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DURABILITY STUDY OF PVD - CATALYSED CATHODE FOR POLYMER ELECTROLYTE FUEL CELLS

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ITALIAN NATIONAL AGENCY FOR NEW TECHNOLOGIES,
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DURABILITY STUDY OF PVD - CATALYSED CATHODE FOR POLYMER ELECTROLYTE FUEL CELLS

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Riassunto

Questo lavoro riporta uno studio sulla comprensione delle prestazioni dei catodi per celle a combustibile a elettrolita polimerico, ottenute mediante deposizione PVD del catalizzatore di platino. Per indagare la durata di questo tipo di elettrodo, sono stati effettuati test in celle a combustibile prolungati. I risultati hanno evidenziato una diminuzione del rendimento durante il funzionamento prolungato dell'elettrodo a causa di una diminuzione della zona di reazione nello strato catalitico.

Parole chiave: celle a combustibile ad elettrolita polimerico, PVD, catodo, catalizzatore Pt

Abstract

This paper reports a study on the understanding of the performance of cathodes for polymer electrolyte fuel cells, obtained by means of PVD deposition of the platinum catalyst. To investigate the durability of this kind of electrode, prolonged fuel cell tests have been carried out. The results evidenced a decrease of performance during the prolonged working of the electrode due to a decrease of the reaction zone in the catalytic layer.

Keywords: polymer electrolyte fuel cells, PVD, cathode, Pt, catalyst

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1. Introduction

The polymer electrolyte fuel cells (PEFCs) are considered a leading candidate to replace the traditional power sources like the internal combustion engines. They find applications in the stationary cogeneration power systems, automotive and portable applications (e.g. consumer electronics, etc.). In the cell, fuel (hydrogen) is oxidized at the anode, oxygen (or air) is reduced to water at the cathode and electrical energy is delivered with very high yield in a production process having near-zero emissions. In order to make the PEFCs a commercial reality, a great development work has been aimed on the performance improvement of electrode materials and electrocatalysts [1]. One important goal of the current research is to lower the overall fuel cell cost through several approaches, such as reducing loadings and/or increasing utilization of the electrocatalyst or simplifying the cell design and the manufacturing process.

In the past, several authors have examined the sputter deposition technique as a means to reduce the cell costs by achieving ultra-low levels of catalyst loading and minimizing the loss of precious catalysts [2,3,4,5,6]. Efforts directed to improve the utilization efficiency of the Pt catalyst have been focused on finding an optimal material configuration that fulfil the requirement of a minimum Pt loading while maintaining the access of protons and the electronics continuity. In the past, Alvisi et al [7] focused their investigation to achieve high electrode performance with thin Pt layers sputter-deposited on the un-catalyzed gas diffusion electrodes. In their experiment thin Pt layers (1-6 nm) electrodes were characterised in a three electrodes electrochemical cell for methanol oxidation showing an increase of performance compared to commercial catalyst. Among the characteristics of such electrodes were: (a) localization of the electrocatalyst on the uppermost surface; (b) thin active layer. More recently, Shafiei et al. [8] analysed the improvement of platinum adhesion to carbon surfaces using PVD in order to enhance the Pt catalyst durability in PEMFCs.

Unfortunately, none of the above authors evaluate the performance of these electrodes for prolonged times. Individual tests were carried out only short-lived. Therefore completely missing an analysis of any degradation in the short to medium term. We need to understand whether this technique should be refined or is already sufficient for the technological application.

In this work, thicker Pt-layers sputter deposited (50-150 nm) were studied as cathodes in a complete PEFCs fed with hydrogen and oxygen in order to evaluate the influence of thickness on the membrane electrode assemblies (MEAs) performance. In addition, more prolonged test were achieved using the same kind of electrode as cathode in a MEA for about 300 hours in order to evaluate its short term durability. The best performance was confirmed for Pt layer of 100 nm but also a constant degradation was observed due probably to a weak adhesion of the Pt onto the electrode.

2. Experimental

2.1. Materials and electrode preparation

Commercially available 20 wt.% and 30 wt.% Pt/C catalyst powders on carbon black (Vulcan XC72) were purchased from E-Tek Inc. Two-layer (substrate/diffusive layer) gas diffusion electrodes (GDEs) were prepared using a spray technique described in detail in previous work [9,10]. The substrate was carbon paper (Toray TGPH090). The weight composition of the diffusion layer was 90 wt.% of carbon and 10 wt.% of PTFE, with carbon loading of about 1.2 mg cm^{-2} . These GDEs were used as substrate for the sputtering preparation of PVD platinum cathode. As references, three-layer cathodes were prepared adding on the same above diffusive layer a catalyst layer. They were prepared by mixing appropriate amounts of carbon-supported catalyst (20 wt.%) with 5 wt.% and without Nafion[®] ionomer solution from Aldrich. The platinum loading in this cathode was kept constant at about 0.2 mg cm^{-2} .

Three-layer (substrate/diffusive layer/catalyst layer) gas diffusion anodes were prepared using the same technique described above for cathodes using the same carbon paper substrates. In this case, the weight composition of the diffusion layer was 50 wt.% of carbon and 50 wt.% of PTFE, with carbon loading of about 2.5 mg cm^{-2} . The catalyst layer was prepared by mixing appropriate amounts of carbon-supported catalyst (30 wt.%) and 5 wt.% Nafion[®] ionomer solution from Aldrich. The platinum loading in all anodes was kept constant at 0.54 mg cm^{-2} together with a Nafion[®] amount of 0.9 mg cm^{-2} .

The MEAs were prepared using Nafion[®] 112 as membrane electrolyte. The membranes were used after purification treatment in 5% (w/v) H_2O_2 solution at $80 \text{ }^\circ\text{C}$ for 1 h, followed by a second treatment in 1M H_2SO_4 . The MEAs were formed by hot pressing the electrodes (5 cm^2) onto the membrane at $130 \text{ }^\circ\text{C}$ for 1–5 min and $50\text{--}100 \text{ kg cm}^{-2}$.

2.2. Sputtering preparation of catalysed PVD platinum cathode on GDE

Before insertion into the PVD chamber the GDEs substrates were only gently flushed in pure nitrogen. The Pt films were deposited on GDE substrates at room temperature by using a commercial RF magnetron sputtering system (DDC 300). The distance between the target and the substrate was 5.0 cm. The sputtering deposition system used for the experiments consists of a stainless steel chamber, which was evacuated down to $8 \times 10^{-5} \text{ Pa}$ with a turbomolecular pump backed up by a rotary pump. Before sputtering deposition, the Pt target (60 mm diameter, purity 99.95%) was sputter cleaned in pure Ar. The Ar working pressure (2.2 Pa), the power supply (36 W) and the deposition rate were kept constant throughout these investigations. By varying the deposition time we have deposited samples with different equivalent film thickness ($50\div 150 \text{ nm}$). The equivalent thickness was deduced by measuring the deposition rate on thicker film by

profilometry and the Pt loading was calculated by overestimation using the Pt density and the equivalent thickness [7].

Table 1 – Calculated Pt loading for the Pt-PVD electrodes.

Cathode type	[Pt] mg cm ⁻²
Pt-PVD-50 nm	0.11
Pt-PVD-75 nm	0.16
Pt-PVD-100 nm	0.21
Pt-PVD-125 nm	0.27
Pt-PVD-150 nm	0.32

2.3 Physicochemical characterisation

Some galvanostatic polarisation were carried out in a conventional three-electrode cell (anode/counter electrode/reference electrode) containing a 1 M H₂SO₄ solution at 25 °C [11]. The working electrode disc was inserted in a Teflon holder with an exposed area of 1 cm², gas diffusion electrode was placed inside a Teflon holder provided with a platinum-ring current collector and gas back-feeding. A stream of 10 ml min⁻¹ of O₂ was used in all the experiments of oxygen reduction. A large-area platinum flat electrode was used as a counter electrode and an Hg/HgSO₄ reference electrode was connected to the cell through a Luggin capillary. The potential values quoted are reported with respect to the reference hydrogen electrode (RHE). The electrochemical cell was connected to a Solartron mod. 1287 potentiostat/galvanostat interfaced with a GPIB card to a personal computer.

Full cell electrochemical tests have been tested at 50°C in a 5 cm² active area single cell. The cell was inserted in a Globe Tech Inc. mod. 850C station equipped with an integrated frequency resistance analyzer (FRA) for high frequency resistance (HFR) and an electrochemical impedance

spectroscopy (EIS) equipment. The cell was fed with humidified H₂ at 250 ml min⁻¹ (R.H. 152%) and O₂ at 200 ml min⁻¹ (R.H. 64%) under a pressure of 2 abs bar. The MEAs were submitted to galvanostatic polarization by means of a programmable power supply interfaced with a computer for data acquisition. All the measurements were performed in the same operative conditions. The total cell resistance was also measured continuously by the periodic current pulse transient. In this kind of measurement, the cell current is rapidly removed and the cell voltage sampled after a time delay of 20 μs. [12,13]

Prolonged tests were carried out on the same cell with Pt-100-nm cathode at 70 °C and 140 mA cm⁻² in H₂/O₂ flux at 100 and 200 ml min⁻¹ and R.H (152 and 64%), respectively. In order to simulate periodic start-ups, the cell was switched off and left in air at room temperature overnight or sometimes for an entire weekend. Cell voltage vs. time plots were recorded continuously and impedance spectra were periodically acquired in the galvanostatic mode with a DC current of 0.5 A and 0.7 A and AC 10%DC between 15 kHz and 1 Hz frequency. Electrochemical impedance spectroscopy (EIS) was used to characterize the electrodes using an impedance model already well described elsewhere [12]. Periodically, polarization curves were performed in order to evaluate changes in the whole cell performance.

Water samples were collected periodically at the exhausts using plastic containers and stored for analysis and their conductivity was measured by means of a Crison mod. 525 conductivity meter. Images of the Pt cathode with different thicknesses were acquired with a FE-SEM (field emission scanning electron microscope) LEO mod.1530, equipped with a high-resolution secondary electrons detector (in-lens detector).

3. Results and discussion

Figure 1 shows the polarization plots for the MEAs with different Pt-PVD cathodes. Particularly, the best performance is obtained with the Pt-100-nm cathode, while an increase or a decrease of the thickness brings a performance reduction.

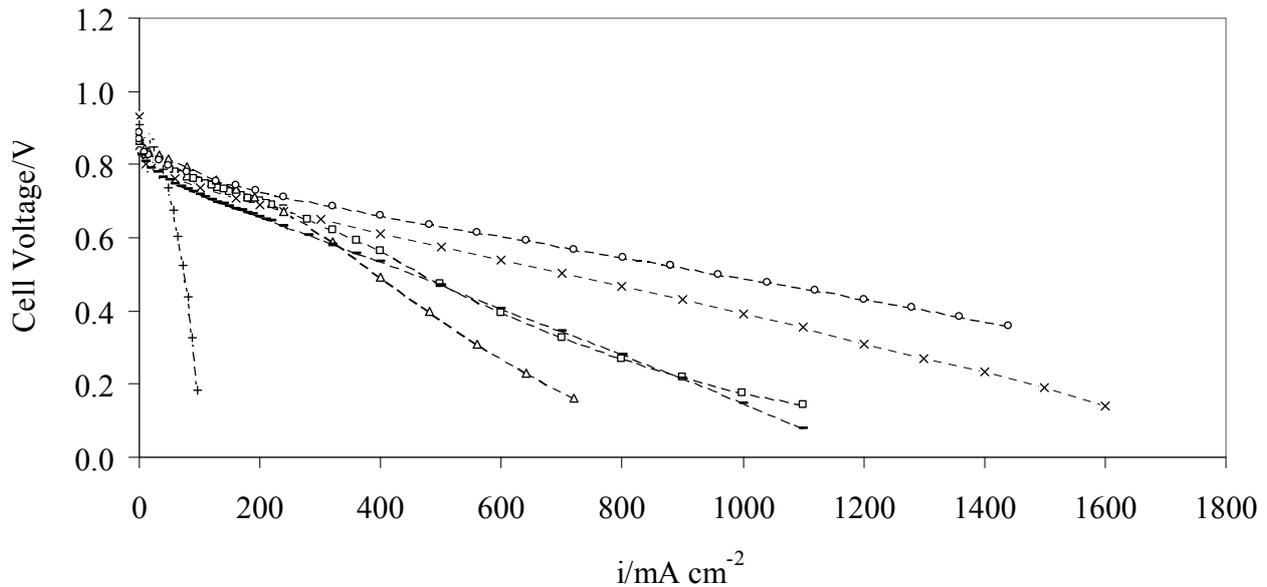


Figure 1 - Cell voltage vs. current density plot of MEAs with different PVD-cathode thickness. Pt-50-nm (Δ), Pt-75-nm (\square), Pt-100-nm (\circ), Pt-125-nm (—), Pt-150-nm (-+) and reference E-tek (\times) at 50°C.

The figure 2 reports the total cell resistance (R) and the maximum power density (P_{max}) for the MEAs as a function of the Pt equivalent thickness used on the cathode. A maximum of P_{max} which corresponds to the minimum in total resistance is evidenced. The bad performances of Pt-50 and Pt-75 can be ascribed to the low Pt loading. On the other hand, the decrease of performance for Pt-125 and mainly for Pt-150 seem to be related to a limitation in the oxygen diffusion inside the cathode at too high catalyst load.

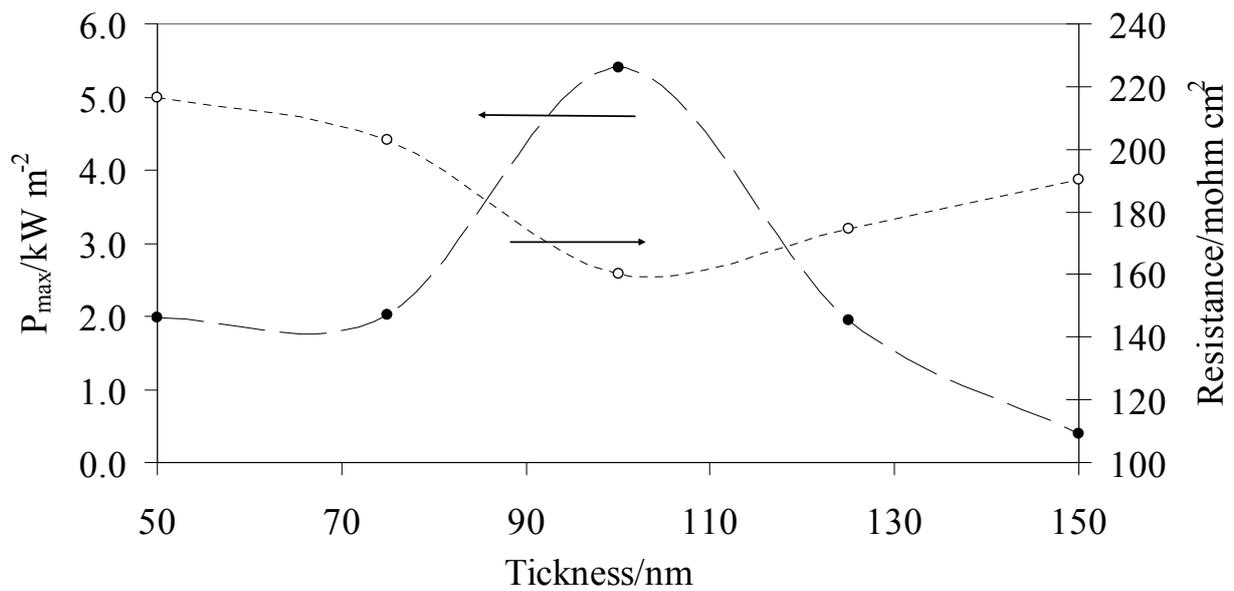


Figure 2 - Maximum power density and total cell resistance of MEAs as a function of the different PVD-cathode thickness.

The P_{max} trend plot reflect that obtained by Gruber et al. [14] at 21°C on other kinds of GDEs. These authors explained that in thicker Pt layers, only pores of large diameter are kept open for the gases supply. In addition, these thicker sputtered layers become more dense. This structure limits the water transport in the only few pores remained. That results in a higher resistance and in a lower electrochemically active surface of the sputtered catalyst. In addition a reduced activity in a thicker catalyst layer widens the gap between proton-conducting membrane and gas supply. This may also decrease cell voltage at higher current densities.

These assumptions were confirmed by our SEM images on three Pt cathodes with different thickness that show a more dense structure with the increase of thickness (Fig. 3).

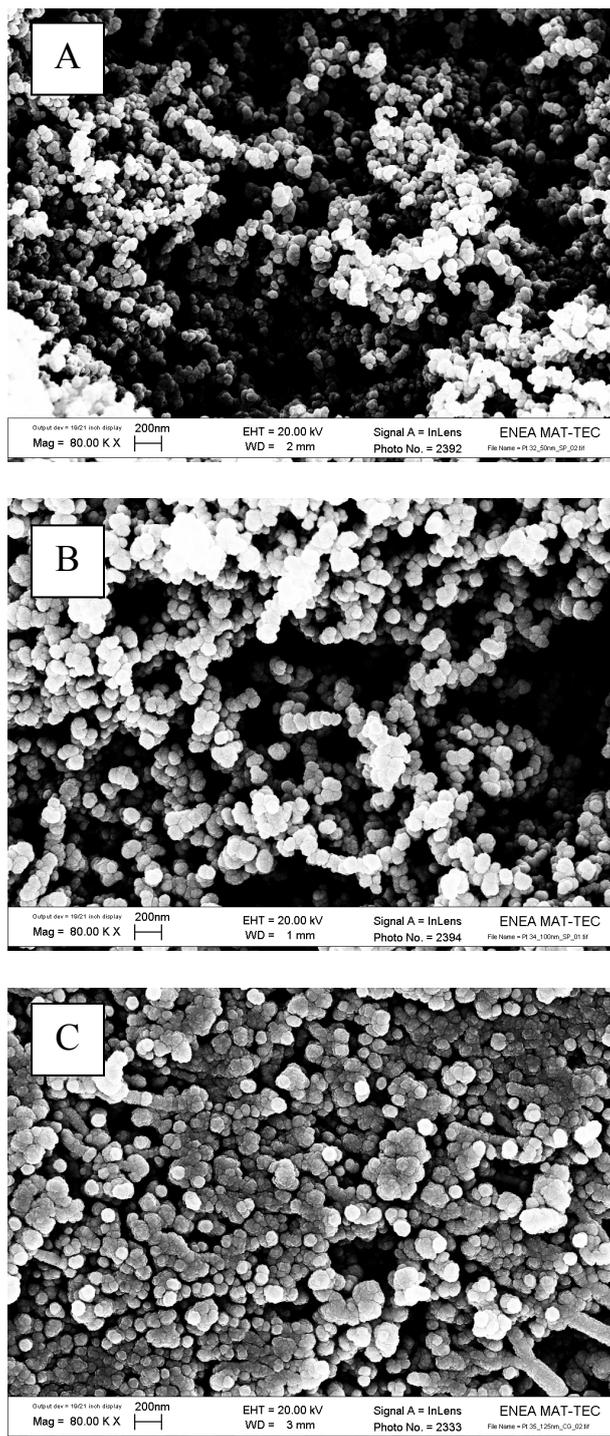


Figure 3 – FE-SEM images of Pt cathode thickness Pt-50-nm (A), Pt-100-nm (B), Pt-125-nm (C).

The Nyquist plot (Fig. 4) for the MEAs with different Pt-PVD cathodes confirms these hypothesis. Impedance data are characterised by a single loop at high frequency that is a fairly good indicator of the properties of the cathode, such as the catalyst surface area, catalyst loading and catalyst

utilization [12]. In particular, the increase of the high frequency circle's diameter, implies an increase of charge transfer resistance R_{ct} related to the oxygen reaction. Thus, the decrease of the Pt layer under 100 nm produce a decrease of active area of the catalyst's load. On the other side, the increase in Pt thickness produces also a typical second low frequency arc in the Nyquist plot, that is related to slow mass transport phenomena and it can be described by the finite-length Warburg impedance [12]. This effect begin to be evident for the Pt-125 nm cathode that shows a small hump at low frequency and it appears clearly for the Pt-150 nm. It is important to observe that the thickness increase over a certain limit (>100 nm), also decreases the active area (higher R_{ct}) because it limits the contact of the three boundary zone (gas, membrane, catalyst).

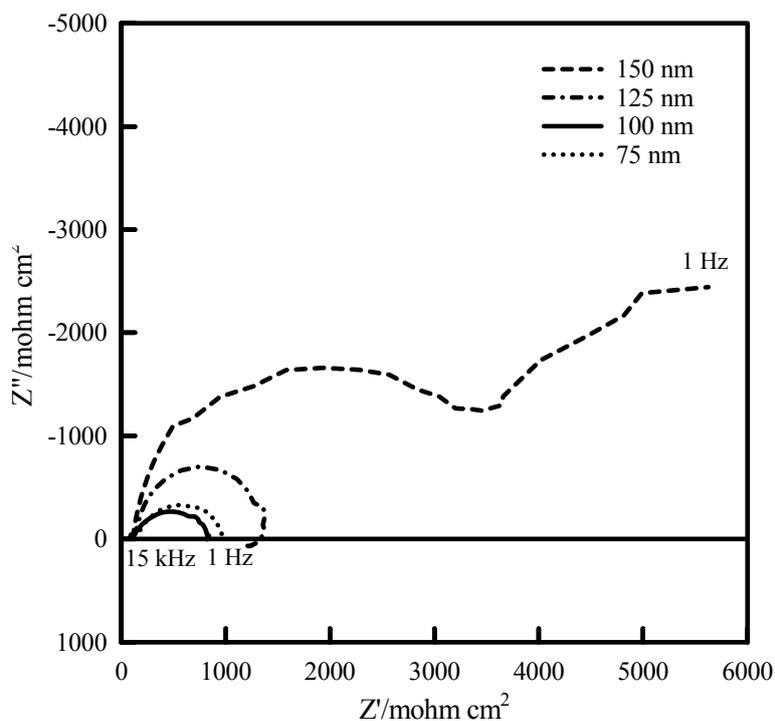


Figure 4 - Nyquist plot of MEAs with different Pt cathode thickness. Pt-75-nm (···), Pt-100-nm (—), Pt-125-nm (---), Pt-150-nm (---) at 0.5 A DC and 0.05 A AC and 50°C.

The half-cell measurements on the Pt-100 nm cathode and E-tek (0.20 mg cm^{-2}) were performed in order to compare the electrodes at the same Pt load. In this case, the load amount on Pt-100 nm was exactly measured by UV-Vis spectrometry in 0.17 mg cm^{-2} [15]. Figure 5 shows the galvanostatic

polarisation in oxygen evidencing similar performance of Pt-100-nm with respect to the commercial reference catalyst ($0.20 \text{ mgPt cm}^{-2}$). Thus, in a short time working, the two preparative methods seem comparable.

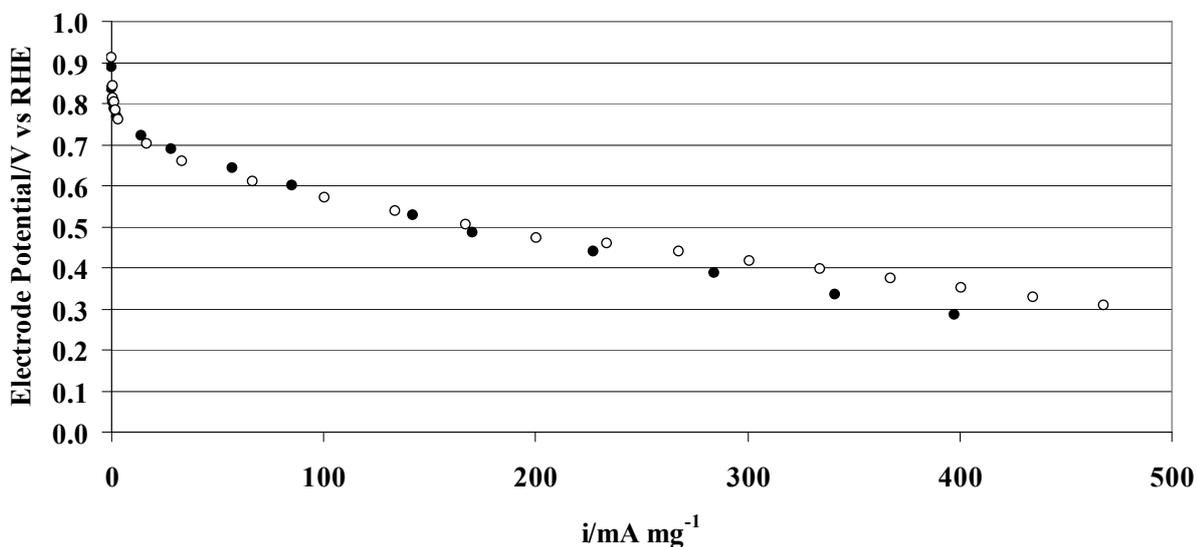


Figure 5 – Electrode potential vs. current density plot of Pt-100-nm (○) and reference E-tek (●) at 25°C.

In contrast, figure 6 depicts the cell voltage vs. time curve of MEA prepared with Pt-100-nm cathode. The MEA performance at 70 °C and current density of 140 mA cm^{-2} decreases at a rate of $-75 \text{ } \mu\text{V h}^{-1}$ after 315 h with an average voltage of $0.72 \pm 0.03 \text{ V}$. In previous work [12], we have obtained, in similar condition a decrease of only $-2.3 \text{ } \mu\text{Vh}^{-1}$ for a 2,000-h long-term test on a cell with a commercial E-tek cathode.

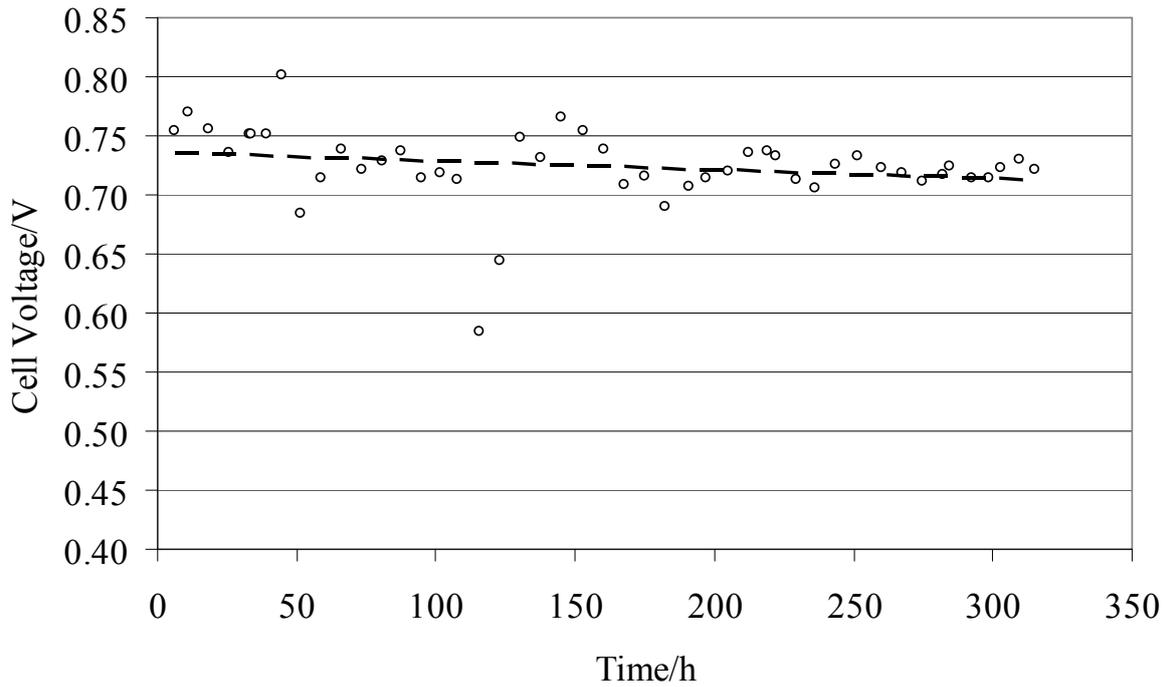


Figure 6 - Cell voltage vs. time plot of MEA with Pt-100-nm (-o-) cathode thickness at 140 mA cm⁻² and 70°C.

Figure 7 show the polarization graph, of the MEA with the Pt-100 nm cathode at 6 and at 150 hours with a well distinguished decrease of performance.

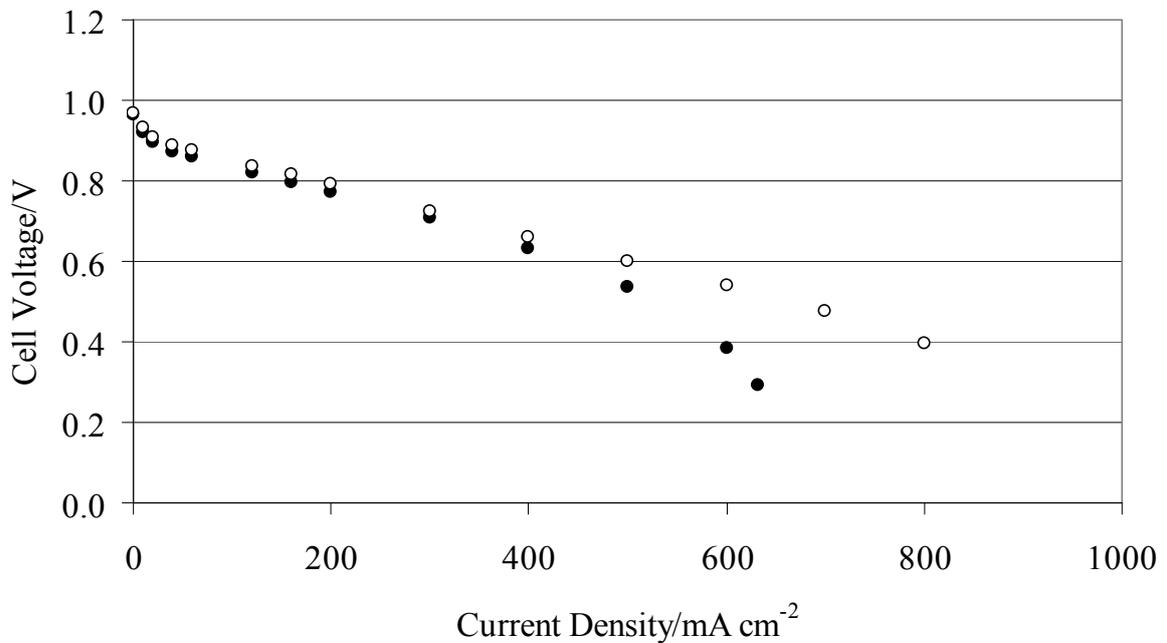


Figure 7 - Cell voltage vs. current density plot of MEA with Pt-100-nm cathode after 6 (○) and 150 (●) hours at 70°C.

In order to better evaluate the degradation mechanism that acts on the cell, impedance data were collected during the time. As example, the figure 8 shows the Nyquist plot of the cell after 6 and 150 hours. The ohmic resistance, that can be obtained as the intersection of the impedance spectrum with the real axis at 15 kHz is constant [12]. This resistance is due mainly to the proton transport resistance of the membrane and the resistance of the current collector plates. On the opposite, the charge transfer resistance (R_{ct}), that can be obtained as the circle diameter amongst 15 kHz and 1 Hz, clearly grows. In a PEFC, the R_{ct} of the cathode is much larger than that of the anode; therefore, this resistance must be referred mainly to the cathode and is a measurement of the charge transfer resistance of the oxygen reduction reaction (ORR).

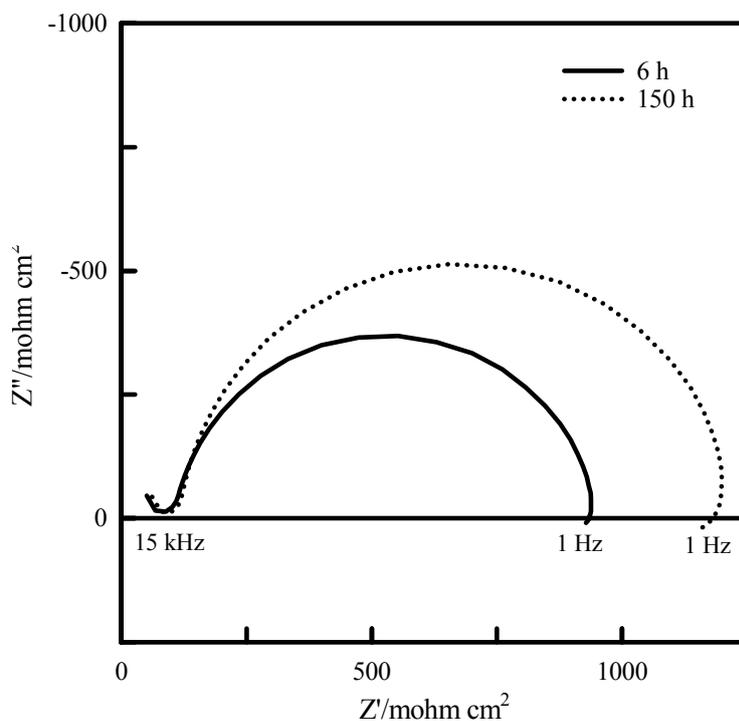


Figure 8 - Nyquist plot of MEAs with Pt-100-nm cathode after 6 (—) and 150 (---) hours at 0.7 A DC and 0.07 A AC and 70°C.

Figure 9 shows the charge transfer resistance and the ohmic one obtained by the Nyquist plot executed periodically on the cell. As above observed, the performance loss is not due to an increase in the membrane cell resistance, but to a growth of the R_{ct} . This increase indicates a degradation

mechanism due to a decrease of cathode active area during the time. The reduction of the reaction zone can be ascribed to the growth of platinum particles onto the cathode surface or to desegregation of catalytic layer, as has been demonstrated extensively by many authors [16]. The water conductivity measured in the exhaust-collected water at the cathode and anode were very low, respectively 6 ± 1 and $7\pm 1 \mu\text{S cm}^{-1}$. As showed in previous works [12,13,17], this parameter is strictly related to the release of fluoride by the membrane and, in this case confirmed the absence of membrane degradation.

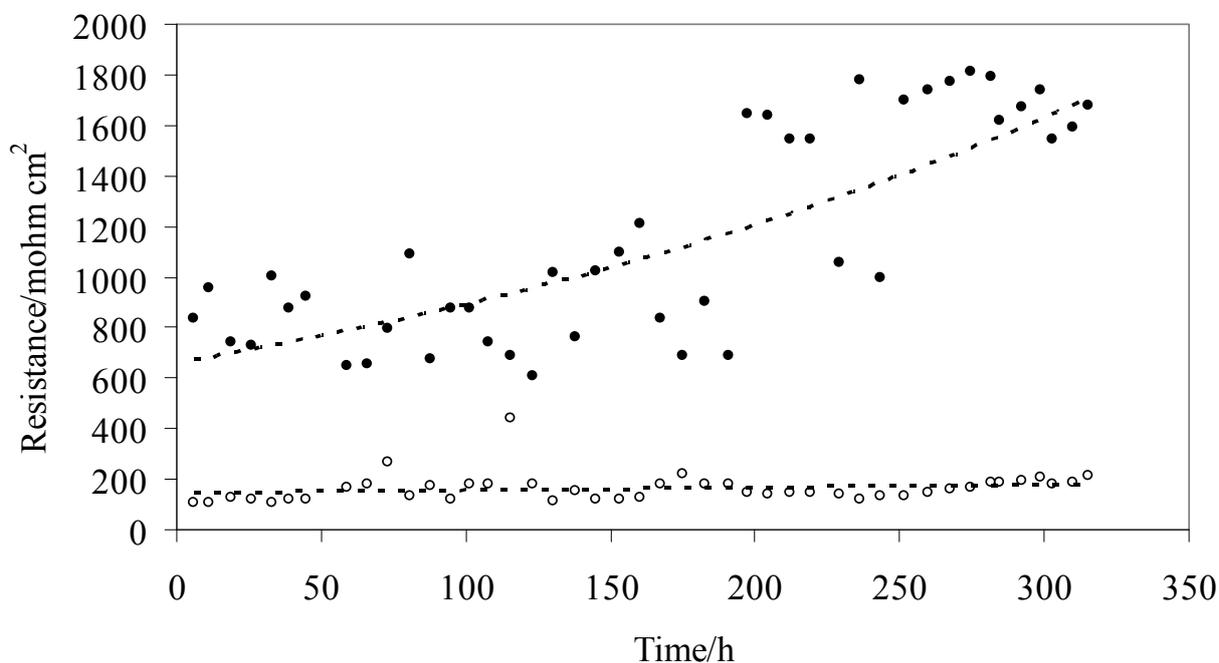


Figure 9 – Ohmic (-o-) and charge transfer (-●-) resistance vs. time of MEA with Pt-100-nm cathode thickness at 140 mA cm^{-2} and 70°C .

To have a more understanding on the reason of the performances reduction, an investigation by means of FE-SEM was carried out. The figure 10 shows the cross-section of MEA after the prolonged test. These post mortem image highlight a partial spread of the cathode platinum catalyst inside the electrolyte membrane with respect the same interface before. The breaking up of this catalytic layer leads to formation of islands inside the membrane. Thus, the catalyst fragmentation

reduce the platinum loading and consequently the catalytic surface available for the electrochemical reaction.

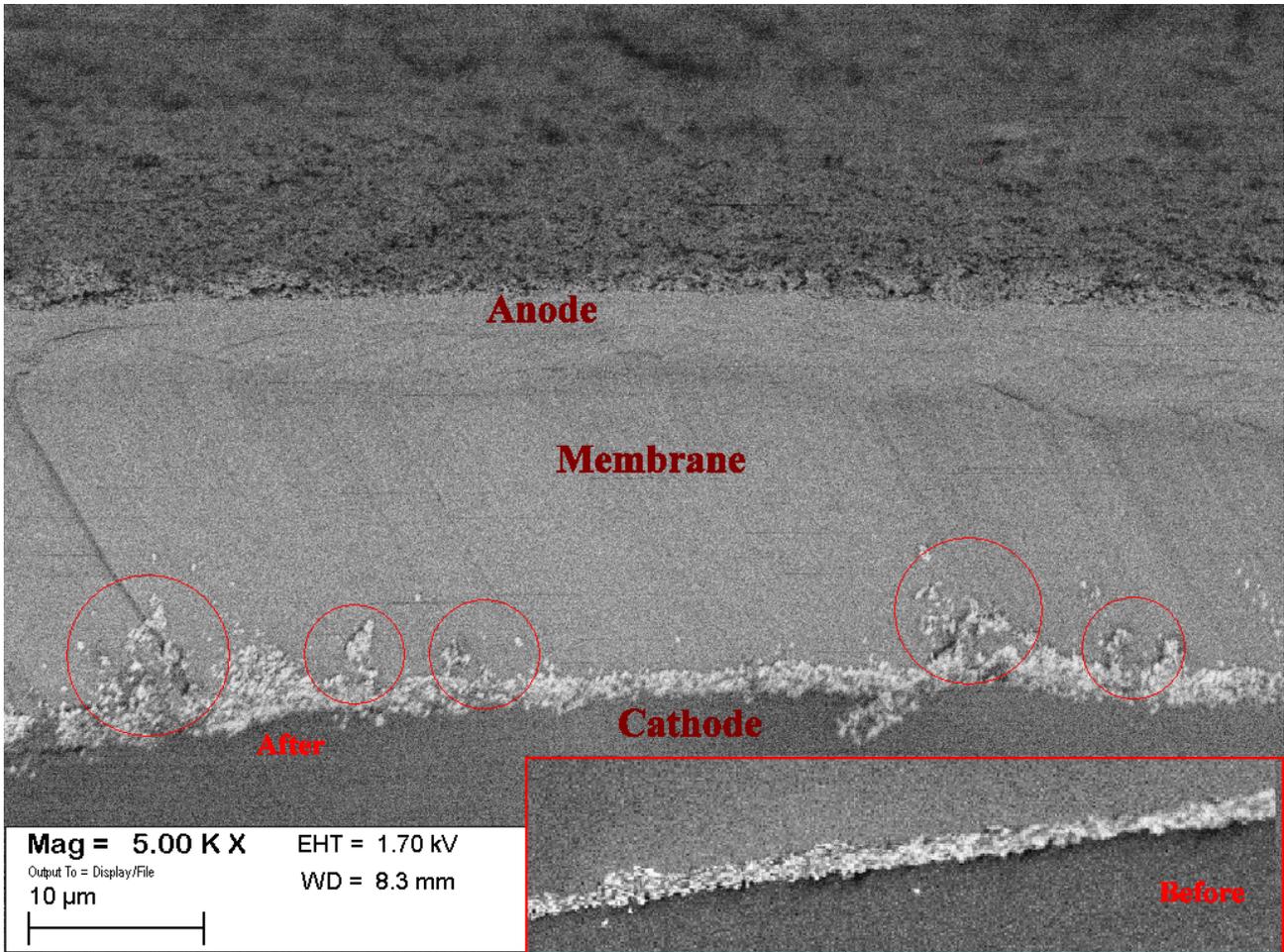


Figure 10 – FE-SEM images of MEA after the prolonged stability test. The red circular areas evidence the fragmentation of Pt-100 nm catalytic layer. In the insert the MEA cathode/membrane interface before the test.

4. Conclusion

This paper has evaluated the performance of cathodes for polymer electrolyte fuel cells, obtained by means of platinum PVD deposition and their durability. Fuel cell tests confirmed: 1) an optimal thickness for the platinum layer of about 100 nm; showed 2) an electrochemical performance of the Pt-sputtered cathode similar with respect to the commercial reference catalyst, for the same Pt load; evidenced 3) a decrease of performance during the prolonged working of the electrode due to a

decrease of the reaction zone in the catalytic layer. Further studies are needed in order to produce more stable Pt-PVD electrodes with improved Pt adhesion and an higher durability.

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