

Wednesday Afternoon, November 1, 2017

Nanometer-scale Science and Technology Division

Room: 19 - Session NS+MN+MS+SS-WeA

Nanopatterning, Nanofabrication and 3D Nanomanufacturing

Moderator: Brian Borovsky, St. Olaf College

2:20pm **NS+MN+MS+SS-WeA1 Site-controlled Si Nanodot Formation for a RT-SET via Ion Beam Mixing and Phase Separation**, *Xiaomo Xu**, *G. Hlawacek, D. Wolf, T. Prüfer, R. Hübner, L. Bischoff*, Helmholtz Zentrum Dresden-Rossendorf, Germany, *M. Perego*, Institute for Microelectronics and Microsystems (IMM-CNR), France, *A. Gharbi*, Laboratoire d'électronique des technologies de l'information (CEA-Leti), France, *H.-J. Engelmann, S. Facsko, K.-H. Heinig, J. von Borany*, Helmholtz Zentrum Dresden-Rossendorf, Germany

The increased use of personal computing devices and the Internet of Things (IoT) is accompanied by a demand for a computation unit with extra low energy dissipation. The Single Electron Transistor (SET), which uses a Coulomb island to manipulate the movement of single electrons, is a candidate device for future low-power electronics. However, so far its development is hindered by low-temperature requirements and the absence of CMOS compatibility. By combining advanced top-down lithography with bottom-up self-assembly of Si nano dots (NDs) we will overcome this barrier.

In this work, Si NDs – suitable as RT Coulomb islands – are formed via ion beam mixing followed by thermally stimulated phase separation. Broad-beam Si⁺ and Ne⁺ beams followed by a rapid thermal annealing (RTA) treatment were utilized to create a layer of NDs, which are subsequently visualized by Energy-Filtered Transmission Electron Microscopy (EFTEM). The conditions for ND formation, namely the dependence on ion type, primary energy, irradiation fluence, layer thickness and thermal budget during RTA, are optimized based on an extensive survey of this multidimensional parameter space. The presented work is guided by TRIDYN simulations of the Si excess in a SiO₂ layer due to ion beam mixing and 3D Kinetic Monte-Carlo (3DKMC) simulation for the phase separation during the thermal treatment. To tailor towards a single Si ND, the focused Ne⁺ beam from the Helium Ion Microscope (HIM) is utilized to create user defined patterns of NDs in planar layer stacks. This allows to achieve a mixing volume small enough for restricted Ostwald ripening and successful single ND formation. The existence of the formation of spatially controlled single NDs with a diameter of only 2.2 nm is confirmed by comparing the EFTEM Si plasmon-loss intensity with simulated plasmon loss images.

In the future – by combining conventional lithography, direct self-assembly (DSA) and ion beam mixing – nanopillars with a single embedded ND will be integrated in a CMOS-compatible way. EFTEM and electrical characterization techniques will be used for realizing this novel pathway towards a room-temperature SET device.

2:40pm **NS+MN+MS+SS-WeA2 Scanning Tunneling Microscope Fabrication of Atomically Precise Devices**, *Richard Silver*, NIST, *X. Wang*, University of Maryland, College Park, *P. Nambodiri, J. Wyrick, S.W. Schmucker, M.D. Stewart, R. Murray, J.A. Hagmann, C. Richter*, NIST

Atomically precise device fabrication is a technique that enables a new class of atom-based electronic structures with applications ranging from novel low dimensional materials to devices for quantum information processing. Deterministic placement of individual dopant atoms in the Si lattice is achieved using hydrogen-based scanning probe lithography. Controlling the position and electronic or quantum state of deterministically placed atoms in a solid state environment enables novel devices such as single atom transistors and solid state qubits.

However, fabricating functional atom-based devices is particularly challenging because of the need for exceptional ultra-high vacuum, near perfect atomic order, and low temperature epitaxial silicon overgrowth. This, coupled with sensitivity of atomic positional accuracy to thermal processing, and variability in scanning tunneling microscope patterning conditions, make exquisite control of process conditions essential.

In this presentation, we will focus on measurements and characterization of ultra-thin, atomically abrupt, highly doped low-dimensional devices and strategies for contacting these devices. We will describe our methods to align and contact buried devices and address significant challenges in making robust electrical contact to buried devices. We will present low-temperature electrical measurement results from atomically abrupt wires and tunnel

junctions with coplanar gates. We have studied the effects of process conditions on device dimensionality and electrical performance in the context of extensive analysis of delta layer formation with optimized locking layer epitaxial growth techniques to enhance the confinement of Phosphorus dopant atoms. Low temperature transport measurements are used to investigate materials properties, effects from atomic imperfection and quantum transport phenomena.

3:00pm **NS+MN+MS+SS-WeA3 Contacting Buried Atomic-Precision Devices in Si using Kelvin Probe and Optical Microscopy**, *Jonathan Wyrick, P. Nambodiri, X. Wang, R. Murray, J.A. Hagmann, K. Li, S.W. Schmucker, M.D. Stewart, C. Richter, R.M. Silver*, NIST

STM based hydrogen lithography has proven to be a viable route to fabrication of atomic-precision planar electronic devices. These devices are realized by a patterning step followed by dopant deposition and incorporation, and ultimately encapsulation with epitaxial Si. Atomically precise tunnel junctions, SETs, and Quantum Dots are examples of components that can be fabricated using hydrogen lithography.

The strength of this technique is the ability to control the lateral placement of phosphorus atoms in a single atomic layer of Si with sub-nanometer precision. At the same time, it presents challenges that must be overcome if devices are to be interfaced to the outside world. Locating and then fabricating aligned electrical contacts to buried devices is non-trivial, and becomes easier as the size of buried features is increased, but this is done at the expense of increased writing times and exposure to potential contamination.

We present a strategy for contacting buried devices aimed at minimizing the write-times associated with STM based fabrication by maximizing the positional accuracy with which we can locate subsurface structures. This is done by employing STM fabricated fiducials, AFM topography scans, Kelvin Probe Microscopy, and dark field optical microscopy. The data from each technique can be aligned and corrected for distortions, allowing us to determine buried device locations to better than 200nm accuracy.

3:20pm **NS+MN+MS+SS-WeA4 Quantifying Liquid Transport and Patterning using Atomic Force Microscopy**, *N. Farmakidis, Keith Brown*, Boston University

Atomic force microscopy (AFM) provides unique insight into the nanoscale properties of materials. It has been challenging, however, to use AFM to study soft materials such as liquids or gels because of their tendency to flow in response to stress. Here, we propose an AFM-based technique for quantitatively analyzing the transport of soft materials from an AFM probe to a surface. Specifically, we present a method for loading an AFM probe with a single 0.3 to 30 pL droplet of liquid, and subsequently measuring the mass of this liquid by observing the change in the vibrational resonance frequency of the cantilever. Using this approach, the mass of this liquid was detected with pg-scale precision using a commercial AFM system. Additionally, sub-fL droplets of liquid were transferred from the probe to a surface with agreement found between the real-time change in mass of the liquid-loaded probe and the volume of the feature written on the surface. To demonstrate the utility of this approach in studying nanoscale capillary and transport phenomena, we experimentally determine that the quantity of liquid transported from the tip to a surface in a given patterning operation scales as the mass of liquid on the probe to the 1.35 power. In addition to providing new avenues for studying the dynamics of soft materials on the nanoscale, this method can improve nanopatterning of soft materials by providing *in situ* feedback.

4:20pm **NS+MN+MS+SS-WeA7 Positioning and Manipulating Single Dopant Atoms Inside Silicon**, *Andrew Lupini, B.M. Hudak, J. Song*, Oak Ridge National Laboratory, *H.R. Sims*, Vanderbilt University, *M.C. Tropichevsky, Oak Ridge National Laboratory, S.T. Pantelides*, Vanderbilt University, *P.C. Snijders*, Oak Ridge National Laboratory **INVITED**

The ability to controllably position single atoms inside materials could enable production of a new generation of atomically precise artificial materials with direct relevance for many areas of technology. For example, spins from individual donors in a semiconductor comprise one of the most promising architectures for quantum computing. However, fabrication of the ‘qubits’ that would make up a quantum computer is still unreliable and many fundamental materials science questions remain unanswered. Perhaps the key technical difficulty is the task of accurately positioning single atom dopants inside a solid with control, or at least understanding, of their local environment.

* NSTD Student Award Finalist

Silicon is the ideal substrate to explore such ideas because of the ability to obtain isotopically purified samples (a “spin-vacuum”) and compatibility with existing electronic components and manufacturing technologies. Group V elements are promising candidates for use as single-atom qubit dopants in Si, and it has recently been argued that bismuth could be an excellent dopant for such applications, because of its anomalously high spin-orbit coupling. Bi, in particular, has a large atomic number relative to Si, making it an ideal candidate to study using Z-contrast scanning transmission electron microscope (STEM). However, both precise doping and the imaging of single dopant atoms present many scientific challenges. For example, Bi is not very soluble in Si, meaning that the dopant atoms tend to migrate out of position during sample growth.

Here we will show examples of sample growth including novel nanostructures and single atom dopants. We will show dopant atoms imaged inside Si samples, and demonstrate electron-beam directed movement of single dopants.

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5:00pm **NS+MN+MS+SS-WeA9 Characterization of Butyl Tin Photoresists for Nanoscale Patterning**, *J.T. Diulus, R.T. Frederick*, Oregon State University, *M. Li*, Rutgers University, *D. Hutchison, M.R. Olsen, I. Lyubinetsky, L. Arnadóttir*, Oregon State University, *E.L. Garfunkel*, Rutgers University, *M. Nyman*, Oregon State University, *H. Ogasawara*, SLAC National Accelerator Laboratory, **Gregory Herman**, Oregon State University
Inorganic photoresists are of interest for nanomanufacturing due the potential for high resolution patterning with low line edge roughness, while having high sensitivity to extreme ultraviolet (EUV) radiation. The combination of high absorption coefficient elements and radiation sensitive ligands can improve inorganic photoresist sensitivity while providing high contrast. Inorganic clusters are ideal candidates for photoresists since they have nanometer particle sizes with high particle size uniformity, and the ligand chemistries can be tuned for radiation induced chemistries that control relative solubility differences. In this presentation, characterization of a promising inorganic cluster-based EUV photoresist will be presented, where the goal of the studies is to better understand patterning mechanisms. In these studies, we are investigating butyl tin Keggin cluster that has recently been synthesized, and has shown promising properties as an inorganic photoresist. Key to these clusters, for application as an EUV photoresist, are the high EUV absorption coefficient for Sn, and the radiation sensitive carbon-tin bond. Removal of the organic ligand changes the polarity of the film, which provides the necessary solubility contrast for nanopatterning. We have used temperature programmed desorption, electron stimulated desorption, and ambient pressure X-ray photoelectron spectroscopy to characterize both thermal and radiation induced processes in thin films formed from these clusters. We have found that butyl group desorption occurs through both thermal and radiation induced processes, and have determined both the carbon-tin bond strength and electron desorption cross-sections. Studies performed in different ambient conditions, and photon energies, have shown large effects on the radiation induced chemistries, where a significant enhancement in carbon decay was observed for O₂ pressures up to 1 torr. These studies provide a means to better understand the radiation induced processes that result in the solubility contrast of these materials, and may guide in the development of improved EUV photoresists for nanolithography.

5:20pm **NS+MN+MS+SS-WeA10 Impact of Polymer Templated Annealing on Gold Nanowires**, *Tyler Westover, R.F. Davis, B. Uptrey, J. Harb, A. Woolley, S. Noyce*, Brigham Young University

The formation of gold nanowires using bottom up nanofabrication has resulted in wires of small dimension or high conductivity, but not both. We form nanowires on DNA origami through directed assembly of nanoparticles or nanorods followed by electrochemical plating. These metal deposition processes result in non-ideal microstructure and correspondingly low conductivities. To remedy this we have sought to reduce the grain boundary density and surface roughness through annealing. However annealing causes

the wires to coalesce into beads. We have found that through the use of a polymer the wires can be templated to retain their overall morphology, while improving surface roughness, throughout a low temperature anneal. We have measured these wires to have less than 1kOhm resistances by electron beam lithography, in a two point configuration. Using electron beam induced deposition we have successfully made four point contacts to measure the change in resistance due to annealing. We will present results on polymer templating, showing that the wires maintain their overall morphology with improved conductivities during low temp (200° C) annealing.

5:40pm **NS+MN+MS+SS-WeA11 Dynamic Growth of Nanopores on Graphene via Helium Ion Microscope**, *S. Kim, Anton Ievlev, M.J. Burch, I. Vlasiouk, A. Belianinov, S.V. Kalinin, S. Jesse, O.S. Ovchinnikova*, Oak Ridge National Laboratory

Controlling atomic-to-nanoscale defect formation on graphene is of significance as defects can modify properties as well as functionality of graphene. Especially, controlled formation of nanopores in graphene can be used for energy harvesting/storage, analysis of biomolecules and the separation of gases or liquids. Nanopores can be fabricated either by using high energy focused electron beam or by focused helium ion beam with high precision. However, focused electron beam has very low throughput to form nanopores despite its superiority in pore size control. On the contrary, focused helium ion beam has much higher throughput in nanofabrication with its capability to form sub-5nm pores. In this study, we utilized the focused helium ion microscope to fabricate nanopores on graphene and demonstrated atomic scale control in growth of nanopores by helium ion irradiation. We demonstrated the size control of nanopores down to ~ 3nm in a diameter. Formation and growth kinetics of nanopores by different helium ion irradiation conditions were explored and analyzed using the image data analytics. Also, Raman spectroscopic measurements was performed to demonstrate the effect of a helium ion dose on the change of initial defect density on graphene which leads to different behaviors and growth kinetics of nanopore formation.

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Authors Index

Bold page numbers indicate the presenter

— A —

Árnadóttir, L.: NS+MN+MS+SS-WeA9, 2

— B —

Belianinov, A.: NS+MN+MS+SS-WeA11, 2

Bischoff, L.: NS+MN+MS+SS-WeA1, 1

Brown, K.A.: NS+MN+MS+SS-WeA4, **1**

Burch, M.J.: NS+MN+MS+SS-WeA11, 2

— D —

Davis, R.F.: NS+MN+MS+SS-WeA10, 2

Diulus, J.T.: NS+MN+MS+SS-WeA9, 2

— E —

Engelmann, H.-J.: NS+MN+MS+SS-WeA1, 1

— F —

Facsko, S.: NS+MN+MS+SS-WeA1, 1

Farmakidis, N.: NS+MN+MS+SS-WeA4, 1

Frederick, R.T.: NS+MN+MS+SS-WeA9, 2

— G —

Garfunkel, E.L.: NS+MN+MS+SS-WeA9, 2

Gharbi, A.: NS+MN+MS+SS-WeA1, 1

— H —

Hagmann, J.A.: NS+MN+MS+SS-WeA2, 1;
NS+MN+MS+SS-WeA3, 1

Harb, J.: NS+MN+MS+SS-WeA10, 2

Heinig, K.-H.: NS+MN+MS+SS-WeA1, 1

Herman, G.S.: NS+MN+MS+SS-WeA9, **2**

Hlawacek, G.: NS+MN+MS+SS-WeA1, 1

Hübner, R.: NS+MN+MS+SS-WeA1, 1

Hudak, B.M.: NS+MN+MS+SS-WeA7, 1

Hutchison, D.: NS+MN+MS+SS-WeA9, 2

— I —

Ievlev, A.V.: NS+MN+MS+SS-WeA11, **2**

— J —

Jesse, S.: NS+MN+MS+SS-WeA11, 2

— K —

Kalinin, S.V.: NS+MN+MS+SS-WeA11, 2

Kim, S.: NS+MN+MS+SS-WeA11, 2

— L —

Li, K.: NS+MN+MS+SS-WeA3, 1

Li, M.: NS+MN+MS+SS-WeA9, 2

Lupini, A.R.: NS+MN+MS+SS-WeA7, **1**

Lyubinetsky, I.: NS+MN+MS+SS-WeA9, 2

— M —

Murray, R.: NS+MN+MS+SS-WeA2, 1;
NS+MN+MS+SS-WeA3, 1

— N —

Nambodiri, P.: NS+MN+MS+SS-WeA2, 1;
NS+MN+MS+SS-WeA3, 1

Noyce, S.: NS+MN+MS+SS-WeA10, 2

Nyman, M.: NS+MN+MS+SS-WeA9, 2

— O —

Ogasawara, H.: NS+MN+MS+SS-WeA9, 2

Olsen, M.R.: NS+MN+MS+SS-WeA9, 2

Ovchinnikova, O.S.: NS+MN+MS+SS-WeA11, 2

— P —

Pantelides, S.T.: NS+MN+MS+SS-WeA7, 1

Perego, M.: NS+MN+MS+SS-WeA1, 1

Prüfer, T.: NS+MN+MS+SS-WeA1, 1

— R —

Richter, C.: NS+MN+MS+SS-WeA2, 1;
NS+MN+MS+SS-WeA3, 1

— S —

Schmucker, S.W.: NS+MN+MS+SS-WeA2, 1;
NS+MN+MS+SS-WeA3, 1

Silver, R.M.: NS+MN+MS+SS-WeA2, **1**;

NS+MN+MS+SS-WeA3, 1

Sims, H.R.: NS+MN+MS+SS-WeA7, 1

Snijders, P.C.: NS+MN+MS+SS-WeA7, 1

Song, J.: NS+MN+MS+SS-WeA7, 1

Stewart, M.D.: NS+MN+MS+SS-WeA2, 1;
NS+MN+MS+SS-WeA3, 1

— T —

Troparevsky, M.C.: NS+MN+MS+SS-WeA7, 1

— U —

Uptrey, B.: NS+MN+MS+SS-WeA10, 2

— V —

Vlassioux, I.: NS+MN+MS+SS-WeA11, 2

von Borany, J.: NS+MN+MS+SS-WeA1, 1

— W —

Wang, X.: NS+MN+MS+SS-WeA2, 1;
NS+MN+MS+SS-WeA3, 1

Westover, T.: NS+MN+MS+SS-WeA10, **2**

Wolf, D.: NS+MN+MS+SS-WeA1, 1

Woolley, A.: NS+MN+MS+SS-WeA10, 2

Wyrick, J.: NS+MN+MS+SS-WeA2, 1;
NS+MN+MS+SS-WeA3, **1**

— X —

Xu, X.M.: NS+MN+MS+SS-WeA1, **1**