Fall M3S Meeting @ Baxter

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Talk Abstracts

TEM Examination of Armor Ceramic Materials

Scott D. Walck, U. S. Army Research Lab – MAS Invited Tour Speaker

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Armor ceramics are perhaps the most important component in ceramic armor systems. The armor ceramic spreads the extremely high dynamic loads during a ballistic impact event and blunts or abrades the incoming round. The most important attributes of the armor ceramic are its hardness, fracture toughness, density, and cost of manufacture, with these being somewhat at odds with each other. Our work centers on identifying and understanding the inelastic deformation mechanisms that are active in armor ceramic materials and how to improve their material design. Understanding the deformation mechanisms in these materials is crucial for developing and optimizing next-generation ceramic materials in body and vehicle armor systems. In an effort to better understand the mechanistic response of polycrystalline boron carbide and silicon carbide materials to large contact stresses, transmission electron microscopy (TEM) methods were used to examine thin cross-sections of the inelastically deformed regions beneath Knoop indents of various loads and load-dwell times. Indentation allows the study of deformation of microstructural features as a function of distance and depth from the loading and allows for comparison to mechanistic modeling. We have developed new, multi-step techniques for the TEM sample preparation below the indented region of ceramic materials that preserve the state of deformation of the material. We report here the microstructural observations of TEM cross-sections that were prepared from 0.3, 1, and 2 kgf Knoop indents in commercially available a hot-pressed polycrystalline boron carbide and three silicon carbide variants (hot-pressed, liquid-phase sintered, and solid-state sintered) that illustrate the complexity of the task at hand.

Microanalysis of Soft and Hard Matter in the Next Generation AEM

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The evolving state of detector technologies and new geometries in electron-optical instruments is providing opportunities for x-ray microanalysis at not only near atomic level characterization in crystalline materials, but now also studies of non-crystalline beam sensitive soft-matter. Increasing the solid angle and drift compensation technologies has afforded the possiblity of analysis of low dose rate analysis in many systems during: in-vacuuo , in-situ as well as in cryo-EM configurations. The current status microanalysis in these areas will be presented and future prospects outlined.

Acknowledgements
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FIB Development and Applications Through the Years

Lucille A. Giannuzzi, EXpressLO LLC – MSA Invited Tour Speaker

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Focused ion beam (FIB) microscopy, specimen preparation, and nanoprototyping has witnessed numerous advances over the past 20+ years. First perceived as an expensive novelty, the FIB currently offers necessary and indispensable capabilities for any major research university, company, or national laboratory. FIB usage for site specific milling and deposition is now status quo. Standard specimen preparation protocol exists for high resolution transmission electron microscopy and other characterization analyses requiring minimal surface damage. Over the years, these techniques improved with an understanding and application of the fundamentals of ion-solid interactions. Successive FIB slicing followed by imaging and associated analytical methods enable 3D tomographic materials characterization containing morphology, microstructure, chemistry, and crystallography. The automation of these functions improves reliability, statistics, and throughput. Despite its maturity, FIB instrumentation and applications continue to develop. New sources emitting different ions species and beam currents allow materials characterization across the nano-, micro-, and macro- length scales. In addition, easier and faster micromanipulation methods performed outside the FIB optimize FIB instrumentation usage. In this lecture, FIB development and applications characterization and prototyping will be presented. Attention will be given to discoveries of structure/property relationships in materials possible only by FIB. In addition, the future of FIB will be discussed. Examples from metals, ceramics, polymers, composites, integrated circuits, minerals, biomaterials, and nuclear irradiated materials will be provided.

Serial Block Face Imaging – First Experiences

Chris Gilpin, Life Science Electron Microscopy Facility, Purdue

Chris Gilpin Ph. D., Director of the Life Science Electron Microscope Facility at Purdue, and campus-wide coordinator for electron microscopy

Purdue took delivery of a Thermo Fisher Volumescope at the beginning of 2018. After practicing on vendor supplied tissue blocks it was our turn to prepare samples and to collect some data. I will talk briefly about the sample preparation but wanted to try and give a real-world view of what it is like to set up the instrument and collect data. Our first real data set from ovules from Arabidopsis. I plan to show videos of the complete setup and data collection process.

Development of Complementary Methods in TEM Tomography to Answer Outstanding Questions in Cell Biology and Virology

Ranjan Sengupta, Department of Medicinal Chemistry and Molecular Pharmacology, Purdue

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One of the biggest challenges for cell biology in the 21st century is the visualization of membrane remodeling in situ. Membrane remodeling controls a vast array of critical cellular events such as organelle biogenesis, membrane trafficking and cell division. However, intricate visual details on these topological pathways are lacking due to various challenges. First, subcellular membrane structures are large, convoluted, and often span over a large volume. Secondly, determining the identity of these intermediates away from their site of biogenesis is a formidable challenge. Finally, these transformations are driven on cue by changing functional states of organelles much of which is still unknown, making it hard to predict and capture these transformations. Thus, dissection of such a dynamic pathway of membrane reshaping and organelle biogenesis entails capturing a statistically significant number of three-dimensional and high-resolution snap-shots of key transitional events over a large volume while also identifying these intermediates in a sea of unrelated membrane structures. We are starting to dissect these key cellular processes by developing two complementary methods, namely large volume TEM tomography and APEX-tag tomography. In in this talk, I will demonstrate the development and application of these two complementary technologies for a step by step reconstruction of a virus induced transformation of the Golgi apparatus into unique trafficking vesicles and to ascertain the 3D localization of a novel mammalian protein within a well preserved intracellular architecture.

Directly Revealing the Chemical Ordering in M_3B_2 and M_5B_3 Borides

Xiaobing Hu, Department of Materials Science & Engineering and NUANCE, Northwestern

Xiaobing Hu Ph.D., Research Assistant Professor, Department of Materials Science and Engineering, The NUANCE Center, Northwestern University

Interstitial phases formed in commercial engineering alloys involve an enormous family of compounds ranging from simple binary compounds to complex multi-elements solid solutions. The technological importance stems from their dominative effect on the properties of many industrially important engineering materials (esp., steels and superalloys). As composed by many different elements, they are generally described by the formula M_aX_b, where M represents the (transition) metal elements and X the light non-metal elements, and *a* and *b* are integer numbers. Determining the distributions of different metal atoms within the crystal lattice of an interstitial particle is particularly important for deep understanding of their dominative effects. Conventional reciprocal space-related diffraction technique is widely used, but only limited to a description of average structures with a low spatial resolution. Hitherto, probing the chemical distribution at atomic scale, however, is still extremely challengeable. Here we use atomic-resolution energy-dispersive X-ray spectroscopy to determine the chemical distribution within the crystal lattice of the representative interstitial phases M_3B_2 and M_5B_3. Different from the widely accepted knowledge that metallic atoms are randomly distributed within the lattice as a solid solution state, we found these metallic atoms prefer to occur in an ordered form. By means of the newly developed crystal structure prediction calculations, we further rationalize the observed chemical ordering in terms of differences in free energy. The combined technique described here provides a direct and reliable way of uncovering the chemical features at atomic scale within any phases with complex compositions regardless of their grain size and volume fraction.

Electron Backscattered Diffraction as a Key Technique in Studying the Effects of Microstructure on Electrical Properties

Jann Grovogui, Dravid Research Group, Department of Materials Science & Engineering, Northwestern

Jann Grovogui, PhD Candidate, Dravid Research Group, Northwestern University, Department of Materials Science and Engineering

Electron Backscattered Diffraction (EBSD) is a well-known Scanning Electron Microscopy (SEM) technique that has been used in metallurgical applications to identify crystallographic phases and grain orientations for the purposes of drawing connections between microstructure and mechanical properties. In EBSD Kikuchi bands are indexed to reveal crystallographic orientation and texturing, as well as grain size and shape anisotropy. This has been particularly useful in identifying the mechanical properties of metals undergoing a variety of deforming and processing techniques, such as rolling, welding, or 3D printing. However, crystal anisotropy is not only important in determining mechanical properties, but for electronic and thermal properties as well.

Thermoelectric materials are capable of converting waste heat into usable electricity and depend on the optimization of both electrical and thermal conductivity, as well as material Seebeck coefficient. These properties are intricately tied together, and the manipulation of microstructure can help decouple these properties to improve thermoelectric performance. Specifically, grain boundaries are of interest in thermoelectrics because they are known to reduce electrical and thermal conductivity, yet few experimental studies exist that investigate the effect of grain boundary morphologies on thermoelectric properties. By using conventional metallurgy as our motivation, we seek to use EBSD as a method to assist in classifying grain boundaries while drawing direct relationships between processing, structure, and properties in thermoelectric systems.