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Electron Energy Loss Spectroscopy for Aqueous *in Situ* Scanning Transmission Electron Microscopy

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Analysis of materials within a liquid environment by transmission electron microscopy (TEM) has produced successful results using both fluid cell specimen holders, [1-4] FIG 1, as well as liquid jets within an environmental transmission electron microscope (ETEM) [5]. Chemical and structural changes during these experiments have yet to be quantified due to thick fluid path lengths varying from 100 nm to several micrometers in thickness [1-4]. The sample imaging thickness for in situ TEM is a combination of the fluid thickness itself, as defined by spacer height, and the thickness of the two electron transparent windows making the cell. The standard thickness for electron transparent windows is 50 nm SiN although successful experiments have been preformed with 25 nm SiN membranes [1]. Electron energy loss spectroscopy (EELS) quantifies the chemical and electronic structures of materials within an electron microscope with optimal specimen thickness defined by a single scattering event of the incident electron, known as the inelastic mean free path. To obtain structural information from materials suspended within a fluid cell holder the fluid path length should be at or below the inelastic mean free path. In aqueous environments the fluid cell exhibits a standard background signal from the water as well as the SiN membranes. Characterization of the oxygen K-edge within the fluid cell at a nominal 50 nm fluid path length provided evidence that the water is characteristic of atmospheric conditions with the introduction of oxygen gas caused by radiolysis damage by the electron beam as seen in FIG 2. Experimental EELS measurements acquired on a spherical aberration corrected scanning TEM (JEOL JEM-2100F/Cs) of the fluid thickness can be compared to theoretical calculations of the expected inelastic mean free path for a certain fluid cell configuration. The scanning modality is optimal to select the region of interest and acquire spectra from individual particles within fluid environment.

The ability of obtaining chemical and structural information from nanomaterials within fluid environment of an electron microscope will aid in understanding the reaction mechanisms as well as basic interactions of the material within the solvent. A review of the experimental conditions for obtaining EEL spectra from a fluid cell with scanning transmission electron microscopy will be presented. Core loss spectra from nanoparticles contained within the fluid cell will also be reviewed along with the experimental parameters necessary to obtain the spectra.

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FIG. 1. Hummingbird Scientific fluid stage holder equipped with inlet and outlet lines for microfluidic pumping. The window region well holds two 2.6 x 2.6 x 0.3 μm windows, composed of electron transparent SiN thin film membrane supported by Si substrate. 50 nm Au spacers were deposited at the window corners.

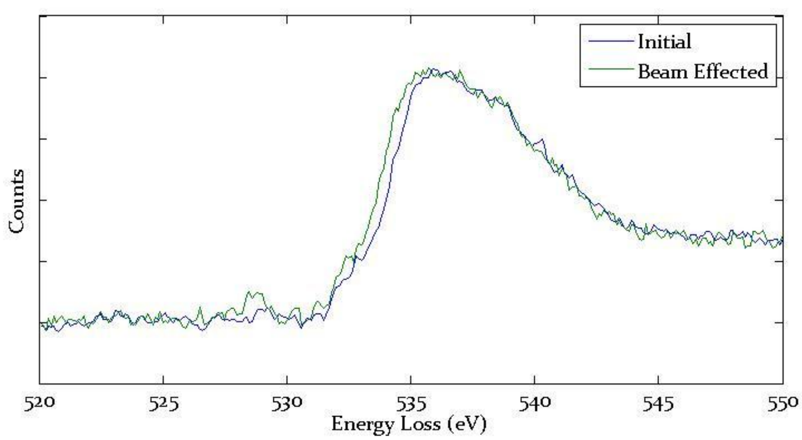

FIG. 2. Electron energy loss spectra of deionized water within fluid stage, two spectra were acquired on a Gatan Quantum spectrometer sequentially. Change in spectra due to radiolysis damage of the beam is seen in the O_2 (g) peak observed at 528eV.