# Comparing Different Cross-Section Cutting Methods for SEM Analysis of Membrane-Electrodes Assemblies


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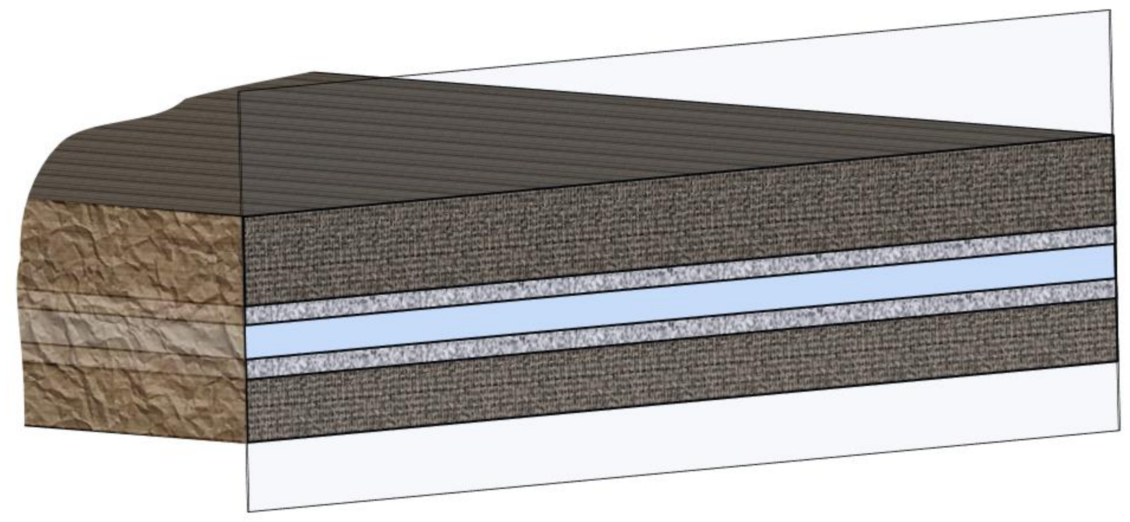
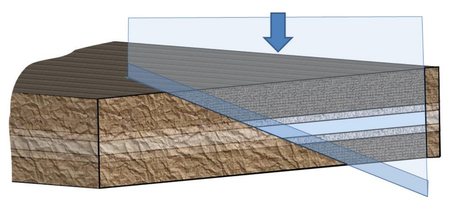
The correct cross-sectional visualization of membrane-electrode assemblies (MEAs) with scanning electron microscopy (SEM) requires preparation of planes where the different phases can be distinguished with minimal modification of their morphology and thickness. Such preparation is complicated by the different mechanical properties of the layers of an MEA, including a plastic membrane, carbon cloths, and porous carbon layers like the catalyst layers and the microporous layers. This communication compares four techniques for the cutting of MEAs for cross sectional visualization with SEM: the sharp-edge cutting, $CO_2$ laser, embedded-mechanical polishing, and ion-milling. From the results of the cross section visualization of the MEAs, the methods are compared attending to reproducibility, phases thickness preservation, and phase morphology preservation.

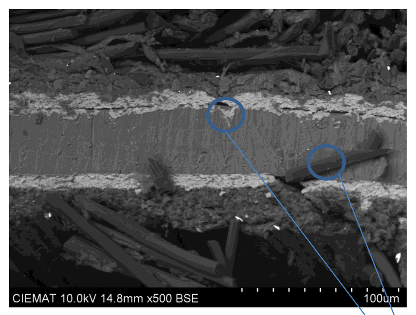



**Experimental:** MEAs prepared with Nafion NR212 membrane (Ion Power Inc.), and two types of commercial gas diffusion electrodes, BASF (ELAT GDE LT250EWALTSI, BASF, 0.25 mgPt·cm$^{-2}$), and Fuel Cell ETC (0.3 mg/cm² 40% Platinum on Vulcan - Cloth GDE). Also electrodes with catalyst layers deposited by the electrospray technique were observed in cross-section. For SEM a Hitachi FE-SEM SU-6600 microscope was used.

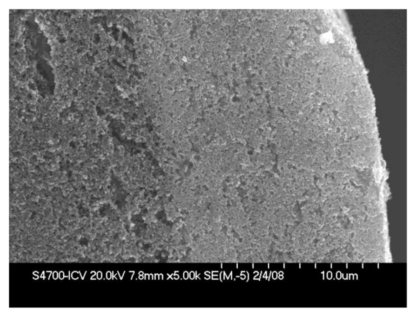
## Sharp-edge cutting



standard laboratory snap-off blade

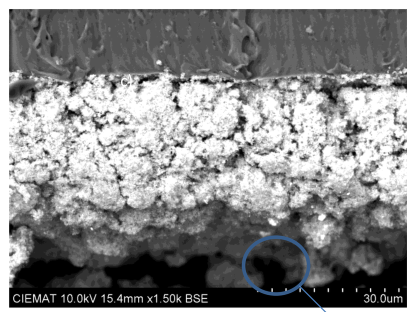


*MEA with two commercial electrodes (BASF, 0.25mgPt·cm$^{-2}$) and Nafion 212NR membrane*

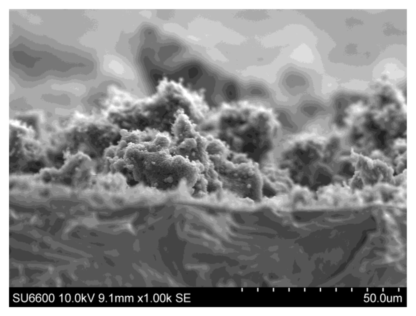


*Catalyst layer of a commercial electrode (90º rotated).*

phase mixing



*Catalyst layer in MEA, prepared by electrospray on Nafion membrane (in the upper part) with Pt/C 20wt% and 0.25mgPt·cm$^{-2}$.*



*Detail of the agglomerates and dendritic structures of an electrosprayed catalyst coated membrane.*

delamination

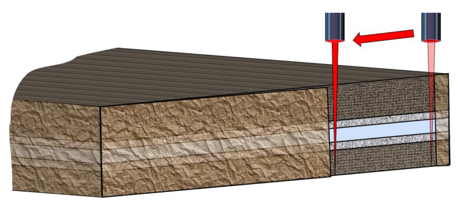
**Advantages:**

- Fast and low cost method
- May preserve the porous morphology of the layers rather unaltered

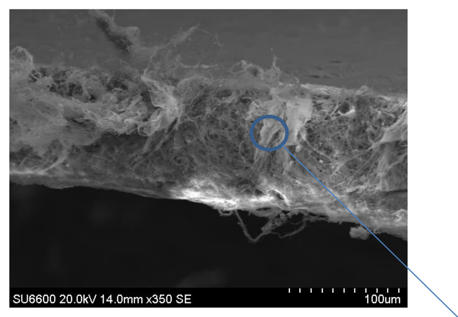
**Disadvantages:**

- Obtaining a good cross-section in a wide area is stochastic and with low probability.
- Frequent intermixing and delaminating of layers

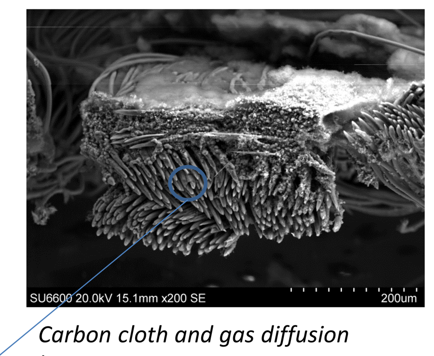
## $CO_2$ laser



laser cutting machine, Gravograph LS100, 35 W, 50-500DPI

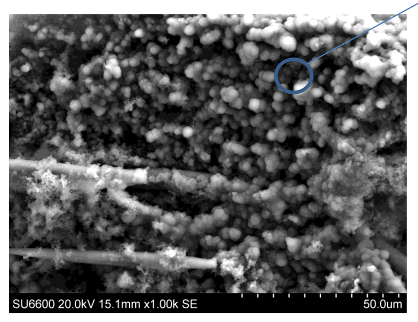


*Nafion 212NR membrane.*

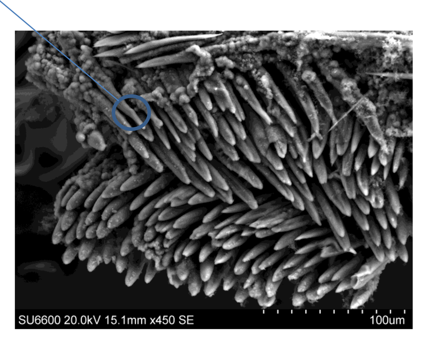


*Carbon cloth and gas diffusion layer.*

burned phases



*BASF catalyst layer.*



*Detail of carbon filaments of a carbon cloth.*

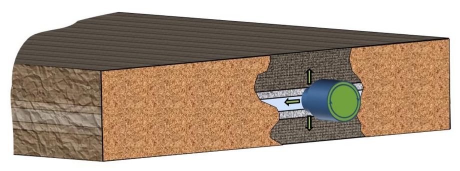
**Advantages:**

- Fast method, with automatic selection of the cutting line
- Commercially available machines at relatively low price

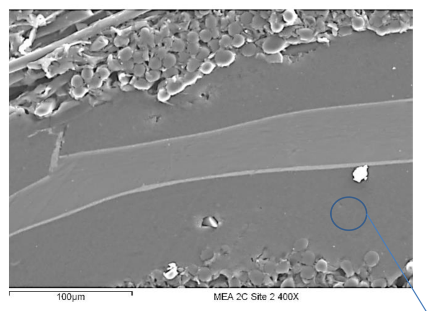
**Disadvantages:**

- Alters severely the microstructure of carbon and Nafion layers as a consequence of the burning of carbon based materials.

## Embedded-mechanical polishing

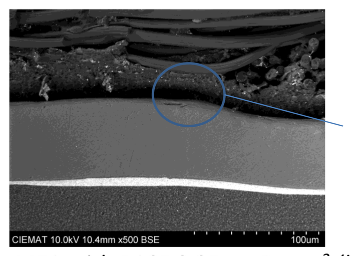


Embedded in EPOMET® G resin, compression under 80 bar at 150 °C. Grinding with SiC papers (P320, P600, P1200) and water, and polishing with diamond paste (6 microns).



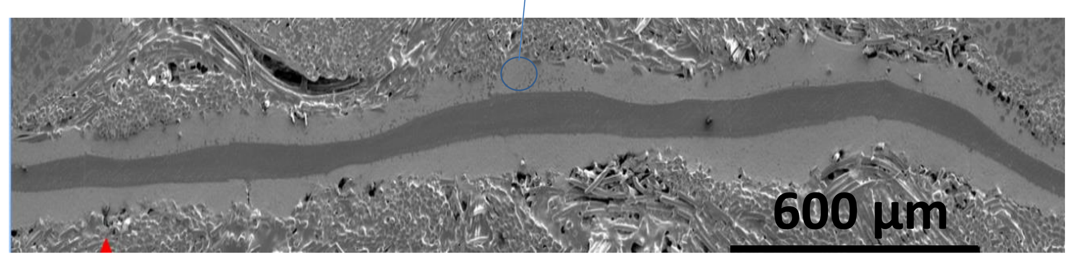
*MEA with BASF 0.25 mgPt·cm$^{-2}$ (lower electrode),Fuel Cell Etc. 0.30mgPt·cm$^{-2}$ (upper), and Nafion 212NR.*

porosity smoothed



removed catalyst layer

*MEA with BASF 0.25 mgPt·cm$^{-2}$ (lower electrode), and electrosprayed 0.25mgPt·cm$^{-2}$ (upper, removed), and Nafion 212NR.*




*Wide area cross section imaging of an MEA with two BASF 0.25 mgPt·cm$^{-2}$ electrodes and Nafion 212NR.*

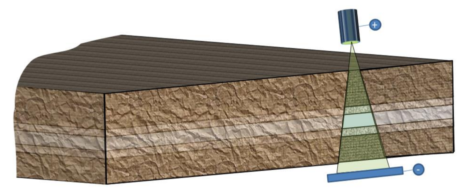
**Advantages:**

- Well established metallographic method
- Reproducible and accurate thickness of the layers

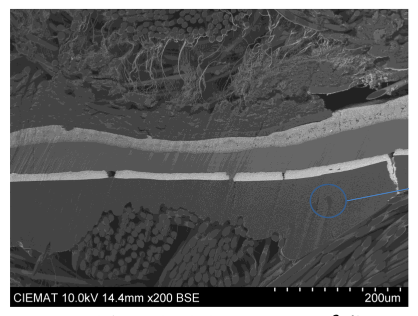
**Disadvantages:**

- Porous layers may appear smoothed or removed
- Time consuming preparation

## Ion-milling

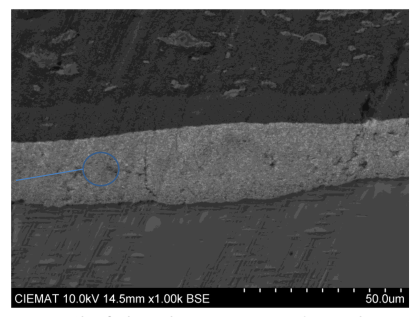


Hitachi High-Tech IM4000 system, using Argon beam with accelerating voltage of 5 kV during 6 h, using 2.3 reciprocation·min$^{-1}$ and a swing angle of ±40 °C. For catalyst coated membranes, the same conditions were used reducing the time to 3 h.

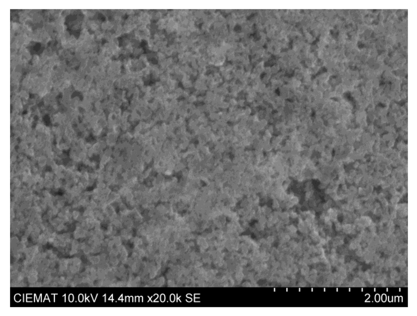


*MEA with BASF 0.25 mgPt·cm$^{-2}$ (lower electrode), and electrosprayed 0.25mgPt·cm$^{-2}$ (upper), and Nafion 212NR.*

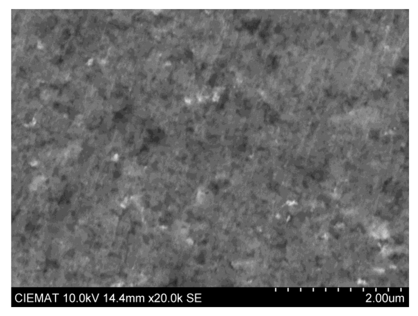
porosity smoothed



*Detail of the electrosprayed catalyst layer.*



*Electrosprayed catalyst layer 0.25mg Pt·cm$^{-2}$.*



*Commercial catalyst layer, BASF 0.25mgPt·cm$^{-2}$.*

**Advantages:**

- Accurate and reproducible thicknesses measurements

**Disadvantages:**

- Smoothing morphologies
- Expensive machinery
- Time consuming preparation
- Short observation length (mm)

| Cutting Method | Reproducibility | Thickness preservation | Morphology preservation | Preparation time | Comments |
|---|---|---|---|---|---|
| **Sharp edge** | low | medium-low | high | minutes | Low cost, preserves porosity |
| **$CO_2$ laser** | high | low | low | minutes | laser machine, alters morphologies |
| **Embedding-polishing** | high | high | low | 2-3 hours | laborious, aggressive with soft material |
| **Ion-milling** | high | high | medium-low | 4-5 hours | expensive equipment, short observation length (mm) |

## Conclusions

1. The four methods tested here for cross-sections preparation of MEAs show differences in thickness and morphology preservation of the samples.
2. For the analysis of porous morphologies, the sharp-edge cutting appears most adequate since it may leave rather unaltered porosities of the layers. However the method is stochastic and relies largely on the experience and skills.
3. For the analysis of the thickness of the layers , the embedding-polishing method and the ion-milling are more reliable and reproducible. However, both methods alter porous morphologies, yielding smoothing of porosities of the carbonaceous layers.
4. The ion-milling method is more conservative with the porosity than the embedding-polishing but it can only be applied to small, millimeter wide, areas of the MEA.
5. It is not discarded that the future optimization of all these methodologies may improve the shortcomings observed in this work.


Portable fuel cell

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236th ECS Meeting Atlanta, GA October 13-17, 2019

**Symposium**: I01A: Polymer Electrolyte Fuel Cells & Electrolyzers 19 (PEFC&E-19) - Diagnostics/Characterization Methods, MEA Design/Model

**Abstract:** #I01A-1393