Fall M3S Meeting @ Baxter Round Lake

Friday, November 22nd, 2019

Talk Abstracts

Examination of Museum Specimens Using Atmospheric XRF and SEM-based Micro-XRF Analysis
Ed Vicenzi, Smithsonian Institute – MAS Invited Tour Speaker

Instrumentation for X-ray fluorescence (XRF) ranges from routine commercial applications (e.g. scrap metal sorting) with handheld spectrometers to quantitative nanoscale imaging of advanced materials at synchrotron beamlines. Between the those vastly different end-members are 2 varieties of laboratory-based XRF instrumentation recently installed at the Smithsonian's Museum Conservation Institute: 1) portable imaging XRF, and 2) SEM-based scanning micro-XRF. An effort has been made to understand the capabilities of the new systems, and illustrations of the method, including: Maya jade, Aztec? obsidian mirrors, and 18th century metal-bearing textiles will be discussed.

Microanalysis in Forensic Paint Investigations
Ethan Groves, Microtrace LLC

The analysis of paint for forensic purposes is multifaceted, combining several microanalytical techniques appropriate to the specifics of the investigation. Whether it's the physical examination of a paint's layer structure, Raman analysis of a pigment package, infrared spectroscopic characterization of binder chemistry, or an elemental survey to characterize the composition of a paint, modern analytical instrumentation can be used to characterize, compare, or source microscopic particles of paint for forensic purposes. This presentation will discuss case examples that illustrate the probative value of microanalysis for microscopic particles of paint, as well as current research intended to advance the practice of elemental analysis by SEM/EDS in forensic paint investigations.

While SEM/EDS has become commonplace for the analysis of materials and approaches for forensic paint comparisons have been validated, little attention has been given to the implementation and significance of SEM/EDS data in forensic comparisons of paints. Our ongoing research intends to explore and define analytical variables such as sample preparation and acquisition parameters to evaluate their impact on the resulting data. Furthermore, through the analysis of 300 automotive paint samples, we can begin to define compositional ranges of automotive paint and develop a formalized approach to the interpretation of SEM/EDS data in relation to the probative value of a particular paint composition. This talk will also discuss the implementation of ion milling and EBSD and the potential value that these techniques can provide in the analysis of multi-layered paint samples.

3-D, In-situ, and Ultrafast Electron Microscopy
Ilke Arslan, Center for Nanoscale Materials, Argonne National Laboratory

With recent advances in in-situ microscopy, a new era in microscopy has arrived that allows for the dynamic imaging of materials under reaction conditions in the (scanning) transmission electron microscope. It is no longer sufficient to image materials under vacuum conditions, but to get closer to the conditions in which the material will be used, such as high/low temperature, liquid environments, gas environments, or a combination thereof. Within these environments, moving beyond "real time" imaging to ultrafast imaging can provide yet another level of fundamental understanding of nanomaterials. Further, combining an in-situ or ex-situ experiment with electron tomography is a very powerful method for materials characterization as this provides the 3-D morphology/chemistry of the materials in more relevant environments. This talk will focus on newer developments such as in-situ heating in liquid, reconstruction algorithms for electron tomography with significantly fewer images, and the future of ultrafast transmission electron microscopy at the Center for Nanoscale Materials.

Confirming Phase Identities with Quantitative Analysis to Ensure Accurate EBSD Mapping
Bil Schneider, University of Wisconsin – Madison

In Geology, a common sample preparation technique for EBSD entails polishing one side of a 30 µm thick slice of rock adhered to a glass slide called a thin section. A very fine vibratory finish is conducted on the thin section to reduce deformation from grinding and polishing so the beam electrons can interact with the undeformed uppermost 10-50 nanometres of the sample surface. Several small EBSD maps were run on the same area of a sample using different plagioclase feldspar phases as the phase to match. Maps for albite, oligoclase, bytownite, and anorthite all solved at a high rate with similar success. Point and ID EDS was used taking five spectra from within the area that was mapped by EBSD.

One Animal, Two Glues: Insights on How Oysters Stick
Jon Wilker, Purdue University

Oyster reefs are constructed by shellfish cementing together into extensive communities, often several meters deep and kilometers long. These reefs become a major factor in determining the viability of coastal ecosystems by filtering large volumes of water and providing places for other organisms to live. With recent news of intense hurricanes, particularly important is the ability of oyster communities to absorb storm surge energy and protect coasts. We are working to understand the nature of oyster cement by studying larvae, juveniles, and adults. Earlier, we showed that adult oyster cement is built from a hybrid of organic and inorganic components, with inorganic calcium carbonate being the most dominant constituent. More recently, we have been examining how larval oysters first attach to surfaces. Larvae eject a premade adhesive that was described in the 1970's by Cranfield. We also see that larval adhesive is a fully organic, hydrated, and fibrous material. What came as a surprise was how the animals change their adhesion strategy so closely with metamorphosis from larvae to a juveniles. At 1-2 days after settlement and initial attachment, a quite different adhesive emerges from in between the two shells. Looking down from the top, this material appears to be much like shell, with inorganic columns packed together closely and an organic binder in between. Underneath these columns, however, is a thick layer of organics providing interfacial contact between animal and substrate. The second, top shell is thicker and does not have such a layer of organics. Further growth shows the more homogeneous organic-inorganic system found in adults. These results show how oysters use strikingly different adhesives at progressive stages of life.

Finding Grains in a Mountain of Sand

Gerry Olack, University of Chicago

Studies on meteorites have increasingly focused on smaller and smaller grains. The same holds true in other fields of study. Our ability to see grains and inclusions in grains in various samples has also driven the development of new instrumentation to analyze those grains and inclusions. One of the difficulties in doing these studies is finding the grains of interest. High resolution mapping using an scanning electron microscopy (SEM) with x-ray data (energy dispersive spectroscopy, EDS) is the one of the first analytical steps in that search. Focused ion beams (FIB) can then be used to mill out the section/species of interest and prepare them for further analysis, e.g. transmission electron microscopy (TEM) or atom probe tomography (APT). If isotopic information is needed, then samples need to be prepared for ion microprobe secondary ion mass spectrometry (SIMS) or resonance ionization mass spectrometry (RIMS). This presentation will focus on how to find grains of interest, e.g. pre-solar grains, and prepare them for further analysis.

Probing Structural Diversity in Beam Sensitive Materials Using Electron Dose Efficient Methods

Roberto dos Reis, Dravid Research Group, Northwestern University

The field of transmission electron microscopy (TEM) witnessed a drastic jump in the resolving power anchored by major advances in both hardware and software over the last few years. However, to obtain such high resolution often requires an electron dose unbearable by many structures of interest. Dose efficient TEM methods are needed in order to overcome damaging and enable imaging from a broader range of structures, from healthcare and biology to chemistry, physics and materials science. For beam sensitive subjects, the practical resolving power of an imaging system can depend as much on the signal to noise obtained before the sample is damaged as on the imaging optics.

In this talk, I will show practical ways to develop a high-resolution low-dose images with focus on application of phase contrast imaging with electron ptychography and discuss some of the issues that need to be resolved to maximize efficiency of this technique. I will demonstrate recent advances in direct-detection electron-counting cameras and how the high detective quantum efficiency allows images to be performed with an extremely low electron beam dose, making it possible to obtain convergent beam electron diffraction of beam-sensitive materials with minimal structural degradation. In addition, I will show examples of collection of electron diffraction patterns at many probe positions with millisecond dwell times (4D-STEM) to obtain many imaging modalities for structural characterization with high spatial resolution of sensitive samples.