

Nanoscale Science and Technology Division

Room B113 - Session NS1+2D+BI+SS-MoM

Combined Nanoscale Microscopy

Moderators: Adina Luican-Mayer, University of Ottawa, Canada, Sergej Kalinin, Oak Ridge National Laboratory

8:20am **NS1+2D+BI+SS-MoM-1 Combined Metrology at the Nanoscale: Advanced Scanning Probe Microscopy to Evaluate Complex Semiconductors**, *Fernando A. Castro*, National Physical Laboratory, UK
INVITED

The performance of semiconductors is strongly affected by spatial variations that can be introduced during manufacturing or due to degradation processes. In addition to the impact of microstructure and defects on electrical and optical properties, complex semiconductors, such as some compound semiconductors, perovskites or 2D materials, can present dynamic changes in properties during operation. Combining metrology methods is critical to better understand and characterise such complex samples as individual methods provide insufficient information. Ideally these combined measurements should be either co-localised or simultaneous in order to reduce uncertainty associated with post process image registration, spatial heterogeneity, or sample contamination. NPL has been developing a suite of spatially resolved measurement methods to understand critical factors that impact semiconductor performance and reliability. In this presentation, we'll focus on nanoscale methods under controlled operational or environmental conditions, including advanced modes of scanning probe microscopy (SPM) such as time-resolved scanning kelvin probe (tr-SKPM) and tip enhanced optical microscopy (TEOS). After introducing the challenges and recent results from the European project PowerELEC, we'll present two examples of how these combined measurements are applied. First, we'll describe the application of SPM to understand degradation mechanisms in state-of-the-art perovskite solar cells (PSCs). Time-resolved SKPM can be used to distinguish the impact of ionic and electronic charges on dynamic processes and in-situ co-localised measurements under controlled environmental conditions can identify nucleation of nanoscale grains on the perovskite film surface at the start of the degradation process, allowing us to link degradation to the local electrostatic environment. The second example will focus on 2D transition metal dichalcogenide (TMD), which present promise for optoelectronic applications but are often limited by Fermi level pinning effects and consequent large contact resistances upon contacting with bulk metal electrodes. A potential solution for near-ideal Schottky-Mott behavior and concomitant barrier height control has been proposed in the literature by contacting TMDs and (semi-)metals in van der Waals heterostructures. We will show how combined nanoscale measurements allows to directly access interface parameters relevant to the Schottky-Mott rule on a local scale and how we use SKPM and TEOS measurements under simulated operational conditions (e.g. electrostatic doping induced Fermi levels) to enable decoupling and quantification of contributions from the interface dipole and electrode work function.

9:00am **NS1+2D+BI+SS-MoM-3 Correlated Functional Imaging of Printed and Ferroelectric 2D Devices for Ubiquitous Sensing and Neuromorphic Computing**, *J. Kim, Z. Zhu, T. Chu, H. Choi, M. Moody, Lincoln Lauhon*, Northwestern University

The unique properties of 2D materials stimulate the design of devices that exhibit useful new behaviors. However, the correspondence of expected and actual operating principles of devices cannot always be established from simple analysis of temperature-dependent current-voltage characteristics. As a result, the rational optimization of even simple devices such as thin-film transistors, as well as the successful realization of novel neuromorphic devices, benefits from spatially resolved characterization of nanoscale structure and properties to discern the relative contributions of device geometry and 2D material structure and chemistry to device performance. This talk will describe case studies in which Kelvin probe force microscopy (KPFM) and scanning photocurrent microscopy (SPCM) are used to investigate the operating principles of thin-film transistors (TFTs) and source-gated transistors (SGTs) fabricated from MoS_2 and In_2Se_3 . In the case of n-type semiconducting 2H MoS_2 , model devices constructed from overlapping exfoliated flakes are analyzed to identify factors limiting the performance of printed thin-film transistors (*ACS Nano* 2023, **17**, 575). KPFM analysis is used to isolate the contact, channel, and junction resistances and calibrate a resistor network model of printed thin films. Simulations of the effective mobility and on-current dependence on flake

thickness, size, and degree of overlap suggest that the performance of printed TFTs are limited by resistance arising from unpassivated edge states.

In the second use case, KPFM, SPCM, and piezoresponse force microscopy (PFM) are used to pinpoint the origin of resistance modulation in $\alpha\text{-In}_2\text{Se}_3$ transistors that exhibit tunable non-volatile channel conductance. Memristive behavior in In_2Se_3 TFTs has been attributed to switching of the channel polarization, but the lack of an obvious threshold for switching raises questions about the evolution of domain structure and the contribution of trap states. Furthermore, the presumed modulation of the Schottky barrier has yet to be confirmed experimentally. We address this gap in understanding through correlated PFM, KPFM, and SPCM measurements. We then fabricate $\text{MoS}_2\text{-In}_2\text{Se}_3$ transistors with a geometry that induces depletion at the source electrode, i.e. a source-gated transistor, and observe non-volatile switching of the low output current. KPFM, SPCM, and finite element simulations are used to confirm source pinch-off and non-volatile multi-level modulation of the effective source resistance. The quantitative correlation of device behaviors with the changes in channel potential at key interfaces usefully constrains the interpretation of the operating principles and builds a foundation for rational design of novel neuromorphic devices and systems.

9:20am **NS1+2D+BI+SS-MoM-4 A Unique New Correlative Microscopy Platform for Combined Nanoscale Microscopy by Combination of AFM and SEM**, *Chris Schwalb*, Quantum Design Microscopy GmbH, Germany; *K. Arat*, Quantum Design, Inc.; *H. Alemansour, A. Alipour*, Quantum Design, Inc., Iran (Islamic Republic of); *A. Amann*, Quantum Design, Inc., Germany; *L. Montes*, Quantum Design, Inc., Colombia; *J. Gardiner*, Quantum Design, Inc.; *H. Frerichs, L. Stuehn, S. Seibert*, Quantum Design Microscopy GmbH, Germany; *S. Spagna*, Quantum Design, Inc.

The combination of different analytical methods into one instrument is a powerful technique for the contemporaneous acquisition of complementary information. This is especially true for the in-situ combination of atomic force microscopy (AFM) and scanning electron microscopy (SEM), two of the most powerful microscopy techniques available. This combination gives completely new insights into the nanoscale.

In this work, we introduce a highly integrated new correlative microscopy platform, the FusionScope, that seamlessly combines AFM and SEM within a unified coordinate system. The self-sensing piezoresistive cantilever technology used for the AFM scanner results in a purely electrical measurement of the cantilever deflection signal. This allows for concurrent, correlated acquisition of both SEM and AFM images at the region of interest. In addition, a three-axis sample stage and a trunnion provide unique experimental capabilities such as profile view – an 80-degree tilt of the combined sample stage and AFM giving full SEM access to the cantilever tip region.

We will present a variety of novel case studies to highlight the advantages of this new tool for interactive, correlative, in-situ nanoscale characterization for different materials and nanostructures. First results will focus on hard-to-reach samples. FusionScope allows for fast and easy identification of the area of interest and precise navigation of the cantilever tip for correlative SEM and AFM measurements. We demonstrate that approach for analysis of blade radius of razor blades and the characterization of lacunae structures on bone surfaces.

In addition, we will present first results for the in-situ characterization of individual nanowires that will be used for energy harvesting applications. The SEM enables the easy location of individual or multiple nanowires, whereas the in-situ AFM allows the characterization of topography, surface roughness, mechanical, and electrical properties of the nanowire.

Based on the broad variety of applications regarding the inspection and process control of different materials and devices, we anticipate that this new inspection tool to be one of the driving characterization tools for correlative SEM and AFM analysis in the future.

9:40am **NS1+2D+BI+SS-MoM-5 Correlative in-Situ Nanoscale Microscopy Using AFM and FIB-SEM for Nanomechanical Property Mapping Throughout a 3D Volume**, *Prabhu Prasad Swain, M. Penedo, N. Hosseini, M. Kangül, S. Andany, N. Asmari, G. Fantner*, Ecole Polytechnique Fédérale de Lausanne (EPFL), Switzerland

In this work, we present results obtained with an atomic force microscopy (AFM) integrated in a focused ion beam- scanning electron microscope (FIB-SEM). The FIB-SEM is a powerful instrument, capable of automated structural analysis and prototyping at nanometer resolution, while the AFM

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is a well-established versatile tool for multiparametric nanoscale characterization. Combining the two techniques allows unprecedented *in-situ* correlative analysis at the nanoscale. Nanoprototyping and enhanced multiparametric analysis can be performed without contamination of the sample or environmental changes between the subsequent processing steps. The power of the combined tool lies in the complementarity of the two techniques. The AFM offers nanomechanical property mapping with electrical and magnetic characterization of the sample, while SEM offers elemental analysis and FIB enables thin slicing of the of the sample for block face imaging. This enables 3D tomographic imaging of complex samples mapping composition and mechanical properties throughout the 3D volume. Controlling both these instruments with open-hardware controller (OHC), allows us to perform automated *in-situ* AFM-FIB-SEM characterization. The setup is aimed to provide true 3D correlative information and mapping, with increased resolution for a larger volume. We will demonstrate the capabilities of correlative AFM/SEM/FIB imaging through a series of correlative experiments on polymers, 2D materials, nanowires and rock sediments.

10:00am **NS1+2D+BI+SS-MoM-6 Anisotropic Friction Effects of Perovskite Nanoplatelets on a vdW Substrate**, *Sidney Cohen, N. Itzhak, I. Rosenhek-Goldian, O. Brontvein, E. Joselevich*, Weizmann Institute of Science, Israel

Interest in 2D materials can be attributed to their unique properties such as electrical, optical, and mechanical characteristics, which can be harnessed in small devices. Assembly of these materials can be challenging. vdW epitaxy is a promising approach, in which nano-sized crystalline structures are grown on a 2D vdW substrate which has minimal interaction energy, resulting in low strain. The epitaxial growth still provides sufficient interaction to favor specific geometries according to lattice directions. In this presentation, the system is CsPbBr₃ platelets grown on vdW ReSe₂. This combination is of fundamental and applied interest due to special optoelectronic properties of these 2D-3D mixed semiconductor systems. The mechanism of the nanoplatelet growth leading to their shape and orientation on the surface remains to be fully revealed. Here, we present tribological studies performed by monitoring the force required to push the platelets along the surface. We observed a significant directional effect expressed in the lateral forces required to slide the platelets along the surface. In particular, forces 4-5 times those required to push rectangular platelets along the ReSe₂ surface along the long axis were insufficient to move the same platelets along their short axis. STEM images showed that this correlated with commensurability of the two lattice structures. Some of the experiments were performed in an ambient AFM system. Because sliding along the surface can be hindered by atomic steps and defects, unbiased analysis of this effect requires searching for small steps of atomic height along the sliding path. Scanning electron microscopy is a convenient way to search for these defects: thus, comparative experiments were performed *in-situ* in a combined AFM-SEM system. This combination had the additional advantage of allowing rapid overview of the surface to locate regions of interest.

In the process of evaluating the measurements, those performed in vacuum required much higher (by as much as an order of magnitude) forces to support pushing along the surface in comparison with comparable measurements made in the ambient AFM system. These measurements will be presented in the context of the characterization of the 2D substrate and platelet nanostructure as revealed by the two correlative measurement techniques.

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