# Synthesis of novel Platinum/Carbon nanofiber electrodes for Polymer Electrolyte Membrane (PEM) fuel cells

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## Introduction

PEM fuel cell electrodes have been traditionally fabricated by deposition of finely divided platinum catalyst particles on a support material such as activated carbon, carbon black, etc. However Vapor Grown Carbon Fibers (VGCF) or carbon nanofibers also possess the ability to efficiently support the platinum catalyst. These graphitic fibers (average diameter of 70-100 nm) hold immense promise due to their excellent storage characteristics<sup>1</sup>, superior electrical conductivity and relatively low cost. Also the fact that their surface can be functionalized makes them suitable candidates as catalyst support in fuel cell electrodes, with the potential to replace the traditionally used carbon support material. This work examines the possibility of employing the "Colloidal Method" of Pt deposition and activation using nanofibers as the support material<sup>2-3</sup> and subsequent characterization of the supported electrocatalyst.

## Experimental

Carbon nanofibers (Applied Sciences Inc., Cedarville OH) were used as carbon support for the platinum catalyst particles. Prior to platinum deposition, the nanofibers were treated using two different oxidizing regimes with sulfuric and sulfuric/nitric acid. Subsequently a platinum precursor complex, hexachloroplatinic acid (Aldrich Chemicals) was reduced to finely divided colloidal platinum, which was then adsorbed on the surface of the oxidized nanofibers and activated in a furnace under nitrogen atmosphere. A similar procedure was carried out using Vulcan® XC-72 carbon (Cabot Corporation) as the control support. The supported electrocatalyst studied by X-Ray Diffraction (XRD), was Transmission Electron Microscopy (TEM) and X-Ray Absorption Spectroscopy (XPS). XRD provided information about the prominent diffraction planes of platinum on the support. Also average platinum particle size was determined from XRD and compared with similar data obtained by TEM. XPS was employed for elemental analysis of the supported catalyst surface and to gain an insight into the state of platinum. Subsequently Cyclic voltametric studies were carried out to determine the Electroactive surface area (EAS) of the supported catalyst samples.

### **Results and Discussion**

XRD analysis in Figure 1 confirms the existence of Pt on the nanofibers and reveals the prominent Pt crystallite planes to be [111], [200], [220] and [311] respectively, indicating a Face-Centered Cubic (FCC) lattice structure characteristic of the Pt crystal.



Figure 1. X-Ray Diffraction (XRD) pattern for Pt deposited on HNO<sub>3</sub> oxidized nanofibers.

A comparison of particle sizes obtained from XRD analysis of the various samples is indicated in Table 1.

Table 1. Mean particle size of Pt on different carbon support (from XRD analysis)

Catalyst	Average particle size (nm)
Pt-HNO <sub>3</sub> oxd. nanofibers	12
Pt-HNO <sub>3</sub> /H <sub>2</sub> SO <sub>4</sub> oxd.	22
nanofibers	
Pt- Vulcan Carbon	26

The average particle size of the Pt/HNO<sub>3</sub> sample obtained from TEM was ~9 nm which is quite close to the value obtained by XRD. Figure 2 indicates the XPS spectrum of platinum (Pt<sub>4f</sub>) region on the HNO<sub>3</sub> oxidized nanofibers. The presence of two prominent sets of Pt<sub>4f</sub> peaks (corresponding to the.  $4f_{5/2}$  and  $4f_{7/2}$  orbital states) is a further confirmation of the platinum being present on the nanofiber surface. The peak regions can be fitted with two sets of peaks at 1) 71.2 eV and 74.6 eV (These correspond to platinum in metallic state), and 2) 71.8 eV and 75.3 eV (These correspond to Pt in oxide form). As seen in Figure 2 relative areas of the two sets of peaks are nearly identical. Thus platinum is present on surface of the nanofibers equally in elemental and oxide state.



Figure 2. X-Ray Photoelectron Spectroscopy (XPS) spectrum of  $Pt_{4f}$  core level for Pt deposited on  $HNO_3$  oxidized nanofibers

### References

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