

## Graphene Topical Conference

Room: 306 - Session GR+TF+NC-MoM

### Graphene and 2-D Carbon Nanostructures

Moderator: B.C. Holloway, Luna Innovations Incorporated, M.Y. Zhu, College of William and Mary

8:20am **GR+TF+NC-MoM1 Electrical Transport Properties of Two-Dimensional Carbon Nanostructures**, *Y. Wu*, National University of Singapore, *H. Wang*, National University of Singapore and Data Storage Institute, Singapore, *S.S. Kushvaha*, National University of Singapore, *S.Y.H. Lua*, National University of Singapore and Data Storage Institute, Singapore

**INVITED**

Recently 2D carbon nanostructures have attracted great interest due to their peculiar structural and electronic properties, especially after the successful mechanical exfoliation of single and few layer graphene from bulk graphite and epitaxial growth of graphene on SiC. We have reported previously the growth of well-aligned 2D carbon nanostructures – carbon nanowalls(CNWs), using MPECVD. Although the CNWs are structurally not as perfect as graphene sheets obtained by other techniques, its free-standing structure makes it an excellent candidate for studying electrical transport properties of 2D carbon nanostructures using end-point contacts of various types of materials including normal metal, semiconductor and superconductors. In this talk, we will present our results on (1) lateral electrical transport measurements using end-point Ti and Nb electrodes with a gap length in the range of 200 nm – 3000 nm (2) vertical transport measurements using nanopores in a UHV system. In (1) we focus on conductance fluctuation and proximity effect and in (2) we investigate the contact resistance between 2D carbon and different types of materials. In lateral transport measurements using Ti electrodes, excess conductance fluctuations with peculiar temperature-dependence from 1.4 to 250 K were observed with an electrode gap length of 300 and 450 nm, whereas the conductance fluctuation is greatly suppressed above 4.2 K when the electrode gap length increases to 800 and 1000 nm. The former is discussed in the context of the presence of both a small energy bandgap, while the latter is attributed to the crossover from mesoscopic to diffusive transport regime. On the other hand, in the case of Nb electrodes, we have observed proximity effect in samples with an electrode gap length of 200 – 500 nm and a reentrant behavior for samples with smaller gap length. Strong conductance oscillation was observed in samples using four-point probes which is attributed to the formation of SNS structures in CNW. In the vertical transport measurement using nanopores, it was found that the end-point contact resistance of carbon nanowalls on stainless steel is the lowest, followed by graphite, Si, Cu, Au (100 nm) / Ta (1 nm) / Si and Au wire. Both the contact resistance and I-V characteristics are in good agreement with the results of first principles calculations reported in the literature. The results may serve as the guides for selecting metal contacts in graphene-based devices.

9:00am **GR+TF+NC-MoM3 Characterization of Functionalized Carbon Nanosheets as a Step Towards the Development of Advanced Graphene Devices**, *R.A. Quinlan*, College of William and Mary, *E. Widenkvist*, Uppsala U., Sweden, *J. Groff*, College of William and Mary, *M. Rooth*, Uppsala U., Sweden, *M. Kelly*, Stanford U., *R.A. Outlaw*, College of William and Mary, *V. Coleman*, *O. Karis*, *B. Sanyal*, *O. Eriksson*, *H. Grenberg*, Uppsala U., Sweden, *B.C. Holloway*, Luna Innovations, Inc., *U. Jansson*, Uppsala U., Sweden

Carbon nanosheets (CNSs) are novel free-standing carbon nanostructures, composed of horizontal basal planes and vertical sheets which are made up of 1-7 graphene layers.<sup>1</sup> Previous work described the successful creation of defects in the graphene lattice via an acid-treatment and the characterization of these defects with x-ray absorption near-edge spectroscopy (XANES). The presence of a peak in the carbon K-edge spectra at 287eV was assigned to a C-O  $\sigma^*$  resonance. Theoretical modeling of a di-vacancy in a graphene bilayer system reproduced the spectra and predicts metallic conductivity of the states surrounding the defect.<sup>2</sup> Here we report the further characterization of functionalized carbon nanosheets (f-CNSs) via high resolution x-ray photoelectron spectroscopy (XPS) and thermal desorption spectroscopy (TDS). Measurements were made at room temperature up to 700K with as-deposited, water- and acid-treated CNSs. XPS measurements indicate an increase in the oxygen content of the nanostructures from 1-2% in as-deposited up to 3-4% in the acid-treated samples. The decrease of sub-peak areas in the C1s and O1s spectra with heating correlates well with the decrease in atomic oxygen content. The partial pressures of H<sub>2</sub>, O<sub>2</sub>, CO<sub>2</sub>, CO and H<sub>2</sub>O measured desorbing from the f-CNSs support the model of

oxygen and hydrogen decorated defects in the graphene system. The XPS and TDS results support XANES measurements of similar samples. These results support the use of a non-oxidizing acid-treatment for controlled defect formation in a graphene system, a first step towards the development of graphene based devices.

<sup>1</sup> Wang et al., Free-standing subnanometer graphite sheets. *Applied Physics Letters*, 2004, 85(7): p. 1265-1267

<sup>2</sup> Coleman et al., Defect formation in graphene nanosheets by acid treatment: an x-ray absorption spectroscopy and density functional theory study. *Journal of Physics D: Applied Physics*, 2008(6): p. 062001.

9:20am **GR+TF+NC-MoM4 Application of Carbon Nanowalls to Negative Electrode in Lithium-Ion Battery for High-Rate Use**, *N. Kitada*, *H. Yoshimura*, Yokohama City University, Japan, *O. Tanaike*, Advanced Industrial Science and Technology, Japan, *K. Kojima*, *M. Tachibana*, Yokohama City University, Japan

Recent design and fabrication of electrode in Lithium-ion battery have been focused for the high rate use since rapid charge and discharge of the battery with high power density are usually required for quick start and stop of electric vehicle or hybrid electric vehicle in next generation. One of the ways to develop the electrode for high rate use is to use nano-sized active materials since the diffusion distance of lithium ions in the solid during charge and discharge is decreased by using them. Here, we reports a new nano-sized and graphitized carbon material, two-dimensional carbon nanostructures called carbon nanowalls (CNWs) [1-5], as a promising one for negative electrode material of lithium ion battery in high rate use. CNWs were synthesized at very low temperature of 973K by a dc plasma-enhanced chemical vapor deposition. They were mixed with binder Polyvinylidene fluoride in N-methylpyrrolidone, pressed and then dried to produce carbon sample electrodes. Conventional three electrodes test cells were constructed for electrochemical measurements by carbon sample as a working electrode and two lithium foils as counter and reference ones. Lithium insertion properties were studied using these test cells by cyclic voltammetry (CV) and charge/discharge measurements. CNWs is found to be an interesting graphitic active material for negative electrode of lithium ion battery having reversible capacity of more than 200 mAh/g with relatively stable charge/discharge potential which is very similar to the properties of graphite, even though CNWs are synthesized at very low temperature of 973K without any graphitization process at high temperature. These CNWs are well layered particles in nano scale having large exposure surface of graphene edge, which is expected to be suitable for lithium insertion at high rate charge/discharge, and present results of CVs and charge/discharge tests support it. [1] Y. Wu et al., *Adv. Matter.* 14, 64(2002). [2] M. Zhu et al., *Carbon*, 42, 2867(2004). [1] S. Kurita et al., *J. Appl. Phys.* 97, 104320 (2005). [2] K. Kobayashi et al., *J. Appl. Phys.* 101, 094306 (2007). [3] I. Kinoshita et al., *Chem. Phys. Lett.* 450, 360 (2008).

9:40am **GR+TF+NC-MoM5 Carbon Nanosheets: Novel Two-Dimensional Materials**, *A. Beyer*, *C.T. Nottbohm*, *X. Zhang*, *A. Turchanin*, Universität Bielefeld, Germany, *A. Sologubenko*, *J. Mayer*, RWTH Aachen, Germany, *A. Götzhäuser*, Universität Bielefeld, Germany

1 nm thick, mechanically and thermally stable carbon nanosheets are fabricated by combining molecular self-assembly with lithography. Self-assembled monolayers of aromatic molecules are cross-linked via electron or EUV irradiation. The cross-linked monolayers are released from the substrate, resulting in “free-standing carbon nanosheets” with the thickness of a single molecule.<sup>1</sup> We show that the nanosheets can be transferred onto arbitrary surfaces and we determine their elastic constants by AFM. We have found that nanosheets possess an extraordinarily high mechanical and thermal stability.<sup>2</sup> Carbon nanosheets can be used as ultrathin sample supports for transmission electron microscopy (TEM). Free-standing nanosheets were transferred onto TEM grids and nanoparticles were deposited onto them. When imaged by high-resolution TEM, the nanoparticles show a much higher contrast than those made with a conventional carbon film.<sup>3</sup>

<sup>1</sup> W. Eck, A. Küller, M. Grunze, B. Völkel, A. Götzhäuser: Free-standing nanosheets from cross-linked biphenyl self-assembled monolayers, *Adv. Mat.*, 17, 2583-2587, (2005)

<sup>2</sup> A. Turchanin, M. El-Desawy, A. Götzhäuser: High thermal stability of cross-linked aromatic self-assembled monolayers: Nanopatterning via selective thermal desorption. *Appl. Phys. Lett.*, 90, 053102 (2007)

<sup>3</sup> C. T. Nottbohm, A. Beyer, A. S. Sologubenko, I. Ennen, A. Hütten, H. Rösner, W. Eck, J. Mayer, A. Götzhäuser: Novel carbon nanosheets as support for ultrahigh resolution structural analysis of nanoparticles. *Ultramicroscopy*, in press.

10:20am **GR+TF+NC-MoM7 Enhanced Field Emission of Vertical Aligned Carbon Nanosheets**, *M.Y. Zhu*, *R.A. Outlaw*, *H. Chen*, *M. Bagge-Hansen*, *D.M. Manos*, College of William and Mary

Field emissions from two-dimensional free-standing carbon nanosheets with ultra-thin edges synthesized in radio frequency (RF) plasma enhanced

chemical vapor deposition from CH<sub>4</sub>/H<sub>2</sub> gas mixture have been previously reported. When C<sub>2</sub>H<sub>2</sub>/H<sub>2</sub> gas mixture was used, the growth temperature was lowered by 100-150 °C and well aligned vertical sheets with uniform height distribution were obtained. Typical C<sub>2</sub>H<sub>2</sub> nanosheets were deposited on Si substrates using 80% C<sub>2</sub>H<sub>2</sub> diluted in H<sub>2</sub> gas (5 sccm total gas flow rate) at 600 °C substrate temperature and 35 mTorr total pressure with 1000 W RF power for 10 min. The growth rate of the nanosheets measured from cross-sectional scanning electron microscopic images was 10.8 μm/hr. In this report, field emission measurements were taken from nanosheet samples 1.8 μm in height. Custom LabView software controlled automatic diode I-V measurements of C<sub>2</sub>H<sub>2</sub> nanosheets yielded a threshold field, for an emission current density of 10 μA/cm<sup>2</sup>, of ~3 V/μm, which was ~2 V/μm lower than CH<sub>4</sub> nanosheets. At an applied field of 5.3 V/μm, the current density was 1.1 mA/cm<sup>2</sup> compared to the CH<sub>4</sub> nanosheets of ~0.007 mA/cm<sup>2</sup>. Calculations from the Fowler-Nordheim plots and their linear fits indicated that the effective emission area of C<sub>2</sub>H<sub>2</sub> nanosheets was ~15 times that of CH<sub>4</sub> nanosheets and the β factor of C<sub>2</sub>H<sub>2</sub> nanosheets was ~26% higher than CH<sub>4</sub> nanosheets. The results are consistent with the morphology differences between the two kinds of nanosheets. The lifetime tests of C<sub>2</sub>H<sub>2</sub> nanosheets conducted in a DC mode with a base pressure lower than 5×10<sup>-9</sup> Torr showed a conditioning from an emission current of ~1.1 mA to a current level of 0.65 mA after 200 hour operation without any drop-outs or arcing failure. These tests were conducted with minimal anode cooling, which may account for the decay of the current on this timescale. The vacuum pressure of the testing system was recorded along with the emission current, and found to be closely correlated to the emission current.

10:40am **GR+TF+NC-MoM8 Control of Structures and Electrical Properties of Carbon Nanowalls Using Plasma Enhanced CVD Employing N<sub>2</sub>/O<sub>2</sub> Addition to C<sub>2</sub>F<sub>6</sub>/H<sub>2</sub> Gases.** *W. Takeuchi*, Nagoya University, Japan, *M. Hiramatsu*, Meiji University, Japan, *Y. Tokuda*, Aichi Institute of Technology, Japan, *H. Kano*, NU Eco-Engineering Co., Ltd., Japan, *M. Hori*, Nagoya University, Japan

Carbon nanowalls (CNWs), that is two-dimensional carbon nanostructure of freestanding vertically oriented graphitic sheets, attract great attentions because of several applications such as electrical devices. Recently, it was reported that the 2-D multilayer graphene sheet devices offer the high mobility and the huge sustainable currents. Therefore, CNW films would have high performances of electric devices, since the CNWs basically consist of graphene sheets. In order to realize the CNW devices, it is necessary to control structures and properties. In this study, we have successfully controlled structures and electric properties of CNWs. CNWs were fabricated on the quartz substrate by plasma enhanced CVD (PECVD) employing C<sub>2</sub>F<sub>6</sub> gas with H radical injection. The influences of N<sub>2</sub> and N<sub>2</sub>/O<sub>2</sub> addition to C<sub>2</sub>F<sub>6</sub>/H<sub>2</sub> gas mixtures on structures and electric properties of CNWs were investigated. The cross-sectional scanning electron microscopy (SEM), Hall measurement and secondary ion mass spectrometry (SIMS) were used to evaluate structures, electric properties and atomic compositions of CNWs, respectively. The SEM image of the CNWs film synthesizes by N<sub>2</sub>/O<sub>2</sub> mixture gas addition indicated that the size of the individual graphene of CNWs increased with less branching. The Hall coefficient indicated the positive value for the CNW film synthesized without N<sub>2</sub> addition. In the case of N<sub>2</sub> addition, it displayed the negative one. The positive or negative value means p- or n-type conduction, respectively. CNW films doped with N atoms were characterized by SIMS to investigate the effect of the N<sub>2</sub> addition on the atomic composition of CNWs. The carrier and N concentration in the CNW films formed by N<sub>2</sub>/O<sub>2</sub> mixture gas addition were almost the same as those in the CNW film by N<sub>2</sub> addition. It is considered that the N atom in the CNWs act as a donor and the electrical property of N-doped CNWs can be controlled by N atom concentration. These results will be crucial to fabricate the electrical devices of CNWs without deteriorating crystallinity.

11:00am **GR+TF+NC-MoM9 Wafer-Scale Synthesis and Electrochemical Property of Graphene Formed by Microwave Plasma Enhanced Chemical Vapor Deposition.** *L.C. Chen*, National Taiwan University **INVITED**

The recent experimental breakthroughs on graphene-based nanoelectronics have triggered enormous academic activities. Among the production methods of graphene, mechanical exfoliation of graphite and surface evaporation of SiC are widely used. Despite the successful fabrication in surface evaporation of SiC, process integration of graphene into the well-established Si-based technology remains a challenging task. In view of this, it is also desirable to develop a reliable and large-scale (or wafer-based) production of graphene for practical applications. In this presentation, we will demonstrate a novel synthetic route for graphene growth on silicon substrate by microwave plasma enhanced chemical vapor deposition (MPECVD). The resultant nanoarchitecture exhibits a wall-like morphology with a high density of sharp edges. Structure wise, each nanowall is composed of SiC decorated by a few layer of graphene (i.e. concrete paint

on concrete wall). Moreover, direct deposition of Pt nanostructures onto these graphene samples is also performed to explore their potentials for electrochemical energy applications. Preliminary cyclic voltammetry studies for electro-oxidation of methanol show a high ratio of forward anodic peak current to reverse peak current, suggesting an efficient oxidation of methanol to CO<sub>2</sub> on the Pt/graphene electrode. The effective suppression in the reverse cathodic peak current probably suggests a selective growth of Pt on graphene. Other electrochemical properties (such as electron-transfer kinetics and chemical reactivity) of MPECVD-grown graphene will also be addressed in this presentation. Our synthetic approach offers an alternative route toward wafer-scale fabrication of graphene for fundamental research and opens up various potential applications in nanoelectronics, sensing, catalysis, and energy production.

11:40am **GR+TF+NC-MoM11 Growth and Structures of Carbon Nanowalls during Plasma-Enhanced Chemical Vapor Deposition.** *H. Yoshimura*, *N. Kitada*, *K. Kojima*, *M. Tachibana*, Yokohama City University, Japan

Recently two-dimensional carbon nanostructures called carbon nanowalls (CNWs) have been fabricated by plasma-enhanced chemical vapor deposition.<sup>1</sup> The CNWs are vertically grown on the substrate. According to our previous studies of Raman spectroscopy<sup>2</sup> and transmission electric microscopy (TEM),<sup>3</sup> and Ultraviolet photoelectron spectroscopy,<sup>4</sup> CNWs are composed of small crystallites with a high degree of graphitization. And these small crystallites so-called "nano-graphite domains" were slightly rotated each other. Such shapes and structures of CNWs may provide us with various applications, such as gas storage, membranes for electrochemical energy storage, and field emitters. The understanding of growth process and structures in CNWs is required for the practical applications. In this paper, we report the detail of growth process and structures of CNWs. CNWs were grown by using a dc plasma-enhanced chemical vapor deposition method with a gas mixture of CH<sub>4</sub>, H<sub>2</sub>, Ar. Typical flow rate of CH<sub>4</sub>, H<sub>2</sub> and Ar were 10, 10, and 80 sccm (standard cubic centimeters per minutes at standard temperature and pressure). To investigate the growth process, specimens were prepared for plasma reaction times of 1, 2, 3, 4, 5, 7 and 15 minutes. The shapes and structures of the specimens were investigated by means of atomic force microscopy (AFM), grazing incidence X-ray diffraction (GIXD), and Raman spectroscopy. In addition, the change in the size and quality of crystallites was observed. As reported previously,<sup>5</sup> it was observed that CNWs changes from parallel to perpendicular to the substrate with deposition time. In addition, the transition structure from parallel to perpendicular was clearly observed. From these results, the growth process of CNWs will be discussed.

<sup>1</sup>Y. Wu, et al., Adv. Matter. 14, 64 (2002)

<sup>2</sup>S. Kurita, et al., J. Appl. Phys. 97, 104320 (2005)

<sup>3</sup>K. Kobayashi, et al., J. Appl. Phys. 101, 094306 (2007)

<sup>4</sup>I. Kinoshita, et al., Chem. Phys. Lett. 450, 360 (2007)

<sup>5</sup>B. L. French, et al., Thin Solid. Film, 494, 105, (2006).

## 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  $\sim 18$  nm/s and lateral dimensions of  $\sim 250$  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.<sup>1-3</sup> 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.

<sup>1</sup>J. 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).

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

<sup>3</sup>S. Latil, V. Meunier, L. Henrard, Phys. Rev. B. 76, 21402(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's<sup>1</sup> 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  $200 \text{ mm}^2$  to  $4000 \text{ mm}^2$  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 ( $> 10,000 \text{ cm}^2/\text{Vs}$ ).

<sup>1</sup>C. 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  $\pi$ -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.<sup>1,2</sup> 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.

<sup>1</sup>G. M. Rutter, et al., Phys. Rev. B 76, 235416 (2007).

<sup>2</sup>G. 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.<sup>1</sup> 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).<sup>2</sup> 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

<sup>1</sup> C. Berger et al., J. Phys. Chem. B 108, 19912 (2004).

<sup>2</sup> 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.

## Graphene Topical Conference

Room: 306 - Session GR+EM+NC-TuM

### Graphene and Carbon Electronics

Moderator: B.D. Schultz, International Technology Center

8:00am **GR+EM+NC-TuM1 Two Dimensional Plasmon Behavior in Graphene Sheets on SiC(0001)**, *Y. Liu*, The Pennsylvania State University, *K.V. Emtsev, Th. Seyller*, University Erlangen-Nurnberg, Germany, *R.F. Willis*, The Pennsylvania State University

Using High Resolution Energy Loss Spectroscopy (HREELS), we compare experimental results for the wavevector-dependent behavior of plasmons in a graphene sheet on SiC(0001), with that due to a filled band of surface states on semiconducting silicon. There are significant differences in behavior between the two systems, and the behavior predicted for a classical two-dimensional sheet of electrons. In particular, the damping increases with wavevector beyond the critical momentum for Landau damping by electron-hole pairs. This unusual behavior is compared with that due to the filling of two dimensional surface states on a metallized silicon surface. The graphene results are a consequence of the unusual collective behavior of Dirac fermions. The effect of adding additional graphene layers is discussed.

8:20am **GR+EM+NC-TuM2 Furnace Growth of High Quality Epitaxial Graphene on 4H-SiC(000-1)**, *M. Sprinkle, F. Ming*, Georgia Institute of Technology, *D. Martinotti*, CEA Saclay, France, *P.G. Soukiassian*, Université de Paris-Sud/Orsay and CEA Saclay, France, *C. Berger, E.H. Conrad, W.A. de Heer*, Georgia Institute of Technology

Multi-layer graphene grown epitaxially on the C-terminated (000-1) surface of 4H-SiC in a high vacuum ( $\sim 10^{-5}$  Torr), high temperature ( $\sim 1420$  °C) induction furnace environment has been shown to be of extremely high quality<sup>1,2</sup> and mobility.<sup>3,4</sup> Though multi-layered, the material exhibits electronic properties similar to those of isolated graphene.<sup>1,3,4,5</sup> Here, we characterize the material by atomic force microscopy (AFM), low energy electron microscopy (LEEM), and ellipsometry, and gain insight into growth mechanisms, highlighting growth on 4H-SiC(000-1) in comparison to 4H-SiC(0001), 6H-SiC(000-1), and 6H-SiC(0001). AFM and LEEM images demonstrate  $\mu\text{m}$ -scale graphene terraces. Ellipsometry and LEEM data show that graphene thickness is quite uniform over mm and  $\mu\text{m}$  scales.

<sup>1</sup> J. Hass et al., Phys. Rev. Lett. 100, 125504 (2008).

<sup>2</sup> J. Hass et al., J. Phys. Cond. Matt. 80, (in press)

<sup>3</sup> C. Berger et al., Science 3012, 1191 (2006).

<sup>4</sup> W. A. de Heer et al., Solid State Comm. 143, 92-100 (2007).

<sup>5</sup> M. Sadowski et al., Phys. Rev. Lett. 97, 266405 (2006).

8:40am **GR+EM+NC-TuM3 Toward Carbon Based Electronics. K. Bolotin**, Columbia University **INVITED**

Carbon based graphitic nanomaterials such as carbon nanotubes and graphene have been provided us opportunities to explore exotic transport effect in low-energy condensed matter systems and the potential of carbon based novel device applications. The unique electronic band structure of graphene lattice provides a linear dispersion relation where the Fermi velocity replaces the role of the speed of light in usual Dirac Fermion spectrum. In this presentation I will discuss experimental consequence of charged Dirac Fermion spectrum in two representative low dimensional graphitic carbon systems: 1-dimensional carbon nanotubes and 2-dimensional graphene. Combined with semiconductor device fabrication techniques and the development of new methods of nanoscaled material synthesis/manipulation enables us to investigate mesoscopic transport phenomena in these materials. The exotic quantum transport behavior discovered in these materials, such as ballistic charge transport and unusual half-integer quantum Hall effect both of which appear even at room temperature. In addition, I will discuss electronic transport measurements in patterned locally gated graphene nanoconstrictions with tunable transmission and bipolar heterojunctions. We observe various unusual transport phenomena, such as energy gap formation in confined graphene structures which promise novel electronic device applications based on graphitic carbon nanostructures.

9:20am **GR+EM+NC-TuM5 Spectro-Microscopy of Single and Multi-Layer Graphene Supported by a Weakly Interacting Substrate**, *K.R. Knox, S. Wang*, Columbia University, *A. Morgante, D. Cvetko*, Laboratorio TASC-INFN, Italy, *A. Locatelli, T.O. Mentis, M.A. Niño*, Elettra - Sincrotrone Trieste S.C.p.A., Italy, *P. Kim, R.M. Osgood*, Columbia University

While graphene's distinctive Dirac-cone electronic structure and simple 2D atomic structure have attracted major interest in the physics community, inherent limitations in the size of available exfoliated graphene samples have made it difficult to study this system with conventional UHV probes such as photoemission and low energy electron diffraction (LEED). Thus, previous photoemission and LEED studies of graphene have probed films grown on SiC.<sup>1,2,3</sup> While graphene grown on SiC can form large area sheets, exfoliated graphene on SiO<sub>2</sub> continues to be the system of choice for transport experiments as it is relatively easy to gate and has shown the most interesting and impressive electrical properties.<sup>4,5</sup> Using the high spatial resolution of the Nanospectroscopy beamline at the Elettra synchrotron light source, we have overcome these size limitations by utilizing micro-spot low energy electron diffraction ( $\mu\text{LEED}$ ) and micro-spot angle resolved photoemission ( $\mu\text{ARPES}$ ) to study exfoliated graphene. In this talk, we will discuss our measurements of the electronic structure and surface morphology of exfoliated graphene using low energy electron microscopy (LEEM),  $\mu\text{LEED}$  and  $\mu\text{ARPES}$ . Our LEEM data can be used to unambiguously determine the film thickness of graphene sheets by means of a quantum size contrast effect; a well understood effect in which modulations in the electron reflectivity arise due to quantum well resonances above the vacuum level.<sup>6</sup> Our diffraction measurements provide information about the surface morphology of monolayer and multilayer graphene sheets, which are not atomically flat, but microscopically corrugated. This corrugation increases with decreasing film thickness, reaching a maximum for monolayer graphene. Our photoemission measurements probe the unique massless fermionic dispersion of monolayer graphene, to confirm that the electronic structure of the valence band is well described by the one-orbital tight binding model.

<sup>1</sup> T. Ohta et al., Science 313, 951 (2006).

<sup>2</sup> A. Bostwick et al., Nature Phys. 3, 36 (2007).

<sup>3</sup> S.Y. Zhou et al., Nature Phys. 2, 595 (2006).

<sup>4</sup> K.S. Novoselov et al., Science 306, 666 (2004).

<sup>5</sup> Y.B. Zhang et al., Nature 438, 201 (2005).

<sup>6</sup> M.S. Altman, et al. App. Surf. Sci. 169, 82 (2001).

9:40am **GR+EM+NC-TuM6 Intercalation and Ultrasonic Treatment of Graphite – a New Synthetic Route to Graphene**, *E. Widenkvist*, Uppsala University, Sweden, *R.A. Quinlan*, The College of William and Mary, *S. Akhtar, S. Rubino*, Uppsala University, Sweden, *D.W. Boukhalov, M.I. Katsnelson*, Radboud University of Nijmegen, the Netherlands, *B. Sanyal, O. Eriksson, K. Leifer, H. Grennberg, U. Jansson*, Uppsala University, Sweden

We will demonstrate that ultrasonic treatment of a graphite crystal in water can lead to the formation of small graphene-like flakes in solution. The delamination of the graphite can be increased dramatically by intercalation of bromine from a Br<sub>2</sub>-saturated water solution. After ultrasonic treatment, large amounts of graphene-like flakes with varying thickness are observed in SEM and TEM. They can be adsorbed onto a surface of a suitable substrate by a simple dipping technique. The effect of polar and non-polar solvents as well as adsorption of the graphene on hydrophobic and hydrophilic substrates will be demonstrated and compared. DFT calculations of the intercalation process have been carried out using the SIESTA package and the effect of bromine intercalation on cohesive energy and electronic structure will be discussed and compared with experimental data. Finally, the general approach of using ultrasonic treatment and intercalation as a facile route to graphene synthesis compared to other methods will be discussed.

10:40am **GR+EM+NC-TuM9 Graphene: Exploring Carbon Flatland**, *E.W. Hill, A.K. Geim*, University of Manchester, UK **INVITED**

Graphene is now a bright and still rapidly rising star on the horizon of materials science and condensed matter physics, revealing a cornucopia of new physics and potential applications. We will overview our experimental work on graphene concentrating on its exotic electronic properties and speculate about potential applications.

11:20am **GR+EM+NC-TuM11 The Mechanism of Graphene Growth on Metal Surfaces**, *E. Loginova, N.C. Bartelt, K.F. McCarty, P.J. Feibelman*, Sandia National Laboratories

The structure and defects of graphene sheets have been characterized on many different surfaces, but the mechanisms of graphene growth largely remain unknown. Although simulations have been reported, how carbon atoms attach to the edge of a graphene sheet has not been experimentally determined, owing to limitations of the available experimental techniques. We have used low-energy electron microscopy (LEEM) to study the epitaxial growth of graphene on a representative metal, Ru(0001). The unique capabilities of LEEM allow us to measure simultaneously the growth rate of individual graphene islands and of the local, absolute concentration of vapor-deposited, mobile carbon adatoms. Combining this information, we have learned what controls the nucleation and growth rate of graphene on Ru(0001), and what species transport carbon over the metal surface. Graphene growth differs strikingly from the well-studied case of metal epitaxy: 1) the growth rate is limited by C-atom attachment, not by C-atom diffusion, and 2) the absolute value of the supersaturation required for appreciable growth rates is comparable to that required to nucleate new islands. Thus, a large barrier must exist for monomers to attach to the graphene step edge. We have also discovered that the growth rate as a function of supersaturation is highly nonlinear. Such behavior can be explained if carbon clusters must form, as precursors to carbon attachment. As experiment and theory reveal, this could arise from strong bonding of individual monomers to the metal substrate. We will discuss a model that explains all these observations, and thus provides insight into the molecular processes by which graphene grows. Lastly, we will show that our understanding and ability to monitor the carbon supersaturation allow the shape and position of the growing graphene sheets to be controlled. This research is supported by the Office of Basic Energy Sciences, Division of Materials Sciences, U. S. Department of Energy under Contract No. DE-AC04-94AL85000.

11:40am **GR+EM+NC-TuM12 Conformal Dielectric Layers Deposited by ALD (Atomic Layer Deposition) for Graphene-based Nanoelectronics**, *B. Lee, S.Y. Park, H.Y. Kim, K.J. Cho, E.M. Vogel, M.J. Kim, R.M. Wallace, J. Kim*, The University of Texas at Dallas

To make use of top-gated graphene devices, uniform and thin dielectrics on top of graphene is required. However, the chemically inert nature of graphene basal planes inhibits deposition of high quality and atomically uniform gate dielectric films. Here, we present characteristics of dielectrics employed by atomic layer deposition on top of a highly oriented pyrolytic graphite (HOPG) surface for localized gate applications. It was found that TMA/H<sub>2</sub>O process shows selective deposition of Al<sub>2</sub>O<sub>3</sub> only along with step edges which have high chemical reactivity. Therefore, it is critical to provide uniform and dense nucleation sites on the basal plane in order to achieve conformal deposition of dielectric. In this presentation, we will demonstrate a facile route providing atomically smooth and uniform Al<sub>2</sub>O<sub>3</sub> layers on top of a HOPG by atomic layer deposition (ALD). The physical properties of the deposited Al<sub>2</sub>O<sub>3</sub> layer will be also studied using various characterization techniques including HR-TEM, XPS, and AFM. Acknowledgements: We acknowledge financial supports by KETI through the international collaboration program of COSAR (funded by MKE in Korea) and the SWAN program funded by the GRC-NRI.

# Tuesday Afternoon, October 21, 2008

## Graphene Topical Conference

Room: 306 - Session GR+TF+NC-TuA

### Graphene: Characterization, Properties, and Application

Moderator: Y.J. Chabal, University of Texas at Dallas

1:40pm **GR+TF+NC-TuA1 Computational Modeling of Graphene.** **K.J. Cho, G. Lee,** University of Texas at Dallas **INVITED**

Using the ab initio density functional theory (DFT), tight-binding (TB), and non-equilibrium Green's function (NEGF) methods, we have studied the electronic properties of graphene oxides (GOs) and graphene nanoribbons (GNRs). Dry oxidation of graphene induces epoxide groups on the basal plane, and the corresponding electronic structures of GOs show directional band gap opening. We found that GOs have small energy gaps due to the lattice distortion at low oxygen coverage  $\theta_0$ , and that they become semiconductors for  $\theta_0 \geq 1/2$  with the larger gap at the higher  $\theta_0$  ( $E_g = 3.3$  eV at  $\theta_0 = 1$ ). However, for intermediate coverage, GOs become metallic along one zigzag direction while opening directional energy gaps along other zigzag directions depending on O adsorption patterns. We apply the percolation theory and NEGF method to explain the electron transport behavior of GOs. The insulating property of GOs is used to explain the inactive edge width of GNRs which are observed in GNR experimental studies. Electronic properties of GNRs are shown to have strong dependence on the edge chemistry indicating a practical challenge in graphene nanoelectronics using GNRs as channel materials.

2:20pm **GR+TF+NC-TuA3 Control of Conductivity in Graphene by Formation of Defects.** **S.H.M. Jafri, T. Blom, E. Widenqvist, K. Carva, B. Sanyal, O. Eriksson, H. Grennberg, U. Jansson,** Uppsala University, Sweden, **R.A. Quinlan,** College of William and Mary, **B.C. Holloway,** Luna Innovations Incorporated, **A. Surpi, K. Leifer,** Uppsala University, Sweden

Due to their large surface areas, the conductivity of graphene and carbon nano-sheets depends strongly on their chemical environment. This is the base for future environmental sensors containing graphene sheets. Here, ab-initio calculations propose a possibility of conductivity increase. In the experiment, a 1-2 orders of magnitude increase of the conductivity is observed experimentally on sub-nanometre carbon nano-sheets by using an in-situ nano-manipulation set-up. The conductivity of the graphene sheets was assessed from first-principle simulations. Insertion of defects in the graphene sheets can lead to a strong increase of the conductivity of single graphene sheets. To study this result experimentally, we carried out conductivity measurements on sub-nanometre graphene nano-sheets that are deposited on W-substrates by radio-frequency plasma-enhanced chemical vapour deposition. This deposition process creates free-standing micrometer-sized carbon nano-sheets with sub-nanometre thickness. These nano-sheets were exposed to an acid treatment. It has been shown recently that such acid treatment creates defects in these sheets. Using a nano-manipulator inside a scanning electron microscope, we individually contacted the nano-sheets and measured their resistance as a function of their functionalization. From more than 1000 measurements we obtain a 1-2 order of magnitude increase of conductivity in the functionalised carbon nano-sheets as compared to just water treated or untreated carbon nano-sheets. This result corresponds well to the conductivity change obtained from theory. This study makes it possible to create environmental sensors based on graphene like carbon nano-sheets.

2:40pm **GR+TF+NC-TuA4 Graphene on Graphite.** **J.W. Choi,** Kyung Hee University, South Korea

Topmost graphene layer of graphite is investigated using scanning tunneling microscopy and spectroscopy. Tunneling gap-distance, gap-voltage and bias polarity play an important role in the atomic image contrast and site-dependent tunneling spectra. The study revealed that the coupling and decoupling of the topmost graphene layer to the underlying graphite is occurred because of the weak physical interaction between graphene layers, the electronically active and mechanically soft beta-carbon atoms of graphite and the strong tip-sample interaction.

3:00pm **GR+TF+NC-TuA5 Using Templates to Assemble Graphite Oxide (GO) and Graphene Nanostructures.** **P.E. Sheehan, Z. Wei, J.T. Robinson, D.E. Barlow, E.S. Snow,** Naval Research Laboratory

Graphene and graphite oxide (GO) are new nanoscale building blocks that have generated widespread interest in both basic and applied research. The rapid, inexpensive, and reproducible generation of graphene and GO samples would expedite this work. To this end, we have directed the assembly of single-layered GO sheets using chemical templates patterned

via micro contact printing.<sup>1</sup> Single-layer GO was inexpensively produced using the Hummer method and redispersed in water. Templates of 11-Amino-1-undecanethiol SAMS were created using microcontact printing. Unlike prior work in fullerene templating, the GO sheets could be captured electrostatically without the aid of surfactants. This process yields isolated, single-layer graphene sheets that are arbitrarily located on a patterned substrate. The electrostatic capture mechanism was verified by varying the pH to turn capture on and off. We will discuss the parameters (pH, time, etc.) that affect GO adsorption as well as the surprising resistance of the unpassivated Au substrate to adsorption of the GO sheets. Finally, the adsorbed GO and graphene were electronically and spectroscopically characterized to determine the effect of capture on the reduction process.

<sup>1</sup> submitted to Nano Letters.

4:00pm **GR+TF+NC-TuA8 Studies of Graphene Oxidation and Graphene Oxide Reduction by In-Situ FTIR.** **L. Goux, R. Guzman, J.-F. Veyan, Y.J. Chabal,** University of Texas at Dallas

Graphene oxide (GO) is being investigated by the graphene community because it represents one of the most promising ways to produce graphene single sheets on a large scale.<sup>1,2</sup> Indeed graphene oxidation followed by exfoliation and reduction has been recently demonstrated to give single graphene layers in solution<sup>3</sup>. In addition, in any practical electronic device systems, electron transporting materials need to be controlled by insulating materials which can function as gate dielectrics or separator between device structures. Thus, the role of GO in graphene-based nanoelectronics may be comparable to that of SiO<sub>2</sub> in silicon-based microelectronics. We have therefore developed in-situ IR characterization to monitor graphene oxidation and GO reduction, in order to facilitate the development of graphene-based nanoelectronics. Graphene oxidation is being achieved using a remote oxygen plasma generator. We have designed a vacuum IR-cell (10<sup>-7</sup> Torr base pressure), connected to the oxygen plasma and a Nicolet 6700 FT-IR spectrometer. Preliminary experiments have been carried out using HOPG. The GO reduction is performed in-situ by high temperature annealing in a Specac high temperature cell. In-situ FTIR studies of GO upon thermal reduction have shown a production of CO<sub>2</sub> gas concomitant with the disappearance of the vibrations associated to carboxyl, hydroxyl and peroxide groups in the 120°C-230°C temperature range. Interestingly the vibrational lineshape suggests that CO<sub>2</sub> is incorporated in GO. Around 290°C, there is a strong increase of the absorbance associated with structure changes of GO, resulting from an increase in scattering due to a higher refractive index. The change of refractive index most likely arises from an increase of electrical conductivity after reduction of GO.

<sup>1</sup>Stankovich, S. et al. Carbon 45, 1558-1565 (2007).

<sup>2</sup>Stankovich, S. et al. J. Mater. Chem. 16, 155-158 (2006).

<sup>3</sup>Li, D. et al. Nature Nanotechnology 3, 101 - 105 (2007).

4:20pm **GR+TF+NC-TuA9 Growth of Few Layer Graphene by Microwave Plasma Enhanced CVD.** **R.G. Vitchev, A. Malesevic, A. Vanhulsel, R. Kemps, M. Mertens,** Flemish Institute for Technological Research (VITO), Belgium, **G. Van Tendeloo,** University of Antwerp, Belgium, **C. Van Haesendonck,** Catholic University of Leuven, Belgium, **R. Persoons,** Flemish Institute for Technological Research (VITO), Belgium

Graphene has recently attracted considerable attention as a potential material for nanoelectronic devices. A promising method for its mass production is microwave plasma enhanced chemical vapour deposition (MW PECVD). The main advantage of this technique is that few layer graphene (FLG) can be grown without the need of a catalyst on different substrates that can withstand high temperature (up to 700°C). However, the growth mechanism of PECVD synthesized graphene is not well understood. The aim of this work was to investigate the growth process of FLG deposited by MW PECVD on several substrates (quartz, silicon, platinum). The resulting thin films were characterized by X-ray diffraction, scanning and transmission electron microscopy, Raman spectroscopy and angle resolved X-ray photoelectron spectroscopy (ARXPS). Three stages of film growth on silicon were identified by ARXPS: formation of a carbide layer on the substrate, deposition of an amorphous carbon layer and finally formation of a graphitic layer parallel to the substrate surface. It was established that crack edges in this graphitic layer serve as nucleation sites from which FLG flakes, only four to six atomic layers thin, grow perpendicular to the surface. This growth mechanism appears to be substrate dependent since no intermediate carbide layer was formed on both quartz and platinum surfaces. Furthermore, no amorphous carbon layer was detected on the platinum substrates, even for the shortest deposition time intervals.

4:40pm **GR+TF+NC-TuA10 Uniform Transparent and Conducting Solution Processed Graphene Thin Films for Large Area Electronics, M. Chhowalla, Rutgers University** **INVITED**

The integration of novel materials such as single walled carbon nanotubes and nanowires into devices has been challenging. Similarly, although fundamental research on graphene has been prolific since its discovery, reports on making it technologically feasible for integration into devices have only recently appeared. In this presentation, a solution based method which allows uniform and controllable deposition of reduced graphene oxide thin films with thicknesses ranging from a single monolayer up to several layers over large areas will be described. The opto-electronic properties can thus be tuned over several orders of magnitude, making them useful for flexible and transparent semiconductors or semi-metals. The thinnest films exhibit graphene-like ambipolar transistor characteristics while thicker films behave as graphite-like semi-metals. Controllable p-type doping via exposure to SOCl<sub>2</sub> vapor is also demonstrated. Cl doping leads to breakup of symmetry in ambipolar field effect characteristics, providing a route for unipolar devices. In addition, composite graphene/polymer thin film devices exhibiting on/off ratios >10 will also be reported. Collectively, our deposition method could represent a route for translating the interesting fundamental properties of graphene into technologically viable devices.

5:20pm **GR+TF+NC-TuA12 Electronic Manipulation in Graphene Formed by Proton-irradiated Method, C.-H. Chuang, National Taiwan University and National Synchrotron Radiation Research Center, Taiwan, C.-H. Chen, H.-W. Shiu, National Synchrotron Radiation Research Center, Taiwan, X. Gao, M.B.H. Breese, F. Watt, S. Chen, A.T.S. Wee, National University of Singapore, M.-T. Lin, National Taiwan University, Taiwan**

Graphene is referred to the honeycomb lattice of carbon atoms formed as a 2D flat single layer. It is also the building block to construct 0D fullerenes, 1D carbon nanotubes, and 3D graphite in graphitic materials. Similar to carbon-based materials, the strong C-C bonding with sp<sup>2</sup> hybridization is stable and rigid in ambient atmosphere. In this letter, we introduce the simple method to modify its electronic character and use Scanning Photoemission Microscopy (SPEM) to study its electronic structure. The previous reports about ion- or proton-irradiated graphitic materials present the new physical and magnetic property, e.g. induced ferromagnetic behavior. The reasons are mainly related to the disorder lattice, the vacancy density, or hydrogen-absorbed carbon atoms in the graphene network. However, it is necessary to provide the evidence about electronic structure after proton irradiation. SPEM provides the spatial mapping image with different element and the chemical bonding environment of XPS at the individual location. As compared with the graphite, we can know the C 1s state of graphene is similar. After the proton-irradiated impact on the graphene, the irradiated area shows the broad FWHM of C 1s state and chemical shift of 0.4 eV up to the high binding energy. Besides, we find the satellite peak with binding energy 291 eV, related to  $\pi$  to  $\pi^*$  state transition, is decreased after irradiation. The observed result in our irradiation sample may be due to the lattice reconstruction of C-C bond. Our experimental findings open up a new field in electronic manipulation in graphene-based electronics.



# Tuesday Afternoon Poster Sessions

## Graphene Topical Conference

Room: Hall D - Session GR-TuP

## Graphene Poster Session

### GR-TuP1 Growth Mechanism of Carbon Nanowalls Synthesized by Irradiation of Independently Controlled Ar Ions and CF<sub>x</sub>/H Radicals.

*S. Kondo, O. Stepanovic, Nagoya University, Japan, K. Yamakawa, S. Den, Katagiri Engineering Co., Ltd., Japan, M. Hiramatsu, Meijo University, Japan, M. Hori, Nagoya University, Japan*

Carbon nanostructures such as carbon nanotubes, fullerenes, etc. are investigated intensively in the world. Among a nanocarbon family, we are focusing on carbon nanowalls (CNWs). The CNWs can be described as two-dimensional carbon nanostructures with edges comprising stacks of plane graphene sheets standing almost vertically on the substrate. The CNWs have several unique characteristics, e.g. high aspect ratio and large surface area. Hence, the CNWs attract great attentions for nanoscale electronic devices, storage materials for hydrogen gas, and fuel cells. Previously, we have fabricated CNWs using parallel-plate capacitively coupled plasma with hydrogen (H) radical injection. Considering the practical applications of CNWs, further investigations are required to clarify the growth mechanism for control of their morphologies and properties. In this study, two radical sources, inductively coupled plasmas (ICPs) using 13.56 MHz, and an ion source, a ICP using 13.56 MHz, were used in order to understand which radicals or ions contributed to the CNWs formation. The heated Si substrate was exposed to fluorocarbon (CF<sub>x</sub>) and H radicals as well as Ar ions, of which densities were controlled in each source independently. The correlation between the characteristics of irradiated species, such as Ar ion energy and H radical density, and the CNWs morphologies was investigated employing a spectroscopic ellipsometry for in-situ observation on the substrate surface. It was found that CNWs were not formed with Ar ions of energies below 100 eV with CF<sub>x</sub> and H radical injection. On the other hand, CNWs were successfully formed by Ar ions of energies between 100-200 eV, which were confirmed by SEM and Raman spectroscopy. The deposition rate of CNWs was proportional to the acceleration voltage of the Ar ions. As a result, not only fluorocarbon and hydrogen radicals but also high energy ions accelerated at energies of more than 100 eV were required for the nucleation of CNWs because higher energy ions created dangling bonds on the edge of CNWs, which will be a key for their growth. Furthermore, it was found out that the best value of hydrogen gas flow rate for the highest deposition rate and the better morphology of CNWs existed. The growth of high quality of CNWs with a high growth rate is determined by the balance of the ratio of H radicals to CF<sub>x</sub> radicals. These results will be crucial to elucidate the mechanism of CNWs.

### GR-TuP2 Enhanced Field Emission from Carbon Nanosheets by Thin Film Coatings, *M. Bagge-Hansen, R.A. Outlaw, M.Y. Zhu, D.M. Manos, The College of William and Mary*

Carbon nanosheets (CNS) are a promising two-dimensional carbon allotrope for high current field emission cathodes and are grown by radio frequency (RF) plasma-enhanced chemical vapor deposition from a C<sub>2</sub>H<sub>2</sub>/H<sub>2</sub> gas blend at substrate temperatures of ~ 600 °C. The resulting film consists of vertically oriented, honeycomb sp<sup>2</sup> carbon arrays terminating in single graphene sheets that serve as field emission cathodes. Conditioned films have previously been shown to provide high emission current density (~ 2 mA/mm<sup>2</sup>), and stable lifetime (less than 5% variation over >200 h). The Fowler-Nordheim equation predicts that the field emission current density is a strong function of the local effective work function, e.g., a reduction of 1 eV in work function results in an increase in emission current of up to two orders of magnitude. Since the work function of graphite, carbon nanotubes, and amorphous carbon is relatively high, 4 - 5 eV, selective thin film coatings can substantially lower the effective work function at active emission sites. Mo<sub>2</sub>C and NbC films of ~1 nm have been grown on CNS by e-beam physical vapor deposition (PVD) in very high vacuum and subsequently characterized by Auger electron spectroscopy (AES) and x-ray photoelectron spectroscopy (XPS) in UHV. Comparison of the as-grown CNS to coated CNS show large increases in current density; for example, a 2 nm coating of Cr<sub>2</sub>O<sub>3</sub> /CNS gave an enhanced emission of a factor of 135. The emission shows a strong film thickness sensitivity that may be a consequence of band bending in the surface potential barrier. Coatings of ThO<sub>2</sub>, Cr<sub>2</sub>O<sub>3</sub> and NbO<sub>x</sub> on CNS have also been similarly grown and demonstrate similar improvements in current density.

### GR-TuP3 Thermal Desorption Study of Graphene Oxide, *D.A. Field, C.A. Ventrice, Texas State University, I. Jung, D. Yang, A. Velamakanni, R.D. Piner, R.S. Ruoff, University of Texas*

Graphene oxide is an electrical insulator that shows potential for use in nanoscale electronic devices. An understanding of the thermal stability of graphene oxide sheets is important since the electrical, chemical, and mechanical properties of graphene oxide will change as it is reduced at elevated temperatures. In this study, graphene oxide films were grown by deposition of an aqueous solution of graphene oxide onto oxygen plasma cleaned silicon nitride on silicon substrates. The thermal stability of these films was studied by temperature programmed desorption under ultra-high vacuum conditions. The primary decomposition components of the films are CO and CO<sub>2</sub>. Analysis of the post-anneal chemical composition of the films was performed with x-ray photoelectron spectroscopy. Peaks associated with the C-C bond the C-OH, C=O, and C-O-OH functional groups are monitored at anneal temperatures up to 900 °C.

### GR-TuP4 Photoemission-Assisted Plasma Chemical Vapor Deposition Synthesis of Nano-Grain Graphene on Mica and Silicon, *T. Takami, Tohoku University and CREST JST, Japan, E. Ikenaga, Japan Synchrotron Radiation Research Institute and CREST JST, M. Nihei, Fujitsu Limited and CREST JST, Japan, Y. Takakuwa, Tohoku University and CREST JST, Japan*

Multilayer graphene with nano-scale grain size has been formed on mica and silicon (100) surface with a plasma chemical vapor deposition method at the temperatures lower than 700 C from the mixture gas of methane and argon. The plasma was assisted with an irradiation of ultraviolet light to the sample, induced photoelectric effect. The grown nanographene has been confirmed by transmission electron microscopy and core-level X-ray photoemission spectroscopy with synchrotron radiation. The lattice images with transmission electron microscope and the diffraction patterns indicated that nanographene was formed on the substrates. The peak position of C1s band and pi-pi\* plasmon loss band on the X-ray photoemission spectra also revealed the growth of graphene.

### GR-TuP5 Adsorption Kinetics of Alcohols on Single Wall Carbon Nanotubes, *J. Goering, M. Komarneni, U. Burghaus, North Dakota State University*

Carbon nanotubes can be used as supports for applications such as direct liquid fuel cell catalysts. The adsorption kinetics of alkanes (butane, pentane, hexane, trimethylpentane) and alcohols (methanol, ethanol, propanol, 2-propanol, butanol, pentanol, and hexanol) on single wall carbon nanotubes (HiPco-CNTs) supported on silica has been studied by thermal desorption spectroscopy (TDS). Multi-mass TDS indicate molecular adsorption/desorption with low coverage binding energies increasing linearly with the size of the alcohols. The coverage dependence of the heat of adsorption has been determined by a Redhead analysis. The heat of sublimation, determined by a leading edge analysis, increases linearly with the size of the alcohols whereas the zero order pre-exponential factor is approx. independent of the molecular size. Coadsorption TDS of alcohol-alkane mixtures indicate adsorption of these molecules inside of the CNTs. Adsorption probabilities of alkanes measured by molecular beam scattering increase while opening the CNT tube ends by annealing fresh samples. First results on CoMoCAT CNTs are presented in order to address a possible structure activity relationship.

### GR-TuP6 Fabrication, Structural Characterization, and Surface Modification of Carbon Nanoballs, *T. Ishizaki, National Institute of Advanced Industrial Science and Technology, Japan, N. Saito, O. Takai, Nagoya University, Japan*

Carbon related materials, eg, C60, CNT, diamond, and DLC, have attracted much attention and can be widely used in practical engineering fields. Synthesis of the carbon materials by chemical vapor deposition (CVD) have been considered as one of synthesis methods toward mass production. Carbon nanoballs (CNBs) can be also synthesized by the techniques that are normally used to produce carbon nanotube (CNT). CNBs have been expected to apply polymer composites and biological systems. In the case of application to the polymer composites, it is necessary to tailor the chemical property of the CNBs surface in order to covalently bind the polymer to the CNBs. In order to fabricate the CNBs-polymer composite, it is crucial to establish the surface modification method of the CNBs. In this study, we aimed to fabricate the CNBs by thermal CVD and to chemically introduce carboxylic acid groups to the CNBs surface. Fe-sputtered silicon wafer was used as a substrate. A target of Fe (99.99%) was used as received. A quartz tube in a ringed furnace was used as thermal CVD reactor. The substrate was placed on the center of the quartz tube. Reaction

temperatures were changed from 750 to 990°C. The source gases were H<sub>2</sub> (100 sccm) and CH<sub>2</sub>=CH<sub>2</sub> (25,75, 100, and 175 sccm). The samples obtained were characterized by FE-SEM, TEM, and Raman spectroscopy. The introduction of carboxyl groups to CNBs' surface were carried out by chemical wet process and vacuum ultra-violet (VUV) irradiation. The CNBs were successfully fabricated at the temperature of 850 and 990 °C. Raman spectra showed that the CNBs had two peaks at around 1350 and 1590 cm<sup>-1</sup>, which correspond to D and G bands, respectively. The carboxyl groups were introduced to the CNBs surface by chemical wet process. The COOH-terminated CNBs were uniformly dispersed in water, ethanol, and acetone.

**GR-TuP7 Exposure of Epitaxial Graphene on SiC(0001) to Atomic Hydrogen.** *N.P. Guisinger*, National Institute of Standards and Technology, *G.M. Rutter*, Georgia Institute of Technology, *J.N. Crain*, National Institute of Standards and Technology, *P.N. First*, Georgia Institute of Technology, *J.A. Stroscio*, National Institute of Standards and Technology

Graphene films on SiC exhibit coherent transport properties that suggest the potential for novel carbon-based nanoelectronics applications. Recent studies suggest that the role of the interface between single layer graphene and silicon-terminated SiC can strongly influence the electronic properties of the graphene overlayer. In this study, we have exposed the graphitized SiC to atomic hydrogen in an effort to passivate dangling bonds at the interface. We have used scanning tunneling microscopy to investigate the interface surface structure following exposure to atomic hydrogen for a range of sample temperatures. Initial results indicate that regions of clean SiC were successfully passivated with atomic hydrogen below 400 °C, while the underlying interface of the graphitized regions appear to be unchanged for all temperatures studied. The threshold temperature for passivating clean SiC suggest that the passivated dangling bonds are primarily from Si atoms that are present within the SiC surface reconstruction. Although the hydrogen does not appear to penetrate below the graphene layer, initial results suggest that it does adsorb to the graphene. \*This work was supported in part by NSF grant ECS-0404084.

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