Chemical Analysis and Imaging at Interfaces
Room 321 - Session CA1-FrM

Chemical Analysis and Imaging at Interfaces Oral Session I

Moderators: Dr. Andrei Kolmakov, National Institute of Standards and Technology (NIST), **Gabriel Parker**, Oak Ridge National Laboratory

8:15am **CA1-FrM-1 Semiconductor Manufacturing Metrology*, Alexander Liddle,*** Canon Nanotechnologies Inc. **INVITED**

The old adage from optics that, if it can be measured, it can be made, is nowhere more true than for semiconductor manufacturing.Leading-edge devices contain nanoscale features, and require sub-nanometer dimensional, compositional, and interfacial control over length scales up to the diameter of a 300 mm wafer.Every step of the fabrication process, from deposition, to lithography, to etch, depends on sophisticated metrology, often in-line and at speed, to achieve the level of precision and accuracy needed to flawlessly produce the billions of transistors that make up today's high-performance integrated circuits.In this talk, I will provide a high-level overview of some of the outstanding measurement challenges that must be overcome in order to maintain the semiconductor industry's extraordinary progress in manipulating and controlling matter at the atomic scale.In particular, I will focus on the metrology needed to enable the transition to highly three-dimensional structures, such as gate-all-around (GAA) transistors, high-capacity NAND flash memory, and hybrid-bonded high-bandwidth memory.

8:45am **CA1-FrM-3 High Performance Computing and Artificial Intelligence Enabled Materials Characterization and Experimental Automation*, Mathew Cherukara,*** Argonne National Laboratory **INVITED**

The capabilities provided by next generation light sources along with the development of new characterization techniques and detector advances are revolutionizing materials characterization (metrology) by providing the ability to perform scale-bridging, multi-modal materials characterization under *in-situ* and *operando* conditions. For example, providing the ability to image in 3D large fields of view (~mm^3) at high resolution (<10 nm), while simultaneously acquiring information about structure, strain, elemental composition, oxidation state, photovoltaic response etc.

However, these novel capabilities dramatically increase the complexity and volume of data generated. Conventional data processing and analysis methods become infeasible in the face of such large and varied data streams. The use of AI/ML methods is becoming indispensable for real-time analysis, data abstraction and decision making at advanced, high-data rate instruments. I will describe how high-performance computing (HPC) along with AI on edge devices enables real-time data analysis and self-driving experiments, creating the next generation of AI-powered materials characterization tools.

As instrument and analysis workflows increase in complexity, large language models (LLM) have the potential to assist and enhance the productivity of scientists. I will describe early experiments with AI-powered scientific co-pilots that can provide assistance through every stage of an experiment; planning, execution, analysis and even instrument operation.

9:15am **CA1-FrM-5 Challenges in Real-Time Plasma-Surface Diagnostics*, Vincent Donnelly,*** University of Houston **INVITED**

An array of quantitative surface diagnostic methods has been used over the past 65 years to gain detailed insights into the physics and chemistry of surfaces. These include Auger electron spectroscopy (AES), x-ray photoelectron spectroscopy (XPS), low-energy electron diffraction and similar methods. These techniques required high- or ultrahigh vacuum to detect electrons without collisions with background gas, as well as to keep surfaces clean of unwanted contaminants. Similar constraints are imposed by mass spectrometry methods such as temperature programmed desorption (TPD) and secondary ion mass spectrometry (SIMS). When the gas pressure exceeds a level where the collision mean-free-path becomes comparable to a characteristic distance (e.g. 1 mTorr at ~5 cm), the primary information carried by the electron energy, and neutral or ion desorption product is degraded or lost. The problem is exacerbated in a plasma, where large electron and ion densities, electric fields and magnetic fields dilute and distort the surface analysis signals. Despite these issues, considerable advances have been made in the application of conventional surface analysis methods for real-time probing of plasma-surface interactions. This talk will review some of this work from our laboratory and elsewhere, where AES, XPS and TPD/SIMS-like desorption spectroscopy have been used to obtain information such as adsorbate coverages, reactive sticking coefficients, surface catalyzed reactions, and primary desorption products, all while operating a plasma. Examples will include operando XPS, spinning wall AES and desorption mass spectroscopy, ion-desorption optical emission spectroscopy and laser-desorption spectroscopy.

9:45am **CA1-FrM-7 Gaede Langmuir Award Talk: Investigation of Solid-Solid and Solid-Liquid Interfaces under Electrical Potential via XPS and EIS*, Sefik Süzer[1],*** ÜNİVERSİTELER MAH, Turkey **INVITED**

X-ray Photoelectron Spectroscopy (XPS) and Electrochemical Impedance Spectroscopy (EIS) are well established analytical tools for interrogating surface and interface chemistry of mostly solid materials and their interfaces. Herein, we report using both techniques together, our observations on slow dynamics of devices with Semi-Conducting n- and/or p-doped Si Electrodes, having also ~3-4 nm oxide layer on top, separated by a Porous Polyethylene Membrane (PEM) soaked by an Ionic-Liquid as the electrolyte. As such, the device has two different interfaces: (i) Ionic Liquid / Silicon, and (ii) Silicon / Silicon Oxide. Using XPS, time dependent shifts of well-separated Si2p peaks of the semi-metallic (Si^0) as well as the oxide (Si^{X+}), together with O1s peak have been recorded under external electrical bias, to reflect the local electrical potential developments. For the electrolyte (Ionic Liquid), C1s, O1s and F1s peaks have been used to record the electrical potential developments at different locations. Complementary Electrochemical Impedance Spectroscopic measurements have also been recorded on the very same device within the XPS instrument before and after XPS measurements to follow the various changes, in addition to similar measurements under ambient conditions. Electrochemical findings, regarding the two interfaces, obtained from measurements of these two powerful spectroscopic techniques will be presented and discussed.

[1] **Gaede-Langmuir Award Winner**

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