Wednesday Morning, October 21, 2015

Scanning Probe Microscopy Focus Topic
Room: 212A - Session SP+AS+NS+SS-WeM

Advances in Scanning Probe Microscopy

Moderator: An-Ping Li, Oak Ridge National Lab, Saban Hus, Oak Ridge National Laboratory

8:00am **SP+AS+NS+SS-WeM1 Designer Electrons: Quantum Information and New Particles in Atomically Assembled Matter**, ***Hari Manoharan***, Stanford University **INVITED**

The observation of massless Dirac fermions in monolayer graphene has propelled a new area of science and technology seeking to harness charge carriers that behave relativistically within solid-state materials. Using low-temperature scanning tunneling microscopy and spectroscopy, we show the emergence of Dirac fermions in a fully tunable condensed-matter system—molecular graphene—assembled via atomic manipulation of a conventional two-dimensional electron system in a surface state. We embed, image, and tune the symmetries underlying the two-dimensional Dirac equation into these electrons by sculpting the surface potential with manipulated molecules. By distorting the effective electron hopping parameters into a Kekulé pattern, we find that these natively massless Dirac particles can be endowed with a tunable mass engendered by the associated scalar gauge field, in analogy to the Higgs field. With altered symmetry and texturing of the assembled lattices, the Dirac fermions can be dressed with gauge electric or magnetic fields such that the carriers believe they are in real fields and condense into the corresponding ground state, as confirmed by tunneling spectroscopy. Using these techniques we ultimately fabricate a quantum Hall state without breaking time-reversal symmetry, in which electrons quantize in a gauge magnetic field ramped to 60 Tesla with zero applied laboratory field. We show that these and other chiral states now possible to realize have direct analogues in topological insulators, and can be used to guide or confine charge in nontrivial ways or to synthesize new particles [1,2].

[1] K. K. Gomes, W. Mar, W. Ko, F. Guinea, H. C. Manoharan, "Designer Dirac Fermions and Topological Phases in Molecular Graphene," *Nature* **483**, 306–310 (2012).

[2] M. Polini, F. Guinea, M. Lewenstein, H. C. Manoharan, V. Pellegrini, "Artificial Honeycomb Lattices for Electrons, Atoms, and Photons," *Nature Nanotechnology* **8**, 625–633 (2013).

8:40am **SP+AS+NS+SS-WeM3 Scanning Quantum Dot Microscopy**, ***Ruslan Temirov***, *C.W. Wagner, M.F.B.G. Green, P.L. Leinen,* Forschungszentrum Juelich GmbH, Germany, *T.D. Deilmann, P. Krueger, M.R. Rohlfing,* Muenster University, Germany, *F.S.T. Tautz*, Forschungszentrum Juelich GmbH, Germany

Interactions between atomic and molecular objects are to a large extent defined by the nanoscale electrostatic

potentials which these objects produce. Consequently, a tool for nanometre scale imaging and quantification of

local electrostatic fields could help in many areas of nanoscience research. In this contribution we introduce a

scanning probe technique that for the first time enables truly three-dimensional imaging of local electrostatic

potential fields with sub-nanometre resolution. Registering single electron charging events of a molecular

quantum dot attached to the tip of a tuning fork atomic force microscope operated at 5 K, we image the

quadrupole field of a single molecule adsorbed on a metal surface. To demonstrate quantitative measurements,

we investigate the Smoluchowski dipole field created by a single metal adatom adsorbed on a metal surface. We

show that because of its high sensitivity the technique can probe electrostatic potentials at large distances from

their sources, which should allow for the imaging of samples with increased surface roughness.

Reference

[1] C. Wagner, M. F. B. Green, P. Leinen, T. Deilmann, P. Krüger, M. Rohlfing, R. Temirov, F. S. Tautz

arXiv:1503.07738 (2015)

9:00am **SP+AS+NS+SS-WeM4 Local Probing of the Photo-carrier Lifetime by Kelvin Probe Force Microscopy**, ***Nicolas Chevalier***, *S. Pouch, D. Mariolle,* Univ. Grenoble Alpes/ CEA, LETI, MINATEC Campus, France, *B. Grevin,* Univ. Grenoble Alpes/ CEA, INAC, SPrAM, LEMOH, France, *Ł. Borowik*, Univ. Grenoble Alpes/ CEA, LETI, MINATEC Campus, France

The photo-carrier lifetime plays a major role in the overall efficiency of a solar cell because it limits the proportion of photo-generated charges collected at the electrodes. This lifetime, which should be ideally as large as possible in an organic or inorganic solar cell, is rather difficult to measure in nanostructured materials or in more complex hybrid systems, indirect band-gap semiconductors, and ultra-thin layers. Identifying the losses mechanisms is one of the main objectives for increasing the performances of solar cells. Most of the experimental approaches developed so far consist in studying recombination by techniques such as transient photovoltage measurements or charge extraction. All these techniques average sample properties over macroscopic scales, making them unsuitable for directly assessing the impact of local heterogeneity on the recombination process. In this paper, we propose a steady method to measure the photo carrier lifetime by photo-modulated techniques based on Kelvin probe force microscopy (KPFM). [1] Additionally, KPFM technique provides a spatially resolved measurement, which is applicable on the overall of solar cells.

We will present the principle of this original method based on the measurement of the surface potential by KPFM under an illumination with a rectangular waveform light modulation. Photo-carrier lifetime down to µs scale is reachable with our experimental setup. The modulation-dependent surface potential is plotted as a function of the frequency. Assuming an immediate generation time under illumination and an exponential decay of the surface potential during the dark condition, the averaged surface potential over a cycle can be fitted as a function of the frequency by simple equation where the only fit parameter is the photocarrier-lifetime. [2] Instrumental aspects as well as data treatment will be reviewed. Measurements obtained on silicon nanocrystals embedded in 30 nm film of silicon dioxide [3] and on organic donor-acceptor blend (PBTFB and PCBM) [4] will be presented to illustrate the potential of the technique.

This work was supported by the French "Recherche Technologique de Base" Program and performed in the frame of the trSPV Nanoscience project. The measurements were performed on the CEA Minatec Nanocharacterization Platform (PFNC).

1. Ł. Borowik *et al.* Phys. Rev. B 82, 073302 (2010).

2. Ł. Borowik *et al.* Nanotechnology 25, 265703 (2014).

3. D. Asakura *et al.* Phys. Rev. Lett. 93, 247006 (2004).

4. N. Delbosc et al. RSC Adv 4, 15236 (2014).

9:20am **SP+AS+NS+SS-WeM5 Nanoscale Capacitance-Voltage (C-V) Curves: Using Scanning Microwave Impedance Microscopy (sMIM) to Characterize Local Electrical Properties of Linear and Non-Linear Materials**, ***Stuart Friedman***, *Y. Yang, O. Amster*, PrimeNano, Inc.

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.

When sMIM is applied to non-linear materials, changing the tip sample bias changes the local electric field thereby changing the local electrical properties of the sample just under the AFM tip. The electric field induced changes in the sample create changes in the tip-sample impedance that can be measured by sMIM. For example, when imaging doped semiconductor samples, the tip sample interface forms either a metal-semiconductor junction or a metal-insulator-semiconductor junction. Plotting the sMIM measured capacitance as a function of the tip sample bias voltage produces the equivalent of a typical capacitance-voltage curve, but from nanoscale regions selected from an AFM image. C vs V results from doped silicon samples that closely match theoretical calculations will be discussed. The talk will also present results from advanced and novel materials and devices, such as III-V semiconductors, 2D materials and 1D structures

where sMIM data has been used to assess non-linear behavior and characterize dopant type and distribution.

9:40am **SP+AS+NS+SS-WeM6 STM Study of the Correlation between Structural, Magnetic, and Electronic Properties of Co Nano-Islands on Cu(111)**, ***Jewook Park***, *C. Park, M. Yoon, Z. Gai, A.P. Baddorf, A.-P. Li*, Oak Ridge National Laboratory

An epitaxially grown Co nano-island on Cu(111) surface is a model system to study the correlation between structural, magnetic, and electrical properties of nanophase materials. We carried out an extensive study on Co islands by using spin-polarized scanning tunneling microscopy and spectroscopy (SP-STM/S) at low temperatures (130 K and 38 K). Two structurally different island types are clearly distinguished, rotated by 180 about the surface normal due to a stacking fault in one type of the islands. The triangular Co islands are 5-20 nm wide and 4 Å high. Regardless of the structural asymmetry, both faulted and un-faulted Co islands possess two distinctive spin orientations. With Cr-coated W-tip as a spin-polarized probe, bias-dependent tunneling conductance maps are measured on Co islands. An antiparallel spin-orientation between magnetized tip and Co islands display higher conductance compared to a parallel relation at -400 meV and *vice versa* at around Fermi-level, which is verified by density functional theory calculations. Furthermore, by recording 23 hours of time-lapse images from the same Co islands, we demonstrate a time-dependent correlation between structural, magnetic, and electrical behaviors. We find that a contamination-induced structural change modifies the magnetic properties of Co islands and is confirmed by theoretical calculations.

This research was conducted at the Center for Nanophase Materials Sciences, which is a DOE Office of Science User Facility, and supported by the Laboratory Directed Research and Development Program of Oak Ridge National Laboratory, managed by UT-Battelle, LLC, for the US DOE.

11:00am **SP+AS+NS+SS-WeM10 Probing Electrostatic Field Effect in Quantum Materials by Microwave Impedance Microscopy**, ***Keji Lai***, University of Texas at Austin **INVITED**

The research of complex quantum materials, in which a dazzling number of emergent phenomena take place in the nanoscale, is a major theme in modern condensed matter physics. For real-space imaging of complex systems, electrical impedance microscopy fills an important void that is not well represented by the existing local probes. Using shielded cantilever probes and sensitive microwave electronics, we can now perform non-invasive electrical imaging with sub-100nm resolution and sub-aF sensitivity.

Combining the cryogenic microwave impedance microscopy (MIM) and a spin-coated thin ionic gel layer, we are able to visualize the metal-insulator transition of functional materials in electrolyte-gated electric double-layer transistors. The microwave images acquired at different gate voltages clearly show the spatial evolution of channel conductivity and its local fluctuations through the transition. By applying a large source-drain bias above the glass transition temperature of the gel, an uneven conductance profile is established across the EDLT channel, which can be visualized by the MIM and further investigated by transport measurements and numerical simulations. The combination of ultra-thin ion-gel gating and microwave microscopy paves the way for studying the microscopic evolution of phase transitions in complex materials induced by electrostatic field effects.

11:40am **SP+AS+NS+SS-WeM12 Subsurface Visualization of Soft Matrix using 3D-Spectroscopic Atomic Force Acoustic Microscopy**, ***Kuniko Kimura***, *K. Kobayashi, A. Yao, H. Yamada*, Kyoto University, Japan

Nondestructive visualization of subsurface features of various materials with nanometer-scale spatial resolution is strongly demanded in a wide variety of scientific research fields such as nanoelectronics, nanomechanics and life science. Recently, many research groups have demonstrated the visualization of nanometer-scale subsurface features using various techniques based on atomic force microscopy (AFM) [1-4]. (All references and figures are given in Supplement.) We recently demonstrated the imaging of Au nanoparticles buried under 900 nm from the surface of a polymer matrix by atomic force acoustic microscopy (AFAM), as shown in Fig. 1 [5]. In AFAM, the amplitude and phase of the cantilever vibration at the contact resonance frequency induced by the sample excitation are measured, which allows us the quantitative evaluation of surface stiffness [6]. The AFAM images in Fig. 1 show that the surface viscoelasticity of the soft matrix is affected by subsurface hard objects such as the Au nanoparticles buried even roughly 1 micro-meter below the surface. However, only from AFAM images, it is difficult to determine which the dominant mechanism for the subsurface imaging is viscosity variation or elasticity variation, because AFAM images were taken at a single excitation frequency near contact resonance.

In this presentation, we discuss the origin of the visualization of subsurface features in soft matrix based on spectroscopy of AFAM [7]. We recorded the amplitude and phase spectra at every pixel of the AFAM image as represented in Fig. 2, which we call 3-dimensional spectroscopic atomic force acoustic microscopy (3D-spectroscopic AFAM). A schematic diagram of the 3D-spectroscopic AFAM is shown in Fig. 3. After the tip was brought into contact with the surface, we first measured the contact resonance frequency (fc). Then we recorded the amplitude and phase spectra measured by a lock-in amplifier, while the tip was raster-scanned with the contact mode. At each scanning pixel, the excitation frequency was swept with the span of 25 kHz which was centering around fc, whose sweep time was 35 msec. The total acquisition time for 128 x 128 pixels took about 20 min.

Using this method, we can compare the frequency spectrum measured on the subsurface Au nanoparticle with that on another position having no subsurface particle, as shown in Fig. 4. We can also reconstruct AFAM images of arbitrary frequencies within the sweep frequency range, which is the meaning of "3-dimensional". Moreover, the 3D-spectroscopic AFAM enables us to characterize the amplitude and phase spectra and to detect the variation that may be caused by the nonlinear tip-sample interactions.

12:00pm **SP+AS+NS+SS-WeM13 Quantifying the Effects of Cantilever Modes Shapes on Studies of the Liquid-Solid Interface**, ***Aleks Labuda***, *M. Viani, D. Walters, R. Proksch*, Asylum Research, an Oxford Instruments company

At the core of most AFM measurements is the assumption that the motion of the cantilever probe can be well quantified. However, most AFM systems use a "beam bounce" optical beam deflection (OBD) method which, because it is fundamentally an angular measurement, only provides accurate tip position information when the mode shape of the cantilever matches the calibration conditions. For example, if the OBD sensitivity is calibrated with a force curve, the calibration holds true only for experiments where the mode shape is similar to an end-loaded cantilever. This assumption is quickly violated when the cantilever is oscillated at frequencies different from the calibration. This is especially true in liquids, where Q~1 and the combination of significant base motion and hydrodynamic effects lead to a variety of different mode shapes that are strongly frequency dependent (see Figure). This clearly demonstrates that the sensitivity (nm/V) is actually a frequency dependent quantity. Worse, it may also drift with time. Another consequence is that the effective stiffness of the cantilever, which depends on mode shape, is also highly frequency dependent. Both of these effects cause quantitative misinterpretation of the tip-sample interaction and artifacts in imaging contrast. These problems affect both dynamic AFM modes (such as AM-AFM and FM-AFM) as well as sub-resonance modes such as fast force mapping and force modulation.

To quantify this effect, we present measurements based on Ref [1-2] using a modified commercial AFM that combines a standard OBD detector with an integrated laser Doppler vibrometer (LDV) system that directly measures displacement. As shown in the Figure, The OBD and LDV can be used simultaneously, such that the cantilever base motion or tip motion can be accurately monitored with the LDV during an AFM experiment – independent of the OBD and any feedback loops. In the Figure, the ~2 μm LDV laser spot was scanned along the cantilever for high-resolution in situ mapping of its dynamics across a wide spectrum of frequencies and showing significant deviations from ideal mode shapes over the entire frequency range.

The effects of these frequency-dependent mode shapes are then quantified by appropriate modeling for a variety of experimental conditions, and demonstrated experimentally using stiff levers for AM-AFM at the calcite-water interface and soft levers for fast force mapping of polymeric materials.

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