

2D Materials Focus Topic

Room 201B - Session 2D+MN+NS+SS-WeA

IoT Session: Surface Chemistry, Functionalization, Bio and Sensor Applications

Moderator: Daniel Walkup, National Institute of Standards and Technology (NIST)/ University of Maryland, College Park

2:20pm **2D+MN+NS+SS-WeA1 Impact of Hydrogen on Graphene-based Materials: Atomistic Modeling and Simulation of HRSTEM Images**, C. Guedj, Univ. Grenoble Alpes, CEA, LETI, France; L. Jaillet, F. Rousse, Stéphane Redon, Univ. Grenoble Alpes, CNRS, INRIA, Grenoble INP*, IJK, France

The hydrogen energy transition is highly probable, because hydrogen is the most abundant element in the universe and represents an ideal “green” source of energy. Meanwhile, the safe hydrogen production and storage remains a major challenge still in progress. Potential production and storage materials include graphene. In terms of electronic and optoelectronic applications, hydrogen can tune the bandgap of graphene [1]. Hydrogen also plays a major role during the Chemical Vapour Decomposition (CVD) growth of graphene [2]. Hence, hydrogenated graphene-based materials are potentially relevant for various technological applications.

To understand and optimize the device efficiency and the interface engineering, it is advantageous to perform advanced nanocharacterizations, linked to numerical modelling and simulations. This task is particularly difficult, because hydrogen is labile and prone to rapid reorganization. This structural evolution may be monitored with transmission electron microscopy (TEM) techniques [3,4,5], but in spite of significant progresses, the direct detection of hydrogen with High Resolution Scanning Transmission Electron Microscopy (HRSTEM) or energy-loss spectroscopy still remains a serious challenge.

We investigate here the interaction of hydrogen with graphene using the Brenner module of the SAMSON software platform <https://www.samson-connect.net> and we propose an original methodology to characterize its structural arrangement at the atomic scale by simulating HRSTEM images to interpret experimental results. In particular, we compare the effect of hydrogen on dark field (DF), bright field (BF), high-angle annular dark field (HAADF) and annular bright field (ABF) images, to estimate the best technique suited to hydrogen detection.

In addition, we present the effect of carbon vacancies and adatoms on the stability of hydrogen coverage, associated to the HRSTEM signatures of the most stable configurations. These results provide the necessary building blocks to analyze the structure and energetics of hydrogenated graphene-based materials at the atomic scale.

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2:40pm **2D+MN+NS+SS-WeA2 High Density H₂ and He Plasmas: Can They be used to Treat Graphene?**, Hasan-Al Mehedi, Laboratoire des Technologies de la Microélectronique, CNRS-UJF, France; D. Ferrah, Cea, Leti, Minatec, France; J. Dubois, C. Petit-Etienne, Laboratoire des Technologies de la Microélectronique, CNRS-UJF; H. Okuno, Cea, Inac/sp2m/lemma; V. Bouchiat, Institut Néel, CNRS-UJF-INP; O.J. Renault, CEA/LETI-University Grenoble Alpes, France; G. Cunge, Laboratoire des Technologies de la Microélectronique, CNRS-UJF, France

Since graphene and other 2D materials have no bulk, a major issue is their sensitivity to surface contaminations, and the development of cleaning processes is mandatory. High density plasmas are attractive to treat (clean, dope, pattern) 2D materials because they are a mature industrial technology adapted to large area wafer. However, in these plasmas the substrate is bombarded by a high flux of both thermal radicals and reactive ions with typical energy above 10 eV, which can easily damage atomic layer thin materials. We have investigated systematically the interaction of H₂ and He inductively coupled plasmas (ICP) with graphene in industrial reactors. We report a specific issue associated with the use of H₂ plasma: they etch the inner part of plasma reactor walls, thus releasing impurities in the plasma, most notably O atoms that etch graphene and Si atoms which stick on it. The presence of parasitic oxygen presumably explains the

discrepancies found in the literature regarding the impact of reactive plasmas on graphene damages. To get rid of this issue we propose to use a fluorinated aluminum chamber. In this case, fluorine atoms which are shown to be harmless to graphene are the only impurity in the plasma. Under such conditions H₂ ICP plasma is shown to clean graphene without damages if the ion energy is kept below about 15 eV.

3:00pm **2D+MN+NS+SS-WeA3 Novel Binder-free Ag@Ni(OH)₂ over Graphene/Ni Foam and Glucose Sensing**, Tong-Hyun Kang, J.-S. Yu, DGIST, Republic of Korea

Graphene combining with metal nanoparticles or other compounds is widely recognized to be a viable strategy to assemble high-activity catalysts. Unique properties of high conductivity and transparency, 2D morphology, and high stability in acid and alkaline solutions make graphene an excellent electron transfer medium on the interface of graphene/active materials as catalysts. Among them, graphene/metal nanoparticle (G/MNP) composites have been attracting more interest because of remarkably enhanced catalytic property, which is ascribed to a synergic effect from the interface of graphene and active sites. In general, reducing agents and electrodeposition methods have been employed to *in-situ* reduce metal ions such as Au³⁺, Pt⁴⁺, Ag⁺, and Cu²⁺ (Mⁿ⁺) to MNPs on the graphene to form G/MNP composites. In this study, graphene is grown on nickel foam (NF) by chemical vapor deposition (CVD), which is directly used for MNP deposition. Different from bare NF, special phenomenon is observed that the graphene-coated nickel foam (GNF) composite can greatly speed up the electrodeless reduction of Mⁿ⁺ ions on the surface of the graphene. Interestingly, the MNP deposition and Ni(OH)₂ nanosheet assembly simultaneously occur on the GNF. Binder-free Ni(OH)₂-wrapped Ag hybrid developed on the GNF (Ag@Ni(OH)₂-GNF) is found to serve as an efficient electrochemical sensor because of its unique structure. A low detection limit of 0.3 μM and high sensitivity are achieved for the glucose detection, which confirms that the hierarchical electrode structure of Ag@Ni(OH)₂-GNF composite is highly effective to have extensive applications.

3:20pm **2D+MN+NS+SS-WeA4 Surface Modification and Magnetization of Carbon Based Nanostructures**, Rina Tannenbaum, University of Stony Brook; I.T. Kim, Gachon University, Korea; S. Sharma, University of Stony Brook

We describe here a novel synthesis for the facile decoration of carbon nanomaterials (CNM) with monodisperse γ-Fe₂O₃ magnetic nanoparticles.

These procedures were developed for multi-walled carbon nanotubes (MWNTs), reduce graphene (rGO) and reduced graphene nanoroses (rGO-roses). The decoration of these carbon nanomaterials with γ-Fe₂O₃ induces the magnetization of these structures and opens up the potential for their use in novel applications.

CNM/γ-Fe₂O₃ magnetic nanostructures were fabricated through a modified sol-gel process using ferric nitrate nonahydrate, Fe(NO₃)₃·9H₂O as a starting material. Nucleation sites for the iron oxide were generated at the CNM surface due to the electrostatic interaction between Fe (III) ions and the carboxylate surface groups of acid-treated CNMs. The occurrence of gelation was inhibited by the addition of the NaDDBS surfactant, before the addition of propylene oxide, which is a gel promoter. The surfactant interfered in the growth stage of the iron oxide nanoparticles (gel phase) through to the coordination of the NaDDBS molecules to the iron (III) centers due to the attraction between the negatively-charged hydrophilic head of the surfactant and the positively-charged iron. The rGO-roses were further fabricated from decorated rGO via a novel emulsion process.

Various characterization methods were used to confirm the formation of well-defined maghemite nanoparticles, and show that they were tethered to the walls of the CNMs. The tethered γ-Fe₂O₃ nanoparticles imparted magnetic characteristics to the CNMs, which in turn, became superparamagnetic. The magnetic carbon nanotubes and magnetic rGO were introduced into a polymer matrix [8] and were oriented parallel to the direction of an externally-applied magnetic field. The anisotropic nanocomposites were then used as anodes in lithium ion batteries. The magnetic rGO-roses were used as nuclear magnetic resonance contrast material.

Wednesday Afternoon, October 24, 2018

4:20pm **2D+MN+NS+SS-WeA7 Chemical Modification of Graphene and Carbon Nano Tubes as viewed by XPS and NEXAFS Spectroscopies underpinned by DFT Spectra Simulation**, C. Ehlert, E. Donskoy, Bundesanstalt für Materialforschung und -prüfung (BAM), Germany; P.L. Girard-Lauriault, McGill University, Canada; R. Illgen, Bundesanstalt für Materialforschung und -prüfung (BAM), Germany; A. Lippitz, Bundesanstalt für Materialforschung und -prüfung (BAM); R. Haag, M. Adeli, Freie Universität Berlin, Germany; **Wolfgang Unger**, Bundesanstalt für Materialforschung und -prüfung (BAM), Germany

Graphene is a two-dimensional carbon network with unique properties. However, its low solubility, poor reactivity and the limited accessibility of a well-defined basal plane are major challenges for applications. An ideal method to overcome these problems is the covalent attachment of functional molecules to its surface which enable further reactive modifications for specific applications. There are several technologies for surface functionalization of graphene and related CNT materials. To get control on the functionalization process and to optimize the performance of the modified surfaces analytical tools for surface chemical characterization are required. X-ray absorption (NEXAFS) and photoelectron spectroscopy (XPS) have been identified to be rather powerful here [1-3]. Specifically, NEXAFS spectroscopy underpinned by quantum chemical spectrum simulations [4] is unique in a way to address changes of aromaticity and defect formation at the graphene surface during functionalization.

For relevant surface modification technologies, we present examples on how NEXAFS and XPS are fit for purpose. All presented modifications aim on the production of platforms for defined functional 2D nanomaterials, as for example multi-functional hybrid architectures. In detail we investigated:

- A wet chemical method for covalent functionalization of graphene sheets by a one-pot nitrene [2+1] cycloaddition reaction under mild conditions. Here a reaction between 2,4,6-trichloro-1,3,5-triazine and sodium azide with thermally reduced graphene oxide (TRGO) results in defined dichlorotriazine-functionalized graphene sheets.
- Graphene and carbon nanotube functionalized by Vacuum-Ultraviolet (VUV) induced photochemical or r.f. cw low pressure plasma processes to introduce amino, hydroxy or brominated functionalities.

To underpin finger-print information delivered by C K-edge NEXAFS we studied the effects of selected point and line defects as well as chemical modifications for a single graphene layer model by density functional theory based spectrum simulations.

Acknowledgement

We acknowledge support by the team at the BESSY II synchrotron radiation facility in Berlin, Germany, as well as Dr. A. Nefedov (Karlsruhe Institute of Technology, KIT) from the HE-SGM Collaborate Research Group.

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4:40pm **2D+MN+NS+SS-WeA8 Elastic Spongy Graphene-Functionalized Silicon Anode with Excellent Cycle Stability in Li battery**, **Byong-June Lee**, J.-S. Yu, DGIST, Republic of Korea

Graphite plays a prominent role as a typical anode material in the lithium ion batteries (LIBs) because of its high lithiation-dilithiation reversibility and low voltage window. Unfortunately, the capacity is limited to 372 mAh g⁻¹ [1,2]. To search for materials with higher lithium storage capacity, a great number of investigations on metal oxides (or sulfides), Sn, P, and Si have been carried out in recent decades. Among these materials, silicon can make alloy with lithium in the form of Li₂₂Si₅ to deliver a highest theoretical gravimetric capacity of ~4200 mAh g⁻¹, and thus is considered to be one of the most promising anode materials for next generation LIB. It is worth mentioning that its quite low delithiation potential and high lithium storage capacity can provide a wide working voltage window and energy density, which enable promising potential application in electric vehicles. However, those advantages are seriously offset by a great challenge of large volume expansion during lithiation process and the resultant breakage of bulk silicon particles and solid electrolyte interface (SEI), which causes a serious

damage to the electrode structure and thus gives rise to a fast decay of the specific capacity [3].

In this work, novel 3D spongy grapheme (SG)-functionalized silicon is for the first time demonstrated by chemical vapor deposition for a LIB anode, which can overcome the common silicon anode issues such as poor conductivity and volume expansion of Si as well as transfer of Li ion towards the Si. The elastic feature of graphene has excellent function to self-adaptively buffer the volume variation during charge-discharge process. In particular, different from traditional graphene or carbon shells (core-shell and yolk-shell), the spongy 3D graphene networks provide much improved unique functions with excellent long-cycle stability and rate capability. The Si@SG electrode exhibits excellent cycling performance with high reversible specific capacity [4]. A superior 95% capacity retention is achieved after 510 cycles. All the electrochemical performances get benefits from the well-designed functional SG shells, where interconnected nano-graphene structure not only guarantees a high conductive network but also provides more free paths for excellent mass transfer in addition to self-adaptive buffering capability.

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5:00pm **2D+MN+NS+SS-WeA9 Electrical and Structural Changes of Multilayer WSe₂ Transistors: Atmospheric Gas Adsorption and Long Term Aging**, **Anna Hoffman**, M.G. Stanford, C. Zhng, University of Tennessee Knoxville; I. Ivanon, Oak Ridge National Laboratory; A.D. Oyedele, D.G. Mandrus, University of Tennessee Knoxville; L. Liang, B.G. Sumpter, K. Xiao, Oak Ridge National Laboratory; P.D. Rack, University of Tennessee Knoxville

Interest in transition metal dichalcogenides (TMDs) for opto-electronic applications has been growing recently due to their unique properties and layered structure. Surface science and DFT simulations have corroborated p-type doping and n-type suppression of O₂ and H₂O adsorption in TMDs however, electrical characterization has not been fully investigated. This presentation will demonstrate the reversible suppression of n-type conduction in ambi-polar WSe₂ via water adsorption, which logically has a larger impact as the WSe₂ thickness decreases. Additionally, we observe a reversible and irreversible n-type suppression and p-type doping which we attribute to H₂O adsorption and isoelectronic oxygen chemisorption, respectively, at chalcogen vacancies during long term aging in atmosphere over 6 weeks. Finally, controlled oxygen plasma exposure is utilized to oxidize and p-type dope WSe₂. We will overview our device fabrication and electrical testing procedure, and transfer characteristics for our as-fabricated devices for various WSe₂ thicknesses in air and in vacuum will be illustrated. Long-term (6 week) electrical measurements in both air and vacuum are compared to the as-fabricated devices. Finally, complementary atomic force microscopy and Raman Spectroscopy are used to characterize the devices

5:20pm **2D+MN+NS+SS-WeA10 Ion Migration Studies in Exfoliated 2D Molybdenum Oxide via Ionic Liquid Gating for Neuromorphic Device Applications**, **Cheng Zhang**, P.R. Pudasaini, A.D. Oyedele, University of Tennessee Knoxville; A.V. Ilev, K. Xiao, T.Z. Ward, Oak Ridge National Laboratory; D.G. Mandrus, University of Tennessee Knoxville; O.S. Ovchinnikova, Oak Ridge National Laboratory; P.D. Rack, University of Tennessee Knoxville

The formation of an electric double layer in ionic liquid (IL) can electrostatically induce charge carriers and/or intercalate ions in and out of the lattice which can trigger a large change of the electronic, optical and magnetic properties of materials and even modify the crystal structure. We present a systematic study of ionic liquid gating of exfoliated 2D molybdenum trioxide (MoO₃) devices and correlate the resultant electrical properties to the electrochemical doping via ion migration during the IL biasing process. A nearly nine orders of magnitude modulation of the MoO₃ conductivity is obtained for the two types of ionic liquids that are investigated. In addition, notably rapid on/off switching was realized through a lithium-containing ionic liquid whereas much slower modulation was induced via oxygen extraction/intercalation. Time-of-Flight Secondary Ion Mass Spectrometry confirms the Li intercalation. Results of short-pulse tests show the potential of these MoO₃ devices as neuromorphic computing elements due to their synaptic plasticity.

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5:40pm **2D+MN+NS+SS-WeA11 Infrared Absorption of Nanometer-scale Thermally Reduced Graphene Oxide**, *Erin Cleveland, J. Nolde, G. Jernigan, E. Aijfer*, U.S. Naval Research Laboratory

Strong optical absorption is of fundamental importance to infrared (IR) sensors, and it has been well established that graphene is one of the strongest IR absorbing materials, with approximately 2.3% absorption in the IR and visible regions for a single layer. While reduced graphene oxide (RGO) may not have quite the same absorption strength as graphene on a layer-by-layer basis, we believe that by controllably reducing the oxygen concentration within the GO films we can increase the absorption of the RGO film to approach that of graphene. RGO films, unlike graphene, however, can be made arbitrarily thick, allowing for much higher absorbance in a single pass. Here we explore the use of GO films of varying thickness and UHV annealing temperature to achieve near 100% midwave IR absorbance in a quarter-wave reflection filter structure consisting of an RGO film on top of a $\lambda/4$ -thick SiO_2 layer deposited over a Ti/Pd mirror.

Graphene oxide (GO) is a two-dimensional network consisting of a graphene basal plane decorated with oxygen moieties in the forms of carbonyls, epoxies and hydroxyl groups resulting in variable number of sp^2 and sp^3 bonding geometries. Theory indicates that GO bandstructure and transport are strongly dependent on the ratio of the sp^2 and sp^3 bonding fractions, and therefore, by controllably removing specific oxygen groups, one can tune its electronic, optical, and chemical properties. While it is difficult to modify the oxygen concentration using wet chemical processing, e.g. using hydrazine, GO can be thermally reduced in $\text{H}_2\text{-N}_2$ forming gas with more precise control. However, this procedure, like chemical reduction promotes the occurrence of N and H impurities, as well as carbon vacancies within the graphene basal plane, significantly degrading the electronic quality of the film. Here, we use ultrahigh vacuum ($<10^{-9}$ Torr) annealing to controllably reduce the oxygen concentration in GO films while introducing fewer defects. Not only does UHV annealing prevent the introduction of impurities, but after oxygen removal, dangling bonds tend to reform in hexagonal structure. UHV annealing also enables in-vacuo measurement by x-ray photoelectron spectroscopy (XPS) to precisely characterize the overall oxygen concentration and its distribution within alcohol, epoxy and carbonyl species. Following an 800°C UHV anneal for example, we find that the oxygen concentration is reduced to ~5%, and the layer spacing is equivalent to epitaxial graphene grown on the C-face of SiC.

6:00pm **2D+MN+NS+SS-WeA12 Dielectric Properties of Carbon Nanomembranes prepared from aromatic Self-Assembled Monolayers and their application in All-Carbon Capacitors**, *Xianghui Zhang, P. Penner, E. Marschewski*, Bielefeld University, Germany; *T. Weimann, P. Hinze*, Physikalisch-Technische Bundesanstalt, Braunschweig, Germany; *A. Götzhäuser*, Bielefeld University, Germany

Carbon nanomembranes (CNMs) are two-dimensional materials that are made by cross-linking self-assembled monolayers (SAMs) of aromatic molecules via low energy electron irradiation. Previous studies of the charge transport in molecular junction incorporating SAMs and CNMs of oligophenyl thiols has been carried out by using conical eutectic Gallium-Indium (EGaIn) top-electrodes¹ and conductive probe atomic force microscopy (CP-AFM)². Additional investigations of the dielectric properties of pristine SAMs and CNMs were performed by impedance spectroscopy on EGaIn tunneling junctions. Here we demonstrate the fabrication and characterization of all-carbon capacitors (ACCs) composed of multilayer stacks of dielectric CNMs that are sandwiched between two types of carbon-based conducting electrodes: (1) trilayer graphene made by chemical vapor deposition and mechanical stacking; (2) pyrolyzed graphitic carbon (PGC) made by pyrolysis of cross-linked aromatic molecules. The junction area is defined by the width of electrode ribbons, ranging from 1 to 2500 μm^2 , and the separation between two electrodes is tuned by the number of CNM layers. The frequency response of nanocapacitors was measured with an LCR meter. A dielectric constant of 3.5 and a capacitance density of up to 0.5 $\mu\text{F}/\text{cm}^2$ were derived from the junction capacitance. A dielectric strength of 6.2 MV/cm was determined. These results show the potential of carbon nanomembranes to be used as dielectric components in next-generation environment-friendly carbon-based molecular electronic devices.

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² X. Zhang, E. Marschewski, P. Penner, A. Beyer and A. Götzhäuser, *Journal of Applied Physics*, 2017, 122, 055103.

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