Actinides and Rare Earths Focus Topic
Room On Demand - Session AC-Contributed On Demand

Actinides and Rare Earths Contributed On Demand Session

AC-Contributed On Demand-1 Characterization of Uranium Oxide Corrosion Using a Microfluidic Electrochemical Cell*, Jennifer Yao*, *E. Buck, S. Tripathi, N. Lahiri, E. Ilton, S. Riechers, D. Reilly,* Pacific Northwest National Laboratory*; S. Chatterjee,* TerraPower LLC*; X. Yu,* Pacific Northwest National Laboratory

Study of UO$_2$ corrosion is important in understanding the spent nuclear fuel (SNF) disposal. To address the challenges in investigating large amount of SNF materials, we developed a new approach to incorporate UO$_2$ powder onto the working electrode of a microfluidic electrochemical cell, aka the system for analysis at the vacuum liquid interface (SALVI) E-cell, to facilitate UO$_2$corrosion study at the microscale. Instead of using bulk spent fuel pieces as working electrode at the macroscale, UO$_2$ powder was mixed withpolyvinylidene fluoride (PVDF) and carbon black to form the working electrode with mm dimension and included into the SALVI E-cell. The UO$_2$ powder electrode went through electrochemical corrosion in 0.1 M NaClO$_4$ (pH=9.5) aqueous electrolyte using the electrochemical station. Multimodal imaging analysis, including in situ scanning electron microscope (SEM) coupled withEnergy-dispersive X-ray spectroscopy (EDS) as well as ex situ transmission electron microscopy (TEM) and atomic force microscopy (AFM), was applied to reveal the morphological change, oxidation layer distribution, and topographical information of UO$_2$ before and after corrosion.In addition, X-ray photoelectron spectroscopy (XPS) was used to determine the oxidation state of the UO$_2$ electrode surface disassembled from the microfluidic E-cell after anodic oxidation. Our results demonstrate a promising approach to characterizing UO$_2$ corrosion at the microscale using multimodal imaging. Particularly, in situ SEM imaging and EDS mapping allow direct observation of corrosion in liquid. This new approach is useful in studying the interaction between geological repository environments (e.g., groundwater) and SNF to validate and improve the Fuel Matrix Dissolution Model.

AC-Contributed On Demand-4 Observation of Multiple Dirac States in a Magnetic Topological Material EuMg$_2$Bi$_2$*, Firoza Kabir,* University of Central Fllorida*; M. Hosen,* University of Central Florida*; F. Cheenicode-Kabeer, A. Aperis,* Uppsala University, Sweden*; X. Ding,* Idaho National Laboratory*; G. Dhakal, K. Dimitri, C. Sims, S. Regmi, L. Persaud,* University of Central Florida*; K. Gofryk,* Idaho National Laboratory*; P. M. Oppeneer,* Uppsala University, Sweden*; D. Kaczorowski,* Polish Academy of Sciences, Poland*; M. Neupane,* University of Central Florida

Initiated by the discovery of topological insulators, topologically non-trivial materials, more specifically topological semimetals and metals have emerged as new frontiers in the field of quantum materials. In this work, we perform a systematic measurement of EuMg$_2$Bi$_2$, a compound with antiferromagnetic transition temperature at 6.7 K, observed via electrical resistivity, magnetization and specific heat capacity measurements. By utilizing angle-resolved photo emission spectroscopy in concurrence with first-principles calculations, we observe Dirac cones at the corner and the zone center of the Brillouin zone. From our experimental data, multiple Dirac states at Gamma and K points are observed, where the Dirac nodes are located at different energy positions from the Fermi level. Our experimental investigations of detailed electronic structure as well as transport measurements of EuMg$_2$Bi$_2$ suggest that it could potentially provide a platform to study the interplay between topology and magnetism.

AC-Contributed On Demand-10 Tuning Formation of Large Uranium Oxide Cluster Ions from the Surface of Depleted Uranium Under Static Bombardment Using Ga+ During ToF-SIMS*, Shohini Sen-Britain,* *A. Nelson,* Lawrence Livermore National Laboratory

Laser ablation (LA) mass spectrometric and static time-of-flight secondary ion mass spectrometry (ToF-SIMS) of metals produces similar ion distributions of large M$_x$O$_y$+ ions due to gas phase recombination reactions of small M$_x$O$_y$+ ions in the gaseous cloud formed during LA, or selvedge formed in static ToF-SIMS during ion sputtering [1].Specifically in the case of depleted uranium (DU), an expanded understanding of these recombination reactions due to ion sputtering under vacuum and in the presence of oxygen or in atmosphere can further our understanding of nuclear forensics and debris analysis [2, 3].We describe here precise control of large U$_x$+ and U$_x$O$_y$+ ion formation as a function of static ToF-SIMS Ga+ primary ion bombardment parameters.

We report the observation of large U$_x$+, U$_x$O$_y$+, and U$_x$O$_y$H$_z$+ ions (x = 1-14) during 25 keV Ga+ analysis of the DU surface oxide and sputter cleaned metal surface during primary ion bombardment under static conditions.These ions are large enough to suggest that intact cleavage from the DU surface is energetically prohibitive.Rather, collision cascade theory suggests that these large species are the product of post-ionization reactions in the selvedge of DU [4].Here, we report variation in the power law fits of secondary ion counts of U$_x$+, U$_x$O$_y$+ and U$_x$O$_y$H$_z$+ ions as a function of x (x=1-7) due to sputtering 2 nm into the DU surface oxide at ion current densities varying from 20 pA to 20 nA.We also report the variation of power law fits to secondary ion counts of U$_x$+ ions as a function of x (x = 1-14) due to tuning of instrument cycle time.Finally, we observe different rates of conversion of U$_x$+ ions to U$_x$O$_y$+ as a function of x during in-chamber surface oxidation under UHV conditions from residual moisture during static spectrum collection over a period of 3000 s.

Future applications of this work include monitoring of ion formation and variation in recombination reactions in the presence of oxygen, water vapor, and temperature.This work was also performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under contract DE-AC52-07NA27344.

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AC-Contributed On Demand-13 Hybridization Effect on the X-Ray Absorption Spectra for Actinide Materials*, W. Chiu,* University of California at Davis*; R. Tutchton,* Los Alamos National Laboratory*; G. Resta,* University of California at Davis*; T. Lin,* Rutgers University*; E. Bauer, F. Ronning,* Los Alamos National Laboratory*; R. Scalettar,* University of California at Davis*;* ***Jian-Xin Zhu,*** Los Alamos National Laboratory

Studying the local moment and 5*f*-electron occupations sheds insight into the electronic behavior in actinide materials. X-ray absorption spectroscopy (XAS) has been a powerful tool to reveal the valence electronic structure when assisted with theoretical calculations. In this work, we employed the DFT+Gutzwiller rotationally-invariant slave boson method to obtain the local Hamiltonian of the single-impurity Anderson model (SIAM), and used exact diagonalization (ED) method to calculate the XAS spectra from the model. An in-house built computational code was developed for the ED method. By applying this technique to the recently discovered 5*f*-electron topological Kondo insulator plutonium tetraboride (PuB$_4$), we were able to determine the signature of 5*f*-electronic correlation effects in the theoretical X-ray spectra. We found that the Pu 5*f*-6*d*hybridization effect provides an extra channel to mix the *j*=5/2 and 7/2 orbitals in the 5*f* valence. As a consequence, the resultant electron occupation number and spin-orbit coupling strength deviate from the intermediate coupling regime. We have also applied this approach to the δ-phase of Pu.

AC-Contributed On Demand-16 EXAFS as a Probe of Actinide Oxide Formation in the Tender X-Ray Regime*, James Tobin,* University of Wisconsin-Oshkosh*; S. Nowak,* SLAC National Accelerator Laboratory*; S. Yu,* Lawrence Livermore National Lab*; R. Alonso-Mori, T. Kroll, D. Nordlund, T. Weng, D. Sokaras,* SLAC National Accelerator Laboratory

Recent developments in Resonant Inelastic X-Ray Scattering Experiments have vastly improved the resolution in X-ray Spectroscopies such as X-ray Absorption.However, the use of Tender X-rays (~3keV) and the reactivity of actinides such as uranium have given rise to new questions about bulk and surface sensitivity, what constitutes a surface and how to characterize the formation of an oxide.It will be shown that the *in situ* electron scattering features that are part of the RIXS spectrum can be used to quantify the uranium oxide formation and determine cleanliness, independently of the near edge features that provide information about the unoccupied electronic density of states.

*J. G. Tobin,S. Nowak, S.-W. Yu, R. Alonso-Mori, T. Kroll, D. Nordlung, T.-C. Weng, D. Sokaras, "EXAFS as a Probe of Actinide Oxide Formation in the Tender X-Ray Regime," Surface Science **698**, 121607 (2020) https://doi.org/10.1016/j.susc.2020.121607.

AC-Contributed On Demand-19 Detection of Covalency in Pu(IV) Materials: Spectroscopic and Computational Tools*, **Bianca Schacherl,*** Karlsruhe Institute of Technology (KIT), Institute for Nuclear Waste Disposal (INE), Germany*; P. Bagus,* Center for Advanced Scientific Computing and Modeling (CASCaM) Department of Chemistry University of North Texas*; A. Beck,* Karlsruhe Institute of Technology (KIT), Institute for Nuclear Waste Disposal (INE), Germany*; M. Tagliavini,* Heidelberg University, Institute for theoretical physics, Germany*; M. Trumm,* Karlsruhe Institute of Technology (KIT), Institute for Nuclear Waste Disposal (INE), Germany*; M. Haverkort,* Heidelberg University, Institute for theoretical physics, Germany*; T. Vitova,* Karlsruhe Institute of Technology (KIT), Institute for Nuclear Waste Disposal (INE), Germany

Plutonium has a complex electronic structure, where the role of the 5f orbitals in chemical bonding and the level of covalency has not been understood in detail and is a very active field of research. Resonant inelastic X–ray scattering (RIXS) is a valuable tool and can lead to a deeper understanding of the electronic structure of plutonium materials.[1,2] Also high-energy resolution X-ray absorption near edge structure (HR-XANES) has proven powerful in plutonium speciation studies.[3,4]

Relativistic quantum chemical computations for the Pu^{4+} ion and the PuO_2 compound were performed. We will discuss four computational measures of covalency of the Pu 5f orbitals. The $PuM_{4,5}$-edge HR-XANES and Pu $M_{4,5}$-edge core-to-core 3d4f RIXS on PuO_2 and other Pu^{4+} compounds under various experimental conditions were conducted at the INE and ACT beamlines at the Karlsruhe research accelerator (KARA) at KIT in Karlsruhe Germany.[5,6]

We found that for PuO_2 the Pu 5f covalent mixing with O valence orbitals overall is relatively small. It is the largest in the 5f(7/2) a_{2u} orbital with the highest orbital energy. The analysis of the Pu $M_{4,5}$-edge HR-XANES in combination with the calculated results, showed that the spectra can not be described with a simple one electron transition between individual 3d and 5f orbitals applying the dipole selection rule. There is considerable amount of redistribution of 5f electrons involved in both the Pu M_4-edge and the Pu M_5-edge absorption processes, i.e. shake-up excitations (J = 5f(5/2) to 5f(7/2)) take place. From the comparison of the computational results with the HR-XANES spectrum, it was found that the second peak in the Pu M_4-edge and the shoulder feature in the Pu M_5-edge spectrum are probing the 5f(7/2) a_{2u} orbital and are therefore expected to be sensitive to bond variations. It will be discussed how the different interatomic interactions affect the Pu $M_{4,5}$–edge core-to-core 3d4f RIXS map of PuO_2. The here presented spectroscopic and theoretical tools will help to advance the understanding of the electronic structure of Pu materials.

Acknowledgement: We acknowledge funding from the European Research Council (ERC) (grant agreement n° 101003292). We thank the Institute for Beam Physics and Technology (IBPT), KIT for the operation of the storage ring, the KARA.

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AC-Contributed On Demand-22 Interrogating the Surface Chemistry of Nanoscale Uranium Oxides*, **Liane Moreau**, H. Johnson,* Washington State University

A thorough exploration of uranium oxide surface chemistry is critical to understanding UO_2 corrosion in the nuclear fuel cycle and potential for catalytic reactivity. Nanoscale uranium oxides provide a template through which to study the surface chemistry of actinide materials. Synthesis of actinide nanoparticles, however, has not been empirically well-developed, making investigations into their size- and surface-dependent properties relatively unexplored. Synthesis of nanoparticle constructs in the range between 2 and 100 nm utilizing colloidal and template-based approaches will be discussed, along with their structural attributes on the atomic and nanometer length scales. Characterization of such particles, in a manner which resolves surface chemistry, becomes challenging using conventional characterization techniques. In particular, the high curvature of nanoparticle surfaces and complex solution interface chemistry makes surface techniques appropriate to thin film systems insufficient structural probes. We overcome the presented challenge through focusing on the study of ultrasmall nanoparticles, which present particularly high surface area to volume ratios. The high percentage of surface atoms makes bulk characterization techniques such as X-ray absorption spectroscopy (XAFS) become surface-sensitive probes, enabling us to resolve surface structure in atomistic detail. New models will be presented that provide an indication of elemental speciation of surface atoms and sites for surface ligand binding, through combining geometric models with structural parameters extracted from XAFS. Results obtained through this work are broadly applicable towards resolving nanoparticle surface chemistry and provide foundational methodology towards exploring the nanoscale properties of actinide oxides.

AC-Contributed On Demand-25 Broadening of the XPS Spectra of U Oxides*, C. Nelin,* Consultant*; **Paul S. Bagus**,* University of North Texas

It is common to attempt to improve the energy resolution of XPS in order to obtain more information about the electronic structure of the system studied. However, it may not be possible to improve the resolution because unresolved final states features are present which lead to broad features. These unresolved features may arise from closely spaced multiplets for the angular momentum coupling of the core and valence open shell electrons. They may also arise from excitations to higher lying vibrational levels for the final ionic states which may be especially important when bond distances for the core-ionized states are very different from those for the initial state; see, for example, Ref. [1]. When the energy separation of the final states are less than or comparable to the lifetime of the core-hole, it will not be possible to resolve the states and there will only be a broadening, often quite significant, for the observed peak composed of these unresolved features. Thus, for example, the $U(5f_{7/2})$ peak of UO_2 has a FWHM of 1.4 eV although the instrumental resolution was 0.3 eV. [2] Similar large FWHM have been observed for U in different oxidation states. [3] In order to be able to relate the widths of these broadened features to the chemical and physical interactions in the system, it is necessary to understand the separate contributions of the multiplet splittings and the vibrational excitations. It has been shown that, for U(IV) 4f XPS in UO_2, the contributions of the multiplet splitting and the vibrational excitations are comparable, each contributing ~0.5 eV to the FWHM. [2] In the present work, the contributions of these mechanisms are examined for U(V) and U(VI) oxidation states. In addition, the broadening is examined for the XPS of different core levels where the relative importance of multiplet and vibrational broadening is different from that for the U(4f) XPS. The theoretical predictions for these different parameters can be validated paving the way to extract chemical information from the measured FWHM. [4] The theoretical framework for these predictions is based on wavefunctions for embedded cluster models of the oxide.

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AC-Contributed On Demand-28 XPS, UPS Study of Pure and Alloyed U Hydrides*, **Oleksandra Koloskova**, E. Tereshina-Chitrova, M. Paukov,* Charles University, Prague, Czech Republic*; T. Gouder,* European Commission, JRC. Institute for Transuranium Elements, Germany*; J. Kolorenč,* Institute of Physics, Czech Academy of Sciences, Czechia*; L. Havela,* Charles University, Prague, Czech Republic

Besides the fundamental importance, uranium hydride has considerable relevance for nuclear energy and devices, which motivates continuous effort to understand its formation and properties [1], in particular its electronic structure.

Photoelectron Spectroscopy is a method which directly probes the electronic structure. However, surface oxidation is an issue, which we overcome using the strategy of thin film synthesis. Surprisingly, it became possible to stabilize the UH_2 phase (non-existent as a bulk) in a thin film form [2].

XPS was first used for analysis of the samples quality. One of the difficulties is that the H-1*s* line is a part of the U valence band, hence it cannot be used to quantify the H concentration. We followed an empirical approach based on gradual increase of H_2 partial pressure in the working gas (Ar) while monitoring variations of the U-4*f* core-level spectra until saturation is reached [3].

Details of electronic states in the vicinity of the Fermi level are explored by UPS, surpassing XPS both by intensity and energy resolution (about 70 meV). Comparison of UPS spectra of U and UH_3 revealed that the spectra are similar just at the Fermi level (if properly normalized), the maximum for UH_3 is slightly displaced from the Fermi level to ≈140 meV binding energy. The U metal has DOS increasing up to the Fermi level and the Fermi-Dirac cutoff forms the maximum at 90 meV. Another feature of UH_3 (UH_2 is very similar) is the broad shoulder at 0.5 eV (ascribed to 5*f* multiplet from GGA+U calculations). Details of the DOS shape resist to a quantitative description using DFT calculations [1]. However, spectral density obtained from DMFT calculations captures both the maximum just below E_F and the 0.5 eV shoulder. This stresses the importance of electron correlations for the description of U hydrides.

As the XPS spectra of UH_3 and UH_2 are so similar, it is hard to distinguish which species was deposited. Magnetization measurements turned particularly useful. While both species are ferromagnets, their T_C values are different (165 K for UH_3 and 120 K for UH_2) [2]. Using the $M(T)$ dependence, we can assess the phase composition (pure UH_2, UH_3 or mixed-phase) of the samples.

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AC-Contributed On Demand-31 Development of the High-T CDW in *RE*Pt$_2$Si$_2$, ***Volodymyr Buturlim,*** *P. Dolezal,* Charles University, Czechia*; E. Duverger-Nedellec,* Sorbonne Université, France*; A. Andreev,* Institute of Physics of the Czech Academy of Sciences, Czechia*; M. Falkowski,* Institute of Molecular Physics, Poland*; K. Gofryk,* Idaho National Laboratory*; L. Havela,* Charles University, Czech Republic

RPt2Si2 compounds form in the tetragonal non-centrosymmetric CaBe2Ge2 structure type both for lanthanides and actinides. The unifying pattern seems to be the CDW state, coexisting either with magnetic order or conventional superconductivity for rare earths. We identified that the generally recognized low-temperature CDW state setting in the first order transition is probably generally preceded by another CDW state, entered at a critical temperature close to 300 K via the second order transition, leaving less dramatic fingerprints in physical properties [1,2]. Here we will present results obtained using single-crystal XRD and high-precision dilatometry on selected single crystals along a- and c-axis. As one of the two Pt-Si slabs is most likely responsible for the instability towards the CDW formation, we believe that at least phenomenologically similar behavior of UPt2Si2 [3] indicates localization of the U-5f states in the U analogue. Possible involvement of the 5f states in bonding would arguably change the cohesion characteristics and the analogy of U with rare earths would be lost.

This work was supported by the Czech Science Foundation under the grants No. 18-02344S and 21-09766S.

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AC-Contributed On Demand-34 Electronic Structure and Spectroscopy of UH3 from the LDA+DMFT Perspective*, B. Chatterjee,* ***Jindrich Kolorenc,*** Institute of Physics, Czech Academy of Sciences, Prague, Czechia

We employ material-specific dynamical mean-field theory (LDA+DMFT) to investigate the electronic structure of UH3. We use the computed electronic structure to model valence-band photoemission and x-ray absorption spectra. We investigate the sensitivity of our results to the Coulomb U parameter that enters the LDA+DMFT method, since there is a large spread of recommended values reported in the literature, ranging from 0.5 eV [1] to more than 5 eV [2].

To understand the origin of certain spectral features, we analyze how the many-body eigenstates of the DMFT auxiliary impurity model evolve when the hybridization between the uranium 5f states and the hydrogen 1s states is ramped up from zero (ionic 5f3 model) to the realistic value. This theoretical experiment enables us to illustrate how some of the photoemission features relate to the final-state multiplets of the 5f3 to 5f2 transition. The LDA+DMFT method thus provides a solid ground for the earlier empirical interpretation of the photoemission spectra in terms of the 5f atomic multiplets [3].

This work was supported by the Czech Science Foundation under the grant No. 21-09766S.

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