

Nanometer-scale Science and Technology Division Room A222 - Session NS-ThA

SPM for Functional Characterization

Moderators: Volker Rose, Argonne National Laboratory, Renu Sharma, NIST Center for Nanoscale Science and Technology

2:20pm NS-ThA-1 Interatomic Force Laws That Evade Dynamic Measurement, *John Sader*, University of Melbourne, Australia **INVITED**

Atomically-resolved imaging and force measurements using the atomic force microscope (AFM) are performed most commonly in a frequency-modulation (FM) mode. This is achieved by configuring the AFM cantilever as an oscillator, enabling highly sensitive frequency detection with quasi real-time readout. Use of FM-AFM has led to spectacular results, including direct observation of the atomic structure of complex molecules and quantification of chemical and frictional forces at the atomic scale.

In this talk, I will briefly review the theory underpinning FM-AFM force measurements that allows conversion of the measured frequency shift to the interaction force law experienced by the cantilever tip. This will be followed by new research [1] showing that this force conversion capability is directly regulated by the shape of the interaction force law – an effect that depends critically on the oscillation amplitude used. Rapidly varying interatomic force laws, which are common in nature, can lead to unphysical results. A mathematical theory is derived that enables reliable force measurements in practice. The validity of this theory is demonstrated by comparison to atomically-resolved measurements.

Reference:

1. J. E. Sader, B. D. Hughes, F. Huber and F. J. Giessibl, *Nature Nanotechnology*, 13, 1088 (2018).

3:00pm NS-ThA-3 Intermittent Contact Resonance Atomic Force Microscopy (icr-Afm) for Nanoscale Mechanical Property Characterization, *Gheorghe Stan*, National Institute of Standards and Technology **INVITED**

In the last two decades, significant progress has been made on developing new dynamic atomic force microscopy-based methods for nanoscale mechanical properties measurements. The changes in the tip-sample contact mechanics during scanning uniquely modify the high-frequency response of the AFM cantilever and much effort is dedicated to correctly retrieve the sample mechanical properties from the measured signal. Recently in a newly proposed dynamic AFM method, namely the intermittent-contact resonance atomic force microscopy (ICR-AFM), the contact stiffness measurement capability of the conventional contact resonance AFM (CR-AFM) was paired with the less-invasive surface probing of a force-controlled intermittent AFM mode. As an AFM tip goes in and out of contact with the sample during scanning, the change in the tip-sample contact stiffness is observed in the change of the eigenmode frequencies of the cantilever and a fast detection is required to measure the frequency changes during each tap. By collecting the depth dependence of the contact resonance frequency at each point in the scan, a three-dimensional (3D) data volume is generated. This data can be used to obtain nanoscale tomographic views of the sub-surface elastic properties of a material. The involved tip-sample contact mechanics also poses some challenges for samples with edge geometries. A very convenient and robust method to address these types of contact geometries was found in the form of the conjugate gradient method applied to contact mechanics. In this talk, ICR-AFM implementation, measurements, and necessary contact mechanics models will be discussed for mechanical property characterization of thin films and nanostructures.

4:00pm NS-ThA-6 Novel Approaches Towards Cantilevers for Functional Multiparametric AFM Characterization, *Georg Ernest Fantner*, *N Hosseini*, *M Neuenschwander*, *B Ghadiani*, École Polytechnique Fédérale de Lausanne, Switzerland

The cantilever is arguably the most important part in the measurement chain of an atomic force microscope (AFM), because it transduces the interaction with the sample to a measurable quantity. While a large variety of different cantilevers are available for different AFM modes, most of these cantilevers use the same concepts as the first AFM cantilevers developed 30 years ago. The progress in AFM towards techniques such as high-speed AFM and multiparametric imaging puts new demands on the AFM cantilevers. In this talk I will discuss several ways we are exploring to increase the performance of AFM cantilevers by using non-standard

materials, fabrication processes and actuation schemes. Applications of these new cantilevers are high-speed AFM multi-parametric imaging, and correlated microscopy.

4:20pm NS-ThA-7 Fluid Handling using Scanning Probe Lithography for Nanocombinatorics, *V Saygin*, *N Alsharif*, *Keith A. Brown*, Boston University

Scanning probes have been widely applied as characterization tools due to their high resolution and versatility. In parallel with the development of these capabilities, scanning probe lithography (SPL) has been advanced such that it is now capable of directly writing nanoscale domains of soft materials such as polymers, a capability that can be massively parallelized across centimeter scales through the use of cantilever-free scanning probe arrays. In this talk, we will discuss recent advances in the development of nanoscale fluid handling using scanning probes and describe how these advances allow for the realization of libraries for nanocombinatorial studies. Despite the utility of these approaches, operating with nanoscale domains of fluid raises interesting challenges in terms of managing capillary phenomena, evaporation, and deterministically directing fluid transfer. In order to address these, we have performed a series of studies using atomic force microscopy to explore nanoscale fluid dynamics. After being written, polymer features can function as reactors for subsequent chemistry or as samples for further characterization. We explore the opportunities and challenges inherent to this class of experiment and highlight recent discoveries made using such libraries. While the majority of functional explorations using scanning probes center around use of these instruments as characterization tools, the concept that scanning probes can also prepare combinatorial libraries is becoming increasingly common and providing new avenues for nanoscale science.

4:40pm NS-ThA-8 Accuracy of Tip-sample Interaction Measurements Using Dynamic Atomic Force Microscopy Techniques, *O Dagdeviren*, *Udo D. Schwarz*, Yale University

Atomic force microscopy (AFM) is a versatile surface characterization method that can map a sample's topography with high spatial resolution while simultaneously interrogating its surface chemistry through the site-specific high-resolution quantification of the forces acting between the sample and the probe tip. Thanks to considerable advances in AFM measurement technology, such local measurements of chemical properties have gained much popularity in recent years. To this end, dynamic AFM methodologies are implemented where either the oscillation frequency or the oscillation amplitude and phase of the vibrating cantilever are recorded as a function of tip-sample distance and subsequently converted to reflect tip-sample forces or interaction potentials. Such conversion has, however, been shown to produce non-negligible errors when applying the most commonly used mathematical conversion procedures if oscillation amplitudes are of the order of the decay length of the interaction [1]. The degree of divergence from actual values may also critically depend on both the overall strength of tip-sample interaction and the distance at which the interaction is obtained [2]. These systematic errors can, however, be effectively eliminated by using oscillation amplitudes that are sufficiently larger than the decay length of the interaction potential.

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

[2] O. E. Dagdeviren et al., *Review of Scientific Instruments* 90, 033707 (2019).

5:00pm NS-ThA-9 Utilizing AFM to Study the Effect of Malaria-derived EVs on the Mechanical and Morphological Properties of Red Blood Cells, *Irit Rosenhek-Goldian*, *E Dekel*, *Y Ohana*, *S Maihib*, *S Cohen*, *N Regev-Rudzik*, Weizmann Institute of Science, Israel

The deformability of Red Blood Cells (RBCs) is critical for the function of the cell and its viability. RBCs deform substantially and repeatedly when passing through narrow capillaries. There is growing evidence that RBC deformability is impaired in some pathological conditions. This is the case when the human malaria parasite invades these cells, leading to the secretion of Extracellular Vesicles (EVs) whose mechanistic effect on healthy RBCs is unknown.

We have applied atomic force microscopy (AFM) to study the mechanical changes occurring in cells treated with malaria-derived EVs, as well as morphological transformations in the cellular cytoskeleton. Mechanical measurements were made at physiological temperature without covalent linkage of the cells to the substrate to allow closest approximation to their natural state. Indentations were performed using a colloidal tip at applied forces kept sufficiently low to avoid damage to the cells as verified by comparing images taken before and after the mechanical test. Young's

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modulus values computed by Herzian analysis were achieved with sensitivity of 100 Pa. The results revealed a significant drop in compliance of the infected cells, with the mean value falling by a factor of approximately three for the infected ones. Furthermore, high-resolution images of dried cells with exposed cytoskeleton show distinct morphological differences associated with the breakdown and softening of the cell structure.

These results will be discussed with respect to the currently unknown mechanistic role of parasite-derived EVs on the RBC host membrane.

5:20pm **NS-ThA-10 Silicon Oxide for RRAM Application: The SPM Analysis Approach**, *Adnan Mehonic, M Buckwell, W Ng, A Kenyon*, University College London, UK

INVITED

Resistive Random Access Memory (RRAM) has established itself as a promising technology for the next generation of non-volatile memories due to the simple design, high scalability, fast and low-power operation. Additionally, RRAM devices are considered for the implementation of power efficient hardware in applications of artificial intelligence (AI) and machine learning (ML) implemented in non-von Neumann architectures. Redox-based RRAM (ReRAM), based on the formation of conductive filaments in thin metal oxides are particularly popular due to excellent CMOS compatibility. However, significant challenges still exist for the full utilisation of the technology; such as device variability and yield. To better design and optimise the devices it is crucial to understand the physics that underlies the resistance switching processes. Here we present how SPM techniques can be used to characterise silicon oxide-based ReRAM devices. We find these techniques to be invaluable for developing a better sense of the oxide microstructure and the link with resistance switching processes. We also use the method of conductance tomography to directly visualise the shapes and sizes of conductive filaments in three dimensions - this is typically extremely challenging to obtain using conventional microscopy techniques.

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