Characterization Of Fuel Cell Electrocatalysts Using X-ray Methods

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High surface area electrocatalysts are critical components of high efficiency low cost polymer membrane fuel cells. The platinum and/or platinum alloy catalysts are typically prepared as nanocrystalline carbon supported and unsupported anode and cathode materials. The choice of catalyst type depends on whether the application is for hydrogen or direct methanol fuel cells (DMFCs). 2 nm crystallite size Pt supported on Vulcan XC-72 carbon is the anode and cathode catalyst most commonly used for hydrogen fuel cells while Pt-Ru alloys of 3-5 nm are currently being used for anode catalysts in DMFC systems.

Key parameters for successful catalyst design are average alloy composition, crystal structure, crystallite composition crystallite size and size distribution. All of the aforementioned parameters can be efficently and nondistructively measured using laboratory scale X-ray analysis methods. Recent advances in personal computer technology allow for full profile (Rietveld) and Warren-Averbach Fourier transform X-ray diffraction methods to be performed quickly and routinely. Full profile, also known as whole pattern analysis methods, model the entire X-ray diffraction pattern rather than just peak maxima. Highly overlapped diffraction patterns are very common in nanocrystalline materials due to size related line broadening phenomena. Full profile methods allow for the precise determination of lattice parameters and accurate measurement of individual diffraction line intensities. Phase fractions and percentages of amorphous material can also be estimated using full profile analysis techniques.

Warren-Averbach Fourier transform methods allow for the determination of particle size distributions. This method offers advantages in speed and cost over electron microscopic analysis methods to obtain crystallite size distributions. [1]

Fundamental parameter X-ray fluorescence spectroscopy methods allows for the rapid accurate determination of catalyst composition and mass loadings on raw materials and membrane electrode assemblies. Another advantage of this method over older empirical standard methods is the elimination of many calibration standards of different compositions. The fundamental parameter method needs only a single standard per element for calibration.

We have analyzed a large number of Pt and Pt/Ru based catalysts prepared by various synthesis techniques. These methods include unsupported and supported catalysts prepared by: colloidal precipitation, spray pyrolysis and ultrasonic atomization freeze drying methods. As prepared catalysts vary substantially crystallite size and size distribution. The degree of crystallinity, alloy composition and oxidation state also vary substantially with preparation method.

Reference

1. M.S. Wilson, F.H. Garzon, K.E. Sickafus, S. Gottesfeld, J. Electrochem. Soc., Vol .140(#10) pp. 2872-2877 (1993)