Nanometer-scale Science and Technology
Room 101D - Session NS-TuA

Nanoscale Imaging and Characterization

Moderators: Mehmet Z. Baykara, Bilkent University, Turkey, Sidney Cohen, Weizmann Institute of Science, Israel, Rainer Timm, Lund University, Sweden

2:20pm **NS-TuA1 Frontiers of Force Microscopy in Nanoscience and Nanotechnology*, Ricardo Garcia*[*], CSIC, Spain** **INVITED**

This contribution aims to provide an overview of some recent developments as well as some challenges faced by force microscopy in nanoscience and nanotechnology. Specifically, the focus will be oriented to applications to study materials in air and liquid environments. The presentation is divided in three sections. The first section provides an introduction to the physics and key instrumental aspects of advanced force microscopes. The second section describes some applications to generate high resolution (atomic, molecular or nanoscale) maps of soft matter interfaces (polymer and biomolecules). Those maps combined topography and nanomechanical properties. A method to generate three dimensional and atomically-resolved mapsof solid-liquid interfaces will be presented. The third section, illustrates how the nanoscale control afforded by scanning probe microscopes has enabled the development of scanning probe-based patterning methods.

References:

E.T. Herruzo, A.P. Perrino and R. Garcia, *Nature Commun.* **5**, 3126 (2014)

R. Garcia and E. T. Herruzo, *Nature Nanotechnol*. **7**, 217-226 (2012).

R.Garcia, A.W. Knoll, E. Riedo, Nature Nanotechnology 9, 577-587 (2014)

3:00pm **NS-TuA3 Elemental and Magnetic Fingerprinting of Materials at the Nanoscale by Synchrotron X-ray Scanning Tunneling Microscopy*, Volker Rose,*** Argonne National Laboratory **INVITED**

Recently, substantial progress was made on Argonne's Synchrotron X-ray Scanning Tunneling Microscopy (SX-STM) project. In particular, we demonstrated the power of SX-STM for elemental characterization and topography of individual Ni nano-islands on Cu(111) at 2 nm lateral resolution with single atom height sensitivity [1], tested a new probe tip concept based on carbon nanotubes [2], and demonstrated soft x-ray imaging of nanoscale magnetic domains of an iron thin-film by x-ray magnetic circular dichroism (XMCD) contrast [3]. Further substantial advances are expected using the new low temperature (LT) SX-STM system, which has been developed over the last 3 years and is currently under commissioning.

To fully exploit the special capabilities of the new LT x-ray microscope, XTIP, a dedicated beamline for SX-STM is under construction at the Advanced Photon Source. 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 400-1600 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.

This work was funded by the Office of Science Early Career Research Program through the Division of Scientific User Facilities, Office of Basic Energy Sciences of the U.S. Department of Energy through Grant SC70705. Use of the Advanced Photon Source and the Center for Nanoscale Materials was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under contract DE-AC02-06CH11357.

[1] N. Shirato et al., Nano Letters 14, 6499 (2014).

[2] H. Yan et al., J. Nanomaterials 2015, 492657 (2015).

[3] A. DiLullo et al., J Synchrotron Rad. 23, 574 (2016).

4:20pm **NS-TuA7 Quantitative Nanomechanics of Soft Materials with AFM: Old and New Methods*, Igor Sokolov,*** Tufts University **INVITED**

Quantitative study of mechanical properties of soft materials at the nanoscale, such as the Young's modulus, storage and loss moduli, etc. becomes important in the study of nanocomposites materials, polymers, biological tissues, and cells. Nanoindentation techniques are not capable to attain nanoscale resolution for such materials; whereas atomic force microscopy (AFM) techniques do allow quantitative measurements of soft materials at the nanoscale. Although being attractive and simple, direct extrapolation of macroscopic models to the nanoscale is frequently incorrect. In this talk I will describe the AFM methods suitable for measurements of both static and dynamic moduli of soft materials. I will discuss both well-known modes of operation, such as the force-volume and nonresonant modes (e.g, PeakForce QNM) as well as new modes we recently developed (FT-NanoDMA or imaging nanoindentation and Ringing modes). Comparison of all these modes (including the nanoindentation technique) will be given. Studies of elastic moduli of polymers and cells will be exampled.

5:00pm **NS-TuA9 AFM Based Nanoscale Structure-Property Characterization of Nanoporous Organo-Silicates*, Qichi Hu,*** *K. Kjoller,* Anasys Instruments*; G. Stan,* NIST/Material Measurement Laboratory*; S.W. King,* Intel Corporation

The continued advancement of nanostructured materials and exploitation of nanoscale size effects will ultimately require understanding material structure-property relationships at nanometer length scales. Despite a plethora of metrologies capable of characterizing thermal, mechanical, electrical, and optical properties at the nanoscale, combined nanoscale chemical structure-property characterization has only recently become possible with the development of atomic force microscope based IR spectroscopy (ARM-IR). In this regard, we have combined AFM-IR chemical structure and contact resonance AFM (CR-AFM) mechanical property measurements in the investigation of 20 – 500 nm wide fin structures fabricated in a nanoporous organosilicate material. By combining these techniques, we have observed nanoscale modifications in the chemical structure and mechanical properties of the nanoporous fins that correlate with one another, the feature size, and fabrication process. This demonstration should lead the way for nanoscale chemical structure-property characterization of other materials systems where such relationships are deemed essential.

5:40pm **NS-TuA11 Scanning Microwave Microscopy Imaging in Liquids through Ultra-Thin Membranes*, Alexander Tselev,*** Oak Ridge National Laboratory*; J. Velmurugan,* National Institute of Standards and Technology (NIST), University of Maryland (UMD)*; A. Kolmakov,* NIST/CNST

The growing need in operando imaging of submicron objects immersed in liquids relevant to biomedical or energy applications resulted in a significant effort invested into in situ TEM and SEM. In these techniques, objects of interest are incased inside a chamber equipped with ultra-thin electron-transparent but molecularly-impermeable membrane(s) enabling electron or X-ray probing of the chamber interior. However, local radiation damage and radiolysis induced by high-energy electron or X-ray beams often lead to sample deterioration or adversely affect nanoscale chemical processes. Here, we report a novel concept of in situ near-field scanning microwave microscopy of reactive and biological samples in liquids. Microwaves of a few gigahertz frequencies offer photons of energies ~10 μeV, which ensures non-destructive imaging free from radiolysis and radiation damage associated with use of high-energy electron and X-ray beams. In our approach, the nanoscale objects of interest are separated from ambient by a-few-nanometer-thick dielectric membranes transparent for microwave near-fields. The imaging is performed with microwave near-fields formed at a scanning probe of an atomic force microscope in contact with the ultra-thin membrane. In the proposed approach, a liquid and/or a reactive environment of the object of interest are completely isolated from the probe and the rest of the microscope. We performed a comparative, side-by-side study of imaging capabilities of microwave microscopy and SEM in liquids using the same set of biological and inorganic samples. Sensitivity, spatial resolution, probing depth, and probe-induced effects were evaluated and compared. In particular, we demonstrate in situ real-time imaging of growth of metal dendrites at electrode-liquid electrolyte interface during an electrochemical reaction. The demonstrated spatial resolution of the near-field microwave imaging was ca. 250 nm. The resolution can be improved by optimization of probe and membrane geometry, as well as of the membrane material. Under optimal conditions, a resolution of ca. 50 nm can be achieved for metallic objects with commercially available probes. Such resolution is comparable to that demonstrated by in situ SEM in liquids.

The research was supported in part through Scientific User Facilities Division (ORNL), BES, US DOE, US Civilian Research and Development Foundation, and NIST-CNST/UMD-IREAP Cooperative Agreement.

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[*] **NSTD Recognition Award**

6:00pm **NS-TuA12 SnS Nanoplates***, **Nancy Trejo**, A. Hunter, C. Wrasman, S. Ganguly,* University of Minnesota*; J. Dwyer,* St. Catherine University*; E.S. Aydil,* University of Minnesota

Two dimensional (2D) layered materials such as graphene, metal dichalcogenides and black phosphorus are of increasing interest because of their unique electronic properties. Tin monosulfide (SnS) has the same crystal structure as black phosphorus. SnS may have potential applications in photovoltaics, photocatalysis, thermoelectrics, and batteries. We have synthesized 3-60 nm thick and up to approximately 10 micron wide SnS nanoplates via decomposition of tin(IV) diethyldithiocarbamate upon hot injection into oleylamine (300-340 ^{o}C). The reaction products are characterized using a combination of electron microscopy, energy dispersive X-ray spectroscopy, X-ray diffraction (XRD), atomic force microscopy and Raman spectroscopy. All characterization techniques confirm that the final reaction product is orthorhombic SnS (*e.g.,* after 60 minutes at 340 ^{o}C). At low temperatures (300 ^{o}C) and short synthesis times (1 minute) we also observe the presence of SnS_2 which suggests that decomposition of tin(IV) diethyldithiocarbamate first produces SnS_2 . Tin disulfide is subsequently reduced, likely by oleylamine, to SnS. Orthorhombic SnS grows preferentially as plates, with [010] direction normal to the plate surfaces. In fact, when plates are large (>1 micron) XRD from films cast from colloidal dispersions in toluene show predominantly the (040) diffraction. The SnS nanoplate sizes could be altered by controlling the temperature, oleylamine concentration, and reaction time. Nanoplate dispersions in toluene exhibit an optical absorption feature in the visible range of the electromagnetic spectrum, which we surmise to be of plasmonic origin. The nanoplate dispersions in toluene also respond to electric fields.

Author Index

Bold page numbers indicate presenter