Laser Assisted Atom Probe Tomography analysis of nanoscale, energy and environment related materials at EMSL

S. Thevuthasan[*], Satyanarayana V N T Kuchibhatla[*], P. Adusumilli[*, #], M. Olszta[*, $], B. Arey[*], V. Shutthanandan[*], J. P. McKinely[*,$], C. M. Wang[*], S. Bruemmer[*, $], D. R. Baer[*], T. Prosa[**], R. Ulfig[**], D. Seidman,[***] A. Buxbaum[****], Y. C. Wang[****], T. Tessner[****],

[*]EMSL, [$]Pacific Northwest National Laboratory, Richland, WA – 99354

[**]Cameca Instruments Inc., Madison, WI – 53711

[***, #]Northwestern University, Evanston, IL – 60208

[****]FEI Corporation, Hillsboro, OR - 97124

Increasing emphasis on materials with enhanced properties in various functional applications has provided an impetus to develop the tools that will help understand materials with increased spatial resolution, at larger field-of-view and enhanced sensitivity. Laser assisted atom probe tomography analysis fits these objectives well and provides 3D-chemical images. While the atom probe analysis has been used for a few decades, the development of laser pulsing has extended the scope of this technique from metals to semiconductors and very recently to the insulating materials such as bulk oxides. EMSL, a national scientific user facility of the DOE, is developing an atom probe tomography capability complimenting the existing surface and interfacial analysis, microscopy capabilities to provide solutions to various problems of interest to energy and environment, in particular, at the nanoscale.

This presentation examines the feasibility of using atom probe for various challenging materials systems including some that have not been previously examined by atom probe methods. We report the first Local Electrode Atom Probe (LEAP®) analysis of Au nanoparticles embedded in MgO using ion beam synthesis method, in which, 2MeVAu ions were implanted in a MgO(100) substrate at various temperatures and the substrate was subsequently annealed at 1000oC for 10 hours in air. These samples were analyzed using a combination of atom probe tomography and analytical electron microscopy. Also analyzed for the first time, are the samples obtained from the reductive biotransformation of 6-line ferrihydrite located within porous silica (intragrain ferrihydrite) by Shewanella oneidensis MR-1. Imaging and analysis of biologically induced Fe-phosphate precipitates in porous silica using He-ion microscopy and atom probe tomography will be outlined. Selected results from EMSL user projects related to semiconductor and nuclear energy materials will also be discussed. Dopant distribution and the oxidation mechanisms will be predicted based on the atom probe analysis in these systems. Dual beam FIB/SEM systems at EMSL have been extensively used for sample preparations and some of the key observations during sample preparations will also be outlined in this presentation.

Some of the key observations from the Au-MgO system and the oxidation of the Ni-based alloy are presented in figure 1 and 2, respectively.

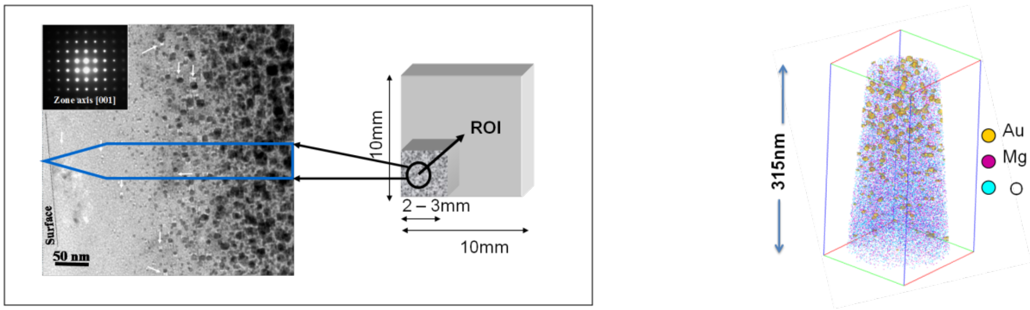

Figure 1: left) The HRTEM image and the region-of-interest (ROI) in the Au-MgO sample are depicted. The blue lines indicate the possible atom probe sample configuration. Figure on the right is the 3-D reconstruction of the sample that indicates the cluster Au-embedded in the MgO matrix

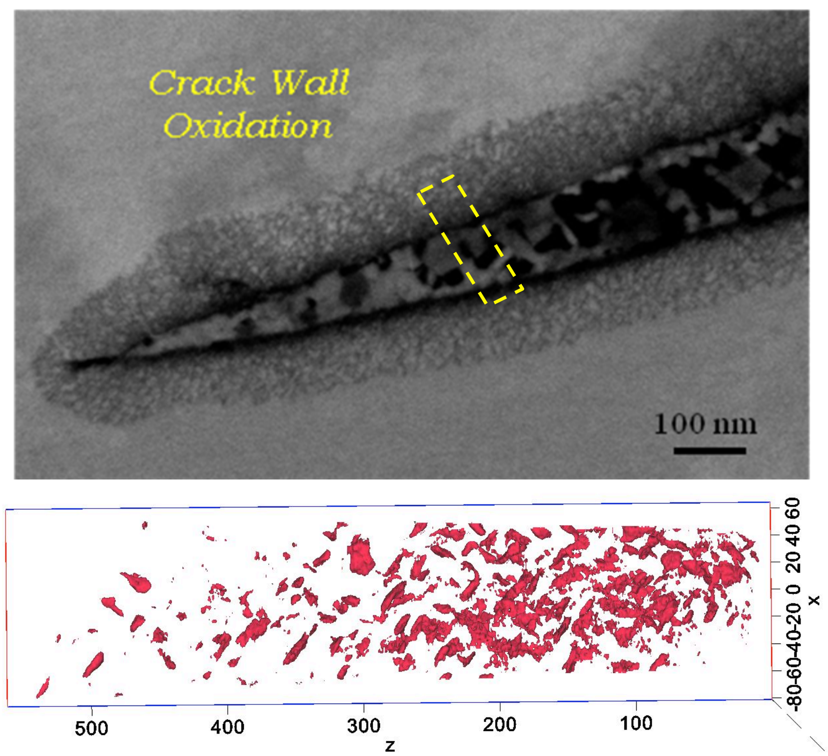

Figure 2: Stress corrosion crack tip in a Ni-30Cr-9Fe alloy. SEM low angle backscatter image (top) illustrates mottled contrast of oxide penetrative attack from the crack wall into the metal matrix. 3DAPT reconstruction (bottom) shown an isoconcentration surface of Cr + O (98%) for a 10 nm lateral slice of a sample removed from the crack wall (see dashed box). Analysis of individual particles reveals nearly pure Cr_2O_3 centered in oxide-filled “tunnels” penetrating into the metal matrix.