STXM Characterization of Nanostructured Thin Film Anode Before and After Start-up Shutdown and Reversal Tests

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In recent years nanostructured thin film (NSTF) technology has been extensively evaluated as a possible path forward for production of low Pt or Pt alloy loading catalyst layers for proton exchange membrane (PEM) fuel cells for automotive application [1-4]. It has been reported that main challenges for NSTF based cathodes vs. conventional carbon based electrodes include long pre-conditioning time, voltage losses under operating conditions involving higher temperatures and low relative humidity and flooding at cold start up conditions [1, 5]. In contrast, no negative effects have been reported for NSTF anode catalyst especially with addition of small amounts (2-10$\mu g/cm^2$) of oxygen evolution reaction (OER) catalysts. It is expected that anode will be qualified for automotive use first [6].

High resolution scanning electron microscopy and transmission electron microscopy techniques with energy dispersive X-ray analysis have been used to study morphology, microstructure and composition in pristine and aged NSTF based electrodes. For cathode application dealloying of Co has been reported [1], while for anode, larger stability of sputtered Ir over Ru in fuel cell operating conditions has been reported [4, 6]. However, these characterization approaches unfortunately are not capable of providing the information on changes that may occur to crystalline perylene red support whiskers.

Soft X-ray scanning transmission microscopy (STXM), a synchrotron based technique, has been recently applied for characterization of fuel cell MEA components [7-11]. Utilizing intrinsic near edge X-ray absorption fine structure (NEXAFS) signal, STXM is capable to quantitatively map chemical components in catalyst layers and membrane with 30 nm spatial resolution. Using the combination of two energy maps at F 1s and C 1s edge, a methodology for ionomer and carbon support imaging in PEM cathode catalyst layer was developed [9]. This was followed by studies on ionomer distribution in different catalyst layer structures [10]. By performing an STXM tomography, the first 3D mapping of carbon support and ionomer was demonstrated in a thin focused ion beam cut cathode section [11].

This paper reports on using the STXM technique to characterize NSTF anode at the beginning of life and after start-up shutdown and reversal tests. Changes related to anode microstructure and chemical composition especially related to perylene red support will be discussed.

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