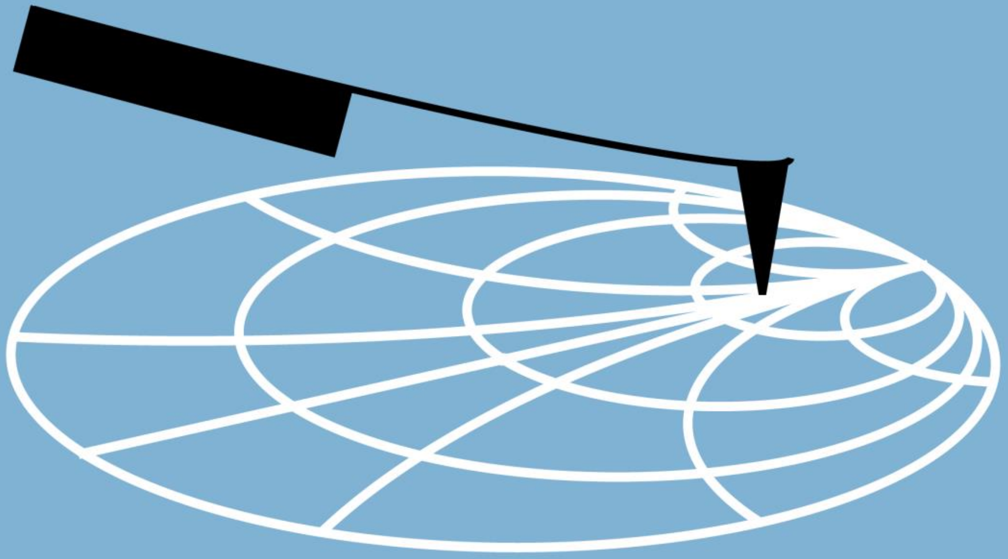
Radio Frequency Scanning Probe Microscopy Workshop



Wednesday, November 29, 2017
St. Julien Hotel, Boulder, Colorado

Held in conjunction with the Fall 2017
ARFTG Symposium (www.arftg.org)

ABSTRACTS

Radio Frequency Scanning Probe Microscopy Workshop

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Session 1 (Mitch Wallis, NIST, Chair)

08:00-08:40 "Microwave Impedance Microscopy as a Probe of Quantum Phenomena in Prospect Topological Phases of Matter" Z. X. Shen, *Stanford University*

Microwave Impedance Microscopy (MIM) as a local transport probe has seen a growing application in quantum phenomena in solids. In this talk, we discuss results of local conductivity investigation on edge states and domain walls in a number of topological materials: i) unusual metallic domain walls in magnetic insulator Nd2Ir2O7[1]; ii) quantum phase transitions in magnetic topological insulators of delta doped Cr/(Bi,Sb)2Te3[2]. The development of MIM and other measurements from quantum materials will also be discussed.

[1] E.Y. Ma et al., Science 350, 538-541 (2015)

[2] M. Allen et al., unpublished

08:40-09:20 "Advances in imaging and quantification of electrical properties at the nanoscale using Scanning Microwave Impedance Microscopy (sMIM)" Stuart Friedman, Oskar Amster, and Fred Stanke, *PrimeNano*

Understanding and optimizing advanced materials frequently requires detailed knowledge of nanoscale electrical properties. Scanning probe techniques such as scanning tunneling microscopy (STM), conductive AFM (cAFM), scanning capacitance microscopy (SCM), and Kelvin probe force microscopy (KPFM) provide such nano-electrical measurements, but are generally limited in the classes of materials they can characterize or the properties they can measure. Scanning microwave impedance microscopy (sMIM) uses GHz frequency microwaves and shielded AFM probes to directly measure the impedance (capacitance and conductance) of the tip sample interface. As such sMIM is sensitive to the permittivity and conductivity of a wide variety of samples including dielectrics, conductors, and semiconductors.

After introducing the theory of operation, we will review the state of the art, including high resolution imaging of electrical properties of quantum structures and 2D materials and devices. Additionally, we will present research on quantification of dielectric properties and semiconductor doping concentration, including nanoscale capacitance-voltage curves. Research results obtained at cryogenic temperatures and high field will also be discussed.

09:20-10:00 “Probing Domain Wall Vibration and Electroacoustic Transduction by Microwave Microscopy” Keji Lai, *University of Texas, Austin*

Microwave impedance microscopy (MIM) probes the nanoscale response to electromagnetic excitation, which is usually dominated by the local conductivity of the material. Here, we report two experiments where the MIM contrast is not associated with the free-carrier conduction. In ferroelectric hexagonal rare-earth manganites, the dissipation of electrical energy of domain walls on the (001) surfaces is substantially enhanced at GHz frequencies than that at DC, whereas the effect is absent on surfaces with in-plane polarized domains. First-principles-based model calculations indicate that the frequency range and selection rules are consistent with the presence of a localized vibrational mode, i.e., the periodic DW sliding around its equilibrium position. In this case, the MIM can map out the nanoscale dielectric loss of the sample. In addition to the domain wall oscillation, the local electroacoustic energy transduction in lithium niobate domains can also be visualized by the MIM. The interference-like features vividly demonstrate the spatial variation of surface-acoustic-wave generation, which can be understood by finite-element modeling of the energy conversion. In contrast to the standing-wave patterns due to wave reflection from hard boundaries, the fringes with one-wavelength periodicity are the consequence of sign reversal of the piezoelectric coefficients in opposite domains. Our work opens up a new frontier to explore various low-energy dynamic phenomena in complex materials and novel devices by nanoscale electromagnetic imaging.

10:00-10:40 Break

Session 2 (Joe Kopanski, NIST, Chair)

10:40-11:20 “Calibrated permittivity and conductivity measurements of nanoscale surface and subsurface structures by scanning microwave microscopy” Ferry Kienberger and Georg Gramse, *Keysight Technologies*

Scanning Microwave Microscopy (SMM) is a nanoscale imaging technique that combines the lateral resolution of Atomic Force Microscopy (AFM) with the high measurement precision of microwave analysis at GHz frequencies, provided by Vector Network Analyzers (VNA). SMM enables measuring complex materials properties for nano-electronics, materials science, and life science applications. SMM operates at broadband frequencies between 1 GHz and 20 GHz. We developed novel calibration workflows for complex impedance imaging[1-2] and dielectric quantification[3]. Various nanodevices are studied including dopant profiling layers and high voltage transistors[4,5]. Based on the high frequency the laborious fabrication of back electrode contacts is not required, making it an easily applicable tool for electrical characterization of nanodevices. The capability of the electromagnetic waves to penetrate the surface of the sample under study allows the technique to be used to selectively sense sub-surface features[6]. The sub-surface and quantitative resistivity measurement capabilities are demonstrated for silicon back-wafer imaging and semiconductor failure analysis. As will be shown as an ultimate limit even atomically thin regions of dopant atoms in silicon can be electrically characterized and localized in 3 dimensions with nm resolution[7]. Such dopant structures are building blocks of quantum devices for physics research and it is anticipated that they will also serve as key components of devices for next generation classical and quantum information processing.

[1] G. Gramse et al, "Calibrated complex impedance and permittivity measurements with scanning microwave microscopy", *Nanotechnology*, 25, 145703 (2014)
[2] M. Kasper et al, "An advanced impedance calibration method for nanoscale microwave imaging at broad frequency range," *IEEE Transactions on MTT 65, 7,* 2017, 2418-24
[3] M.C. Biagi et al, "Nanoscale Electric Permittivity of Single Bacterial Cells at Gigahertz Frequencies by Scanning Microwave Microscopy," *ACS Nano*, 10, 280 (2016)
[4] E. Brinciotti et al, "Calibrated nanoscale dopant profiling and capacitance of a high-voltage lateral MOS transistor at 20 GHz using Scanning Microwave Microscopy," *IEEE Transactions on Nanotechnology*, vol.16, no.2, pp.245-252 (2017)
[5] E. Brinciotti et al, "Frequency Analysis of Dopant Profiling and Capacitance Spectroscopy Using Scanning Microwave Microscopy," *IEEE Transactions on Nanotechnology*, vol.16, no.1, pp.75-82 (2017)
[6] G. Gramse and E. Brinciotti et al, "Quantitative sub-surface and non-contact imaging by scanning microwave microscopy," *Nanotechnology 26, 135701 (2015)*
[7] G. Gramse et al., Non-destructive imaging of atomically-thin nanostructures buried in silicon. *Science Advances 3, 6 (2017)*

11:20-12:00 "Microwave Near-Field Imaging of Nanoscale Electronic Properties"

Sam Berweger, T. Mitch Wallis, and Pavel Kabos, *NIST*

New generations of electronic and photonic devices are emerging that leverage the novel properties of low-dimensional and nanoscale materials. However, the successful application of such systems will require a detailed understanding of the interplay between the nanoscale structure and the associated electronic and optical properties. Scanning microwave microscopy (SMM) provides a powerful tool to study nanoscale electronic structure and properties *in situ* and non-destructively by directly probing local conductivity variations with nanometer spatial resolution. Here we discuss our recent work studying variations in electronic properties in low-dimensional and thin film materials.

We emphasize the capabilities of SMM by studying the electronic variations in GaN nanowires integrated into a simple transistor architecture, where we can simultaneously control the local carrier density by applying a tip bias and image the resulting local carrier density variations. We will then discuss the specific application to select semiconducting 2D transition metal dichalcogenides (TMD's). We modify the sample charge carrier concentration and optimize the related SMM contrast to identify spatial local variations in sample conductivity. Together with finite element modeling we determine the dominant charge carrier type and extract the local dopant concentration. Lastly, we will discuss our work studying local compositional and associated electronic variations in pristine and deteriorated perovskite photovoltaic films.

12:00-13:00 Lunch

Session 3 (Sam Berweger, NIST, Chair)

13:00-13:40 "Near-Field Nonlinear Microwave Microscopy of Superconductors"

Bakhrom Oripov, Tamin Tai, Seokjin Bae, and Steve Anlage, *University of Maryland*

The microscopic origins of superconducting radio frequency (SRF) accelerator cavity breakdown are still a matter of some debate. A means to evaluate the deleterious RF properties of surface defects under conditions experienced in the SRF cavity is most desirable. We have developed a near-field microwave microscope that measures local nonlinear response (harmonic generation) from bulk and thin film superconductor surfaces under conditions approaching Bsurface ~ 200 mT at temperatures down to 2.7 K and frequencies in the multi-GHz range. The microscope reveals significant nonlinear response (3rd harmonic voltage V3f(T, Brf)) beginning at Tc and continuing to lower temperatures. Measurements on bulk Nb show evidence of suppression of Tc, suggesting that fields on the order of the critical field are being developed on the surface of the sample. Measurements on Nb show systematic trends with temperature; for example a periodic nonlinear response as a function of rf magnetic field suggests quantized response associated with rf vortex generation. We will also discuss our efforts to model the nonlinear response and relate it to SRF-relevant quantities.

In another experiment, we have directly imaged the anisotropic nonlinear Meissner effect in an unconventional high-temperature superconductor through the nonlinear electrodynamic response of both (bulk) gap nodes and (surface) Andreev bound states [1]. A superconducting thin film is patterned into a compact self-resonant spiral meta-atom structure, excited near resonance in the radio-frequency range, and scanned with a focused laser beam perturbation. At low temperatures, direction-dependent nonlinearities in the reactive and resistive properties of the resonator create photoresponse that maps out the directions of nodes, or of bound states associated with these nodes, on the Fermi surface of the superconductor. The method is demonstrated on the nodal superconductor YBa2Cu3O7-δ and the results are consistent with theoretical predictions for the bulk and surface contributions. We have recently extended the method to much lower temperatures and to examine other superconducting materials. We discuss ongoing efforts to use microscopic calculations to understand the experimental results.

This work is supported by the US Department of Energy/High Energy Physics (Award No. DE-SC0012036), the US National Science Foundation through grant # DMR1410712, and CNAM.

[1] A. P. Zhuravel, B. G. Ghamsari, C. Kurter, P. Jung, S. Remillard, J. Abrahams, A. V. Lukashenko, A. V. Ustinov, Steven M. Anlage, "Imaging the Anisotropic Nonlinear Meissner Effect in Nodal $YBa_2Cu_3O_{7-\delta}$ Thin-Film Superconductors," Phys. Rev. Lett. 110, 087002 (2013).

13:40-14:20 "Near Field Scanning Microwave Microscopy in Biology for Cellular and Subcellular Characterization" Marco Farina, Davide Mencarelli, A. Morini, and A. Di Donato, *U. delle Marche*

Near field Scanning Microwave Microscopy (SMM) is emerging as an important complementary technique for the experimental characterization of biological samples, down to nano-scale. Working with cells or biological tissues poses several problems, especially if samples are to be scanned in-vivo in physiological environment (liquid buffer). Working with relaxed operating conditions, like scanning dried samples (or samples while drying), could be less difficult, but still highly challenging. For instance, whenever using STM-assisted SMM there may be conflicting constraints, like the use of low bias to avoid electrochemical reactions and to avoid amplifier saturation from ionic currents, which is in contrast with the need of high bias to allow the detection of non-conducting surfaces. From a more general point of view, the present work proposes a wide discussion on some of the main challenges of the SMM technique, in particular: achieving quantitative measurements during imaging, looking "underneath" the surface (sub-surface probing), and imaging transient and dynamical phenomena. Both STM- and AFM-based implementations of microwave microscopy are considered for comparison.

The calibration issue, a central point of any SMM measurement, is also addressed, by considering the capability of the system to provide quantitative data about the surface electrical features, and by trying to disentangle the sample information from topography or from unwanted electromagnetic signals – reflected back to the probe tip. In this context, a possible approach for time-domain calibration is described, starting from a broadband measurement which allows spectroscopic analysis and Time-Domain Reflectometry (TDR).

Several examples about microwave scanning of biological scans are reported.

14:20-15:00 "Recent Progress in Microwave Scanning Probe Microscopy at NIST Gaithersburg," Joe Kopanski and Lin You, *NIST*

The ability of microwave signals from the Scanning Microwave Microscope (SMM) to interact with subsurface features and then be detected in reflection opens the possibility of acquiring three-dimensional information with a scanning probe platform. We have used a SMM based on an Keysight* SPM platform to image subsurface metal lines buried in TEOS dielectric from a multi-level metallization process for integrated circuits. Contrast is obtained from both reflected signal amplitude and the phase. At some frequencies, SMM images showed contrast reversal, that is, sometimes the buried lines reflected more signal than the surrounding background matrix and sometimes they reflected less signal than the background. A finite-element model of the SMM was developed using the electrostatic and RF modules of COMSOL. COMSOL results explains the contrast reversal that sometimes occurs as arising from the shift of the resonance frequency due to varying impedance between the tip and sample as a function of measurement frequency.

We will also describe development of reference materials for determining the spatial resolution and accuracy of electric field gradient measurements with SPMs. Electric field measurements on the nanoscale depend critically on the shape of the electrodes used to generate and detect the field. Fabricated structures include near atomically flat Pt-Au junctions on high grade mica and various structures fabricated with a four-layer metallization process. An electric field gradient reference structure uses a voltage divider to provide four orders of magnitude change in electric field over a short distance with various resolutions. Various electric tip shape profiler structures allow the electrical tip shape to be deduced from electrical field measurements of the test structure. A procedure for extracting electric tip shape from measurements and results on various commercially available electrical SPM tips has been demonstrated.

15:00-15:30 Break

Session 4 (Pavel Kabos, NIST, Chair)

15:30-16:10 "Integrated Atomic Force Microscope/Scanning Microwave Microscope on a Single CMOS-MEMS Chip" Raafat Mansour, *University of Waterloo*

This talk introduces a new class of devices and instrumentations that leverage the myriad economic benefits of having integrated systems with both electrical and mechanical functionality on a single-chip. This is enabled by a CMOS post-processing technique developed at the University of Waterloo to create MEMS devices within CMOS chips. We integrate actuation, sensing and control electronics on a single CMOS-chip. This unique chip-scaled technology platform enables the development of highly advanced devices for a wide range of applications including radio frequency (RF) communication systems and nano-instrumentations. The talk will address the use of CMOS-MEMS technology in the realization of instrumentations such as an integrated atomic force microscope (AFM) / scanning microwave microscope (SMM) on a single CMOS chip.

16:10-16:50 "Radio Frequency Nano-Probing Under Scanning Electron Microscopy" Kamel Haddadi, O. C. Haenssler, K. Daffe, S. Eliet, C. Boyaval, S. Arscott, D. Theron, and G. Dambrine, *University Lille*

To foster the progress of miniaturization of electronic circuits, new metrological issues related to the dimensional and electrical characterization of nanoelectronic devices must be addressed. In particular, the radio frequency characterization of nanoelectronic devices such as attofarad capacitors, nanoscale contacts, 1D and 2D based devices is still challenging. Among the most promising solutions, the introduction of near-field scanning microwave microscopy (NSMM) tools have pioneered the possibility to measure microwave impedances of one-port devices with nanometer-scale spatial resolution. Basically, the NSMM consists of an atomic force microscope (AFM) combined with a microwave signal applied to the tip. The tip scans across the sample, emitting a microwave signal scattered by the material, altering its amplitude or/and phase properties. In this frame, unique hybrid measuring equipments combining guided/near-field radio frequency (RF) probing, nanorobotics and scanning electron microscopy (SEM) is developed for tackling the frontiers between spatial resolution and frequency domain. The systems can produce simultaneously RF complex impedance, AFM and SEM measurements featuring unprecedented capabilities for nanoscale material and device analysis. The theory, techniques, instrumentations and related applications will be exemplary shown.

16:50-17:30 "Scanning microwave microscopy of cristae remodeling of the interior of mitochondria," Peter Burke, *University of California, Irvine*

There is considerable debate and controversy about how the interior ultra-structure of mitochondria changes (called cristae remodeling) in order for the protein CytC to be released from mitochondria, the point of no return for apoptosis, which is programmed cell death in response to DNA damage, ROS, stress, and cell-death signals. Resistance to cell death signals is a hallmark of cancer, and a target for pharmacological manipulation. Optical methods do not have enough spatial resolution for such detection. Electrophysiological methods such as patch clamp have been attempted with only limited success, since the ultra-structure has deep folds and crevasses (cristae) that are 10 nm wide and 500 nm deep. TEM has enough special resolution but the microconidia must be fixed to be imaged. To overcome those problems, scanning microwave microscope (SMM) stands out among many options. It has nanoscale special resolution, the microwave detecting signal can penetrate further in ionic solution before being screened, and it is capable of measuring live organelles in liquid. However, SMM is a rather newly developed tool, and its functioning in liquid is quite challenging to achieve calibrated and high resolution detection, with interpretation of the images also an unsolved challenge. Conventional calibration techniques do not work in our case, due to the presence of a highly conducting ionic solution. We ended up working to fabricate our own calibration standards (metal disks of known diameter) directly on the side of mitochondria on the same substrate. By doing that we can compare the SMM signal from mitochondria with that from the known standards nearby to give a reasonable estimation of absolute tip-mitochondria capacitance. To enhance the signal to noise ratio in the ionic solution, we have used a microwave interferometer designed right after the probe to read out the tip-sample reflection. We tethered single mitochondria onto a graphene substrate, and with those enhancements, we are able to map the tip-mitochondria capacitance pixel by pixel. We are expecting to see the capacitance change due to cristae remodeling occurring in response to BH3 peptides BID and BIM. Beyond the specific purpose of this work, the practice of SMM in a liquid environment can generate more possibilities for nano-scale interfaces with living systems.