

Thursday Evening Poster Sessions, October 25, 2018

Applied Surface Science Division Room Hall B - Session AS-ThP

Applied Surface Science Division Poster Session

AS-ThP-1 Toward an Improved Understanding of the role of soil organic matter in NO_y cycling through Investigation of Heterogeneous Reactions with NO₂⁺, *R Hansen*, Indiana University; **Mark Engelhard**, Pacific Northwest National Laboratory; *J Raff*, Indiana University

Molecular level investigation of compositional changes due to heterogeneous reactions of nitrogen oxides (NO_x, NO_y) with soil organic matter (SOM) is important to develop a fundamental scientific understanding of the soil/atmosphere interface. In addition, interactions between NO_x reservoir species and SOM play a more important role in NO_x and NO_y recycling than previously realized. Despite the importance of soil to the global terrestrial-atmospheric cycling of nitrogen, interactions of N₂O₅ with SOM are not well understood. Uncertainty in these processes is problematic because it means that NO_y is not properly represented in the Earth-systems models used for prediction and regulation. The ultimate objective of this study is to investigate the production of NO_y from the reaction of N₂O₅ with SOM and elucidate the mechanisms that return NO_y back to the atmosphere, where NO_y can contribute to aerosol and O₃ formation. In the initial phase of this study, we reacted SOM standards with NO₂⁺ (produced from concentrated HNO₃), which is an intermediate in the heterogeneous reaction of N₂O₅ with SOM. We then characterized these reaction products using X-ray photoelectron spectroscopy (XPS). XPS was used to measure the nitrogen chemistry before and after reaction of SOM with NO₂⁺. These results will be discussed along with the pros and cons using XPS to characterize SOM chemistry.

AS-ThP-2 Measuring the Damage Depth and Recovery of PEMA Thin Films using Multiple Technique Analysis, *William Sgammato*, *R Simpson*, Thermo Fisher Scientific, UK

Sputtering polymer surfaces with monatomic Ar ions is known to induce changes in the polymer chemistry. PEMA in particular, is a "self-sealing" polymer type which means during Ar⁺ sputtering cross-linking occurs. The advantage of using Ar cluster ions is that such chemical changes are not induced. It has also been demonstrated that Ar_n⁺ ions can be used to remove the damage layer produced by Ar⁺ ions, thus recovering the surface chemistry. In this investigation this ability to recover the polymer surface is used to measure the relative thickness of the damage layer produced by Ar⁺ ions. To facilitate this other techniques were used for film thickness measurement and calibration. AFM was used to accurately measure the thickness of the PEMA film, this data was then used to calibrate a separate scale that was used to estimate the thickness based on the colour of the surface. As the colour of the surface is dependent only on the refractive index of the material, the angle of incidence of the incoming light and the thickness of the film, the measurements can produce a high accuracy thickness estimate to within ± 10 nm. The estimate of the original film thickness, made using the film colour, ~ 400 nm was in good agreement with the average thickness measured using AFM 398 nm.

The results of this investigation showed that the relative depth of damage induced by a 3 keV Ar⁺ ion beam in a PEMA thin film was 245 ± 10 nm. Film thickness measurements were also used to calibrate Ar⁺ and Ar_n⁺ etch rates which were calculated as 0.94 and 2.60 nm/s respectively. The low Ar⁺ etch rate was an indication of sample cross-linking during etching.

AS-ThP-3 Determination of Band Offsets in Semiconductor Heterostructures (2D/3D) by using XPS, *Mohamed Hedhili*, King Abdullah University of Science and Technology (KAUST), Core Labs, Saudi Arabia; *M Tangi*, *P Mishra*, *T Ng*, *B Janjua*, *C Tseng*, Photonics Laboratory, King Abdullah University of Science and Technology (KAUST), Saudi Arabia; *D Anjum*, King Abdullah University of Science and Technology (KAUST), Core Labs, Saudi Arabia; *M Alias*, Photonics Laboratory, King Abdullah University of Science and Technology (KAUST), Saudi Arabia; *N Wei*, King Abdullah University of Science and Technology (KAUST), Core Labs, Saudi Arabia; *L Li*, Physical Sciences and Engineering Division, King Abdullah University of Science and Technology (KAUST), Saudi Arabia; *B Ooi*, Photonics Laboratory, King Abdullah University of Science and Technology (KAUST), Saudi Arabia

Heterojunctions are generally formed at the interface of different energy-gap semiconductor materials due to their mismatching electron energies in the valence bands. The values of valence-band mismatches or offsets hold a great significance for the optoelectronics applications and hence their

accurate determination is of paramount importance. High-resolution X-ray photoemission spectroscopy (HR-XPS) is proven to be a powerful way of measuring the valence band offsets in such semiconductor heterojunctions. In this report, we extend HR-XPS studies for such measurements to 2D/3D types of heterojunctions. These 2D/3D junctions are synthesized by combining 3D semiconductors (e.g. GaN or InAlN) with 2D semiconductors (e.g. WSe₂ or MoS₂). By performing an elaborate XPS analysis, we are able to show the type of heterojunctions formed by these semiconductor materials. For instance, we observed that the heterojunction of GaN with either MoS₂ or WSe₂ is of "Type-II". Whereas, the junction of InAlN with MoS₂ is "type-I". The presented HR-XPS results are both supported and corroborated by performing the analysis of these samples with other techniques including atomic force microscopy, scanning transmission electron microscopy, micro-Raman, absorbance, and microphotoluminescence. The band alignment parameters determined here provide a route toward the integration of group III nitride semiconducting materials with transition metal dichalcogenides (TMDs) for designing and modeling of their heterojunction-based electronic and optoelectronic devices.

AS-ThP-4 Multi-technique Characterization of Nanowire-based Catalysts and Electrodes, *Sarah Zaccarine*, *C Ngo*, Colorado School of Mines; *S Shulda*, *S Mauer*, *S Alia*, *K Neyerlin*, *B Pivovar*, National Renewable Energy Laboratory; *S Pylipenko*, Colorado School of Mines

In response to the increasing anthropogenic impact on the environment, it is vital to implement sustainable solutions to meet global energy demands. Polymer electrolyte membrane fuel cells (PEMFCs) are a promising option but the sluggish oxygen reduction reaction at the cathode leads to issues with cost and efficiency. Pt nanoparticles supported on high surface area carbon (Pt/HSC) are commonly used but suffer performance losses and do not meet Department of Energy targets for durability or cost. Extended surface nanostructures are a promising alternative as they show improved specific activity and durability. We have developed extended surface nanowire-based platinum nickel catalysts with durability, mass activity, and specific activity superior to Pt/HSC. Since the catalyst functions differently under altered conditions, it is crucial to study the catalyst at all stages as it transitions from a powder to a membrane electrode assembly (MEA), which requires a multi-technique approach.

The catalyst was studied as a powder, ink, fresh MEA, and tested MEA to determine the changes that occur as the catalyst is integrated into a full MEA. Several spectroscopy and microscopy techniques were utilized to address all relevant length scales (from atomic to micrometers). First, the catalyst was investigated using a combination of extended x-ray absorption fine structure (EXAFS) spectroscopy, x-ray photoelectron spectroscopy (XPS), scanning transmission electron microscopy (STEM) with energy dispersive x-ray spectroscopy (EDS) hypermapping, and atom probe tomography (APT) to obtain detailed information about distribution of platinum and nickel, discerning differences between surface and bulk speciation at nanometer and sub-nanometer scale. This detailed information about surface speciation was then used to better understand oxygen adsorption behavior of these catalysts, investigated using near-ambient pressure XPS (nAP-XPS). Second, evolution of the catalyst and catalyst-ionomer interface when incorporated in an electrode were examined with STEM/EDS and x-ray tomography. These studies offer invaluable insight into structure-performance relationships of the nanowire-based catalysts and development of efficient electrodes.

AS-ThP-5 Detailed Peak-Fitting Analysis of the Photoemission Spectra of the Early Oxidation Stages of Cobalt Thin Films, *Dagoberto Cabrera-German*, Universidad de Sonora, México; *O Cortazar-Martinez*, *G Vázquez*, *J Torres-Ochoa*, Cinvestav-Unidad Queretaro, Mexico; *A Herrera-Gomez*, CINVESTAV-Unidad Queretaro, Mexico

The quantitative analysis of the X-ray photoemission spectra (XPS) of cobalt and its compounds is a challenge; because an intricate multiplet structure, intense background, and in occasions strong overlapping Auger lines^[1] can lead to inaccurate results. These intricacies have been studied before,^[2,3] but the apparent asymmetry of the Co 2p photoemission line in metallic cobalt and the early oxidation stages of the metallic surface have not been treated in detail via state-of-the-art peak-fitting methods.^[4-6]

In this work, metallic cobalt thin films were prepared using thermal evaporation and the early oxidation of the films was done by exposing the surface to high purity molecular oxygen at high vacuum. The thin film preparation and the XPS measurements were done without the loss of vacuum, ensuring no ambient surface contamination.

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The spectra of the metallic films show asymmetric line-shapes and the reproduction of the background trend on both sides of the spectra requires an individual assignment of Shirley backgrounds to each peak present; a task that was accomplished with the use of the Shirley-Vegh-Salvi-Castle (SVSC) background under the *active background* approach.^[4-6] Additionally, by measuring the energy loss spectra of the films, it is possible to determine the presence of satellite peaks that are not related to an electron energy loss process.

The spectra of the early oxidation stages of cobalt show several satellite peaks corresponding to CoO with a chemical composition of $\text{CoO}_{1.00\pm 0.15}$ throughout increasing oxygen exposures. This suggests that the set of peak parameters employed to resolve the metallic and oxide photoemission signals, can, in fact, be employed to accurately assess the peak intensities required for a proper quantitative analysis.

The close experimental data reproduction and the coherency between chemical composition and the shape of the photoemission spectra strongly indicate that the use of the *active background*^[6] approach is paramount when performing a detailed peak-fitting analysis of the photoemission spectra of metallic cobalt and its oxides.

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AS-ThP-6 Characterization of Laser-Treated Ti-6Al-4V-Surfaces, Harry Meyer, D Leonard, A Sabau, Oak Ridge National Laboratory

Lightweight Al, Mg, and Ti alloys are ubiquitous in aerospace and automotive applications. When these alloy materials are joined, either to similar or dissimilar materials, the chemical and physical state of the surface determines the quality of the bond. Methods for cleaning (chemical) and texturing (physical) metal alloy surfaces prior to joining or painting have traditionally relied on aggressive chemicals that are now considered environmentally unfriendly. Costs for adequately protecting of worker and protecting the environment are high enough that alternate surface processing methods are needed. During the last two years ORNL, began a systematic study of a unique laser processing method for both cleaning and texturing metal surface simultaneously. This novel surface treatment method uses laser interferometry produced by two beams of a pulsed Nd:YAG laser. Operating at 10Hz of frequency, this technique has been used to clean aluminum surfaces, and at the same time creating periodic and rough surface structures. Preliminary results for the Al-alloy laser-based surface treatment process were reported last year at this conference. One of the primary findings for Al-alloy surfaces was the development of an enhanced oxide surface region. Influence of the enhanced oxide on both adhesive joining and corrosion protection are now underway. Recently, we have extended these laser-processing studies to Ti-6Al-V4 (Ti64) alloys, which are widely used in the aerospace industry. Ti64 alloy surfaces can be cleaned using high-energy laser pulses (nanoseconds to milliseconds range) and, as with the Al-alloys, is accomplished mainly by surface melting and ablation. Our method is non-contact, does not rely on surface abrasion and significantly reduces the chemical impact of commonly used solvents and detergents. The process being optimized at ORNL uses a 2-beam method that not only cleans the surface for joining but textures the surface in a periodic manner. This poster present preliminary surface characterization results for the cleaning of Ti64-alloy surfaces. Results from scanning transmission electron microscopies, x-ray photoelectron spectroscopy, and contact angle measurements on as received and laser-treated commercial Ti64-alloy surfaces will be presented. This abstract has been authored by UT-Battelle, LLC under Contract No. DE-AC05-00OR22725 with the U.S. Department of Energy.

AS-ThP-7 Cross-Sectional Mapping vs. Depth Profiling Analysis: Is the Choice Always Clear?, Kathryn Lloyd, J Marsh, DuPont Corporate Center for Analytical Sciences

Differences in chemistry across monolithic or multi-layered films are often critical to product performance. Each component in a formulation serves a

purpose. It is important to be able to monitor the integrity of multi-layered structures, the striation or migration of components, the enrichment of components at interfaces, and the formation of buried defects.

Approaches used to obtain this information can be broadly separated into cross-sectioning (combined with chemical mapping) and depth profiling (with or without chemical mapping). Cross-sectioning calls for more labor up front to prepare the samples – potting in epoxy; microtoming -- but the resulting cross-sectional surfaces are amenable to multiple chemical mapping techniques, such as Raman microprobe, secondary ion mapping, and Energy Dispersive Spectroscopy (SEM/EDS), all of which exhibit comparable lateral resolution (around 1 micron). Depth profiling requires little to no sample preparation and offers higher depth resolution (10s of nanometers).

To a large degree, film and/or layer thickness determine the approach pursued. Practical depth profiling historically has not extended much more than 10 microns into a surface. Thus, for multilayer films with thicknesses on the order of 25 microns or more, cross-sectioning has been preferred. This can present problems for surface chemical mapping techniques such as ToF-SIMS, when even cryo-microtoming can result in a thin overlayer of smeared epoxy components covering the surface.

The advent of new gas cluster ion beam (GCIB) sputter sources has not only enabled cross-sectional ToF-SIMS mapping analysis for layered and heterogeneous organic systems, but has also made the choice between cross-sectioning and depth profiling not as clear. GCIB depth profiling through organic or polymeric material can be very efficient, making the idea of profiling through 25 microns or more not as impractical as it once was.

This presentation will show examples of cross-sectional mapping analysis and sputter depth profiling, exploring which factors can influence the choice of approach and illustrating some of the trade-offs involved.

AS-ThP-8 Investigation on Human Evidences using ToF-SIMS Combined with Advanced Matching Recognition, T Terlier, Korea Institute of Science and Technology; J Lee, M Kang, Yeonhee Lee, Korea Institute of Science and Technology, Republic of Korea

The expansion of the use of forensic sciences requires to develop new techniques providing prompt and reliable information. In terms of chemical analysis, the useful information ranges from trace elements, e.g. the elemental detection of residues in a specimen, to molecular information, e.g. the identification of high mass molecules. Time-of-Flight Secondary Ion Mass Spectrometry is able to provide a detailed 3D view of the elemental and molecular composition of solid samples and has already been used previously to analyze inorganic, organic and biological specimens, showing its strong potential to retrieve more information from trace evidence than the traditional methods.

Several challenges still prevent a widespread application of ToF-SIMS to forensic analysis. Samples from the human body are the most commonly collected specimens and these samples are particularly challenging for analysis due to the variety of specimens, including hair, fingerprints, fingernails and lipstick, and the complexity of the samples that can contain blood, saliva and/or sweat. A common material to almost all of the human specimens is cosmetics, which can provide crucial information about an individual. Through analysis of five different types of cosmetics - hair styling products, lipstick, lotion, nail polish, and foundations, we have collected a large range of ToF-SIMS spectra and build a cosmetic database from these reference samples.

The characterization of different types of human evidences has been performed to illustrate the large range of the possibilities given by the ToF-SIMS. However, to identify the chemical composition of the cosmetic residues and to determine their origins, advanced methods of classification and discrimination need to be applied. For example using Principal Component Analysis, it was possible to identify the cosmetic residues present on hair cuticles and to discriminate the hair product applied from a list of 16 references. Nevertheless, quantitative analysis can be required for identifying the exact nature of the residues. Thus, a new method combining Matching Factor and decision tree has been developed. Fingerprint analysis has been performed to identify the traces of residues contained in the fingerprint, which have also highlighted the fingerprint's ridges. After PCA segmentation, the extracted mass spectrum from the residue's region has been evaluated using a new matching recognition method, which has clearly indicated the origin of the cosmetic product.

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The development of matching recognition method and the analysis performed on the human evidences has demonstrated that the ToF-SIMS is a promising way to help the forensic investigations.

AS-ThP-9 Calculation of Multiplet Structure in a Mixture of Copper Oxides, Diego Fernando Mulato-Gómez, J Torres Ochoa, Cinvestav-Unidad Queretaro, Mexico; D Cabrera-German, Universidad de Sonora, México; A Herrera-Gomez, CINVESTAV-Unidad Queretaro, Mexico

The Cu 2p photoemission spectrum of thermally oxidized ultra-clean copper is very complex. Part of the photoemission structure (i.e., peaks) has been attributed to Cu¹⁺ and Cu²⁺. [1,2] However, there are peaks remaining that cannot be attributed to those species. Therefore, we present atomic multiplet ligand field calculations [3] for Cu³⁺ that show a very good agreement with this extra structure.

X-ray photoelectron spectra using a monochromatic Al K α source ($h\nu = 1486.7$ eV) were recorded from thick-enough (~ 20 nm) copper oxide films that were prepared by thermal sublimation of copper onto Si (100) in an ultra-clean environment (carbon-free and oxygen-free) and a subsequent oxidation at 200°C for 10 minutes. The experimental Cu 2p photoemission spectrum and the atomic multiplet ligand field calculations show a close agreement with each other, sustaining, as mentioned before, the presence of an additional Cu³⁺ state in thermally oxidized ultra-thin copper. Also, the O 1s spectrum shows three components that could be associated to each one of the three copper oxidation states (Cu¹⁺, Cu²⁺, and Cu³⁺). As supplementary evidence for the latter asseveration, we present the chemical composition of each oxide species present in the film. The calculation of the composition required the discrimination of the chemical components through peak-fitting. A very close fitting of the Cu 2p spectrum was achieved through state-of-the-art background modeling. The expected Tougaard contribution to the background [3,4] was combined with species-dependent Shirley-type backgrounds (SVSC-background [5]) by employing the *active background approach* [5]. This peak-fitting and analysis approach has been successfully applied in the characterization of other (Co [6], Fe [7] and Zn [8]) transition metal oxides.

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AS-ThP-10 Wafer Bonding Between LiTaO₃(100) and Alpha-quartz SiO₂(100) via Low Temperature (<220°C) NanoBonding™ Using Surface Energy Modification, Brian Baker, J Kintz, A Yano, N Herbots, Arizona State University; W Lee, Cactus Materials, Inc.; S Narayan, J Day, Arizona State University; R Islam, Cactus Materials, Inc.; Y Watznabe, TDC Coporation; M Koury, M Johnson, R Culbertson, M Magnus, Arizona State University

Wafer bonding is displacing heteroepitaxy in the manufacture of heterostructures, such as tandem solar cells and sensors. High costs and poor yields limit effective production of opto-electronic devices and sensors.

In this work, piezoelectric LiTaO₃(100) and alpha-quartz SiO₂(100) wafer bonding is investigated via NanoBonding™ near 300K [1,2]. NanoBonding™ generates molecular bonds between surfaces at the nanoscale over large interfacial domains, creating a 2D bonding interphase between the two materials. First, electron exchange is created between surfaces by engineering a key hydrophilic-hydrophobic surface pairs (via wet chemical processes, spin, vapor, or plasma etching). This method catalyzes electronic exchange and bonding by modifying native surfaces to a less stable state where electronic displacement is enhanced. Surface Energy Modification is measured via the surface total energy γ_T , and its three components γ_{LW} , γ^+ , γ^- . These values are based on the Van Oss-Chaudhury-Good (vOCG) theory, and using high resolution Three Liquid Contact Angle analysis. A key feature of Surface Energy Modification for NanoBonding™ is the creation of 2D precursor phases on surfaces. Next, for nanocontacting, the surface has

to be planarized at the macro, micro, and nano scales. Last, NanoBonding™ activation can occur on contact and/or after thermal activation.

NanoBonding™ depends thus on the control of surface energy, planarity at three scales, and composition. γ_T can be computed from 3LCAA. The liquids used are 18 MO water, glycerin, and α -bromo-naphthalene (10 10 μ L droplets). The average γ_T , across as received 4" LiTaO₃ wafer is 43.3 ± 2 mJ/m² (hydrophobic). However, the electron acceptor energy γ^- can vary from 43 mJ/m² to 23 mJ/m² ($\sim 50\%$ difference). Regions with low γ^- (low electron transfer) do not bond while those high γ^- do. This correlates directly with bonded interfacial regions visualized when LiTaO₃ wafer is nano-contacted with 4" quartz wafers. In this way, 3LCAA can determine one cause of bonding failures.

Thermal activation (100 and 200°C) does not enhance bonding as significant thermal expansion causes mismatch fractures, interface delamination, or thermal decomposition due to the high mobility of Li ions.

In conclusion, thermally or plasma activated wafer bonding is clearly not optimal for wafer bonding LiTaO₃. To Si-based materials, causing high fracture rates for LiTaO₃ as well Li out diffusion. Instead, Nanobonding(TM) is more appropriate due to a reduced fracture or thermal decomposition chance.

Herbots N. et al. US Pat. No 9,018,077 (2015), US Pat. No 9,018,077 (2017)

Herbots N., Islam R., US Pat. Pending (2018), filed March 18, 2018

AS-ThP-11 Structural, Morphological and Electrical Properties of Multilayer Sequentially Sputtered Nb₃Sn Films for Different Layer Thicknesses, Md. Nizam Sayeed, Old Dominion University; U Pudasaini, College of William and Mary; H E. Elsayed-Ali, Old Dominion University; G Eremeev, Thomas Jefferson National Accelerator Facility

Nb₃Sn is an intermetallic compound of A15 crystal structure that has type II superconductivity. Due to higher critical temperature $T_c = 18.1$ K and upper critical magnetic field of up to 30 T, Nb₃Sn is considered as an alternative of niobium for Superconducting Radio Frequency (SRF) applications in particle accelerators. Nb₃Sn coating on the inner surface of niobium SRF cavities can be operated at 4.2 K, whereas standard niobium cavities are currently operated at 2 K and consequently reduce the operation cost. However, synthesis of Nb₃Sn is challenging due to the availability of other phases of niobium and tin (Nb₆Sn₅ and NbSn₂), which have poor superconducting properties. We have fabricated Nb₃Sn films on sapphire using multilayer sequential sputtering. Several thin layers of Nb and Sn were deposited repeatedly by magnetron sputtering and annealed afterward at 950 °C for 3 hours to form Nb₃Sn. We have varied the Nb and Sn layer thicknesses of the films and characterized their crystal structure by X-ray diffraction (XRD), surface morphology by scanning electron microscopy (SEM), surface topography by atomic force microscopy (AFM), film stoichiometry by energy dispersive X-ray spectroscopy (EDS). The films showed crystalline structures of Nb₃Sn only. T_c up to 17.63 K with sharp superconducting transition has been achieved.

AS-ThP-12 Corrosive and Thermal Properties of ZrO₂- Y₂O₃ Thermal Barrier Coatings, Byung-Koog Jang, Kyushu University, Japan; H Kim, Korea Institute of Ceramic Engineering and Technology

Thermal barrier coatings (TBCs) have received a large attention because they increase the thermal efficiency of gas turbine engines by increasing the gas turbine inlet temperature and reducing the amount of cooling air required for the hot section components. Electron beam-physical vapor deposition (EB-PVD) or plasma spray coatings is a widely used technique for depositing thermal barrier coatings (TBCs) on metal substrates for high temperature applications, such as gas turbines, in order to improve the thermal efficiency. The controlled microstructure of coatings is one of the most important properties for obtaining superior TBCs. In addition, the high temperature capability of TBCs used in a gas turbine is often degraded by deposits of calcium-magnesium-alumino-silicate (CMAS). The CMAS melts are produced when siliceous minerals (volcanic ash, dust and sand) are ingested with the intake air and deposited on the hot surface of TBCs.

This work describes the thermal conductivity and corrosive properties of 2~8mol%Y₂O₃-stabilized ZrO₂ (YSZ) coatings by EB-PVD and plasma sprayed coatings. The thermal conductivity of the coated samples was measured by the pulsed thermal imaging method and the laser flash method. The hot corrosion between YSZ coatings and volcanic ash was examined by isothermal heating at 1200°C in air between 10 min and 100hrs. The thickness of corrosive region at top surface of TBCs by the reaction between YSZ coating and volcanic ash was increased with increasing the oxidation time.

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