Atom Probe Tomography Focus Topic
Room: 230A - Session AP+AS-MoA

Current and New Research Fields for Applications of Atom Probe Tomography
Moderator: Baishakhi Mazumder, Center for Nanophase and Materials Sciences Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA

2:20pm **AP+AS-MoA1 APT Studies of the Embrittlement of Fe-Cr Ferrite**, ***Mattias Thuvander***, Chalmers University of Technology, Sweden, *J. Odqvist, P. Hedström*, KTH Royal Institute of Technology, Sweden **INVITED**

Ferrite containing more than a few percent chromium is susceptible to embrittlement when subjected to elevated temperatures for sufficiently long periods of time. The phenomenon is often called '475°C embrittlement', indicating the temperature at which the effect is most rapid. The embrittlement is an issue for several important types of steel, including corrosion resistant ferritic steel, duplex stainless steel, cast austenitic steel and austenitic weldments, and limits their maximum service temperature (to about 300°C). The volume fraction of ferrite ranges from 100% in ferritic steel, to about 50% in duplex steel, down to 5-25% in austenitic castings and weldments (for which the presence of some ferrite is needed to avoid solidification cracking). The reason behind the embrittlement is the phase separation into two bcc-phases, Cr-rich α' and Fe-rich α, as can be seen in the binary Fe-Cr phase diagram. The phase separation results in an increase in strength and hardness but a decrease in ductility. Depending on Cr-content and temperature, the phase separation occurs by nucleation and growth (typically for low Cr contents) or by spinodal decomposition (typically for high Cr contents). Spinodal decomposition is an interesting phenomenon in physical metallurgy and it has been extensively studied for a long time. The phase separation occurs on the nanometer scale and during the early stages the concentration variations are subtle. Therefore, it is a challenging mechanism to study experimentally. Atom probe tomography (APT) is a well-suited technique, as small concentration variations on a small scale can be measured, also in the case when the phase separation has a complex 3D-structure, which is the case for spinodal decomposition. In this presentation, APT methodology for studying spinodal decomposition will be discussed. Also, results from several investigations concerning phase separation kinetics will be presented. For example, the influence of stress on spinodal decomposition in duplex steels, the influence of homogenization temperature on spinodal decomposition in binary Fe-Cr and the behavior of different ternary Fe-Cr-X systems will be addressed.

3:00pm **AP+AS-MoA3 Comparing APT Mass Spectral Ranging for Compositional Accuracy: A Case Study with Cast Duplex Stainless Steels**, ***Daniel Perea***, *A. Eaton, J. Liu,* Pacific Northwest National Laboratory, *S. Mburu, S. Schwarm, R. Kolli, S. Ankem*, University of Maryland

The mass spectrum of an atom probe tomographic analysis is a superposition of correlated mass-to-charge-state peaks and uncorrelated background counts. The compositional accuracy of a volume of material measured via atom probe tomographic analysis is dependent upon the manner in which the individual mass spectral peaks are ranged for integration. However, an accurate calculation of composition can be challenging due to a combination of complicated peak shapes, peak overlap, and background counts. Using data from both the *α*–ferrite and *γ*–austenite phases of a cast duplex stainless steel , we illustrate a methodology to account for the background counts, as well as to deconvolute overlapping peaks, in order to calculate composition that we compare to energy dispersive x-ray spectroscopy measurements.

3:20pm **AP+AS-MoA4 Chemical Imaging of Atmospheric Aerosols using Atom Probe Tomography and Multi-Modal Characterization**, ***Jia Liu***, *M.I. Nandasiri, L. Gordon, G. Kulkarni, V. Shutthanandan,* Pacific Northwest National Laboratory, *S.A. Thevuthasan,* Qatar Environment and Energy Research Institute, Qatar, *A. Devaraj*, Pacific Northwest National Laboratory

Global climate is significantly dictated by small particulates in the atmosphere known as aerosols. These atmospheric aerosol particles when subjected to certain humidity and temperature conditions can induce heterogeneous ice nucleation, which is directly sensitive to aerosol surface structure and chemistry. These ice nuclei are the precursors to snow fall and precipitation. Often natural atmospheric aerosols are found to be coated with sulfates and organic molecules. Elucidating the mechanism responsible for ice nucleation on coated or bare atmospheric aerosols requires understanding the structure, composition and chemical state of coated and bare aerosols. At EMSL we are developing a multimodal approach for imaging bare and coated aerosols utilizing a combination of atom probe tomography (APT), imaging X-ray photoelectron spectroscopy (XPS), focused ion beam scanning electron microscopy (FIB-SEM) and transmission electron microscopy (TEM). Imaging XPS provides the composition and chemical state of organic or inorganic elements within the top 5nm of the surface of aerosol particles with a spatial resolution of ~3 µm. FIB-SEM aids in understanding the morphology and porosity of particles both on the surface and sub-surface. TEM provides the atomic scale structural information and laser assisted APT provides sub-nanoscale compositional mapping of aerosols. TEM and APT are sub-single particle analysis techniques and can complement the individual aerosol particle measurements provided by the single particle laser ablation time of flight mass spectrometry (SPLAT). All these techniques provide specific multiscale chemical and structural information about the aerosol particles from the macro- to the atomic-scale. Specific examples from multimodal chemical imaging of mineral dust aerosols coated with varying concentration of sulfuric acid or organics will be presented along with the direct insights gained through this approach for improving ice nucleation parameterizations.

3:40pm **AP+AS-MoA5 Combining Atom Probe Tomography with TKD and FiB for Comprehensive Characterization of High Performance Materials**, ***Sophie Primig***, University of New South Wales, Australia, *K. Babinsky, P. Haslberger, C. Hofer, D. Lang, C. Turk*, Montanuniversität Leoben, Austria **INVITED**

Despite the increasing interest in atom probe tomography, this technique has so far almost exclusively been applied for chemical analyses of materials at the atomic scale. As nowadays the frontiers of material science are more and more being pushed towards the nanostructure, advanced comprehensive characterization techniques which provide both chemical and crystallographic information are required. For the crystallographic analysis of atom probe specimens several complementary techniques such as transmission electron microscopy have been applied that all have their advantages and drawbacks. Different approaches try to establish crystallographic information directly from the atom probe data itself which is still computationally challenging and not always possible. Another recently proposed straightforward way of quickly obtaining crystallographic information is the application of transmission Kikuchi diffraction on atom probe tips prior to the atom probe experiment. This procedure has so far only been successfully applied for positioning of grain boundaries close to the apex of the tips via focused ion beam milling.

The aim of the current study is to show applications of transmission Kikuchi diffraction on atom probe specimens of high performance materials and to demonstrate the strengths as well as the limits of these two complementary techniques. Four examples are shown which include boron segregation at prior austenite grain boundaries in a heat treatable steel, interlath retained austenite films with cementite in a bainitic steel, molybdenum carbides in a molybdenum alloy, and the preparation of grains with well-defined crystal directions in the tip axis of an iron-cobalt-molybdenum alloy.

4:20pm **AP+AS-MoA7 Atom Probe Tomography Studies of FeCo Nanocomposite Soft Magnetic Materials**, *A. Leary, V. Degeorge, V. Keylin,* Carnegie Mellon University, ***Arun Devaraj***, *J. Cui,* Pacific Northwest National Laboratory, *M. Mchenry*, Carnegie Mellon University

Nanocomposite soft magnetic materials exhibit high magnetization and low coercivity for application in power electronics, motors, and sensors. The composite nature of these materials, created by thermal annealing of rapidly solidified metallic glasses, offers many options to tune desired material properties. Grain sizes below ~30 nm are relevant to average the magnetocrystalline anisotropy between neighboring grains within an exchange volume. Chemical partitioning during devitrification creates variations in local composition compared to the nominal alloy composition. These variations impact material properties such as magnetostriction, Curie temperature, and grain size. Local composition measurements of annealed nanocomposites by Atom Probe Tomography link observed magnetic properties to the material structure. The impacts of chemical partitioning on diffusion limited grain growth, intergranular coupling at high temperature, and induced anisotropy are discussed.

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