

Nanometer-scale Science and Technology Division Room 102B - Session NS+2D+AS+PC-MoA

SPM - New Imaging and Spectroscopy Methodologies

Moderators: Aubrey Hanbicki, U.S. Naval Research Laboratory, Sidney Cohen, Weizmann Institute of Science, Israel

1:20pm NS+2D+AS+PC-MoA-1 A Connection Between Stability of STM Control System and Local Barrier Height: Implications on Imaging and Lithography, S.O. Reza Moheimani, University of Texas at Dallas **INVITED**
Poor performance of the Scanning Tunneling Microscope (STM) control system may result in tip-sample crash, a prevalent failure in STMs. Since its invention, about thirty-five years ago, few attempts have been made to improve the STM control system. Consequently, nearly all STMs are today operated with experimentally selected fixed-gain PI controllers. Selection of controller gains is often done without much attention to the electromechanical dynamics of the scanning tunneling microscope. Performance of such poorly-tuned controllers is limited and a key contributor to the tip-sample crash.

We perform closed loop system identification on a scanning tunneling microscope and show that the system DC gain is proportional to the square root of Local Barrier Height (LBH), a quantum mechanical property of the sample and/or tip that affects the tunneling current. We demonstrate that during a scan the LBH may undergo significant variations and thus it may adversely affect the closed-loop stability if the controller parameters are fixed. Feedback instabilities increase the risk of tip-sample crash in STMs.

In order to improve the closed loop performance, we estimate the LBH, on the fly, and use this information to adaptively tune the PI controller parameters. Experimental results obtained with the self-tuning PI controller confirm the improved STM performance compared to the conventional fixed gain PI controller. Further experiments confirm effectiveness of the proposed method in extending the tip lifetime by lowering the chance of the tip/sample crash.

2:00pm NS+2D+AS+PC-MoA-3 Distinctive Microstructures in a Complex Polymer Evolve with Time and Composition, X Yu, Worcester Polytechnic Institute; *S Granados-Focil,* Clark University; *M Tao, Nancy Burnham,* Worcester Polytechnic Institute

The diverse microstructures observed by atomic force microscopy (AFM) in asphalt binder – a complex polymer – suggest complicated intermolecular associations. These microstructures contribute to binders' bulk mechanical properties; therefore, it is essential to understand chemical-microstructural-mechanical relationships for optimal design of binder-related applications, which range from roads to roofs. The US market for asphalt binders in 2019 is predicted to reach 148 million barrels. [1]

Binders annealed at room temperature for over two months showed time-dependent microstructures, which correlate well with room-temperature steric hardening behavior as verified by other researchers using modulated differential scanning calorimetry. Microstructures of the binder films stabilized after different annealing durations, depending on the dimensions of the molecular structures and the complexity of the molecular interactions among the multiple phases in each bitumen. Distinctive microstructures were observed for remixed binders with increasing asphaltene concentrations. Consistency between our observations [2] and other literature suggests that microstructures observed by AFM are probably not just a surface phenomenon.

Furthermore, the complex nature of binder and the various influencing factors often lead to practical challenges in investigation of its microstructures and their chemical origins. Some of the main concerns related to AFM characterization of binders' microstructures, namely the dependence of the microstructures on such factors as sample preparation methods, annealing conditions and durations, and chemical composition, were also addressed in this study.

The above findings provide practical knowledge and deeper insights into the establishment of the complicated chemical-mechanical relationships for asphalt binders that pave the way toward tuned binder performance.

[1] <https://www.reportlinker.com/p0158665/US-Asphalt-Industry.html>

[2] "Time- and composition-dependent evolution of distinctive microstructures in bitumen." X. Yu, S. Granados-Focil, M. Tao, and N.A. Burnham, *Energy Fuels* 32, 67-80 (2018).

2:20pm NS+2D+AS+PC-MoA-4 Offering new Characterization Capabilities at the XTIP beamline by Combining Scanning Tunneling Microscopy with Synchrotron Radiation, Volker Rose, H Chang, M Fisher, S Hla, N Shirato, Argonne National Laboratory

The race is on for chemical x-ray imaging with nanoscale resolution. Specifically, there are currently substantial efforts underway at synchrotron facilities worldwide that aim to combine x-rays with scanning probe microscopy. Recently, substantial progress was made on Argonne's Synchrotron X-ray Scanning Tunneling Microscopy (SX-STM) project. SX-STM enables an entirely new view into the nanoworld by combining the best of two worlds: the exceptional chemical, magnetic, and structural sensitivity of synchrotron x-rays combined with the high spatial resolution of scanning probe microscopy accompanied by its ability to engineer and manipulate surfaces down to the level of single atoms.

To fully exploit the special capabilities of a unique new cryogenic x-ray microscope, XTIP, a dedicated beamline for SX-STM will become available at the Advanced Photon Source in early 2019. To meet the scientific objective of the nanoscience and nanomagnetism communities most effectively, we are going to build a soft x-ray beamline with full polarization control operating over the 500-1600 eV energy range. The dedicated XTIP beamline will provide researchers access to a one-of-a-kind instrument. Among the potential breakthroughs are "designer" materials created from controlled assembly of atoms and molecules, and the emergence of entirely new phenomena in chemistry and physics.

2:40pm NS+2D+AS+PC-MoA-5 Scanning Probe Microscopy Based Spectroscopy Measurement for Nanoscale Chemical Identification, Chanmin Su, Bruker-Nano, Inc. **INVITED**

Scanning probe microscopy has been instrumental for physical property characterizations at the nanometer scale, primarily for mechanical, electromagnetic and thermal properties. Recent progresses were focused on chemical identification based on mid-IR spectroscopy, pushing FTIR mapping to a resolution at or beyond 10 nm. This presentation will review technology advances in both scanning near field optical microscopy and photothermal based IR spectroscopy. Each of the techniques is discussed and benchmarked by detection limit, spatial resolution and signal to noise ratio, which ultimately determines the chemical mapping efficiencies. We will highlight techniques that address correlative imaging where physical and chemical properties at the same nanoscale location being acquired either concurrently or sequentially. As an example, PeakForce tapping based chemical and physical measurements will be explained in detail with the applications ranging from 2D materials to polymer complexes. The presentation will also highlight major challenges for scanning probe based measurements to be broadly adopted as the premier tool for nanoscale chemical fingerprint mapping.

3:40pm NS+2D+AS+PC-MoA-8 Quantifying Tip-Sample Interactions in Vacuum Using Cantilever-based Sensors: An Analysis, O Dagdeviren, C Zhou, E Altman, Udo D. Schwarz, Yale University

To achieve as much quantitative information on a surface as possible, the local measurement of tip-sample interaction potentials has recently gained much popularity in particular under well-defined ultrahigh vacuum conditions, where such measurements can be carried out with great accuracy both in terms of spatial as well as force resolution. To this end, either the oscillation frequency or the oscillation amplitude and phase of the vibrating force-sensing cantilever are recorded as a function of tip-sample distance and subsequently converted into quantitative values for the force or interaction potential. Here, we theoretically and experimentally show that the force law obtained from such data acquired under vacuum conditions using the most commonly applied methods may deviate more than previously assumed from the actual interaction when the oscillation amplitude of the probe is of the order of the decay length of the force near the surface, which may result in a non-negligible error if correct absolute values are of importance [1]. Caused by approximations made in the development of the mathematical reconstruction procedures, the related inaccuracies can be effectively suppressed by using oscillation amplitudes sufficiently larger than the decay length. To facilitate efficient data acquisition, we propose a novel technique that includes modulating the drive amplitude at a constant height from the surface while monitoring the oscillation amplitude and phase. Ultimately, such amplitude sweep-based force spectroscopy enables shorter data acquisition times and increased accuracy for quantitative chemical characterization compared to standard approaches that vary the tip-sample distance. An additional advantage is that since no feedback loop is active while executing the

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amplitude sweep, the force can be consistently recovered deep into the repulsive regime.

[1] O. E. Dagdeviren et al., *Physical Review Applied* **9**, 044040 (2018).

4:00pm **NS+2D+AS+PC-MoA-9 AFM + Nanoscale Vis-IR Spectroscopy via Photo-induced Force Microscopy**, *Derek Nowak, T Albrecht, S Park*, Molecular Vista

Photo-induced Force Microscopy (PiFM) [1] combines optical spectroscopy and atomic force microscopy (AFM) via illumination of the tip-sample junction with tunable laser light and mechanical detection of forces acting on the tip in response to interaction of light with the sample. With infrared (IR) source, PiFM can map the IR absorption of the sample as a function of IR wavelength and position and achieve nm-scale resolution in displaying the locations of heterogeneous materials on the surface of a sample. Even for samples without active IR absorption band, PiFM can be used to acquire nanoscale mapping based on the dielectric constant of the sample surface; dielectric constant mapping also allows high resolution sub-surface mapping. With tunable visible and near infrared (VisNIR) laser source, PiFM can map exciton resonances with similar spatial resolution even on monolayer samples. Examples from various classes of samples including organic, inorganic, and 2D materials will be presented. We will also present PiFM spectroscopy data that show excellent correlation with bulk FTIR spectra despite the fact that PiFM acquires local chemical information from regions in the range of 10 nm in extent.

[1] D. Nowak et al., *Sci. Adv.* **2**, e150157 (2016).

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