Dynamic spectro-microscopy of nanoparticle growth and corrosion.

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Traditional microscopy carried out on dry ex vitro samples presents significant challenges to the study of dynamic processes which occur within liquid environments. To date, a variety of liquid cells have been used to study nanoparticle dynamics in the transmission X-ray microscope, though usually with limited liquid volumes in order to meet the transmission requirements of the X-rays [1,2]. Here we present a novel ~30 mL liquid cell developed for the transmission X-ray microscope [3], which has been used to carry out in situ electrochemical spectro-microscopy measurements of nanoparticle growth as well as corrosion.

The first system we consider is the electrodeposition of ZnO. The liquid cell was filled with electrolyte containing Zn(NO₃)₂ and KCl, and heated to 65 °C with O₂ bubbling. Nanostructure morphology was varied from nanoplates to nanorods by altering Zn²⁺ concentration and deposition potential. During electrodeposition of the ZnO nanostructures, X-ray imaging was performed simultaneously using the full-field X-ray microscope at the Stanford Synchrotron Radiation Lightsource (SSRL) beamline 6-2. Using this method, it was possible to observe transient events which occur during electrodeposition such as instantaneous or delayed nucleation, providing insights into the growth mechanisms of electrodeposited ZnO.

We also demonstrate the application of the liquid cell to nanoparticle corrosion, by considering the transformation of nanoparticles of CoCrMo alloy which are formed during the wear of metal-on-metal hip prostheses. To understand the mechanism of CoCrMo nanoparticle corrosion, the liquid cell was adapted to simulate the oxidative biological environments which wear particles are expected to encounter in vivo. Nanoparticle behavior was again investigated by in situ TXM to understand the conditions under which Co is leached from the wear particles.

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