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XPS characterization of a membrane electrode assembly from a proton exchange fuel cell

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XPS was used to investigate the Membrane Electrode Assembly of a proton exchange fuel cell to determine the distribution of platinum in the component. Also, the uniformity of the layers was investigated with large area imaging. The samples were prepared with ultra-low angle microtomy prior to the analysis.

Introduction

Proton exchange membrane fuel cells are devices for the production of electricity from the electrochemical reaction of hydrogen and oxygen, with potential applications as diverse as running cars to powering small electronic devices. The fuel cells are particularly attractive because they have high conversion efficiency, and they are environmentally very clean at the point of use.

Thermo Scientific™ Nexsa™ XPS System

One of the components of a proton exchange fuel cell is the Membrane Electrode Assembly (MEA). The MEA has layers of platinum in carbon black, which catalyzes the reaction of hydrogen and oxygen. When manufacturing or developing an MEA, the aim is to maximize the surface area of platinum that is electrically connected to the conducting support. Any loss of surface area decreases the efficiency of the device. Platinum loss can sometimes occur when high currents effectively corrode the carbon-black support liberating the active metal allowing it to migrate from the electrode surface to the adjacent polymer electrolyte. A typical material for electrolyte is Nafion®. The presence of platinum in the Nafion will hinder hydrogen ion mobility in the electrolyte.

This application note describes how XPS can be used to analyze an MEA and determine if platinum has migrated from the catalytically active layers into the adjacent Nafion electrolyte.

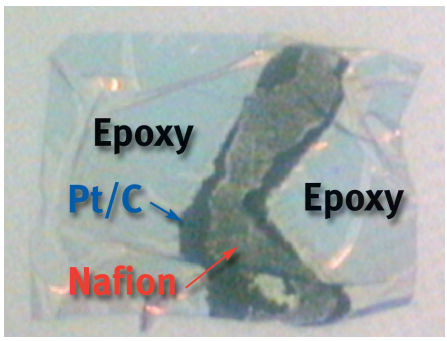

Figure 1: Thermo Scientific Nexsa optical image of ULAM-prepared MEA fuel cell sample

Experimental

The MEA consists of layers which are tens of microns thick. The platinum-containing anode and cathode layers are around the thicker Nafion electrolyte (Figure 1). The Nafion is electrically insulating, but allows transport of hydrogen ions. These layers are too thick for conventional XPS depth profiling so sectioning is required for XPS analysis. Ultra-low angle microtomy (ULAM) was used to cross-section the MEA at an angle of few degrees, allowing effective depth information to be obtained by imaging the cross section. The dimensions of the ULAM-sectioned layers are large enough compared to the X-ray probe area that it is possible to have many data points per layer. This enables the detection of subtle diffusion of platinum in these nanometer scale layers.

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Results

It is possible to use XPS to quantify elemental and chemical states across a wide area of the sample. As the concentration of platinum in the catalytically active layer is very low, it is necessary to use a high performance XPS tool to detect it. Typically the detection limit of XPS for elements is 0.5 atomic percent. Even at low concentration, a high quality, good signal-to-noise spectrum was acquired from the catalyst layer. In the middle of the Nafion layer, there was no detectable platinum.

It is possible to use XPS to quantify elemental and chemical states across a wide area of the sample. A map of epoxy versus Nafion versus platinum (Figure 2) was generated by acquiring full spectral datasets at each mapping pixel. Using advanced processing procedures implemented in the Avantage Data System, it is relatively simple to automatically correlate the data. The principal component's analysis identifies a number of components of the data set. It also allows the data to be reconstructed using a subset of the components. The benefit of the procedure is to remove the noise from the data set but retains all spectral information. This improves the signalto-noise ratio.

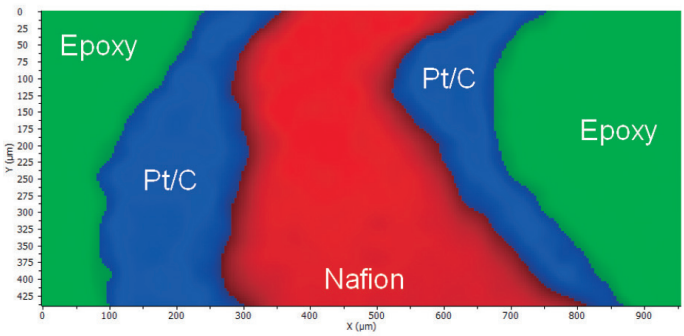

Figure 2: Principal components phase map of MEA sample

It is also possible to take the quantified mapping data and overlay it onto an optical image of the sample (Figure 3) or take a cross-section of the mapping data to generate an atomic concentration linescan (Figure 4). The linescan shows the atomic concentration of platinum along a line across the cathode, Nafion and anode layers, and it demonstrates that there is no large scale diffusion of the platinum into the Nafion.

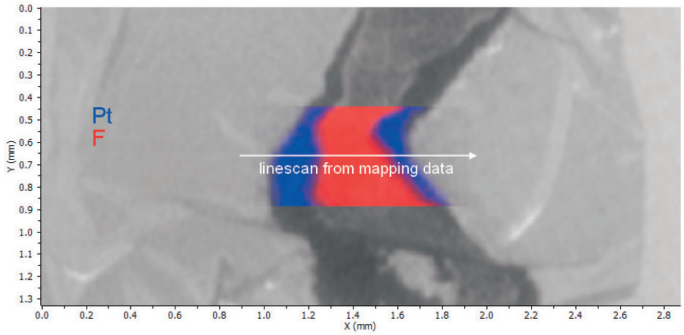

Figure 3: Large area XPS map of Pt/Nafion layers and interfaces in ULAM-MEA fuel cell sample overlayed with optical image

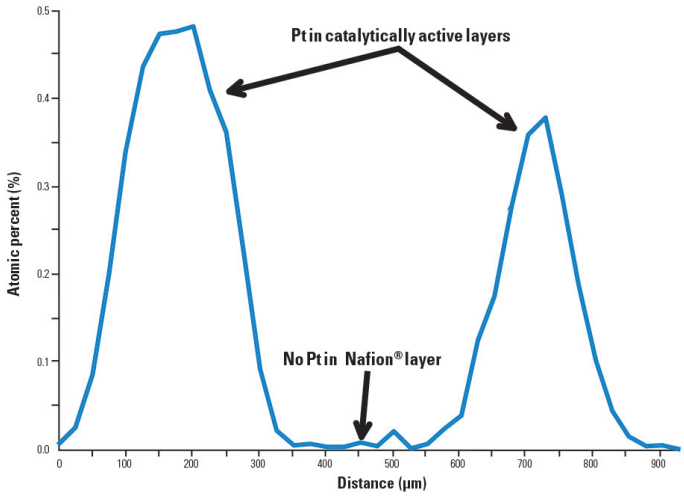

Figure 4: Quantified platinum atomic percent linescan taken from large area XPS map (shown on Figure 3)

Summary

XPS investigation of Membrane Electrode Assembly revealed that the catalytic effect at the anode and cathode on this sample is not detrimentally affected by loss of platinum. No platinum migration from the catalytically active layers into the adjacent Nafion electrolyte was found.

Acknowledgement

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