



H₂/air alkaline membrane fuel cell performance and durability, using novel ionomer and non-platinum group metal cathode catalyst

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ABSTRACT

The development of H₂/air alkaline membrane fuel cells (AMFCs) enables the use of non-platinum group metal (PGM) catalysts which are intrinsically stable and have an activity similar to platinum in alkaline media for the oxygen reduction reaction (ORR). As opposed to PEMFCs, the research and development of these types of catalysts for AMFC has had little attention and even less has been given to the development of alkaline membranes and ionomers. Acta S.p.A. has developed recently new non-PGM ORR catalysts with activity higher than Pt in alkaline media. More importantly, a new anionic ionomer derived from cheap starting materials with optimum performance has been produced. In this paper we demonstrate the use of this new ionomer in H₂/air AMFCs showing the first polarization and durability data, with current densities higher than those recently reported in the literature. Furthermore, we report the effect of CO₂ on AMFC performance, showing the possibility of use of such alkaline devices not only in outer-space applications, but also with atmospheric air for large scale devices.

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1. Introduction

Proton exchange membrane fuel cells (PEMFCs) have been the subject of intensive research over the last 20 years, however the major obstacle to the diffusion of this technology remains the slow kinetics of the oxygen reduction reaction (ORR). This leads to a high loading of precious metal catalysts, usually platinum, on the cathode side which creates a strong limitation to the eventual large scale production of fuel cell devices. In fact, the Pt-specific power density would have to be reduced to $<0.2 \text{ g}_{\text{Pt}} \text{ kW}^{-1}$ at cell voltages of $\geq 0.6 \text{ V}$ (to maintain high fuel cell energy conversion efficiencies $> 55\%$). State-of-the-art Pt loading are $\approx 0.4\text{--}0.5 \text{ mg}_{\text{Pt}} \text{ cm}^{-2}_{\text{MEA}}$, corresponding to Pt-specific power densities of $\approx 0.5 \text{ g}_{\text{Pt}} \text{ kW}^{-1}$. Therefore, to reach the above targets, Pt loadings of $\leq 0.15 \text{ mg}_{\text{Pt}} \text{ cm}^{-2}_{\text{MEA}}$ are needed, which would require improved cathode catalysts with an approximately four-fold higher activity compared to Pt/C [1]. Consequently, many researchers have concentrated on developing alloyed catalysts with low platinum content and non noble metal based catalysts.

The development of alkaline membrane fuel cells (AMFCs) has made possible the use of first row transition metal based cata-

lysts which are intrinsically stable and have an activity similar to platinum in alkaline media. As opposed to PEMFCs, these catalysts are relatively less well studied, as well as anionic exchange membranes and ionomers. In the last few years a number of new anion-exchange polymers [2,3] (membranes and ionomers) have been developed, along with noble metal free catalysts for the ORR, which together offer the possibility of developing MEAs (membrane electrode assemblies) using anionic ionomers [6] as binders analogous to PEMFCs [7].

Research carried out at Acta S.p.A. led to the production of PGM-free catalysts for ORR [4,5] that show higher activity and lower production of peroxides with respect to commercial platinum based catalysts. Recent advances in Acta include the development of a new in house anion-exchange ionomer in collaboration with the University of Pisa, Italy produced from cheap materials and a simple synthetic route, which has shown good performance and durability when used in AMFCs.

This paper describes how this new solvent suspended anion-exchange ionomer has been employed to prepare Pt/Pt catalyst coated membranes (CCM) tested in H₂/air AMFC, using CO₂-free air, with commercially available anion-exchange membranes. Also the non-PGM catalyst HYPERMECTM 4020 prepared by Acta has been tested as a cathode in AMFCs and the polarization curve and durability compared with the Pt/Pt CCMs. The same Pt/Pt CCM has been employed to understand the effect of CO₂ on AMFC

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polarization curves and durability, using non-purified atmospheric air.

2. Experimental

2.1. MEA preparation

For AMFC testing and anion-exchange measurements, a benchmark commercial membrane and an anionic ionomer (I2) suspended in methanol, developed in the collaboration between Acta S.p.A. and the University of Pisa, were used. CCMs were prepared either using a 40 wt% Pt on Vulcan catalyst (BASF) or a transition metal based catalyst supported on carbon (HYPERMEC™ 4020 from Acta [4,5]). The catalyst was mixed with a certain amount of water, lower aliphatic alcohols, and ionomer solution, and then sonicated to obtain a homogeneous ink. The obtained ink was coated on a decal support and transferred to the membrane by hot-pressing to obtain CCMs. SGL carbon paper (25BC and 25BA) with and without a microporous layer were used as diffusion medium.

The ink with Pt on carbon was obtained mixing 0.4 g of C2–40 40% HP Pt on Vulcan XC-72R supplied by BASF, 0.9 g of D.I. water ($\rho \geq 10 \text{ M}\Omega \text{ cm}$) and 2.3 g of 1-propanol (99.95% ACS Aldrich); this mixture was sonicated at 20 kHz, 60 W for 30 s, three times. After the sonication, 2.4 g of ionomer suspension (I2) were added and then the ink was sonicated again at 20 kHz, 30 W for 30 s. Using a mayer rod of 200 μm thickness, the ink was coated on a 0.003" thick PTFE sheet as decal. The metal loading in the coating was ca. $0.5 \text{ mg}_{\text{Pt}} \text{ cm}^{-2}$.

The ink with Acta's catalyst 4020 was prepared using the same procedure with the following quantities: 0.45 g of catalyst, 1.04 g of D.I. water, 4.88 g of methanol (>99%, Fluka Analytical), 3.24 g of ionomer suspension (I2).

2.2. H_2 /air AMFC tests

Cells were assembled using 5 cm^2 stainless steel single cell hardware supplied by Fuel Cell Technologies and MEAs prepared as described above. Pt/C based electrodes were used on the anode side, whereas Pt/C or 4020 based electrodes were used on the cathode side. PTFE gasket thickness was selected to obtain an overall strain of $\approx 25\%$ to guarantee good electrical contact and sealing. H_2 and CO_2 -free air (purity of N 5.0) were provided by Sol Group. Gas flows, humidification, temperature and cell temperature were controlled using a Scribner 850C test station. MEAs were conditioned by cycling between OCV and 200 mV cell potential for 10 min/step (Arbin BT-2000), till stable performance between two cycles was observed. Polarization curves were collected applying a current scan at 10 mA s^{-1} or constant voltage steps in 100 mV intervals from OCV to 200 mV.

2.3. SEM analysis

An MEA with Pt/C at anode side and 4020 at cathode side was subjected to scanning electron microscope (SEM), for morphological and elemental analysis of the electrodes before and after utilization in AMFC, using a JEOL JSM5600LV SEM equipped with Oxford EDS (energy dispersive spectroscopy) detector. Both sides were observed to understand the morphological differences between 4020 and Pt electrodes and correlate it with AMFC polarization and durability results. Each side was analysed before and after test with low vacuum and low magnification ($90\times$), then after sample metallization with high vacuum and high magnification ($20,000\times$) in order to observe the electrode morphology at 100 and 1 μm level. Furthermore, an elemental analysis was carried out with EDS to confirm the electrode composition.

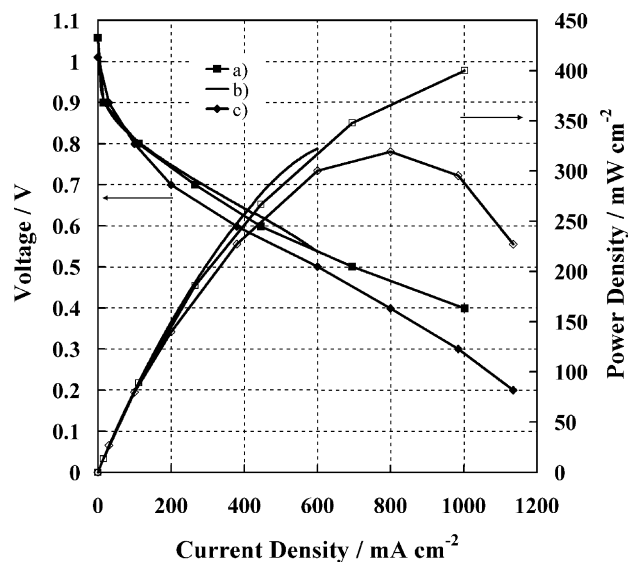


Fig. 1. (a) Polarization and power curves using SGL 25 BA as diffusion medium; (b) polarization and power curves using SGL 25 BC as diffusion medium. Both MEAs with anode and cathode loading of $0.45 \text{ mg}_{\text{Pt}} \text{ cm}^{-2}$, commercial membrane and Acta I2 anion-exchange ionomer with ionomer/carbon 0.3:1. Test conditions: H_2 1 sL min^{-1} T_{hum} : 46°C at the anode, air (CO_2 free) 2 sL min^{-1} T_{hum} : 46°C at the cathode, T_{cell} : 50°C (RH $\sim 85\%$). (c) Polarization and power curves of MEA described in Ref. [7].

2.4. HR-TEM analysis

The catalyst powders used to prepare the MEA were investigated using high resolution transmission electron microscopy (HR-TEM) to determine particle size and elemental composition, using a JEOL 2010 TEM, 200 kV, point-to-point resolution 1.9 \AA , equipped with Oxford EDS (energy dispersive spectroscopy) detector. The elemental analysis with EDS was carried out to confirm the composition and observe the homogeneity of the samples.

3. Results and discussion

3.1. H_2 /air AMFC performance

3.1.1. Pt/Pt CCMs

First of all, the anionic polymer synthesised and suspended in alcohol to obtain the ionomer suspension was analysed to determine fundamental properties like water uptake, ion-exchange capacity and ionic conductivity. Unfortunately, it was not possible to measure them because of its poor mechanical properties after casting and its high water uptake that makes it even less stable in water; in fact, when put in contact with water it is easily fragmented, forming readily a suspension. Nevertheless, this ionomer was used to prepare Pt/Pt MEAs for test in AMFCs. As evident in Figs. 1 and 2 it is effective as an ionomer despite these poor mechanical properties and high water uptake. Fig. 1 shows a comparison between two polarization and power curves in an AMFC with Acta ionomer and a recent data reported in a conference abstract [8]. The ionomer to carbon ratio is 0.3:1 for Acta cells whereas this value is more than twice in the literature data (0.7:1) and the gas humidification used is higher in latter data (95% RH compared to about 85% RH for Acta AMFCs). It is evident that the power and polarization curves are very similar in the kinetic range (low current); both the results obtained in Acta are very close to each other and better than that reported in the literature at higher currents, where contributions from ohmic drops due to membrane and electrodes become important. The difference between curves (a) and (b) is in the gas diffusion layer (GDL) used: in (a) there is SGL 25 BA without a microporous layer while in (b) a SGL 25 BC with microporous

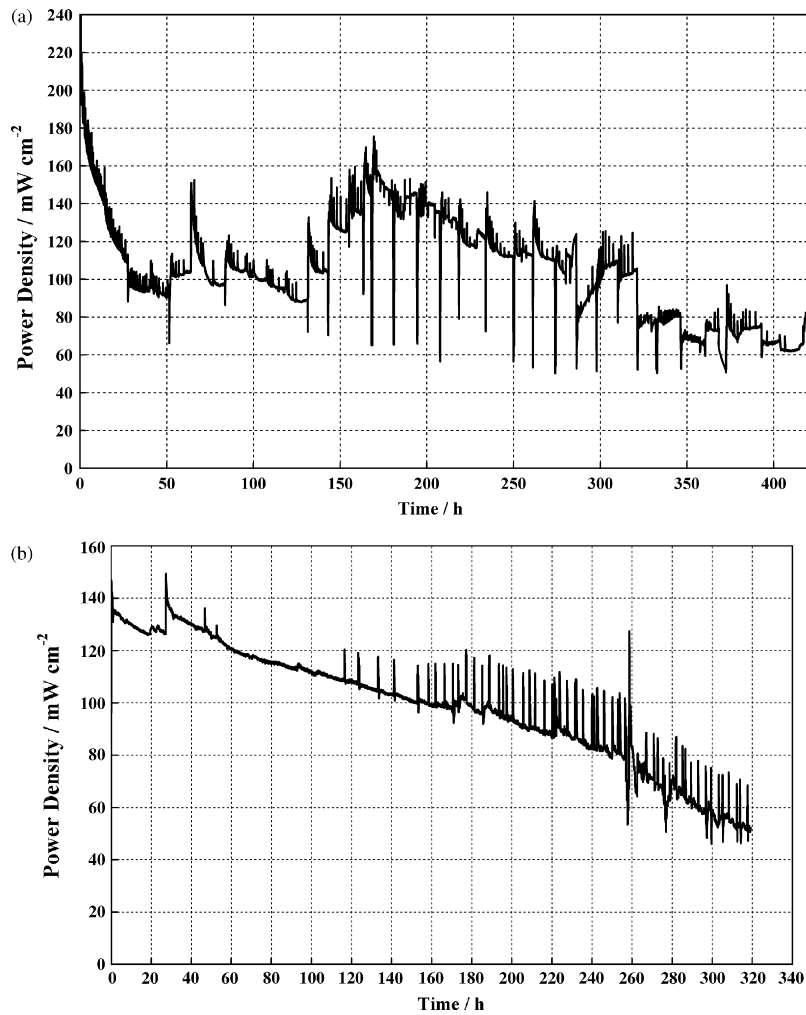


Fig. 2. Durability test for MEAs with anode and cathode loading of $0.45 \text{ mg}_{\text{Pt}} \text{ cm}^{-2}$, commercial membrane and Acta I2 anion-exchange ionomer, ionomer/carbon 0.4:1. (a) Ionomer/carbon 0.4:1; constant potential at 0.4 V with high mass flow. Test conditions: H_2 1 sL min^{-1} $T_{\text{hum.}}$: 46°C at the anode, air (CO_2 free) 2 sL min^{-1} $T_{\text{hum.}}$: 46°C at the cathode, T_{cell} : 50°C (RH $\sim 85\%$) using SGL 25 BC as diffusion medium. (b) Ionomer/carbon 0.3:1; constant current at 240 mA cm^{-2} with low mass flow. Test conditions: H_2 0.1 sL min^{-1} $T_{\text{hum.}}$: 42°C at the anode, air (CO_2 free) 0.3 sL min^{-1} $T_{\text{hum.}}$: 42°C at the cathode, T_{cell} : 50°C (RH $\sim 85\%$) using SGL 25 BC as diffusion medium.

layer is used. The difference between the two results, even though curve (b) is truncated, can be correlated to a better mass transport for curve (a) due to the absence of the microporous layer. The mass transport problem in the literature data can be correlated to the high ionomer to carbon ratio that decreases the amount of porosity in the electrode. From these polarization results we can say that with this ionomer and CCM produced in Acta we can provide excellent peak power density, the best yet to be found in the scientific literature.

The durability curves in Fig. 2 were obtained with a CCM having a higher ionomer to carbon ratio (0.4:1) than that shown in Fig. 1, in order to get better durability, even though slightly decreased power density. Fig. 2a and b shows different durability tests: potentiostatic at 0.4 V and 1 sLm H_2 , 2 sLm pure air for Fig. 2a while for Fig. 2b galvanostatic at 240 mA cm^{-2} that provides a cell voltage of about 0.6 V at the beginning of the test and 0.1 sLm H_2 , 0.3 sLm pure air; the conditions used in the latter test simulate better a real working device. Such preliminary durability tests show a good starting point with a first ionomer with unoptimized properties which include high water uptake and consequent poor mechanical stability. Such durability, of course, is not comparable to the results that can be obtained reliably with Nafion, but this instability can also be explained by poor control of gas humidification control that increases the mechanical stress to the bonded electrode which contains an ionomer that swells too much in the presence of water. The

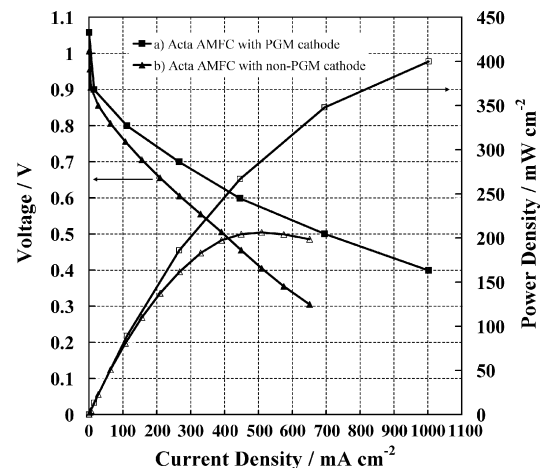


Fig. 3. Polarization and power curves for: (a) MEA with anode and cathode loading of $0.45 \text{ mg}_{\text{Pt}} \text{ cm}^{-2}$, commercial membrane and Acta I2 anion-exchange ionomer with ionomer/carbon 0.3:1; (b) MEA with anode loading of $0.45 \text{ mg}_{\text{Pt}} \text{ cm}^{-2}$ and cathode loading of $0.8 \text{ mg}_{4020} \text{ cm}^{-2}$, commercial membrane and Acta I2 anion-exchange ionomer with ionomer/carbon 0.3:1. Test conditions: H_2 1 sL min^{-1} $T_{\text{hum.}}$: 46°C at the anode, air (CO_2 free) 2 sL min^{-1} $T_{\text{hum.}}$: 46°C at the cathode, T_{cell} : 50°C (RH $\sim 85\%$) using SGL 25 BC as diffusion medium.

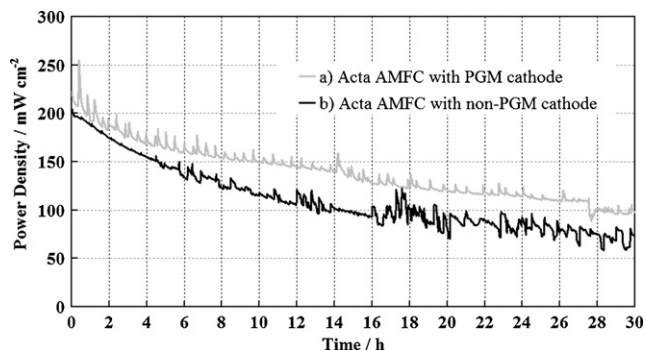


Fig. 4. Durability test for (a) MEA with anode and cathode loading of $0.45 \text{ mg}_{\text{Pt}} \text{ cm}^{-2}$, commercial membrane and Acta I2 anion-exchange ionomer with ionomer/carbon 0.4:1; (b) MEA with anode loading of $0.45 \text{ mg}_{\text{Pt}} \text{ cm}^{-2}$ and cathode loading of $0.8 \text{ mg}_{4020} \text{ cm}^{-2}$, commercial membrane and Acta I2 anion-exchange ionomer with ionomer/carbon 0.3:1; test conditions: H_2 1 sL min^{-1} $T_{\text{hum.}}$: 46°C at the anode, air (CO_2 free) 2 sL min^{-1} $T_{\text{hum.}}$: 46°C at the cathode, $T_{\text{cell.}}$: 50°C (RH $\sim 85\%$) using SGL 25 BC as diffusion medium.

reason for the spikes observed in the durability are due to the necessity of stopping the test to refill the water humidification bottles. All this instrumental factors make the durability tests intrinsically more severe for these CCMs.

3.1.2. Pt/4020 CCMs

Fig. 3 shows a comparison between the polarization curves collected with Pt/Pt (a) and Pt/4020 (b) MEAs. The decrease in peak power for MEA with non-PGM catalyst is evident and from the shape of polarization curve is not due to a kinetic difference

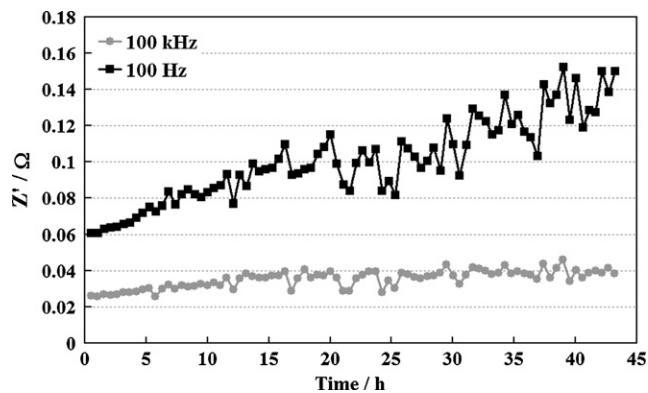


Fig. 5. Real part of impedance at 100 kHz and 100 Hz, voltage amplitude 1 mV, every 30 min during durability test of Fig. 4, curve (b).

between the two catalysts (confirming the RDE data comparison showed in [4]) but an increased ohmic drop due to the non-PGM electrode, since it is the only difference between the two CCMs. Nevertheless, Fig. 3 shows good and promising initial performance of MEAs with the HYPERMEC™ 4020 cathode catalyst (205 mW cm^{-2} of peak power).

In Fig. 4 the first few hours of the durability experiments of Pt/Pt (the same as Fig. 2b) and that of Pt/4020 CCMs are compared. It is evident a faster decrease in power density occurs for the Pt/4020 CCM. Fig. 5 provides evidence that the decrease in performance with time is correlated to electrode resistance: the high frequency real part of impedance, attributable mainly to the membrane

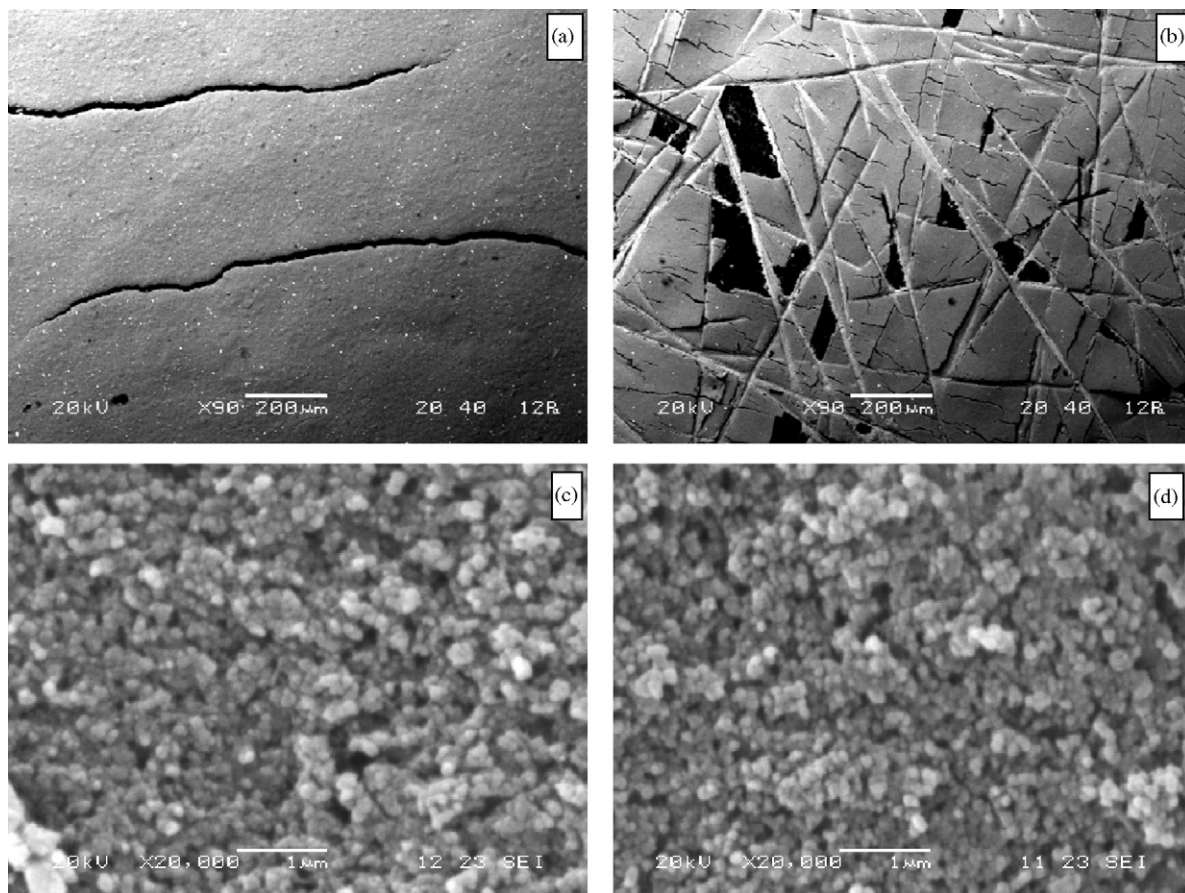


Fig. 6. SEM micrographs of the anode side of CCM (Pt catalyst layer). (a) and (c) before test; (b) and (d) after test. Test conditions: H_2 1 sL min^{-1} $T_{\text{hum.}}$: 46°C at the anode, air (CO_2 free) 2 sL min^{-1} $T_{\text{hum.}}$: 46°C at the cathode, $T_{\text{cell.}}$: 50°C (RH $\sim 85\%$) using SGL 25 BC as diffusion medium.

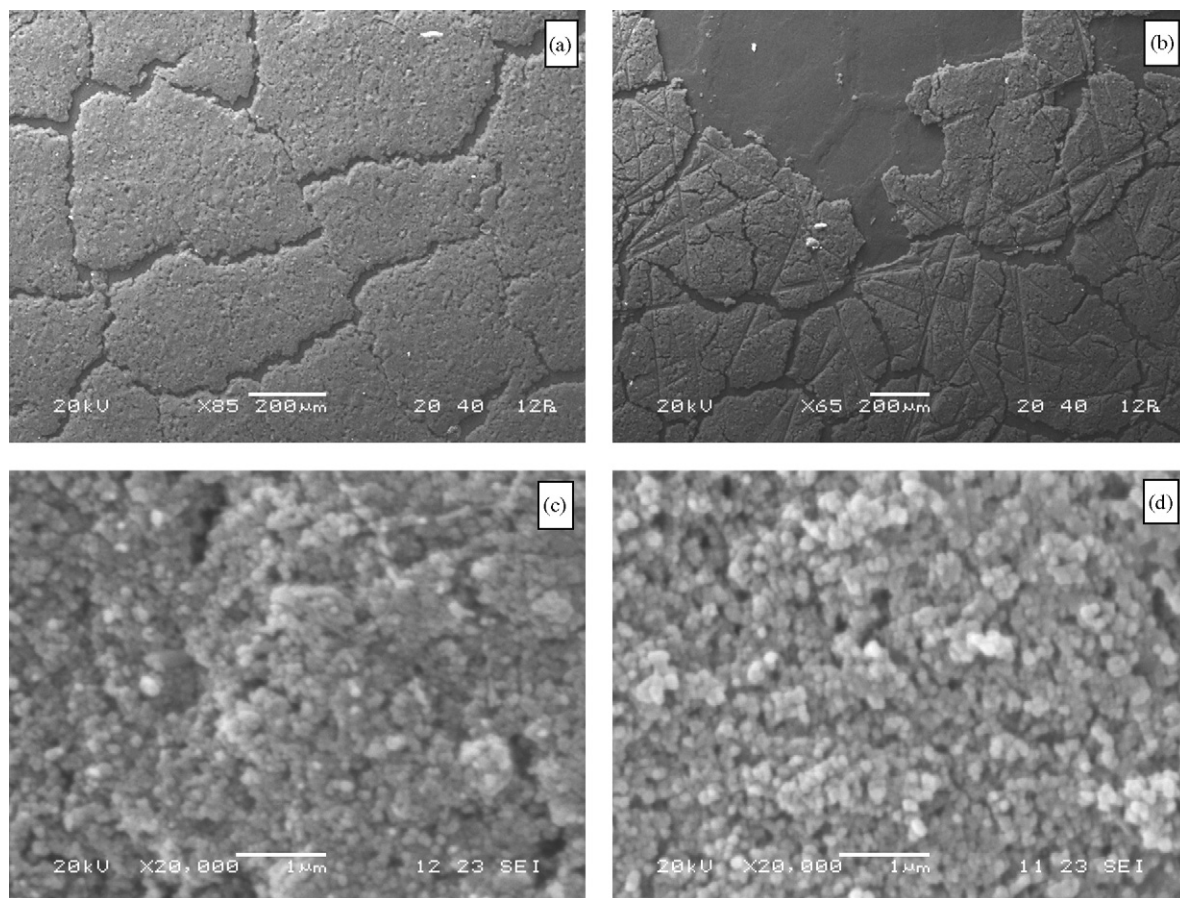


Fig. 7. SEM micrographs of the cathode side of CCM (4020 catalyst layer). (a) and (c) before test; (b) and (d) after test. Test conditions: H_2 1 sL min⁻¹ T_{hum} : 46 °C at the anode, air (CO_2 free) 2 sL min⁻¹ T_{hum} : 46 °C at the cathode, T_{cell} : 50 °C (RH ~85%) using SGL 25 BC as diffusion medium.

resistance is constant with time, while the low frequency real part of impedance, having a higher contribution from electrode resistance, increases with time.

3.2. SEM analysis on CCMs

Figs. 6 and 7 show the SEM analysis on a Pt/4020 CCM before and after testing in AMFC (right column and left column, respectively); the four micrographs in Fig. 6 regard the Pt anodic side of the CCM while the other four in Fig. 7 show the non-PGM side both with high and low magnification. Already at a first glance, also without a microscope, a strong increase in cracking and disomogeneity of the non-PGM cathode side is evident, in comparison with PGM side (Figs. 6a and 7a). The omogeneity of the CCM at 0.1 mm scale on the cathode side decreases further after testing (Fig. 7b) and it is also possible to see on the nude membrane the prints of the fragments of the cathodic coating. Such bad homogeneity and mechanical instability is the first reason of low performance and durability of the non-PGM AMFC, due to increase of electrode resistance. Also PGM side of the CCM shows an increase in cracks and disomogeneity after testing (Fig. 6b); this is, also for Pt, the main reason for decrease in durability. On the contrary, at micron scale there is very good homogeneity for all the coatings after and before the tests, with particle size that is about 100 nm (Figs. 6c and d and 7c and d). Also EDS analysis shows homogeneity of the coatings to submicrometric scale, since it is impossible to discriminate ionomer and catalyst. This is the evidence of a good ink preparation in each case, while the bad homogeneity at macroscopic scale proves bad effectiveness as binder of Acta's I2 ionomer, most of all in the case of non-PGM catalyst.

3.3. CO_2 effect

One of the doubt for the use of an alkaline membrane fuel cell is the carbonation of the MEAs, most of all using non-purified atmospheric air [9]. This doubt is an inheritance from the old H_2/O_2

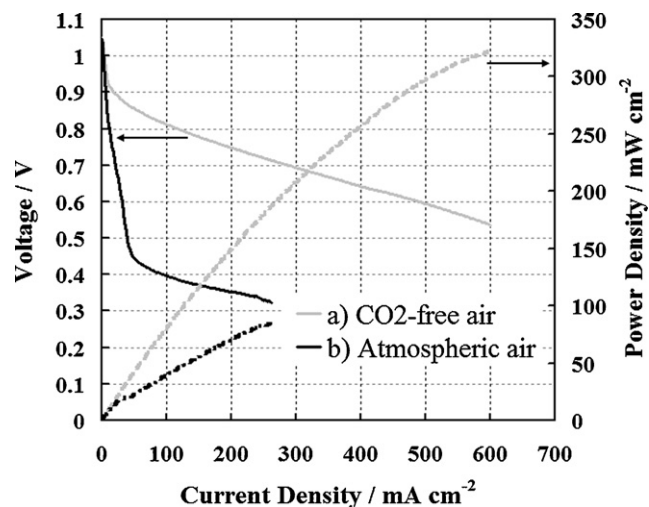


Fig. 8. Polarization and power curves for MEAs with anode and cathode loadings of 0.45 mg_{Pt} cm⁻², commercial membrane and Acta I2 anion-exchange ionomer with ionomer/carbon 0.3:1; test conditions: H_2 1 sL min⁻¹ T_{hum} : 46 °C at the anode, air 2 sL min⁻¹ T_{hum} : 46 °C at the cathode, T_{cell} : 50 °C (RH ~85%) using SGL 25 BC as diffusion medium. (a) Used CO_2 -free air at cathode; (b) used non-purified atmospheric air at cathode.

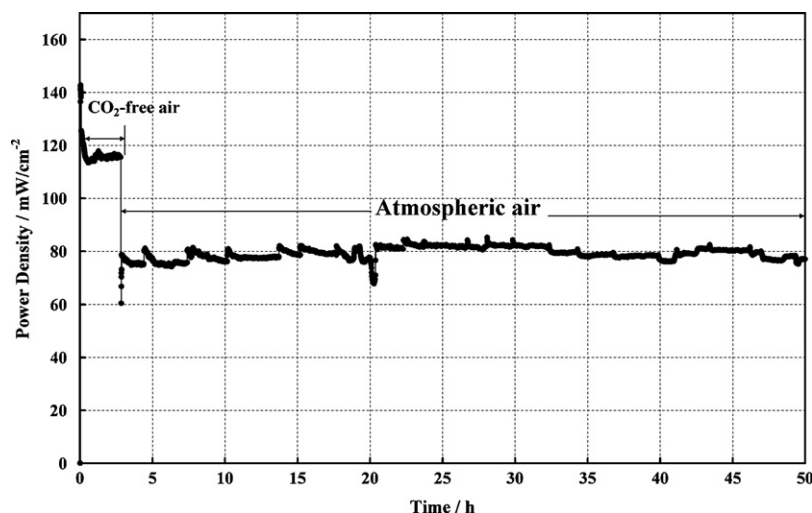


Fig. 9. Durability test for MEAs with anode and cathode loading of $0.45 \text{ mg}_{\text{Pt}} \text{ cm}^{-2}$, commercial membrane and Acta I2 anion-exchange ionomer with ionomer/carbon 0.4:1; test conditions: H_2 0.1 sL min^{-1} $T_{\text{hum.}}$: 42°C at the anode, air 0.3 sL/min $T_{\text{hum.}}$: 42°C at the cathode, T_{cell} : 50°C (RH $\sim 85\%$) using SGL 25 BC as diffusion medium. Galvanostatic test at 240 mA cm^{-2} . After 2 h switched from CO_2 -free air to non-purified atmospheric air at cathode.

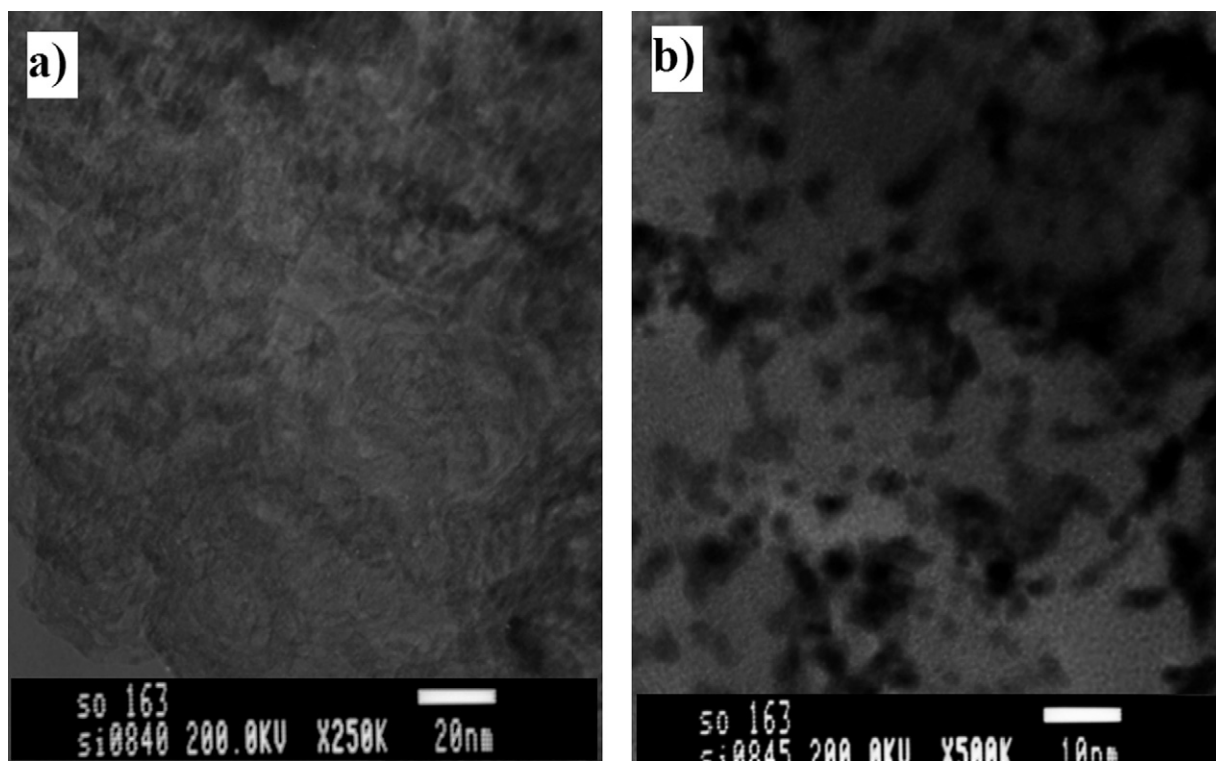


Fig. 10. (a) HR-TEM micrograph on 4020 catalyst powders, showing no evident metal particle but only a dark rim on the carbon edges, evidence of high electron density due to the presence of very finely dispersed metal (definitely less than 1 nm); (b) HR-TEM micrograph on 40% Pt/C evidencing, as expected, Pt particles with approximate size of 5–8 nm on carbon support.

alkaline fuel cells with liquid electrolyte, which had durability issues because of the progressive carbonatation of the electrolyte. In the case of AMFC the situation is slightly different: the electrolyte is a functionalized thin membrane with anionic conductivity which is carbonated in presence of atmospheric air, leading to lower membrane ionic conductivity. When the current increases OH^- ions are produced at the cathode and migrate through the membrane, making its ionic conductivity higher with the so-called self-purging mechanism [3,10]. This behaviour explains the data showed in Figs. 8 and 9. The polarization curve in Fig. 8 shows a strong decrease in potential at low currents due to membrane carbonatation and a fast change in slope at higher currents with a slope similar to the

polarization curve collected using CO_2 -free air. Also the durability in Fig. 9 shows only a small drop in potential and power changing from CO_2 -free air to non-purified atmospheric air, without any poisoning with time. All these results are coherent with the above mentioned mechanism.

3.4. HR-TEM analysis on catalyst powders

The HR-TEM analysis of 4020 cathode catalyst confirmed the presence of very finely dispersed groups of metal atoms, so small that are not directly visible using HR-TEM microscopy, thus having a size definitely lower than 1 nm. Their presence is indirectly

evident from the EDS analysis, which shows an appreciable metal content dispersed over the whole carbon support, even though not homogeneously dispersed, quantified as at least twice the metal content obtained from a background analysis of the surroundings of the sample. Another visual, direct evidence is the presence of a dark rim on the carbon edges, proving high electron density in that region (Fig. 10a). The analysis evidenced other metal phases with much larger particle sizes (about 50 nm), certainly not providing enough surface area and of a quantity to justify the catalytic activity. For sake of comparison, Fig. 10b shows the HR-TEM micrograph of commercial Pt/C 40% used in the AMFC tests. The sample observation evidenced sharp, homogeneously dispersed Pt particles with approximate size of 3–8 nm on a carbon support.

4. Conclusions

This work has confirmed the importance of optimization of the ionomer material for AMFCs improved performance. A family of novel anion-exchange ionomers have been developed in Acta that promote excellent peak power density in AMFC, the best yet to be found in the scientific literature. Together with commercial membranes, these in-house ionomers gave excellent results in H₂/air AMFC using Pt/C in both anode and cathode catalyst. ACTA's own non-PGM cathode catalyst also performed encouragingly in preliminary tests; metal quantification with an EDS detector during HR-TEM analysis on this catalyst provided the first evidence of groups of very finely dispersed metal atoms (size less than 1 nm), visible at direct observation with HR-TEM only as dark rim on the carbon edges. The degradation mechanisms seem to be mainly a physical weathering of the electrode during functioning that leads to a brittle electrode layer that easily separates from the membrane surface. The consequent dramatic increase in electrode resistance was observed to be indeed due to the deterioration of the electrode coating. In order to obtain long term stability in AMFCs (>10,000 h) tuning of important characteristics of the ionomer such as water uptake has yet to be achieved and this will form the basis of fur-

ther research effort in Acta. Furthermore, we have confirmed that feeding atmospheric air at the cathode does not lead to a continuous deterioration in performance due to carbonation but only to a step down in performance. If this is confirmed then AMFCs have a real potential as low cost alternatives to PEMFCs in large scale applications.

Acknowledgements

HR-TEM micrographs on catalyst powders were obtained in the Earth Science Department of the University of Siena. We would like to express our gratitude to Prof. Marcello Mellini for the accurate and prompt HR-TEM analysis.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at [doi:10.1016/j.jpowsour.2009.12.085](https://doi.org/10.1016/j.jpowsour.2009.12.085).

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