characteristics of single fuel cell based on Sample 13 (Table 1) and tested within 550–800 $^{\circ}\text{C}$: (1) 550 $^{\circ}\text{C}$; (2) 600 $^{\circ}\text{C}$; (3) 650 $^{\circ}\text{C}$; (4) 700 $^{\circ}\text{C}$; (5) 750 $^{\circ}\text{C}$; (6) 800 $^{\circ}\text{C}$.**

respectively. At the operating temperature 650 $^{\circ}\text{C}$, the cell power densities were 270 mW cm^{-2} and 220 mW cm^{-2} for oxygen and air, respectively. For comparison, the maximum power density obtained for a cell based on Sample 2 (Table 1) was about 320 mW cm^{-2} at 800 $^{\circ}\text{C}$ in air. These results illustrate the efficiency the using of pulsed electron beam treatment for formatting the thin-film YSZ electrolyte for SOFC operating at reduced temperatures Fig. 7.

4. Summary

In this study, we have investigated the effect of porous anode substrate modification on the thin-film electrolyte characteristics and the performance of an anode-supported solid oxide fuel cell. The method of the pulsed electron beam treatment has been used for the first time to improve the surface of

porous anode substrates. The results observed demonstrate that the pulsed electron beam treatment is an effective and promising method for modifying porous anode substrates of SOFCs. Furthermore, we have chosen the optimal parameters of the pulsed electron beam treatment. It has been found out that the preferable structure of a cell is that of a porous anode substrate and a thin YSZ sublayer (thick of about 0.5–1 μm), the optimal beam energy density is 0.8–2.5 J cm^{-2} and the number of pulses should be within 1–3. The anode/thin electrolyte structure prepared by the proposed method demonstrated a reasonable gas permeability value of $1.01 \times 10^{-7} \text{ mol m}^{-2} \text{ s}^{-1} \text{ Pa}^{-1}$ for an YSZ electrolyte thickness of 2.5 μm . The thin film Ni–YSZ/YSZ/LSM fuel cell exhibited an open circuit voltage of 1.03 V. A power density value of 940 mW cm^{-2} at 800 $^{\circ}\text{C}$ and 270 mW cm^{-2} at 650 $^{\circ}\text{C}$ when operated in oxygen, and 620 mW cm^{-2} at 800 $^{\circ}\text{C}$ and 220 mW cm^{-2} at 650 $^{\circ}\text{C}$ in air were showed.

Acknowledgments

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