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K-Alpha: XPS Characterization of Thin Gold Layers on Steel Separators for Fuel Cell Applications

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Key Words

- K-Alpha
- Characterization
- Depth Profile
- Fuel Cells
- Surface Analysis
- Thin Film
- XPS

K-Alpha, the Thermo Scientific integrated XPS tool, was used for the analysis of the composition and thickness of thin gold films deposited on steel separators for fuel cell applications.

Introduction

A unit of a polymer electrolyte fuel cell is formed by laminating separators to both sides of a membrane electrode assembly (MEA). Multiple units are then pressed together to form a fuel cell stack.

On the anode side, the separator conducts electrons generated by the catalytic reaction of hydrogen gas to an external circuit, whereas the separator at the cathode must have a function for supplying electrons from the external circuit (Figure 1). The separator commonly contains flow channels for feeds into the gas cell and for enabling heat transfer.

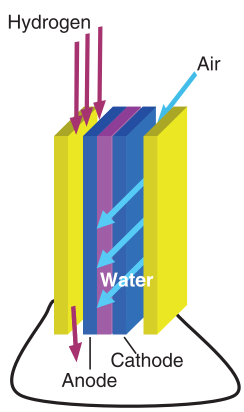

Figure 1: Schematic of a single element of a fuel cell stack. The separators are the outer plates (in yellow) of the cell

A conductive material is required and graphite-based carbon composite polymers or metals have been used in this role. Metals are preferred as they have far superior mechanical strength, allowing a reduction in the size and weight of the stack by using thinner metal plates.

The chosen metal needs to meet requirements of electrical properties and cost. Stainless steel fulfills the cost criteria, but the contact resistance between it and the MEA is greater than that of a graphite separator. This results in a loss of power from the cell. Nickel and chromium can also leech from the steel, poisoning the catalyst. Thus a thin coating of gold is applied to the surface by a method such as plating to reduce the contact resistance to an acceptable level and act as a barrier.

The quantity of gold required needs to strike a balance between cost and performance. A method of easily characterizing the thickness, uniformity and chemical composition of a layer is invaluable. X-ray photoelectron spectroscopy (XPS) has the capabilities to fulfill this requirement. XPS is the only analytical method providing quantitative elemental and chemical information with extremely high surface sensitivity.

Experimental

X-ray photoelectron spectroscopy is a rapid and non-destructive technique for the characterization of the surface of materials. It is quantitive, responsive to changes in chemical state, and extremely surface sensitive. For thin films of around 5 nm or less, it is possible to probe both the outer layer and the substrate without removing the overlayer. The attenuation of the substrate signal by the overlayer enables the thickness of the overlayer to be calculated. Alternatively, surface material can be gradually removed by Ar^+ ion bombardment to expose the substrate.

Results

Figure 2 shows Au 4f spectra of a stainless steel surface which had been coated with gold. The spectra were acquired during a depth profiling experiment where the surface layers are removed by an incident Ar^+ ion beam. By calibrating to a known standard the thickness of the overlayer can be calculated from a depth profile as shown in Figure 3.

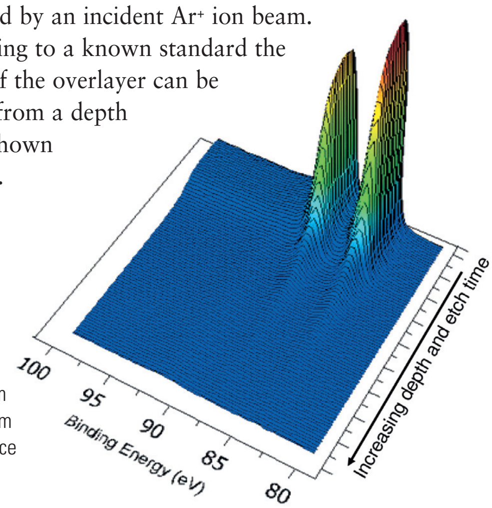

Figure 2: Au 4f spectra as the gold film is removed from the steel surface

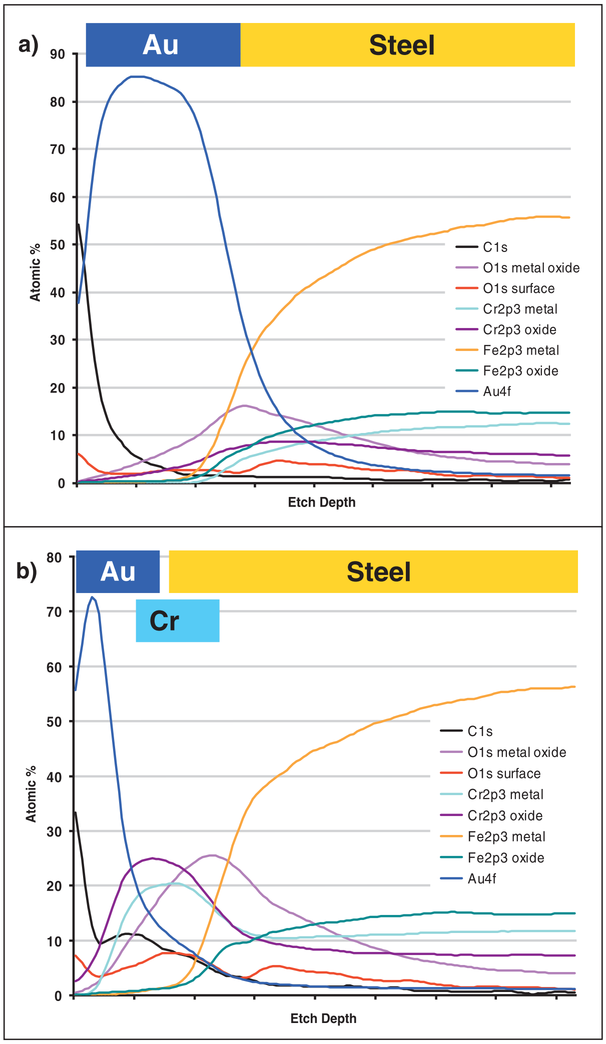

Figure 3: Examples of depth profiling experiments of gold film on a steel substrate. a) The depth profile of a thick gold film. b) Depth profile of thin gold film. It shows Cr migration from the steel substrate to the gold film

Using this information the possibility of migration of material into the film can be investigated. In figure 3b it can be seen that Cr has migrated from the surface of the steel and is forming a layer between the gold and bulk steel.

Alternatively, for thin (<10 nm) layers, a non-destructive approach can be used. This uses a simple model to calculate the thickness of the overlayer that would result in the observed attenuation of the substrate signal. The Thermo Scientific Avantage Data System allows the automation of the thickness calculation, from data collection to processing, enabling a series of analyses to be exported to a spreadsheet and facilitating batch processing of large sample sets. The plot in Figure 4 shows the thickness calculation results for two different analysis methods. There is a clear correlation between the non-destructive and a destructive depth profiling experiments on those samples where both methods were used.

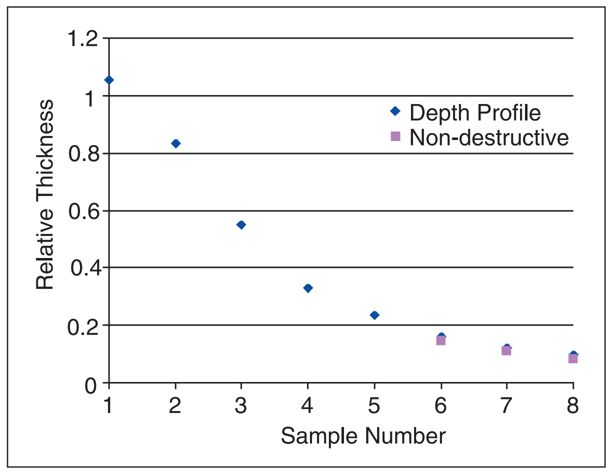

Figure 4: Correlation between depth profiling and a non-destructive calculation method

Summary

Steel substrates coated with thin gold films were characterized with the Thermo Scientific K-Alpha XPS. The thickness of the films was measured using two different methods and clear correlation between the results was found. XPS was also found useful in investigating the composition of the films.

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