Chemical Analysis and Imaging of Interfaces Focus Topic
Room Oregon Ballroom 203-204 - Session CA-TuP

Chemical Analysis and Imaging of Interfaces Poster Session

CA-TuP-1 Combined Spectro-Electrochemical Methods to Investigate Electrochemical Corrosion in Real-Time, *Matteo Olgiati,* CEST GmbH, Austria

Many engineering alloys, ranging from the aerospace grade aluminium alloys 2024 and 7075 to stainless steels, are vulnerable to localised corrosive degradation under certain environmental conditions.

The local nature of corrosion is most of the time dictated by the intrinsic features of the microstructure. The intermetallic second phases in age-hardened aluminium alloys, for example, are known to act as nucleation points for local corrosive attack [1]. Similarly, pitting corrosion in stainless steels was found to initiate in the vicinity of MnS inclusions [2]. Furthermore, the time-dependent evolution and further propagation of corrosive events depends on the local changes in chemistry of the microstructure [3-4], which will ultimately determine the mechanism and rate of such propagation.

For these reasons, characterising and, consequently, understanding the degradation mechanism of these alloys is not only complex, but must also rely on in-situ and highly spatio-temporally resolved techniques [5].

In this contribution, we present a recently developed spectro-electrochemical scanning flow cell, which enables to study corrosive degradation phenomena, as well as corrosion prevention with the use of corrosion inhibitors. Such scanning flow cell allows to induce local corrosion by electrochemically controlling the polarization of the working electrode immersed in 10 mM NaCl solution. Simultaneously, we can characterize the time-dependent appearance of micro- to macroscopic surface features by means of in-situ optical microscopy. This information can be further supported by the online identification and quantification of dissolved mass currents by means of online inductively coupled plasma mass spectrometry (ICP-MS). The combination of this techniques allows us to estimate not only the typical surface-initiated corrosion mechanisms, but also the characteristic corrosion rates of different metal alloys.

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CA-TuP-2 Diamond Hydrogenation Using a Compact and Cost-Effective Low–Power Plasma, *J. Trey Diulus,* NIST Center for Nanoscale Science and Technology; *F. Yi,* NIST-Gaithersburg; *E. Strelcov,* NIST Center for Nanoscale Science and Technology; *D. LaVan,* NIST-Gaithersburg; *A. Kolmakov,* NIST Center for Nanoscale Science and Technology

Device fabrication of field effect transistors (FET)s and other electronic devices are still largely dependent on silicon, which has been the core semiconductor material for over seven decades.[1,2] Newer materials, like SiC and GaN, offer wide-bandgap alternatives to silicon that are required for high power electronics with improved carrier mobility, carrier density, and high operation temperatures.[2,3] Alternatively, diamond is another promising semiconductor material, with its ultra-wide bandgap (5.5 eV), unmatched thermal conductivity, breakdown voltage, and high charge carrier mobility ($\sim$4000 cm^2 V^{-1} s^{-1}).[3] Recently, advances in diamond growth through techniques like chemical vapor deposition (CVD) have demonstrated wafer size of diamond single crystals,[4] overcoming the primary challenge towards industrial implementation. However, hydrogenation of the diamond surface is a necessary process to fabricate high-mobility diamond FETs. The H-terminated surface, however, is sensitive to ambient adsorbates that deteriorate the stability of surface conductivity when using modern device manufacturing processes. The present method for H-terminating a diamond surface is via a high-power (> 1 kW) microwave plasma that typically utilizes a specially designed chamber for high pressure (tens of kPa) exposures at elevated temperatures (>700 °C).[5] Due to the high cost required for such a system, a roadblock exists that prevents broader research community efforts for diamond related devices that is imperative for improving high-power/frequency electronics. Thus, we have developed a recipe for H-terminating diamond based on an H_2 plasma generated by a simple low-power (75 W), low pressure (tens of Pa) RF plasma cleaner for a cost-effective diamond hydrogenation method. Furthermore, we have developed a relevant plasma characterization metrology for H-termination by real time monitoring of the radical flux using catalytically activated and reference metal film samples loaded in a nano-calorimeter,[6] in conjunction with standard optical spectroscopy. We have tested our hydrogenation method on diamond samples by characterizing the surface before and after plasma treatment with *in situ* x-ray photoelectron spectroscopy and *ex situ* 4–point probe measurements. Our efforts will provide researchers a facile route towards diamond research and will broaden the diamond research community.

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CA-TuP-3 A Study of the D-Parameter: Evaluating Measurement Techniques in X-ray Photoelectron Spectroscopy (XPS), *Alvaro Lizarbe, G. Major, B. Clark,* Brigham Young University; *D. Morgan,* Cardiff University; *M. Linford,* Brigham Young University

When looking at carbonaceous material analysis, the D-parameter plays a pivotal role in determining the sp^2/sp^3 ratio via X-ray photoelectron spectroscopy (XPS). Despite its utility, the methodological inconsistency in generating the peak envelope introduces uncertainty in the D-parameter analysis. This study investigates two distinct approaches to measuring the D-parameter, a modified b-spline method and a polynomial regression line fit. The b-spline method entails the creation of synthetic Gaussian peaks in the carbon KLL Auger peak, maintaining a consistent full width at half maximum (FWHM) and equal distance apart but varying areas. The polynomial regression approach, on the other hand, fits the curve by manipulating the degree of the polynomials. Both techniques strive to generate a fit that mirrors the raw data as closely as possible, thereby facilitating a more precise derivative from which the D-parameter can be computed. To evaluate the efficacy of these methods, a comparative analysis was conducted using different carbonaceous materials such as direct current chemical vapor deposition (DC-CVD) diamonds, carbon nanotubes, and highly oriented pyrolytic graphite (HOPG). The results are compared against the traditional D-parameter values for these materials. This research not only contributes to improving the reliability and reproducibility of D-parameter measurements but also offers valuable insights into the optimization of envelope creation methods in XPS.

CA-TuP-4 Proton and Hydroxide Diffusion Within Supercooled Water, *Megan Dunlap,* Pacific Northwest National Lab; *L. Kringle, R. Smith, B. Kay, G. Kimmel,* Pacific Northwest National Laboratory

The molecular mechanisms for the diffusion of protons and hydroxides within liquid water are controversial. To elucidate these processes, we examine proton and hydroxide diffusion within water films under ultra-high vacuum that are heated to temperatures spanning 110-140 K. At these temperatures, the water molecules are arrested, but the charged species move rapidly through the $\sim$40 nm thick films. Their progress is monitored with reflection absorption infrared spectroscopy (RAIRS) via the exchange of the ion with isolated D_2O 'probe' molecules within the film to form HDO. We have investigated the exchange rate as a function of the distance that the ions must travel to reach the D_2O, the temperature, the total thickness of the film, and the ion type. Based on the temporal evolution of the D_2O, we have found that both diffusion and trapping of the ion within the film are required to describe the observations.

Author Index

Bold page numbers indicate presenter