

Graphene Topical Conference

Room: 306 - Session GR+SS+NC-MoA

Materials Issues in Graphene from SiC

Moderator: N.P. Guisinger, Argonne National Laboratory

2:00pm **GR+SS+NC-MoA1 Ultrahigh Vacuum Growth, Electrical Characterization, and Patterning of Graphene Nanostructures on Si- and C-Polar 6H-SiC Surfaces.** *A. Sandin, Z. Wang, J.L. Tedesco, J.E. Rowe, North Carolina State University, R.J. Nemanich, Arizona State University*

We report the growth of graphene films on both C-polar and Si-polar surfaces of 6H-SiC by thermal decomposition in an ultrahigh vacuum (UHV) chamber. Auger electron spectroscopy (AES), low energy electron diffraction (LEED) and scanning tunneling microscopy (STM) have been used in situ to characterize the UHV films. Following growth, focused ion beam lithography has been used to successfully etch the graphene films and control the lateral dimensions of a number of nanostructures on these graphene layers with etch rates of ~ 18 nm/s and lateral dimensions of ~ 250 to 1500 nm. Epitaxial graphene films (1-4 layers thick) have been grown on the Si face. Theoretical reports have recently addressed the bandgap engineering of graphene nanoribbons by altering the physical dimensions, edge structure, and edge atoms of the nanoribbons. However, experimental control of the growth and quality graphene nanostructures is still a challenge. Several experimental studies have shown that annealed SiC does not significantly influence the electronic quality of the epitaxial graphene overlayer. However, these studies also indicate that the thickness of graphene films can be varied by controlling the Si decomposition and carbon aggregation on the surface through control of the annealing. Graphene growth on the Si-face suffers from pit formation located at SiC atomic-height steps and the carbon nanomesh ($\sim 13 \times 13$) can be observed using LEED and STM even for our thickest Si face graphene layers. Our results for graphene film growth on the more chemically reactive C-face find more rapid growth which is difficult to control. Due to the rapid growth, graphene nucleation cannot be mapped relative to the original SiC atomic terraces on the C-face. Scanning tunneling spectroscopy (STS) current-voltage (I-V) curves have been recorded and we observe both metallic behavior and bandgap formation, as well as the local density of states in bulk graphene and etched graphene edges. Future research will be directed to experimentally observe the proposed electronic properties of smaller (< 20 nm in width) graphene nanostructures using STS.

2:20pm **GR+SS+NC-MoA2 Unique Stacking of Multi-Layer Graphene on 4HSiC(000-1).** *J. Hass, J.E. Millán-Otaya, N. Sharma, M. Sprinkle, F. Ming, W.A. de Heer, P.N. First, E.H. Conrad, Georgia Institute of Technology*

Diffraction data have shown that multilayer graphene grown on the polar (000-1) face of 4H-SiC contains large, flat domains and rotational stacking faults approximately every 2 layers. Such faults are particularly interesting because it has been demonstrated that rotational faults in bi- and tri-layer graphene films decouple adjacent sheets, thereby preserving the unique lattice symmetry and linear dispersion found for a single, isolated sheet.¹⁻³ This is in contrast with few-layer graphite, which grows in a Bernal stacking arrangement on the opposite polar (0001) face. Scanning tunneling microscopy (STM) and surface X-ray diffraction (SXRD) data exhibit a number of rotational domains in registry with the SiC substrate. Data will be presented which elucidate particular stacking orientations over micron scale domains. Scanning tunneling spectroscopy (STS) results will be interpreted in the context of symmetry breaking between sheets.

¹J. Hass, F. Varchon, J. E. Millán-Otaya, M. Sprinkle, N. Sharma, W.A. de Heer, C. Berger, P.N. First, L. Magaud, E.H. Conrad, Phys. Rev. Lett. 100, 125504 (2008).

²J.M.B. Lopes dos Santos, N.M.R. Peres, A.H. Castro Neto, Phys. Rev. Lett. 99, 256802 (2007).

³S. Latil, V. Meunier, L. Henrard, Phys. Rev. B. 76, 210402(R) (2007).

2:40pm **GR+SS+NC-MoA3 Ultrahigh Vacuum and RF Furnace Production of Graphene on SiC.** *G.G. Jernigan, B.L. VanMil, D.K. Gaskill, J.C. Culbertson, P.M. Campbell, US Naval Research Laboratory*

INVITED

The electrical, mechanical, physical, and chemical properties of graphene have the scientific community in search of large area samples for technological applications. Since deHeer's¹ initial report of graphene formation by the thermal desorption of Si from SiC, efforts have been underway to use this method to make large area sheets of graphene. Creating graphene by desorbing Si from SiC is conceptually simple, but in practice it is very challenging to produce large area, uniform, electronic grade graphene. In this presentation, I will discuss the two processes we

employ, ultrahigh vacuum (UHV) annealing ($\sim 10^{-8}$ mbar) and RF furnace heating ($\sim 10^{-5}$ mbar), to create graphene in areas ranging from 200 mm^2 to 4000 mm^2 on 4H and 6H SiC and on Si-face and C-face samples. As observed by Raman spectroscopy, both processes are capable of producing graphene, because each set of samples shows the distinctive D, G, and 2D Raman lines. Each process begins with the SiC surface being hydrogen etched to remove polishing damage and to create a uniformly stepped surface. However, the resulting graphene from each process has noticeably different characteristics. UHV production allows us to probe the graphene formation in situ with LEED, XPS, and STM. The UHV method results in single layer and few layer graphene films. Van der Pauw Hall measurements indicate the films have low mobility and the predominant carriers are electrons. AFM studies show that depending on desorption conditions (heating rate, final temperature, and cooling) the surface morphology is roughened due to formation of pits and islands. We believe this roughened surface explains the low mobility. RF furnace production allows us to form graphene in the same system that hydrogen etching is performed, thereby avoiding exposure of the sample to air. Van der Pauw Hall measurements of the RF furnace samples consistently have higher mobility than the UHV samples and the predominant carriers are holes. These films are thicker than the UHV samples and consist of multiple layers of graphene. The surface morphology does not consist of pits and islands, but instead shows lines of built up carbon along step edges. In the end, we want to produce graphene having the best characteristics of the UHV and RF furnace methods with controlled thickness (< 3 layers) and high carrier mobility ($> 10,000 \text{ cm}^2/\text{Vs}$).

¹C. Berger, et al, J. Phys. Chem. B 108, 19912-19916 (2004).

3:20pm **GR+SS+NC-MoA5 Microscopic and Spectroscopic Studies of the Electronic Structure of Epitaxial Graphene on SiC (0001).** *N. Sharma, D. Oh, M. Sprinkle, Georgia Institute of Technology, C. Berger, CNRS Grenoble, France, W.A. deHeer, T.M. Orlando, P.N. First, Georgia Institute of Technology*

Growth of high quality epitaxial graphene (EG) films on the basal plane of hexagonal SiC has been demonstrated previously, providing a potential route to wafer-scale graphene electronics. To realize this promise requires a detailed understanding of the atomic and electronic structure of the EG/SiC interface. For typical EG samples, STM measurements indicate a reconstructed interface (layer 0) covered by a layer (layer 1) that images as graphene (a honeycomb pattern) at low bias voltages, but appears to partially hybridize with interface states. For this layer, we also observe a strong suppression of the 2D Raman peak (also known as D^* or G') that is characteristic of graphene. For the second EG layer, a single Lorentzian-shape 2D peak is observed, which may indicate some isolation of layer 2 from the material below. Graphene films grown on the SiC (0001) surface are intrinsically electron doped. The screening response of the 2D electron gas in EG is expected to be unique due to the small carrier density and novel electronic structure. We use local defects and deposited metal islands to locally change the carrier density, and scanning tunneling spectroscopy to probe the screening response. This is of interest since the screening region in EG could include a transition from hole to electron doping, resulting in a surface PN junction that could influence electrical transport in this system.

4:00pm **GR+SS+NC-MoA7 Structural and Electronic Properties of Epitaxial Graphene on SiC(0001).** *C. Riedl, D.S. Lee, J. Smet, L. Vitali, R. Ohmann, I. Brihuega, MPI for Solid State Research, Germany, A. Zakharov, Lund University, Sweden, C. Virojanadara, now at: Linköping University, Sweden, K. von Klitzing, K. Kern, U. Starke, MPI for Solid State Research, Germany*

Graphene layers can be grown on a solid substrate by the controlled graphitization of SiC surfaces by high temperature annealing in ultra high vacuum (UHV). However, the exact control of the number of layers grown and their quality remains a problem. In the present work, we use angular resolved ultraviolet photoemission spectroscopy (ARUPS) from He II excitation and low energy electron diffraction (LEED) to count the number of layers continuously during the preparation procedure in the home laboratory thus avoiding laborious synchrotron experiments. The layer structure and homogeneity is further analyzed by Raman spectroscopy, core level photoemission spectroscopy (PES) and low-energy electron microscopy (LEEM) measurements. On such precisely prepared graphene samples, we investigate their electronic structure using ARUPS and scanning tunneling spectroscopy (STS). The layer dependent shifting of the energetic position of the Dirac point and the detailed structure of the π -band dispersion are analyzed both with momentum and spatial resolution.

4:20pm **GR+SS+NC-MoA8 Atomic Scale Properties of Epitaxial Graphene Grown on SiC**, *G.M. Rutter, P.N. First*, Georgia Institute of Technology, *J.A. Stroscio*, National Institute of Standards and Technology

Two-dimensional electron systems have been of interest to scientists for many years. From high-electron mobility transistors to novel topological quasiparticles of the fractional quantum Hall effect, the field continues to be rich in scientific possibilities and technological pay-offs. To date, most high-mobility 2D electron systems have been created at an interface between semiconductor heterostructures, making them inaccessible to the electron spectroscopies of surface science. Here we investigate graphene, a new 2D electron system that is accessible to surface studies. Our measurements use scanning tunneling microscopy and spectroscopy to elucidate the properties of epitaxial graphene, resolving heterogeneities at the level of single atoms. In this work, epitaxial graphene is created on silicon carbide wafers by thermal annealing in vacuum. Sequential scanning tunneling microscopy (STM) and spectroscopy (STS) are performed in ultrahigh vacuum at temperatures of 4.2 K and 300 K. These atomic-scale studies address the initial growth of single-layer epitaxial graphene and the role that the interface and defects play in the electronic properties of graphene. Our work shows evidence of graphene formation by mass transfer of carbon, indicated by step edge growth and the nucleation of graphene islands. STM topographic images of single-layer graphene show the atomic structure of the graphene and the graphene/SiC interface, as well as the character of defects and adatoms within and below the graphene plane.^{1,2} STS of lattice defects on single-layer graphene show localized peaks in the spectra. The energy position of such localized states offers a clue to the defect's origin and composition and will be discussed.

¹G. M. Rutter, et al., *Phys. Rev. B* 76, 235416 (2007).

²G. M. Rutter, et al., *J. Vac. Sci. Technol. A* in press, (2008).

4:40pm **GR+SS+NC-MoA9 Layer-Dependent Properties of Epitaxial Graphene on Silicon Carbide***, *P.N. First*, Georgia Institute of Technology **INVITED**

Epitaxial graphene grown on single-crystal silicon carbide has been proposed as a platform for graphene-based nanoelectronics.¹ This new electronic material shows great potential, but also poses a number of challenges. I will discuss results from several surface characterization techniques that determine the structure and electronic properties of this system. In particular, scanning tunneling microscopy and spectroscopy are used to study the electronic and geometric structure versus the graphene layer index for epitaxial graphene on SiC(0001).² Additional measurements show that the structure of graphene grown on SiC(000 -1) differs dramatically from that grown on SiC(0001). Finally, results that address the physics of metal contacts to graphene will be presented.

*Work supported in part by NSF, NRI-INDEX, and the W. M. Keck Foundation. +Work done in collaboration with G. M. Rutter, J. Hass, N. Sharma, T. Li, E. H. Conrad, C. Berger, and W. A. de Heer at the Georgia Institute of Technology, and J. N. Crain, N. P. Guisinger, and J. A. Stroscio at the NIST Center for Nanoscale Science and Technology

¹ C. Berger et al., *J. Phys. Chem. B* 108, 19912 (2004).

² G. M. Rutter et al., *Science* 317, 219 (2007); *Phys. Rev. B* 76, 235416 (2007); *J. Vac. Sci. Technol. A* (in press).

5:20pm **GR+SS+NC-MoA11 Scanning Tunneling Spectroscopy of Epitaxial Graphene on SiC(0001)**, *S. Nie, R. Feenstra*, Carnegie Mellon University

Over the past few years many researchers have investigated the properties of single monolayers of graphite, known as graphene. This material exhibits novel electronic properties arising from its band structure which displays linear dispersion around the band extrema, leading to high carrier mobilities and the potential for high-speed electronic devices. Using scanning tunneling spectroscopy (STS) at room temperature we have studied the electronic properties of graphene formed on the Si-face of the SiC(0001) surface. The substrate was annealed in ultra high vacuum at temperatures ranging from 1200 to 1400°C to form the graphene. With increasing temperature the surface becomes more carbon rich, showing different surface structures including 5×5, 6×6, and graphene-covered 6×6. For the highest annealing temperatures, multiple (>5) layers of graphene were formed. Low-energy electron diffraction was used to monitor the change in surface structure as a function of temperature, and scanning tunneling microscopy (STM) was used to verify the structures. Tunneling spectra were acquired on each of the 5×5, 6×6, graphene-covered 6×6, and totally graphitized surfaces. A distinct spectrum of electronic states was observed on the 5×5 and 6×6 surfaces, containing intense spectrum peaks at about -1.5, -0.5, and +0.5 relative to the Fermi-level. A conductance minimum is seen at the Fermi-level for all spectra. The observed spectra are quite similar for the 5×5 and 6×6 surfaces, indicating that the spectral peaks arise from similar surface-derived dangling bonds and/or reconstructed bonding arrangements in both cases. As graphene forms, covering the 6×6 structure, these spectral peaks diminish in intensity and their energies shift slightly. In particular, near the Fermi-level, a region of minimum intensity forms extending about 0.6 to 0.8 eV below the Fermi-level. We tentatively

associate this region with the 0.8 eV band gap that is known to form for bilayer graphene, with the Fermi-level located at the top of this gap. In any case, even with this evolution in the spectra, the overall position of spectral peaks is quite close for the graphene-covered surface as compared to the 5×5 and 6×6 surfaces. We therefore interpret the features observed in the graphene spectra as primarily arising from the electronic structure at the interface between the graphene and the SiC. This work was supported the National Science Foundation, grant DMR-0503748.

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