Radiation Effects on Materials Focus Topic
Room 318 - Session RE+AS-FrM

Materials Analysis and Characterization with Radiation

Moderators: Scott Dubowsky, University of Illinois at Urbana-Champaign, **Camilo Jaramillo-Correa**, Pennsylvania State University

8:20am **RE+AS-FrM-1 Characterization of Materials and Surfaces with Various Types of Radiation***, Zachary Robinson,* SUNY Brockport **INVITED**

In this talk, I will present an overview of various types of radiation that surface scientists use to characterize materials. In particular, I will focus on my recent work studying materials relevant for post-CMOS neuromorphic computational devices. These materials provide a potential path towards improved power and computational efficiency in a merged biomimetic and CMOS architecture. For this project, we characterized niobium oxides (NbO_2 and Nb_2O_5) using photons ranging from the infrared and visible for quantitative microscopy and spectroscopy through X-rays generated both at synchrotron sources and in benchtop instruments for diffraction and photoelectron spectroscopy. In particular, broad spectrum radiation allows us to study changes in composition and structure brought about in the as-deposited amorphous thin films upon annealing. The various sources of radiation inform our understanding of the material properties by providing information about the crystal structure, stoichiometry, impurities, crystallization percentage, and optical properties. Since undergraduate students were also an integral part of this work, I will describe our efforts in finding ways for them to be productive over a short (8-10 week) summer research internship when using relatively complicated instrumentation.

9:00am **RE+AS-FrM-3 High-Energy (MeV), Heavy Ion Irradiation of Chalcogenide Phase Change Thin Films***, David Adams, E. Lang, T. Clark, C. Sobczak, E. Scott, J. Custer,* Sandia National Labs*; T. Beechem,* Purdue University*; K. Hattar, M. Kalaswad, M. Rodriguez,* Sandia National Labs

Phase change thin films continue to attract interest for applications such as non-volatile electronic memory, sensors, and nanophotonics, because the material can be rapidly switched between amorphous and crystalline states accompanied by large changes in electronic and optical properties. In particular, the germanium-antimony-tellurium system remains a benchmark for studies wherein $Ge_2Sb_2Te_5$ has received much attention. In this study, we have evaluated the response of various chalcogenide thin films to high energy, heavy ion irradiation in order to provide additional insight into phase stability. Crystalline $Ge_2Sb_2Te_5$ and $(Ge_2Sb_2Te_5)_xC_{1-x}$ thin films were irradiated with 2.8 MeV Au ions to different doses and characterized subsequently using Raman spectroscopy, X-ray diffraction, thermoreflectance, Transmission Electron Microscopy and 4-point probe methods. Irradiation experiments were specifically designed to induce disorder while avoiding substantial incorporation of projectile species into films therein preserving the as-deposited film chemistry. Specifically, the 2.8 MeV energy was chosen so that ion range > film thickness which leads to less than 0.01 mol.% of projectile species residing within 100 nm-thick chalcogenide films, according to SRIM estimates. Irradiation led to significant changes in both structure and properties. Pure $Ge_2Sb_2Te_5$ thin films were amorphized when irradiated above a threshold dose, ~1e12 cm^{-2}. Both the thermodynamically-stable trigonal and metastable cubic phases were disordered with concomitant changes in electrical resistivity and thermal conductivity observed. Various $(Ge_2Sb_2Te_5)_xC_{1-x}$ [$0 < 1-x < 0.12$] thin films were also amorphized when irradiated above threshold doses. As with pure $Ge_2Sb_2Te_5$, structural disordering was accompanied by increased electrical resistivity and decreased thermal conductivity. Further insight into collisional-induced disordering was revealed by In-situ Ion Irradiation Transmission Electron Microscopy (I^3TEM). In-situ transmission electron microscopy and electron diffraction mapped the phase-specific response of films confirming different threshold doses for cubic and trigonal phases. Additionally, I^3TEM has been used to study the effects of increasing carbon concentration on phase stability and ion radiation response.

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9:20am **RE+AS-FrM-4 Multiwavelength Raman Microscopy Used to Characterize Surfaces for Plasma-Wall Interaction Study in Tokamaks***, Cedric Pardanaud, C. Martin, P. Roubin,* Aix-Marseille University / CNRS, France **INVITED**

Tokamaks aim to study the possibility to produce energy by exploiting hydrogen isotope nuclear reactions. The fusion plasma is confined magnetically, but a cold plasma is in interaction with the inner walls. This plasma-wall interaction, heterogeneous at the scale of the machine can lead to component melting, surface erosion, element migration inside the chamber, dust production, tritium retention, impurity contamination, mixed material formation… These walls were made of carbon in previous tokamaks (abandoned due to formation of thick hydrogenated rich deposits). Inner walls are now composed of tungsten and/or beryllium, presenting good thermal and chemical properties.

I will demonstrate that multiwavelength Raman microscopy is a suitable technique for *post mortem* analyses of tokamak plasma facing components [1]. It delivers an information related to chemistry, which is complementary to the classically used Thermal Desorption Spectroscopy (TDS) and Ion Beam Analyses (IBA). I will focus on analyses of both laboratory made samples and samples collected inside some tokamaks.

I will illustrate my talk by presenting first how Raman, IBA and TDS of amorphous carbon deposits found inside the Tore Supra tokamak compare [2, 3]. Then, I will focus on the defective beryllium system in the JET tokamak [4- 6] and formation of beryllium hydrides [7]. I will finish by presenting some results about tungsten oxides [8, 9].

[1] C. Pardanaud, et al.

Raman spectroscopy and applications, chapter 1, edited by Khan Maaz, Intech (2017)

[2] C. Pardanaud, et al.

Diamond and Related Materials 34 (2013), 100-104

[3] C. Pardanaud et al.

Thin solid films 581 (2015), 92

[4] M.I. Rusu, et al.

Nuclear Fusion 57 (2017), 076035

[5] M. Kumar, et al.

Nuclear Materials and Energy 17 (2018) 295–301

[6] C. Pardanaud, et al.

Physica Scripta 96 (2021) 124031

[7] C. Pardanaud, et al.

Journal of Physics: Condensed Matter, 27 (2015) 475401

[8] Y Addab, et al.

Phys. Scr. T167 (2016), 014036

[9] C. Pardanaud, et al.

Nuclear fusion 60 (2020) 086004

10:00am **RE+AS-FrM-6 In Situ Optical Characterization of High Temperature Defect Kinetics in Mixed-Conducting Oxide Films***, Nicola Perry,* University of Illinois, Urbana-Champaign **INVITED**

Thin films represent model platforms for the evaluation of new materials for intermediate-to-high temperature electrochemical devices, including electrolyzers and fuel cells. We focus on candidate perovskite-structured mixed- or triple-conducting electrode materials where the bulk mobile ions are oxide ions or protons, and the surface reactions of interest are oxygen reduction/evolution, hydration, and combinations of the two in the form of hydrogenation. Central to their performance is the dynamic defect behavior at temperatures from 300 – 700 °C, particularly the kinetics of defect transport (diffusivities, D) and interfacial reactions (surface exchange coefficients, k). Conventionally, electrochemical or electrical methods that place precious metal current collectors on the electrode surface are applied to evaluate the ion fluxes; however, we have demonstrated that the presence of such metals actually alters the measured parameters (e.g., k) aiming to quantify surface defect kinetics and bulk defect equilibria. Instead, we exploit the coupling between defect concentrations and UV-vis optical absorption to provide contact-free, continuous, and *in situ* evaluation of the films' defect kinetics via isothermal optical transmission relaxation (OTR) responses to step changes in gas-phase chemical potentials. This method has enabled us to observe the evolution of oxygen exchange kinetics during crystallization, where the onset of crystallization corresponds to the initiation of "breathing" by the films, and we demonstrate orders-of-magnitude enhancements in oxygen surface exchange kinetics in films grown by the low-temperature crystallization method vs. conventional high-temperature-grown films. We also have developed a two-dimensional OTR technique to observe defect kinetics across all regions of 1 x 1 cm^2 films simultaneously, of use in cases with spatially heterogeneous responses. We applied this 2D-OTR to model

metal | perovskite heterointerfaces to further underline the catalytic effect of metal current collectors on mixed conductors for oxygen exchange and show the surprisingly large spatial extent of that effect away from the interface. Most recently we demonstrated the ability of the OTR method to evaluate hydrogenation kinetics in proton-conducting thin films for the first time, enabling quantitative comparison of candidate new triple-conductor electrodes. Origins and applications of the optical absorption-defect concentration relationship will be discussed.

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10:40am **RE+AS-FrM-8 Exploring the Effects of Radiation on Planetary Surfaces through the Analysis of Experimental Analogs and Returned Samples from the Moon and Asteroids***, **Michelle Thompson,*** Purdue University **INVITED**

Materials on the surfaces of airless bodies are continually exposed to the harsh environment of interplanetary space through a process known as space weathering. Space weathering alters the microstructure, chemistry, and optical properties of grains on the surfaces of bodies like the Moon and asteroids. This process is driven by two primary mechanisms: hypervelocity dust impacts, and the interaction of surface material with energetic particles from the solar wind. We can investigate the effects of these processes by performing laboratory experiments and by analyzing materials collected by sample return missions. Samples returned from the Moon via the Apollo missions and from near-Earth asteroids Itokawa and Ryugu by the Hayabusa and Hayabusa2 missions, respectively, have demonstrated that the microstructural and chemical characteristics resulting from solar wind irradiation are complex. Solar wind ions work to vesiculate and amorphize the outer rims (<100 nm) of grains on the surfaces of these bodies, and the depth and degree of this amorphization (e.g., presence and distribution of nanocrystalline domains) varies significantly in relation to the grain composition and its exposure timescale to interplanetary space. Similarly, solar wind radiation can cause preferential sputtering and redeposition, radiation-enhanced diffusion, and/or recoil mixing which can develop atomic-scale chemical heterogeneities on grain rims. We observe such multilayer rims with segregated cation/anion structures in solar wind irradiated silicate minerals from both the Moon and asteroids. In addition, high-energy Fe-group nuclei from solar flares create trails of ionization damage called solar flare tracks. The concentration of these tracks can be used as a proxy for the exposure timescale a grain has experienced on an airless planetary surface. We can further understand these microstructural and chemical characteristics by conducting experiments in the laboratory using ions and energies appropriate for the solar wind. This work has revealed fundamental discrepancies between experimental and natural space weathering, including the rate at which samples amorphize and the development of vesiculated textures. Such results suggest that ion flux, grain composition, and microstructure play a significant role in the development of atomic-scale irradiation characteristics in returned samples. A combined approach of sample analysis and experiments enables us to investigate how solar wind irradiation alters materials throughout the solar system which is critical for building an understanding of the evolution of planetary surfaces through time.

11:20am **RE+AS-FrM-10 Ground-Based Space Environmental Testing of Materials and Components for Spacecraft Mission Assurance***, **Sven Bilén,*** *J. McTernan, C. Zawaski,* The Pennsylvania State University **INVITED**

Space presents an extreme environment for spacecraft materials and systems. For example, in the low-Earth-orbit (LEO) space environment, materials and structures are exposed to high vacuum (10^{-4}–10^{-5} Pa), thermal cycling (−150 to 150 °C), ultraviolet light (100–200 nm), space radiation, potential surface impact from micrometeorites and orbital debris (MM/OD), and atomic oxygen (AO). Materials showing promise for future use in space must be assessed and evaluated for their reaction to exposure to the space environment before being baselined for inclusion into critical space infrastructure. Such evaluation can occur via *in-situ* experiments on space vehicles, but such testing is costly, of limited throughput, experiences significant time lags, and provides less specific information to identify points of failure. Ground-based testing of materials and systems under simulated space environmental conditions can address these limitations. Indeed, space hardware, depending on mission assurance requirements, goes through a battery of testing in space-simulation facilities, such as thermal–vacuum, vibration, acoustic, EMI, etc.

Penn State has facilities for full-scale testing of small spacecraft (e.g., 3-U CubeSats) in a LEO–type environment, individual components and materials, spacecraft charging mitigation schemes, and spacecraft–plasma interactions. This facility is comprised of a vacuum chamber capable of thermal cycling through a range of realistic temperatures, a plasma source capable of producing streaming ions (1–4 eV) and low-energy electrons (~0.1 eV), shake tables, anechoic chambers (for EMI testing), outgassing assessment, and radiation (via its Radiation Science and Engineering Center and Breazeale Reactor). This facility is also capable of testing individual components, whose operation is dependent on the plasma environment (e.g., thermionic cathodes, field emitters, plasma diagnostic tools, *in-situ* diagnostics such as Langmuir probes, etc.). System-wide tests that combine multiple tests are beneficial as results can be interdependent. For example, thermal cycling under vacuum that causes outgassing could contaminate sensors or other diagnostic tools such as Langmuir probes. We are working to add atomic-oxygen exposure, increased thermal range, high-energy electron exposure, UV exposure, and simulated MM/OD damage (via short, focused laser pulses) to our capabilities.

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