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CHARACTERIZATION OF PEM FUEL CELL MEMBRANE-ELECTRODE-ASSEMBLIES BY ELECTROCHEMICAL METHODS AND MICROANALYSIS

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ABSTRACT

Characterization of Membrane Electrode Assemblies (MEAs) is used to help optimize construction of the MEA. Characterization techniques include electron microscopies (SEM and TEM), and electrochemical evaluation of the catalyst. Electrochemical hydrogen adsorption / desorption (HAD) and CO oxidation are used to evaluate the active Pt surface area of fuel cell membrane electrode assemblies. Electrochemical surface area measurements have observed large active Pt surface areas, on the order of 50 m²/g for 20% weight Pt supported on graphite. Comparison of the hydrogen adsorption/desorption with CO oxidation indicates that on the supported catalysts, the saturation coverage of CO/Pt is about 0.90, the same as observed in H₂SO₄. The catalyst surface area measurements are nearly a factor of 2 lower than the Pt surface area calculated from the 30 Å average particle size observed by TEM. The electrochemical measurements combined with microanalysis of membrane electrode assemblies, allow a greater understanding and optimization of process variables.

INTRODUCTION

Electricity is the most convenient and widely-used form of energy. However, electrical energy cannot be stored cheaply in large quantities, and in order to provide practical and flexible electrical energy sources, conversion of energy must be carried out to and from another form. Conservation of fuel in energy conversion processes is essential for economical feasibility and has been the major consideration in development of energy conversion technology. Environmental consciousness has also become more of an issue in recent years, and is driving new technologies. Both considerations motivate the development of fuel cells for energy conversion processes.

Proton exchange membrane (PEM) fuel cells generally use platinum catalyzed proton exchange membranes for hydrogen conversion into electrical energy and have shown high power densities. The hydrogen / oxygen electrode reactions occur in the catalyst layer of the membrane-electrode assembly (MEA) which is the efficiency and kinetically limiting region of the fuel cell. For useful fuel cell production, MEAs must be optimized for high proton transfer, hydrogen oxidation and oxygen reduction kinetics, and low platinum catalyst loadings. The production of MEAs for PEM fuel cells can be accomplished by many different methods,¹⁻⁶ with each production technique presenting many process variables which need optimizing. Because testing of these MEAs is a long and complex process with many fuel cell operating variables to be examined, optimization of production variables can be a tedious procedure.

Characterization of MEAs in various phases of the production process can help reduce the testing time required for MEA evaluation. The use of electrochemical techniques such as hydrogen adsorption and desorption, and carbon monoxide oxidation complement microscopic analysis techniques such as scanning electron microscopy (SEM) and energy

dispersive spectroscopy (EDS). These techniques can be used for characterization of the MEA to determine the characteristics of the catalyst layer and membrane interface. Transmission electron microscopy (TEM) is a valuable technique to examine the individual Pt particle size and Pt distribution on the supporting graphite. Examination of the catalyst layers (anode and cathode) in all dimensions helps illustrate whether the production process has been successful in producing a catalyst with desired uniform properties. In this work, electrochemical and electron microscopies to characterize MEAs are used to assist device optimization of MEA's, thus reducing testing time and furthering our understanding of the MEA variables.

EXPERIMENTAL

MEA's were produced using techniques similar to those used by Wilson & Gottesfeld.^{1,2} Pt catalysts presented in this study were obtained from ETEK corporation and were 20%, 40%, 60% weight Pt supported on graphite and platinum black. The MEA was placed in a single cell test apparatus as shown in Fig. 1, similar to the cell used for fuel cell testing of the MEA. The polycarbonate blocks have facilities for gas distribution, voltage and current takeoff and cell temperature regulation. The graphite flowfield inserts have milled grooves on one side to distribute properly the reactant gases to the MEA. The graphite paper, used for current collection and gas distribution, was impregnated with a Teflon[®] suspension. The active area of the MEA examined was 47.5 cm², which is large enough to simulate flowfield patterns of full size MEAs. The hydraulic press was operated at 60 psi to compress the gasket assembly for leak proof operation. This cell can be operated as an operating fuel cell using hydrogen and oxygen (or air), or as in the case of this study, to evaluate the activity of the platinum catalyst. All measurements described here were made at room temperature.

The active surface area of the catalyst was measured by evaluating the hydrogen adsorption/desorption and relative carbon monoxide oxidation. For these measurements, the fuel cell cathode was exposed to nitrogen while the anode was exposed to hydrogen. The gases fed to the cell were humidified. The cathode was made the working electrode and the anode was used as both the reference and counter electrode. The double layer capacitance from the voltammogram was subtracted followed by integration of the hydrogen adsorption/desorption region from 0.0 to 0.45 V. The values for hydrogen adsorption and hydrogen desorption were integrated separately, and have generally agreed within 10%. To alleviate problems caused by excess hydrogen evolution, the reported values were calculated by integrating the hydrogen desorption region. Polarization effects at the counter electrode were neglected since the hydrogen oxidation reaction at the fuel cell anode is nearly reversible. For CO oxidation experiments, 5% CO (or 5% CO₂) was added to the cathode gas feed. After exposure to carbon monoxide or carbon dioxide, the anode was purged with nitrogen to remove all of the gas phase CO or CO₂ before the electrochemical measurements were made. The electrochemical measurements were made using a Pine AFCBP1 Bipotentiostat interfaced with a Macintosh via Labview for data acquisition.

The TEM measurements were made using a Philips CM30 Analytical TEM with a 300 kV electron beam. The catalyst samples were dispersed in petroleum ether, and applied to a commercially available TEM substrate. The SEM measurements were made on a CAMSCAN SEM interfaced with a KEVEX EDS system. The MEA cross-sections were prepared by drying the membrane, mounting in epoxy and polishing to 0.1 μm alumina. The samples were then coated with either graphite or gold to form a conductive surface coating.

RESULTS & DISCUSSION

TEM Catalyst Characterization

For reduction of catalyst loading, a high surface area Pt catalyst is required. To achieve the highest Pt utilization possible, small Pt particles are required. To achieve this, Pt has been supported on high surface area graphite. The Pt particles should be uniform without large agglomerations of Pt, with the smallest possible particle size. Fig. 2 is a transmission electron micrograph of 20% Pt supported on graphite commercially available from ETEK. Mostly uniform Pt particle size was observed for the ETEK catalyst, with an average particle size of about 30 Å. Particle size generally ranges from about 20 Å to 40 Å, although some larger Pt particles were observed. Particle A is about 20 Å in diameter, while particle B, a larger particle, is 75 Å in the longer direction. The edges of supporting graphite are labeled and there are large areas where no Pt particles are apparent. Since the electrons in TEM go through the entire sample, the particles observed are for a volume of catalytic material. Individual Pt and graphite particles are best examined at the interface where graphite particles are next to regions where no material is present, such as Particle A, to keep volume effects to a minimum. Other areas of graphite show larger concentration of Pt particles, which could be due to a large volume of catalyst material imaged. For an average particle size of 30 Å, the platinum catalyst has a surface area of 93 m²/g. These TEM measurements tend to agree with measurements made by x-ray diffraction taken by ETEK which measured a surface area of 88 m²/g for 30% weight Pt.⁷ The measured Pt surface area by these methods assumes that the entire Pt particle is active, even though the Pt particles are supported on carbon.

Electrochemical MEA evaluation:

Electrochemical voltammetry of poly-crystalline and crystalline platinum surfaces have been examined in detail,⁸⁻¹⁶ however little has been published on the electrochemical voltammetry of Pt supported catalysts.^{17,18} Hydrogen adsorption and desorption (HAD) occur essentially reversibly on the platinum surface in the region from 0.0 to 0.4 V_{RHE}. The potential region from 0.4 to 0.5 V is termed the double layer region, as there are negligible Faradaic currents present. During cathodic sweeps, at > 1.0 V_{RHE} oxygen adsorption occurs which is reduced during the anodic sweep at 0.65V.

The surface area of the platinum can be evaluated by integration of the hydrogen adsorption or desorption according to the following:



Each adsorbed hydrogen is considered to occupy one Pt site. We used a charge transfer of 243 μC/cm² for a monolayer of hydrogen adsorption which correlates to the Pt(111) surface.²⁰ While the supported platinum surfaces used in this study are polycrystalline, the Pt(111) surface is the close packed Pt surface and provides an upper limit for the surface area calculation.

The cyclic voltammograms of MEAs with differing weight % Pt catalysts supported on graphite are shown in Fig. 3. The voltammograms have been normalized for the loading of Pt used in the MEAs so that a direct comparison of the charge transfer per unit weight of platinum can be made easily. Fig. 3a is the voltammogram for 20% Pt supported on graphite and has a surface area of 52.8 m²/g of catalyst. While this is a large active surface area, it is almost a factor of 2 less than the value calculated by TEM (or XRD) measurements. The simplest explanation is that about half of the Pt surface area is used to bond the Pt particles to the graphite support. Figures 3b, 3c and 3d are for 40% Pt, 60% Pt and Pt black and have active surface areas of 23.7, 17.9 and 28.7 m²/g respectively. These results are summarized in Table 1.

MEAs utilized by our group typically require several hours of operation before stable high performance is achieved. This is termed the 'burn in' period. This effect has also been observed when making the hydrogen adsorption/desorption measurements. Fig. 4 shows the cyclic voltammogram as a function of time for a newly prepared MEA. As the MEA is potentially cycled, the Pt hydrogen adsorption/desorption and oxygen oxidation/reduction features slowly start to appear. The amount of hydrogen adsorption/desorption gradually increases over time, perhaps due to removal of organics in the catalytic ink due to oxidation. The first curve (Fig. 4a) is after the MEA has been cycled for 1 hour. The Pt features are apparent, but are not as well defined as generally observed. After 2.5 hours the Pt features are more pronounced, and the amount of HAD is greater. Initially the MEA show essentially no hydrogen adsorption or hydrogen desorption. The third curve, shows a greater amount of hydrogen adsorption which occurred after the MEA was reversed, so that the opposite side was used as the working electrode. This side also showed a gradual increase in the amount of hydrogen adsorption/desorption, although it reached a higher limiting value more quickly. This possibly indicates that the lower amounts of observed HAD could be due to organic species which are gradually removed from both the catalyst layer and the membrane.

Fig. 5 shows the voltammogram of an MEA anode and cathode after testing in a single cell test apparatus. The MEA was used for electrolysis, i.e. in reverse bias to simulate regenerative fuel cell operation. The cathode (a) shows markedly higher hydrogen adsorption than the anode (b). The anode catalyst surface area has been reduced by a factor of 4, from 26 m²/g observed for the cathode to 7.0 m²/g for the anode. This decrease in active catalyst area was also evident from the decrease in performance observed after reverse biasing of the MEA. Subsequent SEM analysis (see Fig. 7d) shows that the thickness of the anode catalyst layer has been greatly reduced by the reverse biasing of the electrode. This result indicates that this type of MEA may not be suitable for electrolysis.

The voltammogram of a MEA with 60% Pt (same as Fig. 3c) after (a) exposure to CO and (b) exposure to CO₂ for five minutes is shown in Fig. 6. The hydrogen adsorption in Fig. 6a is essentially completely blocked as evidenced by the initial curve from 0.0 to 0.4 V. On the following cathodic sweep to 1.5 V, carbon monoxide is oxidized from the Pt surface beginning at about 0.8 V. After oxidation of the carbon monoxide, hydrogen adsorption occurs on the following anodic sweep. The amount of adsorbed carbon monoxide can be calculated via the following reaction:



The charge required to oxidize a monolayer of carbon monoxide corresponds to $Q_{\text{CO}} = 484 \mu\text{C}/\text{cm}^2$ (for Pt(111)).²⁰ Using Q_{CO} to calculate the catalyst surface area yields 12.8 m²/g for Fig. 6a. This is only about 70% of the surface area observed by integration of the HAD. Results of the surface area calculations for different catalysts appear in Table 1. From the values in Table 1, we see that the ratio of the surface area calculated by CO oxidation to HAD ranges from 0.72 to 0.93, with three values at $0.90 \pm .03$. Evaluation of the saturation coverage of CO on crystalline platinum surfaces has been the subject of recent discussion,¹⁹⁻²⁵ and the saturation coverage of CO on Pt has been measured to be 0.70 in HClO₄, and 0.90 in H₂SO₄.^{20,24} The values in Table 1 agree with the saturation coverage of 0.90 observed in H₂SO₄. The CO coverage on the 60% Pt catalyst probably did not reach saturation, and thus a lower value was obtained.

Real world applications of PEM fuel cells often require operation on fuels other than pure hydrogen. Fuels considered are typically reformed hydrocarbons, which result in a feed gas of H₂ and CO₂. Fig. 6b shows that exposure to carbon dioxide forms a species similar to carbon monoxide. However after 5 minutes exposure to carbon dioxide only a small portion of the platinum surface is covered (as evidenced by the amount of

hydrogen adsorption present). If the exposure time to CO₂ is increased, a greater portion of the Pt surface is poisoned. However the coverage does not seem to reach that obtained by direct contact with CO. To evaluate the coverage of the reduced CO₂ adsorbed species, the identity of the reduced CO₂ species must be determined. However, there is conflicting evidence as to whether the reduced CO₂ species is COH²⁶⁻²⁸ or CO.²⁹⁻³⁰ Oxidation of CO is a two electron process, while oxidation of a COH species is a 3 electron process:



If oxidation of the reduced CO₂ species is assumed to be a two electron process, the highest coverage obtained by exposure of the MEA to CO₂ was about 0.70. For a three electron process the coverage obtained would be 0.47. The reduction of CO₂ apparently does not fully poison the Pt surface whether the species is oxidized through a 2 or 3 electron process. This also agrees with the fact that hydrogen adsorption / desorption is still observed at the highest coverages of CO₂.

SEM and EDS MEA Characterization

SEM and EDS were used to characterize the cross-sectional properties of the MEA and catalyst layer. This characterization includes not only the physical uniformity of the MEA, but the elemental distributions throughout the catalyst layers of the MEA. A scanning electron micrograph (SEM) backscattered image of a cross-section of a membrane-electrode assembly is shown in Figure 7a. The image shows the anode (a) and cathode (c) catalyst layers, and the proton exchange membrane (b). In a backscattered SEM image, materials with higher atomic numbers appear brighter. The membrane has sulfur present, thus is brighter than the background of the sample mounting epoxy, but not as bright as the platinum present in the catalyst layer. The catalyst layers are made of diffuse catalyst particles, much smaller than the resolution of the image. A different experimental catalyst ink formulation is shown in Fig. 7b. The catalyst layers appear uniform except for a bright particle present in the top catalyst layer, which indicates a higher concentration of platinum. This was confirmed by EDS (energy dispersive spectroscopy) point analysis. This particle appears to be about 30 μm in diameter, and it has poor adhesion between the particle and the rest of the catalytic layer. The poor adhesion is more apparent at higher magnifications. EDS confirmed that the other elemental constituents of the catalytic layer are not as concentrated inside the particle as the rest of the catalytic layer, so the particle was probably present in the catalytic ink before it was applied to the membrane.

A different region of the same MEA as in Fig. 7b is shown in Fig. 7c. Particles that appear dark in the backscattered image and have only about 60% of the platinum as the rest of the catalyst layer. These particles have good adhesion to the rest of the catalytic layer, and the other elemental constituents of the catalyst layer appear uniform inside the particle. These particles appear to have formed after the catalytic ink was formed, or were at least partially solubilized during the mixing process of the catalytic ink.

Fig. 7d is a micrograph of a MEA after testing in a single cell apparatus, and is the same MEA used for the HAD of Fig. 5. This particular MEA was used to examine not only normal fuel cell operation for power production, but was operated in a reverse bias mode, to simulate regenerative fuel cell operation. As can be seen in the micrograph, the anode catalyst layer has mostly been removed due to the reverse bias operation, and the remaining anode catalyst does not have good contact to the membrane.

CONCLUSIONS

MEA characterization has reduced the time required for fuel cell testing by improving our understanding of the MEA production variables. Use of electrochemical evaluation and electron microscopies have been used to characterize MEAs and the catalyst layers of the MEAs.

Electrochemical surface area measurements have observed large active Pt surface areas, on the order of 50 m²/g for 20% weight Pt supported on graphite. Comparison of the hydrogen adsorption/desorption with CO oxidation indicates that on the supported catalysts, the saturation coverage of CO/Pt is about 0.90, the same as observed in H₂SO₄. The catalyst surface area measurements are nearly a factor of 2 lower than the Pt surface area calculated from the 30 Å average particle size observed by TEM.

MEA cross-sectional characterization by SEM and EDS has helped to identify process variables which lead to uniform catalytic layers. SEM and EDS characterization has also identified large catalyst particles in experimental formulations, and identified whether the particles appear to have been solubilized in the ink before application to the membrane. SEM analysis has also identified degradation of MEA catalyst layers under certain operating conditions.

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Table 1: Measured Surface Areas by CO Oxidation and HAD.

	20% Pt [m ² / g Pt]	40% Pt [m ² / g Pt]	60 % Pt [m ² / g Pt]	Pt Black [m ² / g Pt]
HAD	52.8	23.7	17.9	28.7
CO Oxidation	48.9	21.1	12.8	25.9

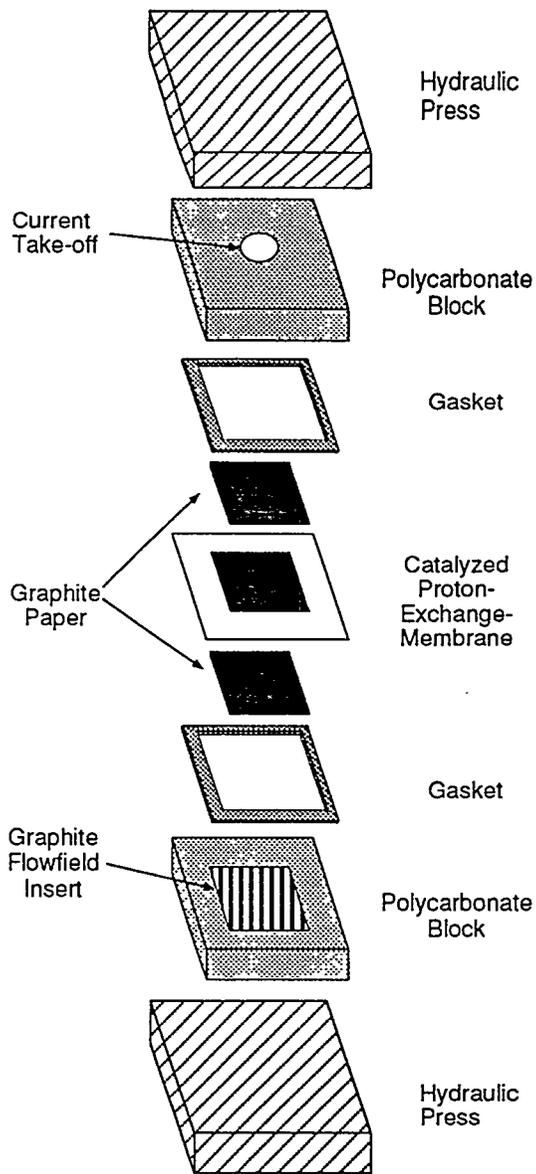


Fig 1. View of single cell testing hardware.

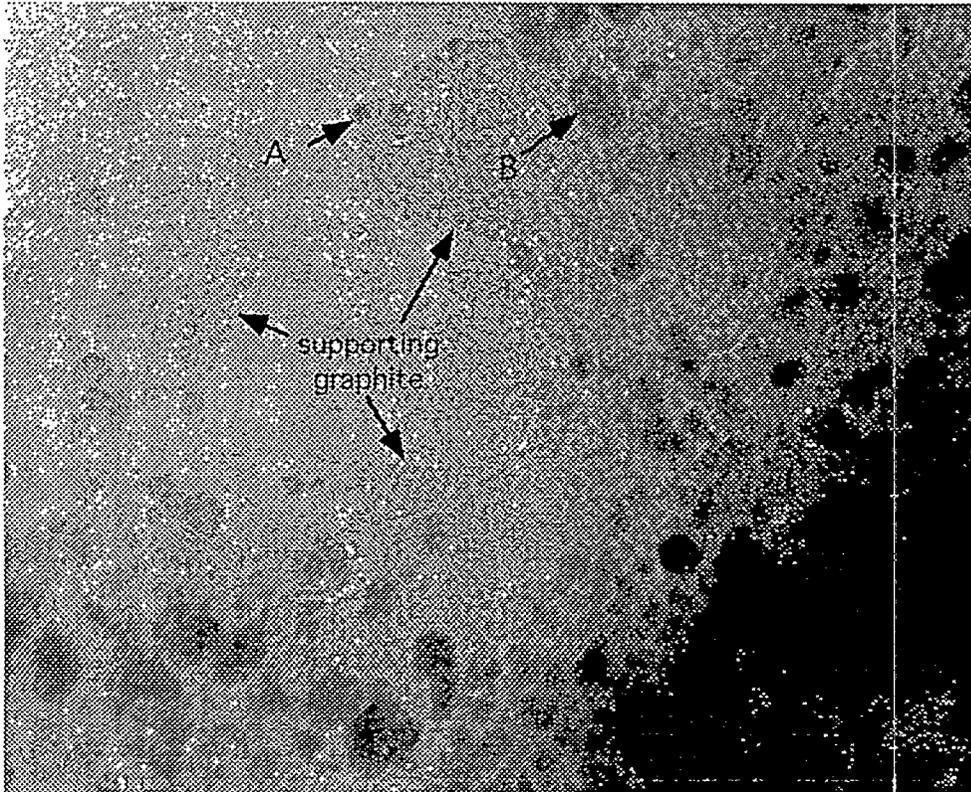


Fig. 2: Transmission Electron Micrograph (TEM) of Pt catalyst. Pt particle A is 20 Å. Pt particle B is 75 Å.

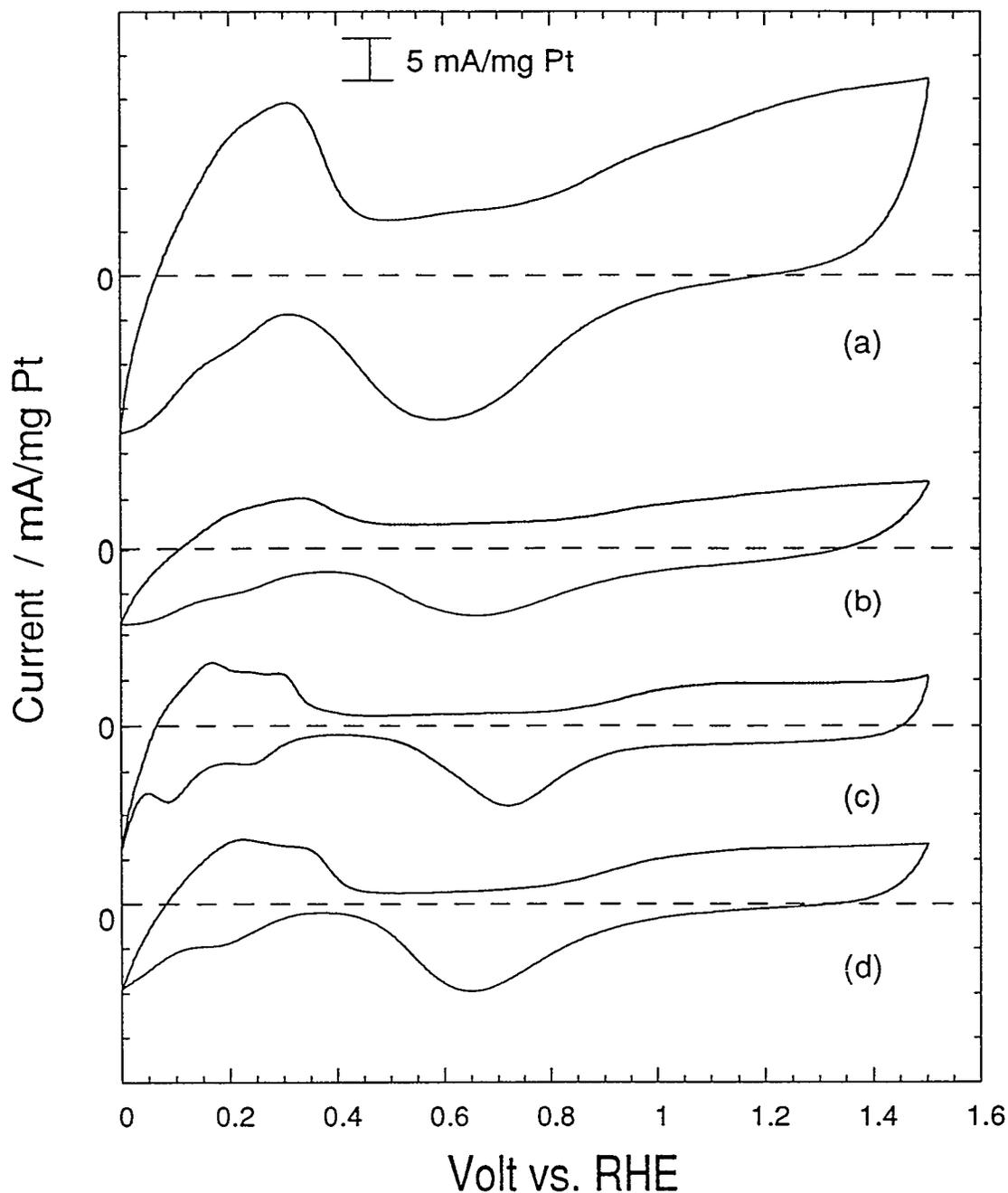


Fig 3: Cyclic voltammogram of the fuel cell cathode showing hydrogen adsorption and desorption and platinum oxidation and reduction. Catalyst used was (a) 20% wt Pt / graphite, (b) 40% wt Pt, (c) 60% wt Pt and (d) Pt black. Scan rate : 20 mV/sec. The reference electrode used was the counter electrode (anode).

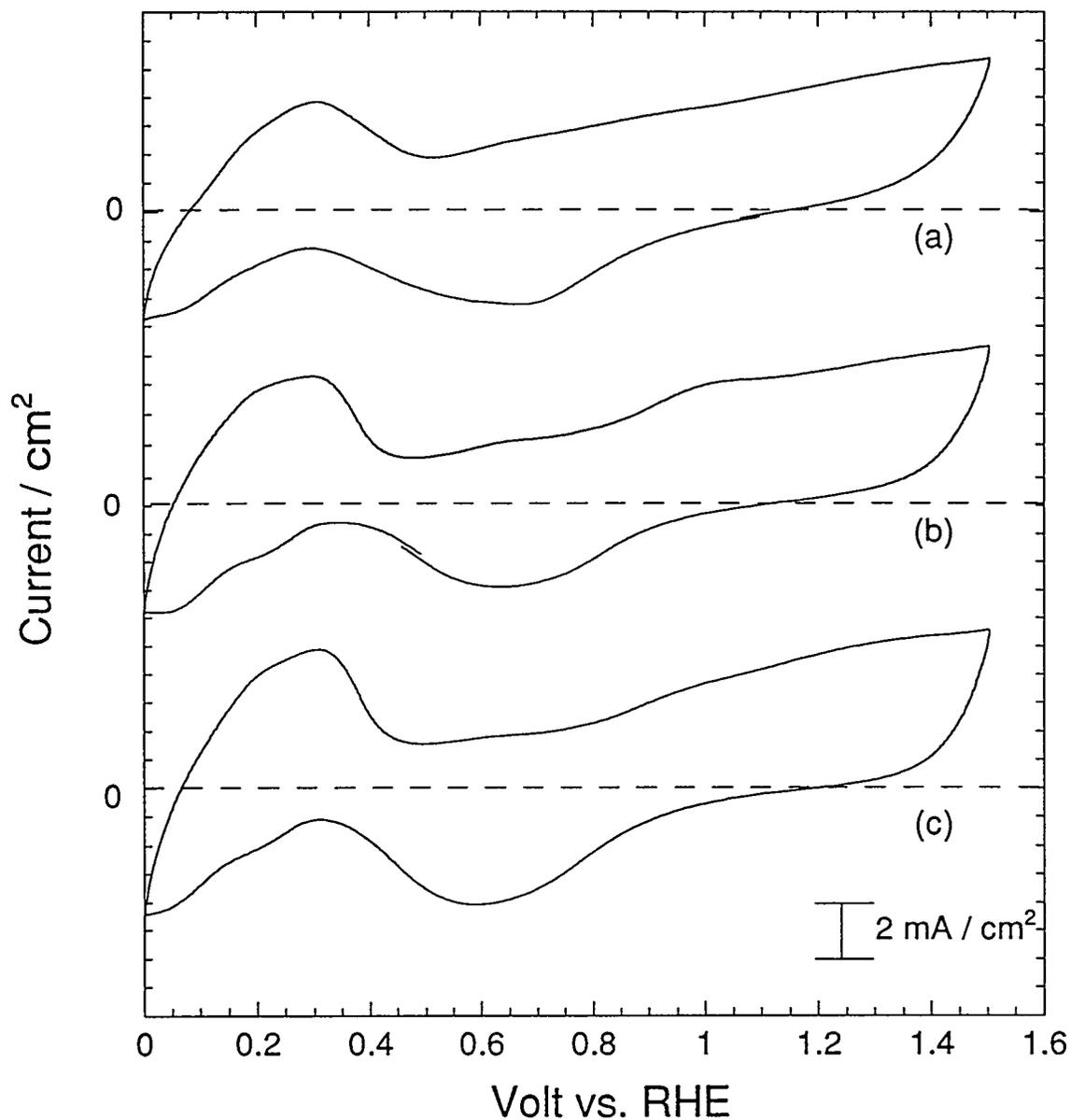


Fig 4: Cyclic voltammograms showing increasing hydrogen adsorption / desorption as MEA is repeatedly cycled. After cycling for (a) 1 hour, (b) 2.5 hours and (c) switching MEA sides and cycling for 1 hour. Catalyst used was 20 wt % Pt / graphite. Scan rate : 20 mV/sec. The reference electrode used was the counter electrode (anode).

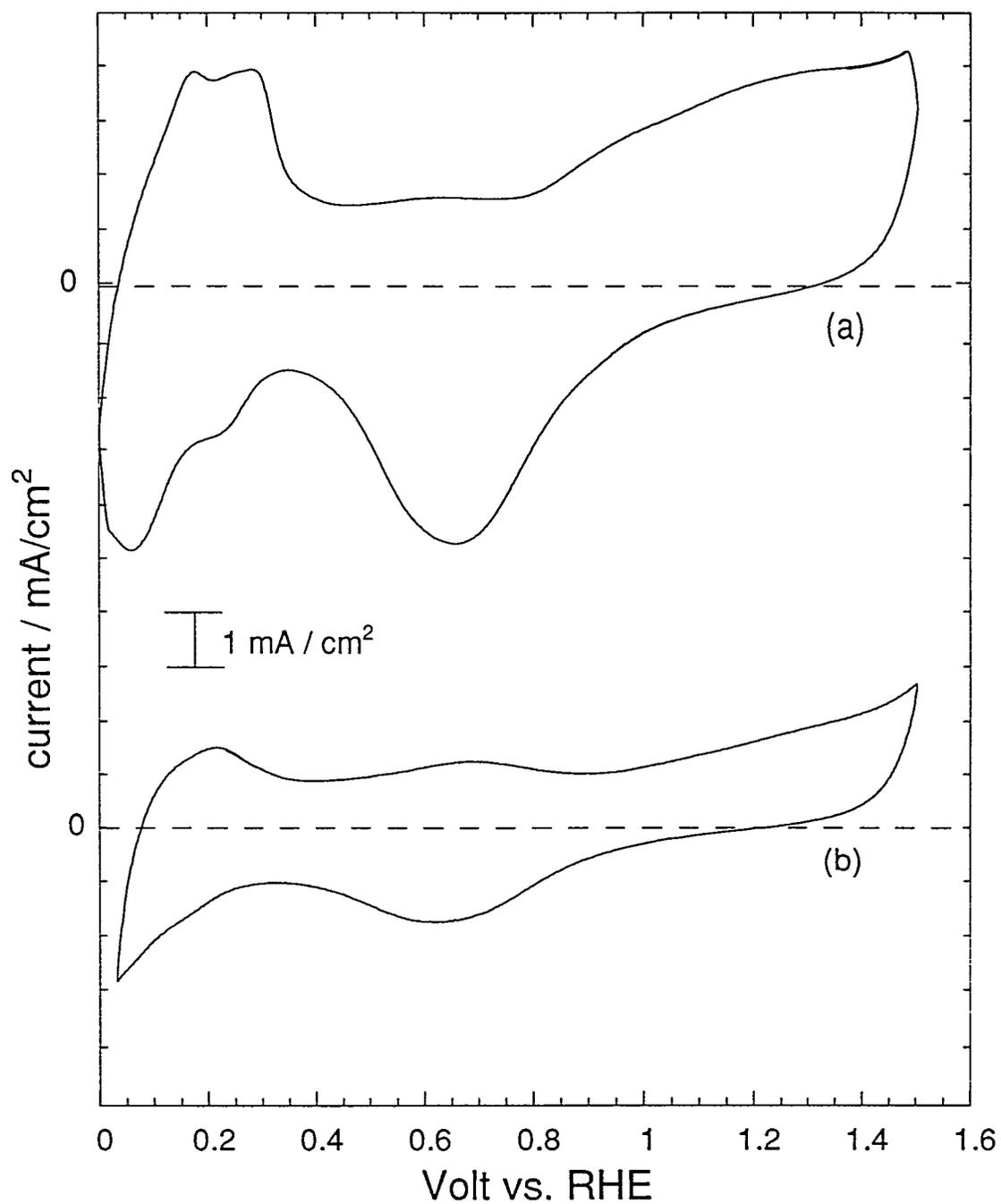


Fig. 5: Cyclic voltammogram of MEA (a) before reverse polarization and (b) after reverse polarization. Sweep rate : 20 mV/sec.

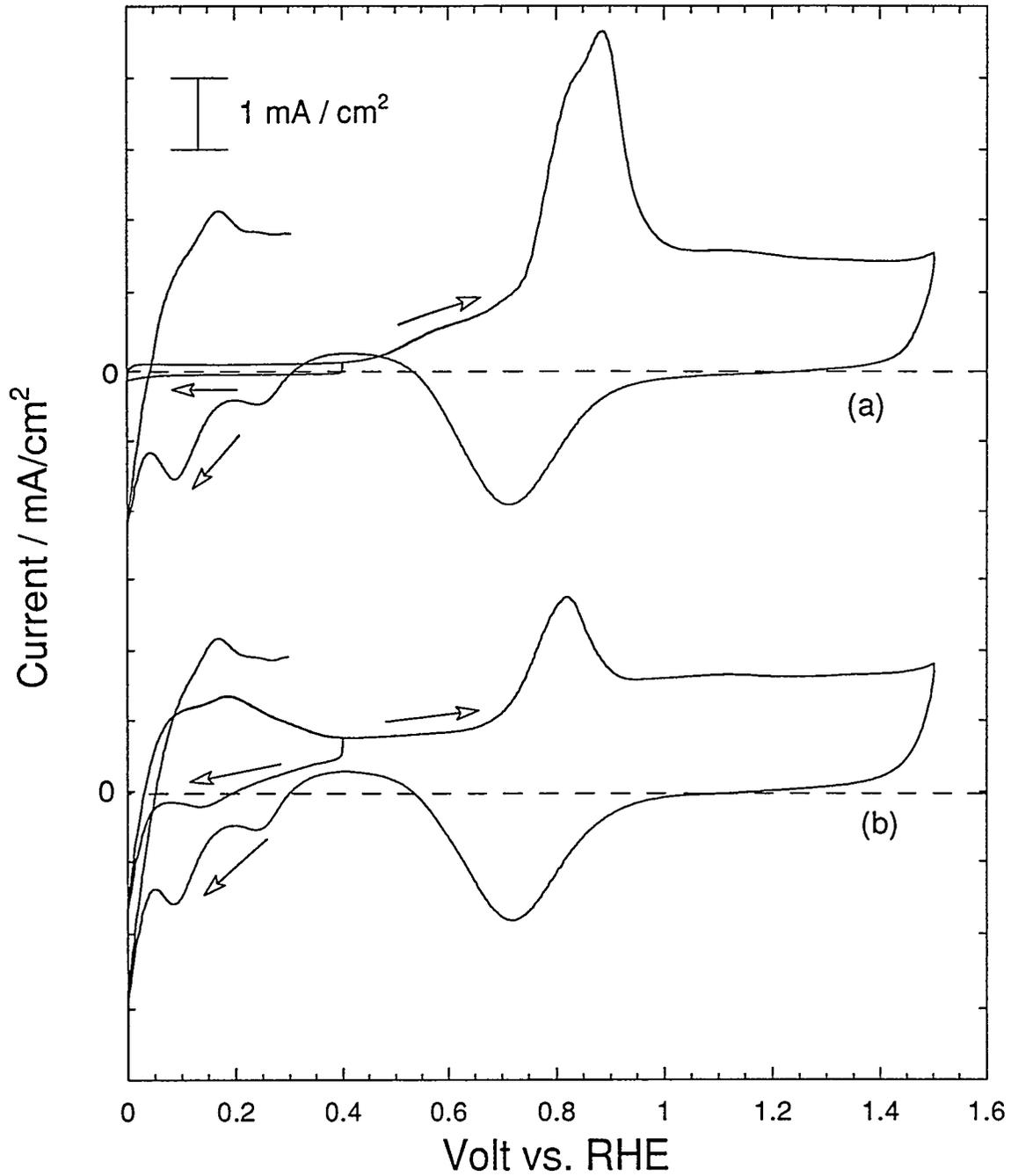


Fig. 6: Cyclic voltammogram of MEA after (a) 5 %CO in gas feed of cathode
 (b) 5% 'CO₂' in gas feed of cathode. CO/CO₂ dosing time : 5 min.
 Sweep rate : 20 mV/sec

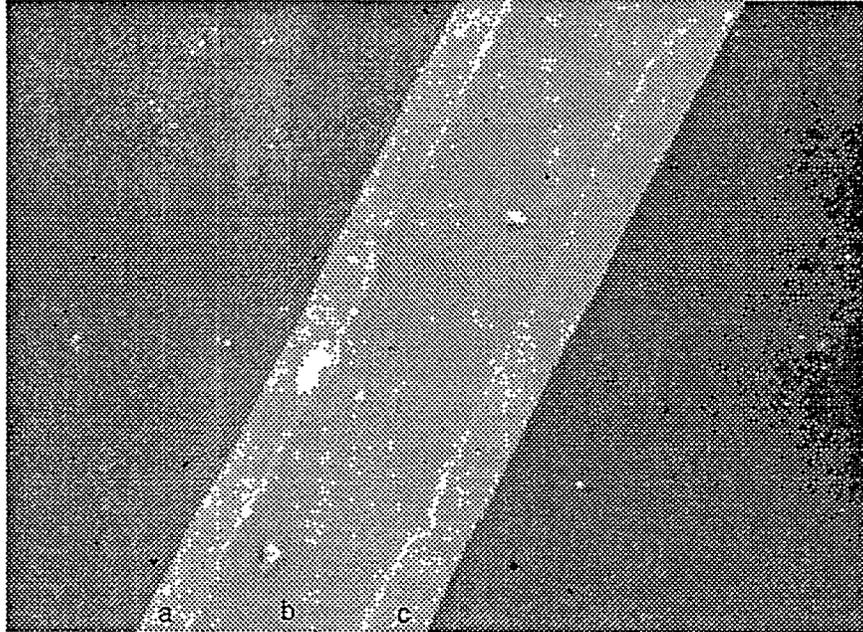


Fig. 7a: Scanning electron micrograph of catalyzed membrane. Image was taken in backscattered mode. Catalytic layer appears to be uniformly distributed, without large particles.

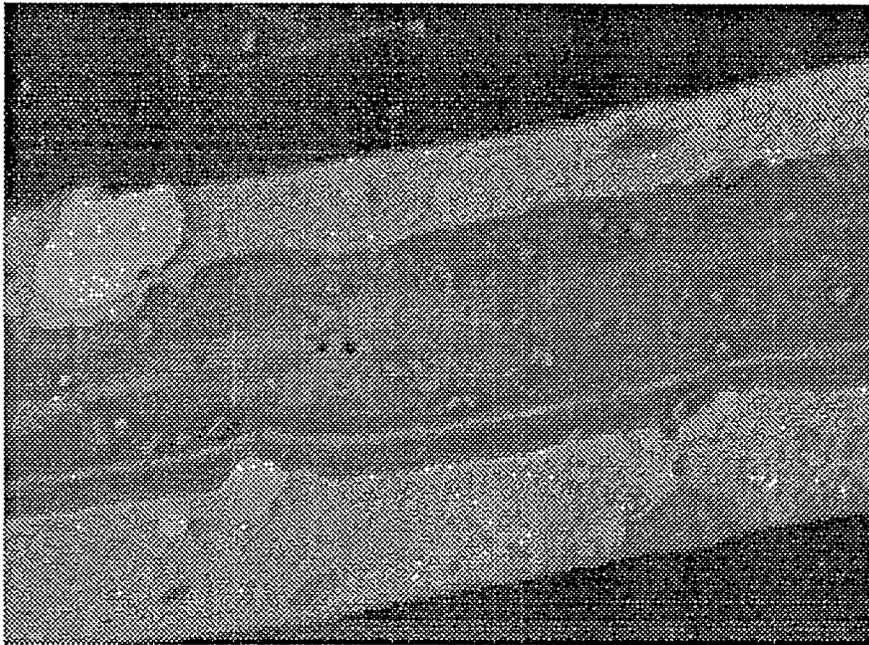


Fig. 7b: Backscattered scanning electron micrograph of catalyzed membrane. Catalytic layer has bright particle of high Pt concentration.

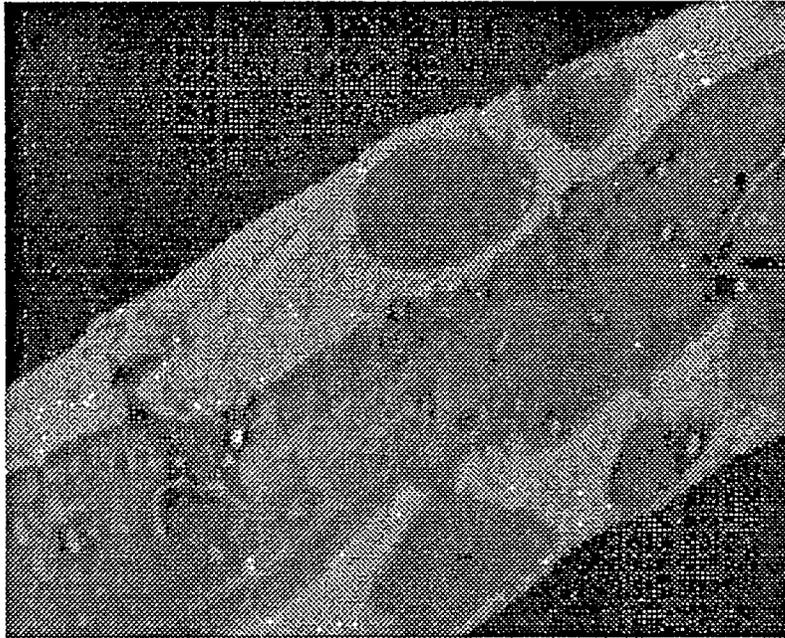
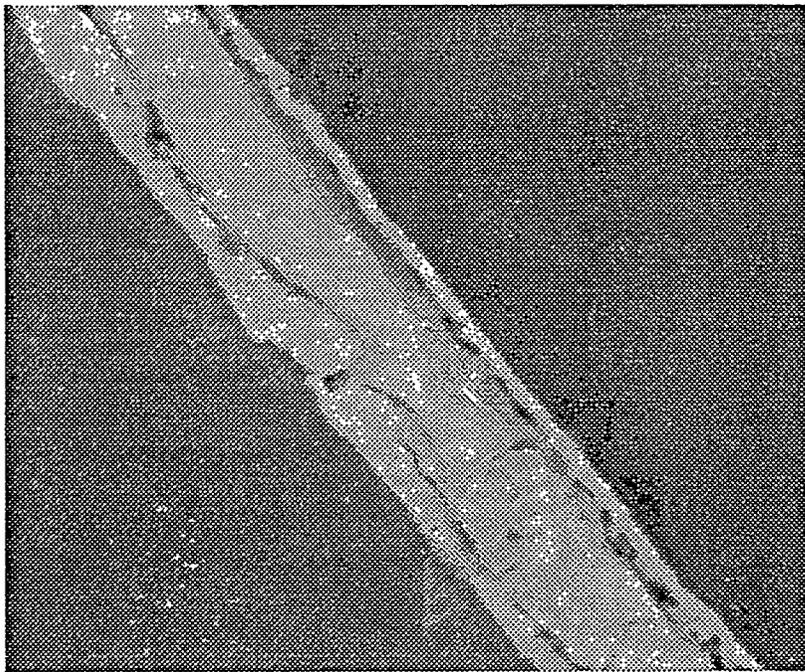


Fig. 7c: Backscattered scanning electron micrograph of catalyzed membrane. Catalytic layer has dark particles of low Pt concentration.



Cathode

Anode

Fig. 7d: Backscattered scanning electron micrograph of catalyzed membrane. MEA was tested in electrolysis mode for regenerative operation.