Planar Solid Oxide Fuel Cell: Electrolyte Deposited by Reactive Magnetron Sputtering and Cell Test

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Abstract

In this work the deposition of a thin YSZ electrolyte layer (< 10 µm) on a porous anode substrate is performed by sputtering Zr-Y metallic targets in argon-oxygen reactive gas mixtures under unstable deposition conditions. A Plasma Emission Monitoring system is used to ensure a high deposition rate of the ceramic film. The deposition parameters are optimised in order to obtain a good compromise between quality, density and deposition rate for coatings realised under unstable conditions.

After a short description of the experimental setup, the characterisation of the YSZ coatings is performed using SEM, XRD and permeability measurements. Then a complete cell incorporating a thin YSZ electrolyte layer (6.2 ± 0.3 µm thick) obtained by magnetron sputtering is manufactured and tested. This single planar solid oxide fuel cell exhibits a power density of 560 mW/cm^2 under a voltage of 0.7 V at 800°C and a maximal power density of 660 mW/cm^2 is reached.

Keywords : SOFC, Magnetron Sputtering, Electrolyte, YSZ, Cell Test

1. Introduction

Solid Oxide Fuel Cell (SOFC) system development involves the decrease of the working temperature and the reduction of the cell cost to better fulfil industrial application requirements. The electrolyte thickness reduction can be exploited to enhance fuel cell performance and lifetime by decreasing the cell core resistance **[1]** and the material degradation. This approach is an alternative to the classical research of new materials having better oxygen conduction properties than YSZ. In this field of investigation techniques based on Physical Vapour Deposition (PVD) represent an alternative to the classical deposition processes like tape casting or screen printing to obtain an electrolyte with a thick thickness (< 10 µm).

Various techniques such as reactive magnetron sputtering can be used to deposit Solid Oxide Fuel Cell electrolytes. Indeed magnetron sputtering technique allows the deposition of thin films that can be used to decrease the cell core resistance and then the operating temperature. This manufacturing process is also particularly adapted to the elaboration of thin coatings in the range 1 – 10 µm. However the deposition conditions of electrolyte coating must be investigated to elaborate a dense coating that can be used in SOFC field. In this work the commonly used materials such as yttria stabilised zirconia is studied. The deposition stage is performed by reactive magnetron sputtering in a chamber allowing a large scale production. Thus yttrium-zirconium oxide coatings are deposited from 84 at.% Zr – 16 at.% Y metallic targets sputtered in argon-oxygen reactive gas mixtures.

In this work, the main sputtering parameters allowing a high deposition rate of the ceramic compound and the improvement of the coating density are more particularly studied. The major problems identified to manufacture SOFC electrolytes by magnetron sputtering are the deposition rate and a sufficient compactness ensuring a physical barrier between anode and cathode **[2]**.

The deposition rate of ceramic compounds can be increased by a Plasma Emission Monitoring system **[3]** which allows a high deposition rate of the coating despite unstable conditions **[4, 5]**. However, it can involve the formation of an oxygen-deficient coating for the higher deposition rates. Thus a full oxidation of the electrolyte can easily be achieved by a thermal post treatment **[6]**.

The compactness of the coatings is also studied. This problem is mainly linked to the energy of the species from the target to the substrate. This energy depends on the sputtering parameters and the phenomena occurring between the target and the substrate. In this study, different parameters impacting the condensation of the sputtering species on the substrate are identified. Thus several parameters such as the decrease of pressure in the sputtering chamber during the deposition stage and the use of a radio frequency bias of the substrate holder can be used to obtain a denser electrolyte layer. These parameters are adapted to achieve a dense coating suitable for SOFC application on porous NiO/YSZ substrates.

As a result, the electrolyte has been implemented in a complete cell whose performances are measured. These performances are mainly discussed regarding the power density of the elaborated cell.

2. Experiments

This work investigates YSZ coatings deposited by magnetron sputtering from two Zr-Y alloyed targets (84 at.% Zr and 16 at.% Y) in argon-oxygen reactive gas mixtures. The experimental device is a 100-litre Alcatel SCM 650 sputtering chamber equipped with two 200 mm diameter targets and with a 610 mm diameter circular rotating substrate holder parallel to the targets at a draw distance of about 110 mm. This sputtering chamber is pumped down via a system combining XDS35i dry pump and a 5401CP turbo-molecular pump. Flow rates of argon and oxygen are controlled by MKS flowmeters and the pressure is measured using a MKS Baratron gauge. In all the experiments (except for the coating presented in Figure 3.a), the argon flow rate is set at 20 $cm^3.min^{-1}$. The targets are supplied with a pulsed DC Advanced Energy® dual generator. The current dissipated on each Zr (84 at.%) – Y (16 at.%) (Zr-Y) target is maintained at a constant value of 3 A pulsed at 50 kHz to avoid electrical instability.

The deposition stage is monitored using a closed loop control system based on the measurement of the optical emission intensity **[7, 8]** of the 360.12 nm Zr emission line on the area close up to the target **[9]**. The signal is sent via an optical fiber to a Ropper Scientific spectrometer, with a 1200 groove mm^{-2} grating and a photomultiplier tube (Hamamatsu R 636). The information is sent to a computer and a program developed at the laboratory monitors the oxygen flow rate to maintain the selected intensity of the optical signal between about 7 (fully oxidised target) and 100 % (in pure argon) of the Zr emission line. In this paper, the regulation percentage corresponds to the intensity of the optical signal (e.g. to the flow rate of oxygen introduced in the sputtering chamber during deposition stage). The experimental setup is presented in **Figure 1**.

The advantage of Plasma Emission Monitoring is to allow the control of the target poisoning under so-called unstable sputtering conditions, which in turns allows the high deposition rate of ceramic compounds **[10]**. With this system equipped with two targets and a regulation percentage of 30 %, a deposition rate of 2.6 $\mu m.h^{-1}$ is obtained. The Alcatel SCM 650 sputtering chamber can be equipped with four targets and in that case, a deposition rate close to 5 $\mu m.h^{-1}$ can be expected. Moreover, even if the deposition rate can be increased with a higher value of the regulation percentage, a low value (< 40 %) is chosen to avoid the problems linked to the post-oxidation of the oxygen deficient coatings realised by a thermal treatment after the deposition stage.

The substrates used are commercial porous NiO/YSZ positioned on the rotating substrate holder at a distance of around 170 mm from its axis. This value corresponds to the minimal draw distance between targets and substrates when both of them are on a same axis parallel to that of the substrate holder.

To carry out gas tightness tests, the reduction of the commercial porous anode substrates previously covered by the YSZ coating is required. This step is performed in a tubular controlled atmosphere furnace Adamel-Lhomargy FN50 equipped to operate under vacuum conditions. A type 818 controller linked to a Pt/PtRh thermocouple allows the temperature regulation at the desired value.

The thickness of the coatings is determined by tacticle profilometry (Altisurf 500) using the step method. The morphology of the coatings is assessed by means of Scanning Electron Microscopy (SEM) using a JEOL (JSM5800LV) also equipped with X-ray Energy Dispersive Spectroscopy (EDS) for composition measurements.

The test bench used to characterise the complete cell has separate atmospheres. The system consists of a furnace and a chemical reactor in which the button cell is loaded at the inner end of an alumina tube. Current collectors, Ni for anode and Au for cathode are sealed using silicone O-rings. The cathode side is exposed to air flow while the anode side to the reactive gas flow H_2 (100 $ml.min^{-1}.cm^{-2}$). The fuel humidification is equal to 3 %. A k-type thermocouple is used to measure the accurate fuel cell temperature. To obtain IV-curves, three phases must be carried out. First the heating process, with an increase of temperature equal to 1°$C.min^{-1}$, under a gas flow of N_2 (30 $ml.min^{-1}$) and air (300 $ml.min^{-1}$). Then the reduction process, under a gas flow of N_2/H_2 (90/10 $ml.min^{-1}$) and air (300 $ml.min^{-1}$) for one hour, followed by a gas flow of H_2 (150 $ml.min^{-1}$) and air (300 $ml.min^{-1}$).

To conclude the measurements are performed with a stabilisation time of 30 seconds with a gas flow of H_2 (150 ml.min^{-1}) and air (300 ml.min^{-1}).

3. Results

3.1 Deposition of dense coatings

One of the main problems to synthesise a thin electrolyte film on a porous anode substrate is to obtain a sufficient permeability to create a physical barrier between anode and cathode **[11, 12, 13]**. Three different conditions must be fulfilled to achieve gas tightness. A dense coating with no cracks covering all the surface of the substrate must be deposited. To fit the requirements, the substrate morphology must also be adapted. Indeed to synthesise a thin electrolyte (< 10 µm) by PVD, the size of the porosity allowing the diffusion of hydrogen in the anode material must be lower than about 5 µm. In our case commercial porous anode substrates exhibiting the required conditions have been chosen.

The production of the metal species, their transport from target to substrate and condensation on the substrate represent the three different steps for a coating elaborated by magnetron sputtering. Different parameters allow a variation of the species energy from their production on the target to their condensation on the substrate.

Thus the initial sputtering species energy is mainly a consequence of the discharge parameters. In our case the intensity applied on each target is set at 3 A. The pressure on the sputtering chamber can change the mean free path of atoms during transport. And the coating growth due to the condensation of the species can be modified with an additional energy (electrical or thermal) applied on the substrate holder in order to increase the diffusion phenomena.

The pressure is mainly conditioned by oxygen and argon flow rates during the deposition stage. The oxygen partial pressure is linked to the reactivity of the target and is varied by the regulation percentage parameter. The low variation of the oxygen inlet flow with time for different regulation percentages is presented in **Figure 2**. A decrease followed by an increase of the oxygen flow is observed. During the deposition stage, the temperature of the sputtering chamber increases from room temperature to around 120 °C due to the sputtering species impacts on the surfaces (substrate holder, sputtering chamber walls). The decrease of oxygen flow rate corresponds to the temperature increase of the chamber walls. This phenomenon leads to an increase of the water vapour desorption that can be observed from 0 to 40 minutes in **Figure 2**. Then, once the steady state temperature is reached, the outgassing of the chamber walls continuously decreases and the oxygen flow rate required to maintain the setpoint then increases.

Thus the global pressure in the sputtering chamber is mainly linked to the argon flow. In **Figure 3** a slight densification of the coating is observed due to a slight decrease of the argon flow from 25 to 20 cm^3.min^{-1}. In our case, this phenomenon described by **[14]** can mainly be observed in the area close to the substrate-coating interface. This last value is close to the lowest argon flow allowing the plasma formation in the sputtering chamber.

However, with a regulation percentage higher than 25 % an oxygen deficient coating is deposited. Thus a thermal post treatment is necessary to form fully oxidised electrolyte. The volume expansion resulting from the oxidation must be particularly studied to increase density and to avoid the desquamation that can appear due to induced mechanical stress. This phenomenon is confirmed by an average increase of the thickness (around 0.2 µm) between a coating realised without and with thermal treatment at 500 °C for 2 hours. The tests performed and presented in **Figure 4** show that the coatings must be deposited with a low oxygen deficit corresponding to a value lower than 50 % for the regulation percentage. Nearly no desquamation appears with a regulation percentage lower than 40 %. However the limit between adhesion and desquamation is not well defined. In our case, it is chosen to point out that no desquamation can appear if the parameters are set under this limit.

Another solution to increase the energy of the species on the substrate is the addition of electrical or thermal energy on the substrate holder **[15]**.This solution is achieved with a radio frequency generator. In this study the substrate bias is set at a value from 4 to 12 W. **Figure 5** shows the results obtained when a radio frequency generator is used in comparison to **Figure 4**. A small shift of the limit can be observed and shows that the regulation percentage must be lower than 35 % when the radio frequency generator is used. This result also confirms the increase of density obtained when the substrate holder is biased.

Moreover to synthesise a dense coating the presence of growth defects must be avoided. Providing an additional energy on the substrate holder can limit the presence of growth defects which mainly results from the substrate surface characteristics. This energy is mainly used to increase the mobility impinging adatoms. **Figure 6** presents coatings realised at a high regulation percentage (60 %) before thermal treatment with (**Figure 6** (**a**)) and without (**Figure 6** (**b**)) radio frequency bias of the substrate holder. On these brittle fracture cross sections, growth defects mainly come from the interface between substrate and coating. However those coatings realised with a high regulation percentage (60 %) present a high sub-stoechiometry in oxygen and their desquamation after thermal treatment is obvious.

Permeability measurements are performed with hydrogen. These tests realised at room temperature with porous substrate have shown an increase of the gas tightness linked to the increase of the coating thickness. The commercial NiO/YSZ substrates from SOFC Power Company are used. After the coating deposition, a first step consists in the reduction of the nickel oxide to allow gas diffusion through the anode material. The reduction is performed with argon and hydrogen flow rates of 1000 ml/min and 400 ml/min respectively and with an internal temperature approximately equal to 650 °C for 2 hours. Then the samples are tested under hydrogen and with a pressure difference of 3 bars to quantify their gas tightness. For coatings realised with a 20 $cm^3.min^{-1}$ argon flow, the hydrogen flow passing through the electrolyte is close to 0.17×10^{-3} $cm^3.s^{-1}$ for 48 hours acquisition time, which is expected to be sufficient for a cell test.

3.2 Characteristics and performances of the deposited coating

The tests performed in previous works **[16, 17]** with this sputtering system allow to identify the parameters commonly used in PVD which can be changed to fulfil the requirements. The solutions proposed in this paper consist of a cell elaborated with an electrolyte deposited by magnetron sputtering. It is realised using a commercial porous anode substrate S-Design (> 75 cm^2) from SOFC Power. First the electrolyte is deposited by magnetron sputtering with the parameters presented in **Table 1**. The synthesised electrolyte has a thickness of 6.2 ± 0.3 µm. Then a 25 × 25 mm^2 half-cell adapted for the single cell test bench is obtained by laser cutting to be further covered by a lanthanum cobaltite based perovskites (LSC) cathode material deposited by screen printing. The deposited cathode active area is 14 × 14 mm^2.

Table 1: Parameters applied for the deposition of the electrolyte.

Target intensity	Draw distance	Regulation percentage	Argon flow	Radio frequency	Deposition time	Substrate

(A)	(mm)	(%)	(sccm)	(W)	(min)	
3 – 3	110 ± 10	25	20 ± 2	6 ± 1	210	Commercial anode

The coating structure and morphology are analysed by X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM), on brittle-fracture cross sections, respectively. Despite XRD spectra are not shown in this study, coatings present fluorite structure of cubic YSZ as demonstrated in previous works **[18]**. Brittle fracture SEM micrograph of the half cell is presented in **Figure 7** and shows that the electrolyte is dense with a slight columnar structure. The adhesion between electrolyte and cathode deposited by screen printing on a surface approximately equal to 20 cm^2 appears sufficient for the complete cell test despite an apparent lack of adhesion compared to that between the electrolyte and NiO/YSZ support.

The cell is tested at 800 °C under hydrogen and air. I-V curve presented in **Figure 8** is plotted with hydrogen and air flows maintained at 150 and 300 cm^3.min^{-1} respectively. An Open Circuit Voltage (OCV) of 986 mV is reached. This value, lower than 1.1 V expected, proves insufficient gas tightness in the electrolyte. In magnetron sputtering technology, this characteristic can be enhanced by RF bias (other sputtering parameters) or with an additional system allowing the substrate temperature increase to improve the species diffusion **[15]**. However, this cell exhibits good performances with a current density higher than 800 mA.cm^{-2} at 0.7 V and an Area Specific Resistance (ASR) of 0.32 Ω.m^2 calculated from the equation (1).

$$ASR = \frac{0.75 - 0.65}{j_{0.65} - j_{0.75}} \qquad (1)$$

These performances underline the good adhesion between electrolyte and electrodes with low resistivity. The plot of power density versus current density presented in **Figure 9** shows that a maximal power density of about 660 mW.cm^{-2} is also reached at 800°C.

Table 2 compares the features of the cell realised in this study with those of a single cell ASC-800 from SOFC Power elaborated with the same anode and with a YSZ electrolyte manufactured by tape casting. Indeed the main difference between those cells is only linked to the electrolyte manufacturing process and cathode composition.

Table 2: Characteristics of the elaborated cell compared to ASC-800 (SOFC Power)

		ASC-800	**This work**
Anode	Manufacturing process	Tape casting	Tape casting
	Composition	Ni/8YSZ	Ni/YSZ
	Thickness	240 ± 20 µm	240 ± 20 µm
Electrolyte	Manufacturing process	Tape casting	Magnetron sputtering
	Composition	8YSZ	8YSZ
	Thickness	8 ± 2 µm	6.2 ± 0.3 µm
Cathode	Manufacturing process	Screen printing	Screen printing
	Composition	LSM/8YSZ	LSC
	Thickness	40 ± 10 µm	40 ± 10 µm
	ASR	0.29 Ω.cm^2	0.32 Ω.cm^2

Cell performance	Tension	0.7 V	0.7 V
	Current density	970 mA.cm^{-2}	800 mA.cm^{-2}
	Power density	680 mW.cm^{-2}	560 mW.cm^{-2}

In this work a power density of 560 mW.cm^{-2} at 0.7 V and 800 °C is also reached. This value is close to the performance presented for the cell ASC-800 which is tested with a similar test bench and under the same operating conditions (hydrogen and air flows, temperature, etc).

The results of the cell manufactured with an electrolyte deposited by magnetron sputtering can also be compared with other YSZ electrolyte cells synthesised using different processes. The performances of those cells characterised at 800 °C and for a voltage of 0.7 V are presented in **Table 3**. Even if the electrode materials and the test conditions are not the same for each cell, this table shows that the performances of our cell manufactured under conditions adapted for industrial mass production, are in the range of state-of-the-art.

Table 3: Comparison of cell performances at 800 °C and 0.7 V for different manufacturing processes and different thickness of the 8YSZ electrolyte

Paper \ Data	**Manufacturing process of the electrolyte**	**Thickness (µm)**	**Current density (mA.cm^{-2})**	**Power density (mW.cm^{-2})**
[19]	Colloidal coating	≈ 10	1430	≈ 1000
ASC-800 SOFC Power	Tape casting	8 ± 2	970	680
[20]	EB-PVD	≈ 8	965	675
[21]	APS	≈ 35	858	≈ 600
This work	**Magnetron sputtering**	**6.2 ± 0.3**	**800**	**560**
[22]	Thermal spraying	≈ 30	585	410
[23]	EPD	17 ± 1	402	282
[24]	Co-sintering	≈ 15	370	260
[25]	EB-PVD	≈ 12	165	115

Conclusion

The aim of this study was to realise and test a single planar cell with a thin YSZ electrolyte for SOFC application. In this work electrolyte deposition by magnetron sputtering on a commercial porous anode substrate is investigated. The coatings are realised with a Plasma Emission Monitoring system which allows the deposition of an oxidised compound with a high deposition rate. With this system and using two targets, the deposition rate is 2.6 µm.h^{-1}. The deposition parameters were optimised in order to obtain a good compromise between quality, density and deposition rate for coatings sputtered under unstable conditions.

The SOFC electrolyte is a physical barrier (dense and covering material) between reactive gases present in anode and cathode side. A thin electrolyte (6.2 ± 0.3 µm) is realised in this study and is further covered by a LSC screen printed layer to obtain a complete cell. A low oxygen deficit combined with a slight radio frequency bias voltage was optimised to avoid the desquamation of the electrolyte after the thermal treatment allowing the full oxidation of the deposited compound and its volume expansion.

To conclude a complete cell is realised and tested under air and hydrogen flows on the cathode and anode sides respectively. This cell exhibits a power density of 560 mW.cm^{-2} under a voltage of 0.7 V at 800 °C. Such performances are comparable to the state of art for cells based on YSZ electrolyte. Moreover this prototype constitutes one of the very first SOFC cell manufactured with a thin electrolyte layer deposited by magnetron sputtering. In future works a new set of parameters will be investigated on this basis to reach a higher permeability of the electrolyte and a value of OCV higher than 1 V.

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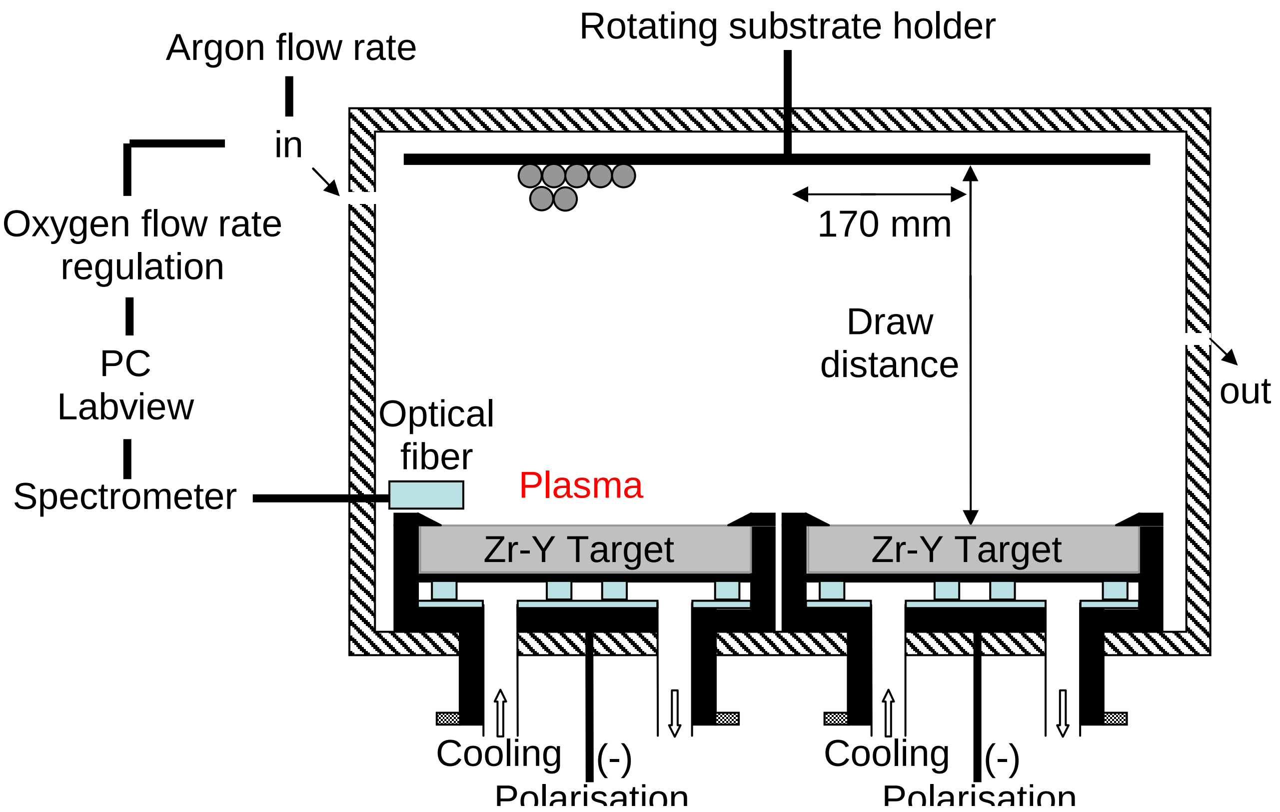


Figure 1: Experimental setup

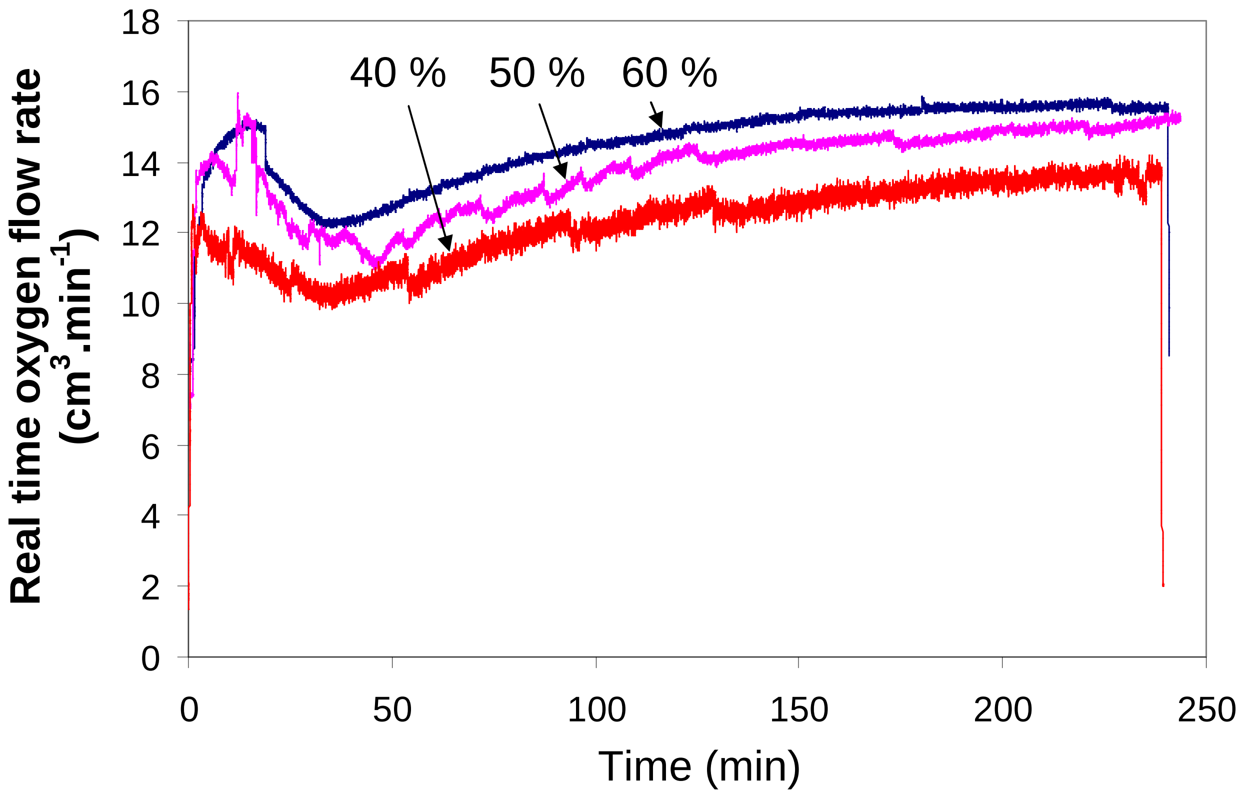


Figure 2: Real time oxygen flow rate in the sputtering chamber as a function of time for coatings realised with different regulation percentages

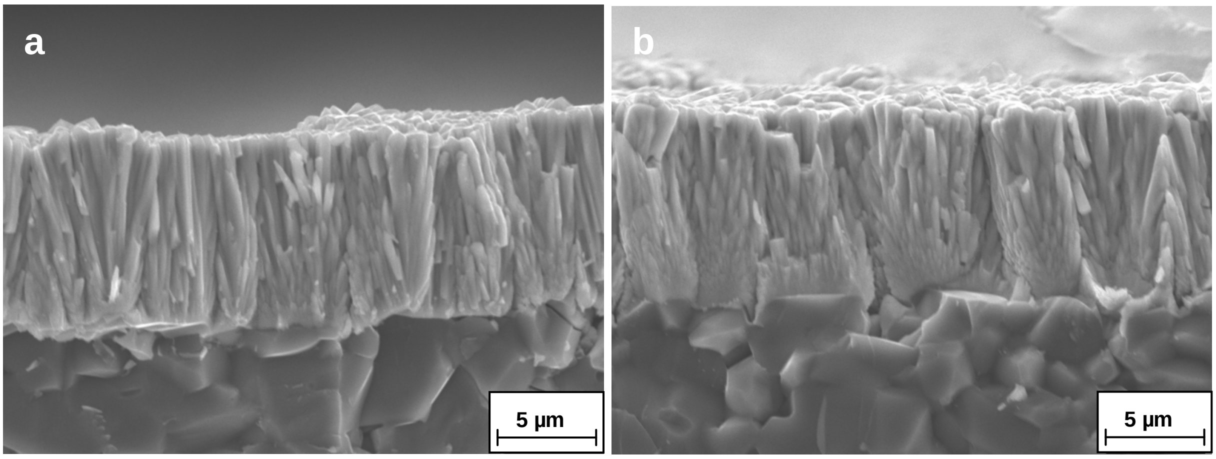


Figure 3: Brittle fracture cross SEM micrographs of YSZ sputter deposited coatings realised on porous alumina substrates with a regulation percentage of 50 % and an argon flow rate of 25 cm^3.min^{-1} (a) and 20 cm^3.min^{-1} (b) before post-oxidation thermal treatment

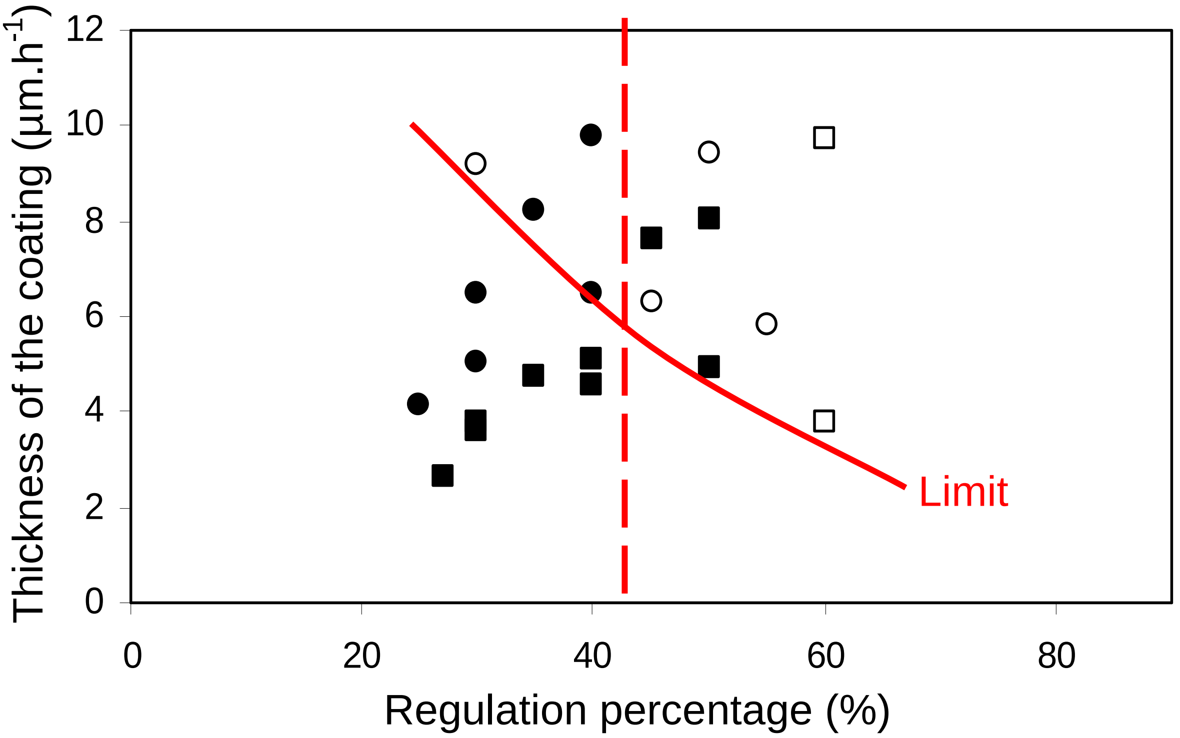


Figure 4: Adhesion (full symbols ● and ■) or desquamation (open symbols ○ and □) after the complete oxidation of the coatings realised with an argon flow rate of 25 (● and ○) and 20 cm^3.min^{-1} (■ and □) versus thickness and regulation percentage

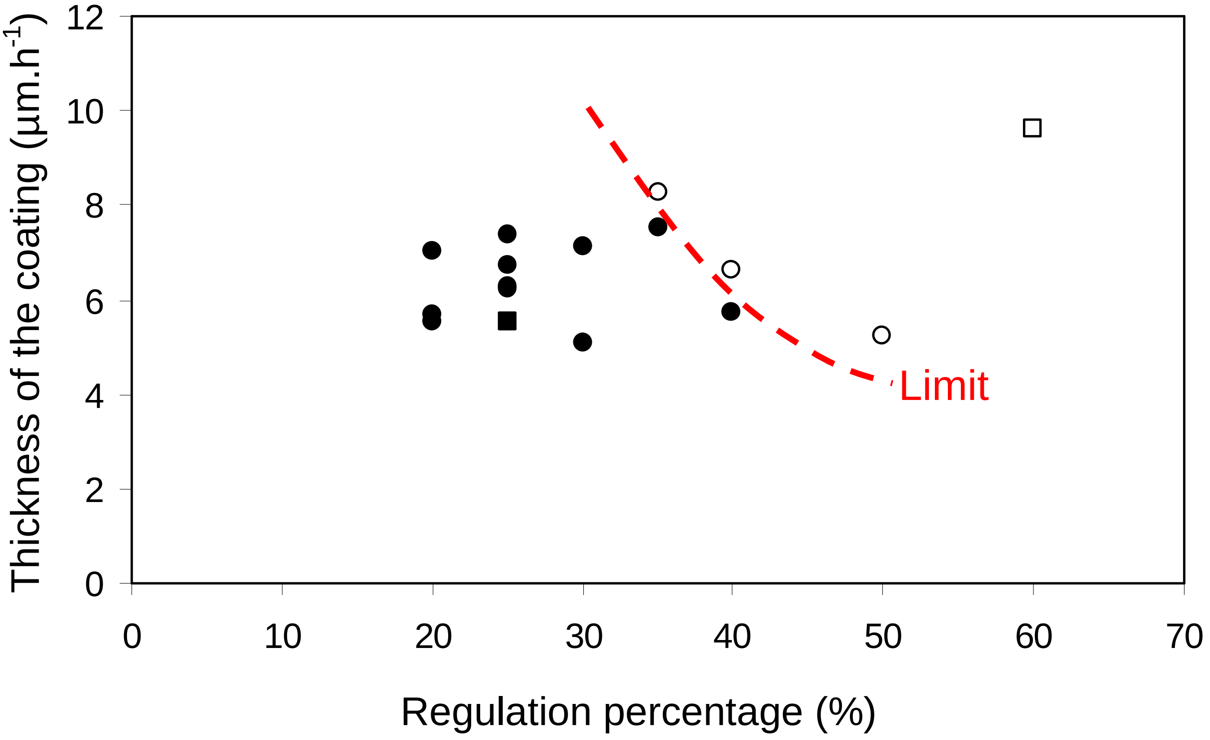


Figure 5: Adhesion (full symbols ● and ■) or desquamation (open symbols ○ and □) after a complete thermal oxidation of the coatings realised with an argon flow rate of 25 (● and ○) and 20 cm^3.min^{-1} (■ and □) versus thickness and regulation percentage

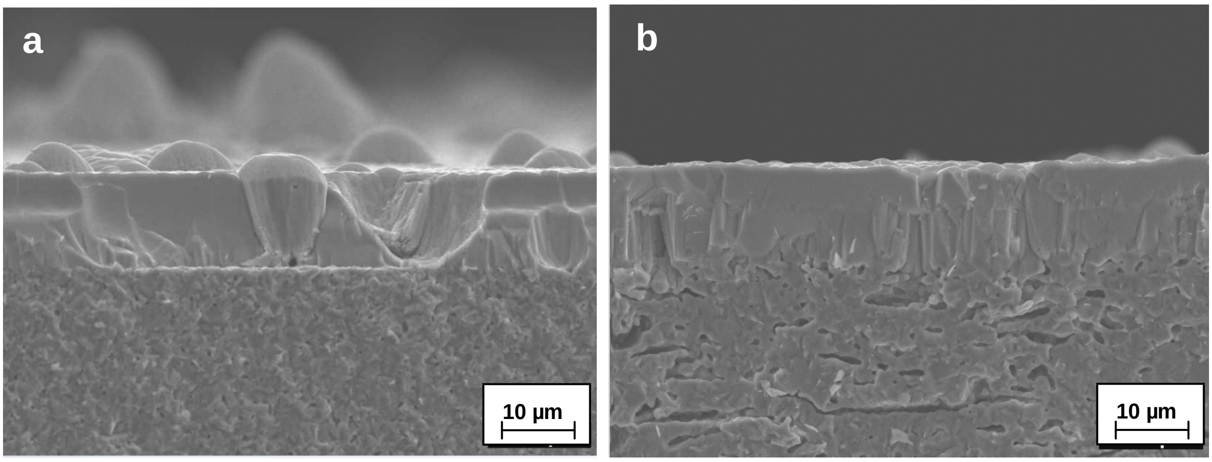


Figure 6: Brittle fracture cross SEM micrographs of as-deposited YSZ coatings realised on porous anode substrates with a regulation percentage of 60 %, an argon flow rate of 20 cm^3.min^{-1} without (a) and with (b) the addition of a substrate holder radio frequency bias

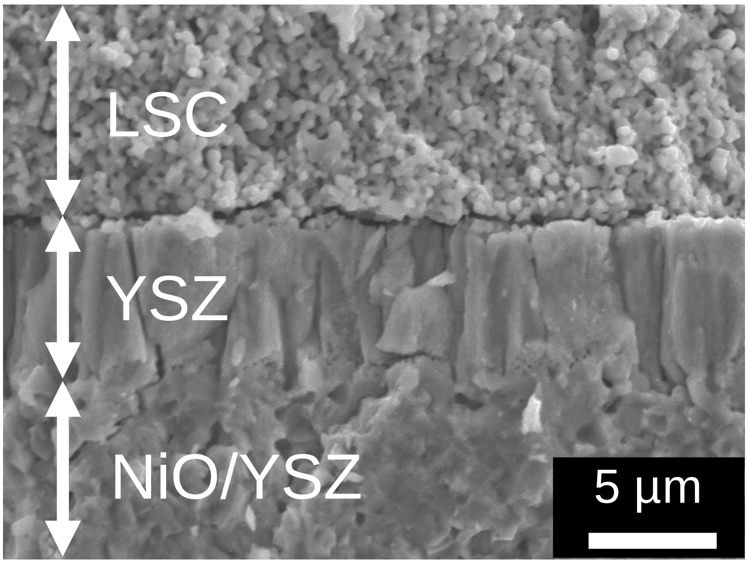
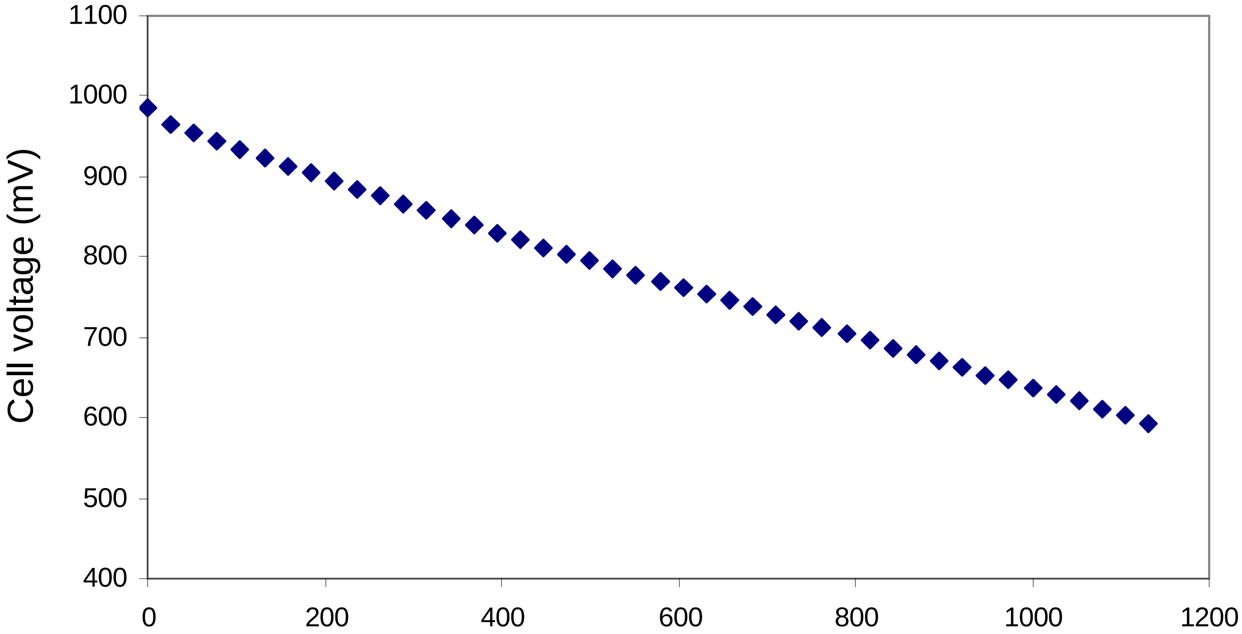


Figure 7: Brittle fracture cross section SEM micrographs of the cell realised in this study



Current density (mA.cm^{-2})

Figure 8: I-V curve at 800 °C

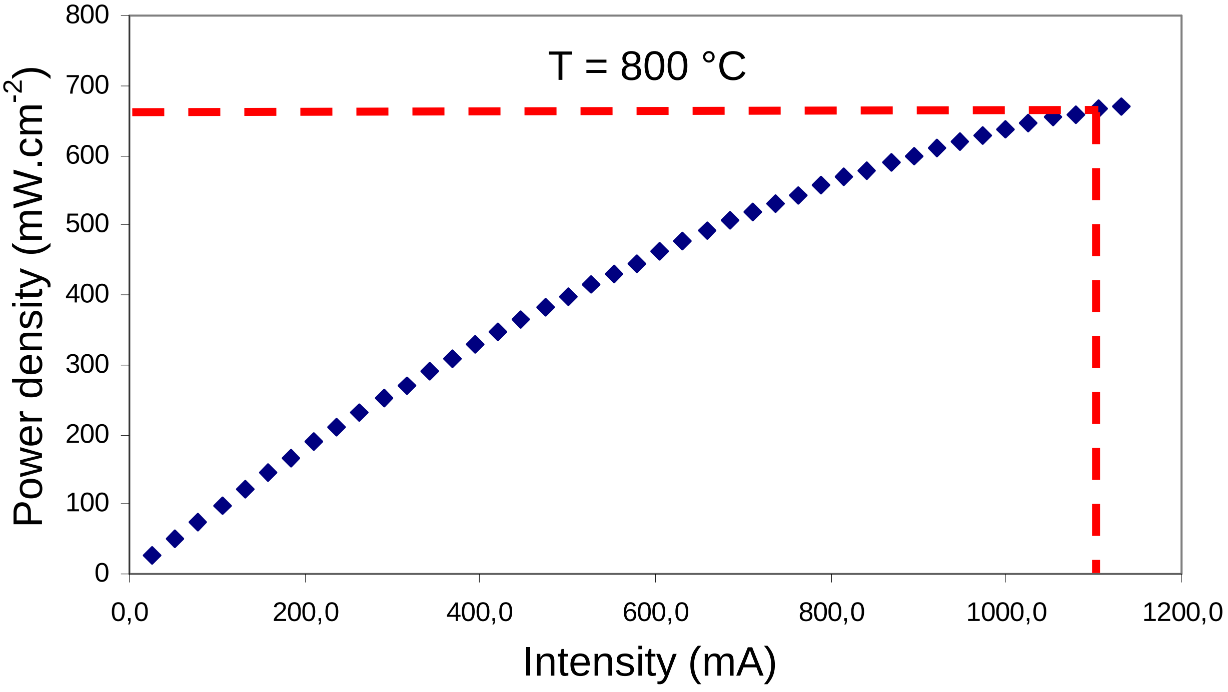


Figure 9: Power density versus intensity at 800 °C