P1: Plenary Session Session Chair: Robert Nemanich Monday Morning, May 21, 2018 Room: Humphreys

8:15 AM Opening Comments Bob Nemamich and William Petuskey

8:30 AM *P1 01

Heteroepitaxial Diamond Wafers—Synthesis and Application
Perspectives Matthias Schreck¹, Stefan Gsell², Martin Fischer², Björn-Christoph Gallheber¹, Michael Mayr¹, Michael Traeger³, Mladen Kiš³,
Patrik Ščajev⁴ and André F. Sartori³; ¹Institute of Physics, University of
Augsburg, Augsburg, Germany; ²Augsburg Diamond Technology GmbH,
Augsburg, Germany; ³GSI Helmholtz-Zentrum für Schwerionenforschung,
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University, Vilnius, Lithuania; ⁵Delft University of Technology, 3mE,
Delft, Netherlands.

Long-standing endeavors to synthesize wafer-scale single-crystal diamond have recently resulted in the successful demonstration of a disc with a diameter of about 3.5 inch and a total weight of 155 carat, created by heteroepitaxial nucleation and growth on Ir/YSZ/Si(001) [1]. Parallel activities on (111)-oriented substrates have revealed that similar improvements in structural quality can be obtained by growth of thick samples also for this crystal direction (so far, on small areas only) [2]. In this presentation, first the basic ingredients for heteroepitaxial diamond nucleation on Ir surfaces and our current understanding of the underlying atomic processes will be briefly reviewed. Next, the structural improvement, i.e. the reduction in mosaic spread, dislocation density and Raman line width will be described, comparing directly the results obtained for (001)- and (111)-oriented growth. In both cases, the strategy of growing thick layers will reach its limits, which we currently estimate to be around 10⁶ - 10⁷cm⁻² for the dislocation density. For further improvement of the crystal quality, modified concepts have to be explored. In the second part of the presentation, the influence of dislocations on material properties relevant for the performance of potential devices, and the perspectives of heteroepitaxial diamond for different applications, will be discussed. [1] M. Schreck, S. Gsell, R. Brescia, M. Fischer, Sci. Rep. 7, 44462 (2017). [2] B.-C. Gallheber et al.

9:15 AM *P1.02

Nanoscale Quantum Sensing <u>Florian Kaiser</u> and Jörg Wrachtrup; Institute for Quantum Science and Technology, University of Stuttgart, Stuttgart, Germany.

The accuracy of measurements is limited by quantum mechanics. Ingenious demonstrations, like measuring gravitational fields or time have explored accuracy limits and reached fundamental obstructions. Yet, precision measurements so far are restricted to macroscale and dedicated environments. In the talk I will discuss spin quantum sensors comprising a single electron spin or multiple electron spins in either bulk or nanocrystalline diamond. With such a system we measure a variety of quantities including electric and magnetic fields, temperature, and force. We use advanced quantum control as well as signal analysis techniques to enhance our measurement accuracy [1-3]. I will present a variety of applications ranging from quantum simulations to imaging of cellular structures. References [1] N. Aslam et al. Science 0.1126/science.aam8697 (2017) [2] L. Schlipf et al. Science Advances 3:e1701116 (2017) DOI: 10.1126/sciadv.1701116 [3] F. F. de Oliveira, et al. Nat. Commun. 8, 15409 doi: 10.1038/ncomms15409 (2017)

10:00 AM *P1.03

Diamane, New Forms of Diamond, Artificial Graphene Crystals, Perfect Graphene and 3D Assembly of Graphene-Oxide by Freeze Casting Rodney S. Ruoff; Center for Mulitdimensional Carbon Materials (Institute for Basic Science), Ulsan National Institute of Science and Technology, Ulsan, Korea (the Republic of).

We discuss: (i) synthesis and analyses proving conversion of multilayer graphene to diamane, (ii) the use of polymer precursors for diamond

and their use to achieve new forms of diamond, (iii) artificial crystals by stacking individual single crystal graphene layers, (iv) growth of 'absolutely perfect single layer and single crystal graphene' with no adlayer, (v) the nature of wrinkles and 'folds' in adlayer-free single crystal graphene on Cu(111) single crystals and the role of friction and stress release in their formation, and (vi) bidirectional freeze-casting to yield new forms of 3D assembled graphene oxide and the structure, properties, and carbonization of such structures.

10:45 AM BREAK

E1: Characterization I Session Chair: Li-Chyong Chen Monday Morning, May 21, 2018 Room: Humphreys

11:00 AM E1.01

Electrical Properties of Polycrystalline Diamond Films as Function of Hydrogen Concentration and Grain Size Effects Jesus J. Alcantar-Peña¹. ², Gerardo Gutierrez-Heredia³.¹, Ali Behroozfar⁴, Diego Barrera¹, Elida de Obaldia¹.⁵, Yuriy Koudriavtsev⁶, Majid Minary-Jolandan⁴, Julia W. Hsu¹, Walter Voit¹.⁴.⁴, Dainet Berman-Mendoza² and Orlando Auciello¹.¹?; ¹Material Science and Engineering, The University of Texas at Dallas, Richardson, Texas, United States; ²Centro de Investigación en Física, Universidad de Sonora, Hermosillo, Mexico; ³Centro de Investigación en Óptica, Leon, Mexico; ⁴Mechanical Engineering, University of Texas at Dallas, Richardson, Texas, United States; ⁵Universidad Tecnológica de Panamá, Panama, Panama; °Electrical Engineering, CINVESTAV-IPN, D.F., Mexico; ¹Bioengineering, University of Texas at Dallas, Richardson, Texas, United States.

In this presentation we will discuss the research of growing polycrystalline diamond films and its posterior integration as active layer for a Schottky diode structure. Therefore, in this work polycrystalline diamond thin filmbased diodes are presented as an alternative to single crystal diamond for commercial high power electronic devices. It is well known that polycrystalline diamond films with large grain size exhibit a high thermal transport (~ 1500 W/Km), high mobility (210 cm²/Vs) and promising sheet carrier concentration (6.5 x 10¹³ 1/cm²). These properties may provide diamond-based diodes with high heat sink thermal layers, as well as the next generation micro/nano electronic power devices on diamond films integrated on large area silicon wafers. Here we are reporting research advances on the electrical properties of polycrystalline diamond films grown on Si substrates with different hydrogen concentration and grain size from ~6 nm up to ~3 µm. Similarly, research results will be discussed on the investigation of the materials structure and effects of H atoms insertion in the lattice on the electrical properties of the diamond films. Polycrystalline diamond films were grown on silicon substrates, using hot filament chemical vapor deposition (HFCVD), and Schottky diodes were fabricated on the surface. Small grain size (\sim 6-100s nm) to large grain size (\sim 1-3 μ m) diamond films were grown with constant CH, flow and changing Ar/H, flow ratios. Electrical conduction trough the grain boundaries of the films were measured using Conductive AFM and Kelvin probe AFM methods, while electrical performance was measured trough Schottky diodes structures fabricated on the surface of the diamond films. In addition, Secondary Ion Mass Spectroscopy (SIMS) studies revealed the distribution of hydrogen concentration as function of depth from the surface into de bulk of the polycrystalline diamond films, indicating a reduction of the hydrogen concentration as function of increasing diamond grain size. Also, different techniques such as X-ray Photoelectron Spectroscopy (XPS), Ultraviolet Photoelectron Spectroscopy (UPS), Kelvin probe and Photoemission Spectroscopy, were used to study and compare the hydrogen concentration and grain size effect. The carbon atom bonds related to the grain boundaries of the polycrystalline diamond films were studied by Raman spectroscopy, using visible 532nm laser wavelength, and the changes on film surface morphology were investigated using Scanning Electron Microscopy (SEM). Results from measurements of modulation of electrical properties of polycrystalline diamond films-based Schottky diodes shows the pathway to approach the electrical performance of single crystal diamond-based Schottky diodes, thus accelerating the practical applications of polycrystalline diamond films to a new generation of micro/nano-electronic power devices on 300mm silicon wafers.

^{*} Invited Paper

11:15 AM E1.02

(Late News) Towards Efficient Quantum Source—Epitaxially Grown Diamond Nano-Pyramids with Embedded Silicon-Vacancy Centers Tzach Jaffe¹, Nina Felgen², Lior Gal¹, Lior Kornblum¹, Cyril Popov², Johann Peter Reithmaier² and Meir Orenstein²; ¹Andrew & Erna Viterbi Department of Electrical Engineering, Technion–Israel Institute of Technology, Haifa, Israel; ²Institute of Nanostructure Technologies and Analytics, University of Kassel, Kassel, Germany.

The negatively charged silicon-vacancy center (SiV) in diamond is a potential high-quality single-photon source of indistinguishable photons for quantum information processing and quantum electrodynamics (QED) applications. In contrast to the more common nitrogen-vacancy (NV) center in diamond, the SiV possesses bright narrow-band and spectrally stable optical transition concentrated (70% of the fluorescence) in its zero phonon line (ZPL) protected by inversion symmetry, even at room temperature. However, when embedded in bulk diamond, this photon source suffers from both, relatively low extraction efficiency attributed to total internal reflection as well as nondeterministic emitter location. On the other hand, the use of nanodiamonds or nanostructures prepared by a topdown approach can mitigate the issue of total internal¹ reflection within the diamond, but inherently at the same time introduces additional decoherence mechanisms attributed to poor surface quality. Here we present and implement a robust and deterministic template-assisted bottom-up process for the creation of arrays of high quality nanoscale diamond pyramids (30 nm top diameter) incorporating SiVs. This method employs a predefinition of high-precision nanosized pillars by e-beam lithography and dry etching, and subsequent epitaxial diamond overgrowth by hot filament chemical vapor deposition (HFCVD) during which Si atoms are introduced into diamond. The overgrown layer that forms the diamond pyramid structures is epitaxial as confirmed by X-ray diffraction (XRD), transmission electron microscopy (TEM) and electron backscatter diffraction (EBSD) measurements. We carried out optical confocal scans for collecting the room temperature photoluminescence (PL) from the nano-pyramid array, filtered at the SiV ZPL (738 nm). A clear signature of SiVs emission was detected with an intensity ratio of 4 between the light emanated from the nano-pyramids and the flat overgrown layer between them. This result is related to both, the higher contents of SiVs incorporated inside the pyramids due to higher growth rate at the pillar site, and the higher extraction efficiency from the diamond substrate. Thus, without compromising the source electronic spin coherence, we achieved deterministic nano-localization, higher extraction efficiency compared to bulk diamond and enhanced emission rate compared to diamond pillars with the same diameter. Next, we proposed, designed and validated by full wave simulations the integration of the nano-pyramids with metaldiamond plasmonic structure. This structure enables simultaneous plasmonic-based focusing of the SiVs excitation wavelength and thus enhancing the excitation efficiency of shallow atomic dipoles polarized normal to the diamond surface (or with significant projection to that direction), as well as an extraction path tuned to their emission wavelength (738 nm) that enhances its photon out-coupling attributed to higher extraction efficiency and increased directivity. These plasmonic structure centered about a deterministic high-quality diamond nano-pyramid hosting the SiVs, can pave the way towards efficient single-photon source arrays with high extraction efficiency and selective and efficient optical addressing. References (1) Zhang, J. L.; Sun, S.; Burek, M. J.; Dory, C.; Tzeng, Y.-K.; Fischer, K. A.; Kelaita, Y.; Lagoudakis, K. G.; Radulaski, M.; Shen, Z.-X.; et al. Strongly Cavity-Enhanced Spontaneous Emission from Silicon-Vacancy Centers in Diamond. Nano Lett. 2018, 18, 1360-1365. (2) Aharonovich, I.; Lee, J. C.; Magyar, A. P.; Bracher, D. O.; Hu, E. L. Bottom-up Engineering of Diamond Micro- and Nano-Structures. Laser Photonics Rev. 2013, 7, L61-L65. (3) Jaffe, T.; Sorias, O.; Gal, L.; Kalish, R.; Orenstein, M. Doubly Resonant Nanoantennas on Diamond for Spatial Addressing of Spin States. Nano Lett. 2017, 17, 4217–4222.

11:30 AM E1.03

(Late News) p-Diamond Plasmonic Sub-Terahertz and Terahertz Detectors Michael Shur¹, Sergey Rudin², Greg Rupper² and Tony G. Ivanov²; ¹Electrical, Computer, and Systems Engineering and Physics, Applied Physics and Astronomy, Rensselaer Polytechnic Institute, Troy, New York, United States; ²U.S. Army Research Laboratory, Adelphi, Maryland, United States.

High values of hole mobility, low contact resistance, and high hole sheet densities in p-diamond make feasible using plasmonic diamond field effect transistors as room and high temperature sub-terahertz, terahertz, and far infrared detectors. For the highest reported values of the hole mobility and hole effective mass, the room temperature momentum relaxation time could be as high as 2.5 ps, in which case the plasmonic resonance quality factor could exceed unity at frequencies as low as 60 GHz. We predict the quality factor of the plasma oscillations in diamond as high 5 to 40 at 1 THz is and from 2 to 10 at 0.3 THz and 0.6 THz, respectively. These two ranges are the possible THz communication windows for THz communication for Beyond 5G WI FI applications. This provides a strong motivation for analyzing the potential performance of the diamond plasmonic FET THz and far IR detectors. The potential performance of the diamond THz plasmonic device is determined by the transistor I-V characteristics. To this end, we measured and evaluated the diamond FET current-voltage characteristics using the Unified Charge Control Model (UCCM). The Ohmic contacts were fabricated on single crystal diamond substrates with 100 orientation. The substrates were provided by Element 6. After appropriate surface preparation, the contact area was hydrogenated, and Au contact metal was deposited. The extracted mobility of the 2DHG in the sub-surface channel is 32 cm²/Vs and the sheet carrier density is $3.4x10^{13}$ cm⁻² corresponding to the sheet resistance of 5.74 k Ω . The TLM measurements yielded ohmic contact resistance of $0.5~\Omega mm$. At such sheet resistance, the channel of 170 nm has the resistance equal to the contact resistance, which could be further reduced using the perforated gate structure. A relatively small contact resistance makes plasmonic operation realistic providing further motivation of our study. We evaluated the expected response to THz and sub-THz radiation for the values of the momentum relaxatuion time ranging from 0.01to 6 ps for the gate lengths of 20 nm, 60 nm, and 100 nm. This was done using the compact analytical model (suitable for the device design and parameter extraction) and using a numerical solution of the hydrodynamic equations describing 2DHG. The computer simulations are in excellent agreement with the analytical model and show a small effect of the hole viscosity (in contrast to more conventional semiconductors used for THz applications, such as InGaAs). The results show that the plasma velocity is relatively small because the hole effective mass is large (as low as 105 m/s). This value is smaller than the hole saturation velocity in diamond, which is high because of the high optical phonon energy in diamond. This makes it possible to achieve the so-called "plasmonic boom" instability in the two-dimensional hole gas (2DHG) in p-diamond (this instability requires the drift velocity values to exceed the plasma velocity). The predicted performance of diamond plasmonic detectors shows that these devices are a viable contender for applications in including the 200 to 300 GHz and 600 GHz atmospheric windows that are of the special interest for the Beyond 5G WIFI THz communications. New design and approaches based on the diamond transistor physics are required to achieve the transistor performance with high hole field effect mobility and for minimizing the effect of parasitics, approaches such as the perforated channel, plasmonic crystal, and plasmonic ratchet designs.

K1: Nanodiamonds I Session Chair: Vadym Mochalin Monday Morning, May 21, 2018 Room: Agassiz/Fremont

11:00 AM *K1.01

Heterogeneous Functionalization of Diamond Nanoparticles Lin Lai², Baichuan Sun¹ and <u>Amanda S. Barnard</u>¹; ¹Data61, CSIRO, Docklands, Victoria, Australia; ²School of Physical Science and Technology, Southwest University, Chong Qing, China.

Coating the surfaces of inorganic nanoparticles with functional linker molecules is an important step in the development of many nanoparticle-based drug delivery systems. The efficiency with which drug molecules can be loaded on to nanoparticle surfaces is contingent on the concentration, distribution and stability of these coatings. For example, functionalization of nanodiamond with carboxyl is the first step in loading these materials with therapeutic agents, and the conjugation with proteins, cytochrome, antigen and DNA. Further, functionalisation with polyethylene glycol (PEG), a process known as PEGylation, has already been shown to facilitate greater control over the loading and stability of doxorubicin (DOX) for selective therapies while retaining the pH-sensitivity that is important for drug release. In this study results of high-throughput electronic structure computer simulations are used to map the distribution of COOH and PEG across the entire surface of a reconstructed nanodiamond, with atomic level resolution. This provides useful insights into how the structure of the host nanodiamond impacts the formation of COOH and PEG surface monolayers, and suggests suitable pre-treatments that increase coverage, homogeneity, and binding probabilities with respect to adventurous, non-functional adsorbates that impact performance.

11:30 AM K1.02

Functionalized Nanodiamond for Water Disinfection Axel R. Arroyo¹, Gerardo Morell², Abelardo Colón³, Brad Weiner¹ and Javier Avalos⁴; ¹Chemistry, University of Puerto Rico Rio Piedras Campus, Toa Baja, Puerto Rico, United States; ²Physics, University of Puerto Rico Rio Piedras Campus, San Juan, Puerto Rico, United States; ³Environmental Sciences, University of Puerto Rico Rio Piedras, San Juan, Puerto Rico, United States; ⁴Physics, University of Puerto Rico Bayamon Campus, Bayamon, Puerto Rico, United States.

Detonation nanodiamond or ultra-disperse diamond (UDD) can be used as an alternative and sustainable nontoxic new disinfection material for water treatment plants, due to his biocompatibility, inertness and antibacterial properties. In this research, we performed UDD surface modification to improve the UDD antibacterial properties against coliforms and fecal Escherichia Coli, by adding functional groups such as carboxylic acid (-COOH) and amines (-NH3), which will promote a better interaction between the material and the bacterial membrane, thereby enhancing the UDD efficiency as a water disinfection agent. UDD was successfully functionalized with -COOH and -NH3 groups by strong oxidation and amine addition. The samples were characterized with infrared spectroscopy (FTIR) to confirm the functional groups and X-ray diffraction (XRD). Bacterial characterization was done using Coliscan MF method. The results indicate that the functionalized UDD has strong antibacterial properties, better than those of pristine UDD. The statistical analyses reveal that the carboxylated groups render the UDD more effective against coliforms in water than the aminated groups.

11:45 AM K1.03

Local Probing of Enhanced Field Electron Emission of Gold-Diamond Nanohybrids Sankaran Kamatchi Jothiramalingam^{1, 2}, K. Panda³, B. Sundaravel⁴, I. N. Lin⁵ and Ken Haenen^{1, 2}; ¹Institute for Materials Research, Hasselt University, Diepenbeek, Belgium; ²IMOMEC, IMEC vzw, Diepenbeek, Belgium; ³Institute for Basic Science (IBS), Center for Nanomaterials and Chemical Reactions, Daejeon, Korea (the Republic of); ¹Indira Gandhi Centre for Atomic Research, Materials Physics Division, Kalpakkam, India; ⁵Tamkang University, Department of Physics, Tamsui, Taiwan.

Field emitters with superior field electron emission (FEE) characteristics are employed as a cold cathode materials in flat panel displays and other electron emitting devices. Diamond possesses high FEE characteristics due to its negative electron affinity (NEA), which makes it a promising material for application as FEE emitters. But owing to the wide bandgap nature of diamond, the electrical conductivity in bulk diamond is low, which decreases its electron emission. Therefore, the material needs to be made conductive. Recently, the main focus in this respect has been directed towards the synthesis of ultrananocrystalline diamond (UNCD) films containing ultra-small diamond grains of 5-10 nm with smooth surface characteristics. Such films exhibit higher electrical conductivity and better FEE properties than any other known form of diamond films, which is believed due to the sp²-bonded carbon in the grain boundaries as acting as electron conduction channels [1]. Hence, direct detection of electron emission sites from diamond grains or grain boundaries on a microscopic scale is crucial to fully understand the underlying emission mechanisms and remaining related questions [2]. In this work, scanning tunneling microscopy (STM) is being utilized to investigate the local FEE behavior and surface electronic properties of diamond films [3]. It allows direct imaging of the emission, leading to understanding of the mechanism behind the enhanced conductivity and FEE properties for Au-ion implanted UNCD films. Such films, implanted with 1×10^{17} Au ions/cm² possess a high electrical conductivity of ~ 5 kS/cm and a low turn-on field of 1.6 V/µm, yielding a high FEE current density of 5.4 mA/ cm^2 (@ 2.65 V/ μ m), and high lifetime stability of 1825 min. Straight imaging of conducting/non-conducting sites is mapped by STM. The local current-voltage measurements illustrate that grain boundaries are indeed the conducting/emitting sites. Further, this fact is supported by the high resolution current imaging tunneling spectroscopy. The formation of abundant conducting sp² nanographitic phases along the diamond grain boundaries, confirmed from transmission electron microscopic examinations, is believed to be the genuine factor that helps for the easy transport of electrons and hence enhances the conductivity/emission properties. The fabrication of these films with high conductivity and superior FEE properties is a direct and simple approach which opens up new prospects in flat panel displays and high brightness electron sources.K. J. Sankaran is a Postdoctoral Fellow of the Research Foundation-Flanders (FWO). References [1] O. Chubenko, et al., ACS Appl. Mater. Interfaces 9 (2017), 33229. [2] R.L. Harniman, et al., Carbon 94 (2015), 386. [3] K. Panda, et al., Carbon. 111 (2017), 8.

> B1: Diamond Electronics I Session Chair: Hiroshi Kawarada Monday Afternoon, May 21, 2018 Room: Humphreys

1:30 PM *B1.01

Deep Depletion Diamond MOSFET <u>Julien Pernot</u>; Univ Grenoble Alpes, CNRS, Grenoble INP, Institut Néel, Grenoble, France.

Diamond is a fascinating semiconductor with exceptional physical properties such as a wide band gap, a high breakdown electric field (10 MV/cm), an outstanding thermal conductivity (20 W/cm/K) and high carrier mobilities. These exceptional properties, or more precisely, the combination of some of these properties makes diamond an ideal semiconductor for high power and/or high frequency electronics which should surpass other materials like silicon, silicon carbide or gallium nitride. Numerous diamond field effect transistors are under investigation:

^{*} Invited Paper

^{*} Invited Paper

H-terminated accumulation FET, O-terminated inversion channel FET, metal-semiconductor FET and junction FET.In this work, we propose a new transistor concept in order to exploit the full potentialities of diamond material¹. First, a stable deep depletion regime will be demonstrated in metal oxide semiconductor capacitors using p-type oxygen-terminated (100) diamond as a semiconductor and Al₂O₃ deposited by atomic layer deposition at 380°C. Current voltage I(V) and capacitance voltage C(V) measurements were performed to evaluate the effectiveness of diamond semiconductor gate control². An effective modulation of the space charge region width is obtained by the gate bias, where the deep depletion regime is demonstrated for a positive gate bias. The deep depletion concept will be described and proposed for MOSFET devices. A proof of concept of deep depletion diamond MOSFETs will be presented^{1,3}. Finally, the recent improvements achieved at Institut Néel in terms of device performances will be discussed. References 1 T.T. Pham, N. Rouger, C. Masante, G. Chicot, F. Udrea, D. Eon, E. Gheeraert, and J. Pernot, Appl. Phys. Lett. 111, 173503 (2017). ² T.T. Pham, A. Maréchal, P. Muret, D. Eon, E. Gheeraert, N. Rouger, and J. Pernot, J. Appl. Phys. 123, 161523 (2017). 3 T.T. Pham, J. Pernot, G. Perez, D. Eon, E. Gheeraert, and N. Rouger, IEEE Electron Device Lett. 38, 1571 (2017).

2:00 PM B1.02

Diamond RF Electronics Technology Development at the US Army Research Laboratory Pankaj B. Shah, James Weil, Mahesh R. Neupane, Kevin G. Crawford, A. G. Birdwell, Frank J. Crowne and Tony G. Ivanov; US Army Research Laboratory, Adelphi, Maryland, United States.

Due to the thermal limits of GaN and other prospective ultra-wide bandgap semiconductor materials, the US Army considers diamond, with its high thermal conductivity and large energy bandgap, as a potential next generation semiconductor electronics material. As a result, the Army Research Laboratory has an in-house program to develop Radio-frequency (RF) field-effect transistors (FETs) on hydrogen-terminated diamond surfaces. These devices are fabricated on single-crystal diamond substrates, and employ both atmospheric adsorbates and MoO₂ as the transfer-doping electron acceptor layer leading to a free-hole current channel.1 Our presentation will discuss device fabrication together with the DC and RF characterization results for FETs fabricated with gate lengths ranging from 4µm to sub-100nm. Starting materials are plasma hydrogenated to achieve a negative electron affinity surface. Following this procedure, a standard four mask fabrication process is used with the last mask being utilized for electron-beam writing of the gate finger. These masks are 1) Ohmic contact / device isolation, 2) Ohmic contact anchor between oxide and hydrogenated regions, 3) source-drain separation / Schottky contact metal and 4) plating-up probed regions. Ohmic contacts adhesion to the hydrogenated surface is improved by deposited metal anchor layers that are held in place via the increased adherence to oxygen terminated diamond. Contact resistance, sheet resistance and mobility data are collected and correlated with device results. A highlight of the atmospheric transfer-doped RF FET performance comes from the devices fabricated at L_c=100nm. These devices resulted in DC drain current densities I_D=600 mA/mm, transconductance g_m=140 mS/mm, current gain cutoff frequency f=41GHz, and power gain cutoff frequency of f_{max}=44GHz. This demonstrated technology compares favorably with reported state-of-the-art results, in terms of DC and small signal performance.^{2,3} Capacitance-voltage measured charge carrier concentration peaked at 1.3×10^{20} cm⁻³ for a 1µm gate length FET compared to 7.5×10^{15} cm⁻³ for a 2µm gate length FET. Sheet resistance below 5 kW/sq are typically seen for the hole conducting channel. To better understand device performance and improve chances for accurately predicting potential for transfer-doped diamond FET devices, we performed both drift-diffusion device and atomistic materials and electronic structure simulations. Driftdiffusion simulation of the FETs indicate that precise modeling of the acceptor states, including energy band offsets, correlates well with device performance.4 Also, our modeling results indicate that dopants preserved under the gate lead to higher on-currents. An atomistic first principle simulation is used to identify stable diamond surface configurations with and without hydrogenation. Furthermore, using the identified stable configuration, the effect of hydrogen surface coverage on device parameters such as mobility, electron affinity and work-function were determined. Exploring the hydrogenation process by means of this atomistic model may assist experimental efforts focused on the preservation of the hydrogen termination layer, and hence to long-term robust device operation. 1 F. Maier, M. Riedel, B. Mantel, J. Ristein, and

L. Ley, "Origin of Surface Conductivity in Diamond", Phys. Rev. Lett., 85, 3472 (2000). ² D. A. J. Moran, S. A. O. Russell, S. Sharabi, and A. Tallaire, "High Frequency Hydrogen-terminated Diamond Field Effect Transistor", 12th IEEE International Conference on Nanotechnology, Birmingham, UK (2012). ³ H. Kawarada, H. Tsuboi, T. Naruo, T. Yamada, D. Xu, A. Daicho, T. Saito, and A. Hiraiwa, "C-H Surface Diamond Field Effect Transistors for High Temperature (400°C) and High Voltage (500V) Operation", Appl. Phys. Lett. 105, 013510 (2014). ⁴ P.B. Shah, J. Weil, A.G. Birdwell, and T. Ivanov, "Charge Trapping Analysis of High Speed Diamond FETs", MRS Advances, DOI: 10.1557/adv.2017.141 (2017).

2:15 PM B1.03

Band Alignment of Al₂O₃/SO₂/H-Diamond Heterointerfaces Using Synchrotron X-Ray Photoelectron Spectroscopy (XPS)/XANES Measurements Niloy C. Saha¹, Kazutoshi Takahashi², Masaki Imamura² and Makoto Kasu¹; ¹Electrical and Electronic Engineering, Saga University, Saga, Japan; ²Synchrotron Light Application Center, Saga University, Saga, Japan.

Diamond possesses the highest band-gap and high breakdown electric field among semiconductor materials. Therefore, diamond is preferred for high-power and high-temperature operations due to its excellent physical properties. We have developing diamond FET using NO, hole doping. Thus, O₃, NO₂, SO₂ and NO adsorption generate holes carriers¹. This mechanism was explained by the first principle calculation that the unoccupied orbitals of inorganic gas molecules attract the electron of H-diamond and induced holes on the surface, thus increasing the sheet concentration². The Al₂O₂/NO₂/H-diamond heterointerface was studied previously and found that type II band-alignment, where the valence band offset was 3.9 eV³. Band offset of the Al₂O₃/H-diamond interface is influenced by the exposed NO₂ gas molecules and a high band offsets were observed than those of H-diamond without any exposure. In this study, we determine energy band alignment using synchrotron X-ray photoelectron spectroscopy (XPS) and X-ray absorption near edge structure (XANES), and investigate the interface states of Al₂O₂/SO₂/H-diamond heterointerface. We used 1-µm-thick (001) H-diamond homoepitaxial layer. H-diamond sample was exposed to the SO, gas for 10 mins. Finally, a 4-nm-thick Al₂O₂ layer was deposited as oxide layer to fabricate the Al₂O₂/SO₂/H-diamond structure. All the measurements were performed at Saga Synchrotron Light Source (SAGA-LS), while Al₂O₂ layer was thinning by Ne⁺ ion beam sputtering. The ratio of C 1s/O 1s was used to determine the thickness of the Al₂O₃ layer.O 1s, C 1s, and Al 2p spectra were taken with the photon energy of 700 eV using XPS measurements. The respective peak of the measured spectrum was fitted by the leastsquare-fitting technique using a Shirley background and Voigt line shape. In this case, O 1s spectra were decomposed into two peaks and C 1s spectra were decomposed into 4 peaks. In addition, photoemission spectra were taken in the binding energy ranges from 155 to 180 eV in order to detect S 2p peak as the H-diamond was exposed to SO₂. However, no peak of S 2p was found. The O 1s peak at higher binding energy and absence of S 2p at the interface indicate that SO₂ gas molecules are decomposed and oxygen adsorbates chemically bonded to H-diamond surface. Measurement of X-ray absorption near edge structure (XANES) spectra at O K-edge and Al L-edge were performed to investigate interfacial states. Energy band alignment was determined from the energy positions of the core-level spectra of C 1s and Al 2p, and valence band maximum (VBM) value of the diamond and Al₂O₃ layer. VBM of Al₂O₃ layer was determined as 3.5 ± 0.1 eV. A type II band (staggered type) alignment is determined from measured values, where a valence band offset of 3.5 ± 0.1 eV and a conduction band offset of 2.3 ± 0.1 eV were found. The valence band offsets are lower than those of NO₃-exposed diamond interface. However, a good agreement of accumulation hole carriers due to SO₂ exposure is demonstrated from the band-offset of the Al₂O₂/SO₂/H-diamond heterointerface. **References** [1] M. Kubovic and M. Kasu, *Jpn. J. Appl.* Phys. 49 (2010), 110208. [2] Y. Takagi, K. Shiraishi, M. Kasu, and H. Sato, Surf. Sci. 609 (2013), 203. [3] K. Takahashi, M. Imamura, K. Hirama, and M. Kasu, Appl. Phys. Lett. 104 (2014), 072101.

2:30 PM B1.04

Hydrogen-Terminated Diamond Field-Effect Transistor with Double Dielectric Layers of HfSiO₄/Al₂O₃ Yan-Feng Wang, Xiaohui Chang, Wei Wang, Dan Zhao, Jiao Fu, Zongchen Liu, Guoqing Shao, Zhangcheng Liu, Shuwei Fan, Renan Bu, Jingwen Zhang and Hongxing Wang; Xi'an Jiaotong University, Xi'an, China.

Diamond film collects all the outstanding properties of mechanics, electronics, heat, and optics etc. together making it have potential applications in the fields of wide range optical transparent window material, super-hard coating tolls, especially, in the field of high power electron devices which can work with high frequency and in high temperature environment Recently, researchers have extensively investigated hydrogen-terminated diamond (H-diamond) field effect transistors (FETs)1,2. H-diamond surface can form a p-type conduction layer with a sheet hole density and hole mobility of 10¹³ cm⁻² and 50-150 cm²V⁻¹s⁻¹,repectively. However, this conduction layer is thermally and chemically instable and vulnerable to the ambient environment such as oxidation of diamond surface or higher annealing even in vacuum. All these would cause the missing of p-type conduction layer and the degradation of FET performance. This problem could be solved by fabricating dielectric layers on diamond surface to maintain the p-type conduction channel, which also acts as a insulators. Various dielectric layers have been used such as SiO2, Al2O3, HfO2, Ta2O5, and ZrO2 on H-diamond surface. However, there are few reports on using dielectric of HfSiO,, which has excellent thermal stability, adequate band gaps (from 15 to 25 depending on hafnium content). In this work, investigation of H-diamond FETs with ALD-Al₂O₂/SD-HfSiO₄ dielectric layers have been carried out. Firstly, gold electrodes were patterned on H-diamond by electron beam evaporation technique. Secondly, UV-ozone was used to insulate p-type conduction channels between source and drain electrodes. The length of channels were 20um. Then, 4nm ALD-Al₂O₃/30nm SD-HfSiO₄ dielectric layers were fabricated on p-type conduction channels by ALD and SD technique. Finally, electrical properties of this diamond FETs were investigated. The detail will be presented in the conference. 1H. Kawarada, H. Tsuboi, T. Naruo, T. Yamada, D. Xu, A. Daicho, T. Saito, and A. Hiraiwa, C-H surface diamond field effect transistors for high temperature and high voltage operation, Applied Physics Letters, 105 (2014) 013510. 2Akira Daicho, Tatsuya Saito, Shinichiro Kurihara, Atsushi Hiraiwa, and Hiroshi Kawarada, High-reliability passivation of hydrogen-terminated diamond surface by atomic layer deposition of Al2O3, J. Appl. Phys. 115 (2014) 223711.

2:45 PM B1.05

Enhanced 2DHG Mobility Using an Al $_2$ O $_3$ Interfacial Layer Between Diamond and V $_2$ O $_5$ Yichen Yao 1 , Yu Yang 2 , Xingye Wang 1 , Franz A. Koeck 2 and Robert J. Nemanich 2 ; 1 School for Engineering of Matter, Transport and Energy, Arizona State University, Tempe, Arizona, United States; 2 Physics, Arizona State University, Tempe, Arizona, United States.

Vanadium pentoxide has been used as a thermally stable electron accepting material for surface transfer doping of diamond. The large difference between the Fermi level of hydrogen-terminated diamond and transition metal oxides (V₂O₅, MoO₃, WO₃, Nb₂O₅ and etc) has been described as a driving force for electron transfer from the valence band of diamond to transition metal oxides (TMO). With its high electron affinity of up to 6.2 eV, V₂O₅ has indicated its potential for electron transfer in diamond-TMO structures compared with its competitors. However, to the experimentally measured hole mobility of H-diamond/ V₂O₅ structures appears to limit its application. Scattering from near interface charges is often described as one of the sources of the low mobility. To address this limitation, we propose to employ a thin layer of Al₂O₂ to retain the 2DHG characteristics and potentially support an increased carrier mobility due to reduced scattering. In this study, H-terminated intrinsic and B-doped diamond substrates were used as starting surfaces. A thin (2nm) Al₂O₂ layer was deposited by plasma enhanced atomic layer deposition (PEALD). A temperature stable surface conducting state was obtained after a 500C remote hydrogen plasma treatment. Thin layers of vanadium oxides were deposited on the Al₂O₃ layer using molecular beam deposition. Each deposition process was followed by an oxygen plasma treatment to obtain fully oxidized V2O5. In situ XPS and UPS measurements were used to establish the presence of the 2DHG and to verify the oxidation state and thickness of the V₂O₅. Downward band bending was observed after Al₂O₃ growth which transitions to upward

band bending and a 2DHG after the post deposition hydrogen plasma treatment. Deposition of vanadium oxides followed by plasma oxidation results in core level features consistent with V_2O_5 and a 2DHG. Moreover, ex situ Hall measurements confirm the presence of a conducting layer after Al_2O_3 and V_2O_5 deposition. In fact, the presence of the Al_2O_3 interlayer, leads to a lower sheet resistance and an increased hole mobility. The band diagram derived from the photoemission measurements supports the presence of the 2DHG in the H-diamond/Al $_2O_3/V_2O_5$ structure. Acknowledgement: this research is supported through grants from MIT Lincoln Laboratory and the National Science Foundation (DMR - 1710551).

3:00 PM BREAK

G1: Graphene and 2-D Materials I Session Chair: Chia-Liang Cheng Monday Afternoon, May 21, 2018 Room: Agassiz/Fremont

1:30 PM G1.01

Advances in Ultra-Low Energy Ion Implantation of Graphene and Other Low Dimensional Materials <u>Hans Hofsaess</u> and Manuel Auge; Faculty of Physics, 2nd Institute of Physics, Göttingen, Germany.

Doping of 2D materials by ion implantation has unique requirements regarding ion energy, ion energy spread, ion beam optics, sample preparation and sample electrical conductivity. Efficient substitutional incorporation of low energy ions into the graphene lattice requires energies well below 50 eV. We use a low energy mass selected ion beam system with a UHV implantation chamber. A 30 keV mass selected ion beam is guided through differential pumping stages and homogenized using a beam sweep. The beam is then decelerated in a UHV-chamber down to energies of as low as 10 eV. With the help of focusing/defocusing lenses an area of several mm² up to about 2.5 cm² can be uniformly irradiated with these ultra-low-energy (ULE) ions with beam current up to several μA. Up to now ion sources are available as isotopically pure beams of B⁺, C⁺ N⁺ ,F⁺, and P⁺, S⁺, Mn⁺, Se⁺, W⁺ and Au⁺. Ion sources for elements like Al, Fe, As and rare earth elements are under development. The implanted areal concentration and elemental composition can be verified with in-situ Auger spectroscopy and more quantitative with Rutherford backscattering by implanting into amorphous carbon films as test samples. The detection limit is below 10¹⁴ ions/cm² for heavier elements. Implanted ¹¹B can be analyzed with the ${}^{11}B(p,2\alpha)\alpha$ nuclear reaction with a detection limit of about 1x10¹⁴ B/cm². Challenges for ULE ion implantation such as nonflat substrates, charging of substrates, cleaning of surfaces and lateral controlled implantation will be briefly discussed. Examples for doping of monolayer graphene with ¹¹B⁺, ¹⁴N⁺ and P⁺ ions and doping of 2D MoS, with Se⁺ ions will be presented.

1:45 PM G1.02 DISCUSSION TIME

^{*} Invited Paper

2:00 PM *G1.03

Unique Aspects of Charge Transfer in Graphene and Their Applications Mario Hofmann¹, Ya-Ping Hsieh² and Kevin Chang³; ¹Department of Physics, National Taiwan University, Taipei, Taiwan; ²Institute of Atomic and Molecular Sciences, Academia Sinica, Taipei, Taiwan; ³Department of Materials, National Cheng Kung University, Tainan, Taiwan.

Graphene has shown superior performance to bulk materials in many areas ranging from sensing to RF amplification. However, the two-dimensional nature of graphene is often neglected in approaches to employ it as a replacement for traditional engineering materials. We here report several unique aspects of carrier transport in graphene under extrinsic doping and the promising applications they enable. Since graphene's carriers are confined to the surface, we observe that the morphology of dopants significantly affects carrier transport. Employing in-situ characterization tools we identify distinct scattering mechanisms at low and high dopant coverage regimes. At low dopant coverage we observe a low charge transfer efficiency between dopant and graphene that is 5% of the values expected from energy considerations. This discrepancy originates from due the limiting geometrical capacitance of small adsorbate clusters. An increase of the cluster size was found to increase the charge transfer efficiency to 70% and lower the sheet resistance to 106Ohm/sq at 97% transmittance which represents highest reported performance for doped single layer graphene and is on par with commercially available transparent conductors. Unfortunately, the achievable enhancement in performance upon increase of dopant coverage is limited by a competing effect of charge carrier scattering. We obseve a capacitive coupling between doped and undoped regions of graphene that suppress ambipolar transport and result in a persistent p-type transfer characteristic. This effect is dominating the carrier scattering and can result in a six-fold decrease in mobility. When dopant clusters reach dimensions where they start merging, charge transport proceeds through percolation in the pristine graphene phase. This transition results in an increase in sheet resistance and a surprising behavior of the Hall effect that had been previously misinterpreted as change in doping type and mobility enhancement. The efficient charge transfer to graphene was demonstrated to enable several novel applications. Electrostatic control over graphene's carrier concentration allowed change of the band alignment in graphene/insulator/semiconductor heterojunctions. Thus, vertical tunnelinginjection light-emitting transistors (VtiLET) were produced where gating allows adjustment of the light emission process. This advance enabled arbitrary color light emission from one single emitter. Moreover, we show that the electrochemical properties of graphene are intimately linked to the position of its Fermi level. Thus, an electrochemical transistor was realized that permits the dynamic modification of the potential and reaction rate of an electrochemical reaction over a wide range.

2:30 PM G1.04

Twisted Bilayer Graphene Based ECG Sensor Angela Luis Matos¹, Samuel Escobar Veras¹, Ruth Adames Méndez³, Tej B. Limbu¹, Frank Mendoza¹, Brad Weiner² and Gerardo Morell¹; ¹Physics, University of Puerto Rico Rio Piedras, San Juan, Puerto Rico, United States; ²Chemistry, University of Puerto Rico Rio Piedras, San Juan, Puerto Rico, United States; ³Interdisciplinary, University of Puerto Rico Rio Piedras, San Juan, Puerto Rico, United States.

Physiological recordings have been one of the most significant tools for today's diagnosis technologies, allowing for non-invasive techniques that deliver useful insights in patient care. Electrocardiogram (ECG) recordings stand among the most important of the diagnosis tools, since according to the Department of Health statistics, for more than a decade heart related diseases have proven to be more lethal than cancer. Nonetheless, ECG recordings electrodes suffer from material degradation which results in high impedance values that limit the usability of the technique for an accurate diagnosis. In order to fix this problem new electrodes most be proposed with low impedance and high chemical stability, properties that graphene has been known for. Therefore, hereby we report results on the implementation of large area bilayer graphene as a long term low impedance electrode for ECG applications. The large area bilayer graphene has been synthesized using the hot filament chemical vapor deposition (HF-CVD) technique on copper substrates. The as synthesized material was later transfer to a more suitable substrate for physiological recordings experiments. Raman spectroscopy, alongside sheet resistance measurements, ensures the high quality of our material, before and after the transfer process. Electrical measurements have been taken to determine the material impedance dependence on frequency, in the interface skin/gel/electrode. Results suggest that graphene offers a practical solution to extend the life cycle of an ECG electrode when interfacing with human skin. Thus, securing long term low impedance reliable recordings.

2:45 PM DISCUSSION TIME

3:00 PM BREAK

C1: Diamond Photonics/Spintronics I Session Chair: To Be Announced Monday Afternoon, May 21, 2018 Room: Humphreys

3:30 PM C1.01

Fluorescence Enhancement of Nitrogen-Vacancy Centres in Diamond Marco Capelli^{2,1}, Philipp Reineck^{2,1}, Desmond Lau¹, Antony Orth^{2,1}, Jan Jeske¹, Marcus W. Doherty³, Takeshi Ohshima⁴, Andrew D. Greentree^{2,1} and Brant C. Gibson^{2,1}; ¹School of Science, RMIT University, Melbourne, Victoria, Australia; ²ARC Centre of Excellence for Nanoscale BioPhotonics, Melbourne, Victoria, Australia; ³Laser Physics Centre, Research School of Physics and Engineering, Australian National University, Canberra, Australian Capital Territory, Australia; ⁴National Institutes for Quantum and Radiological Science and Technology, Takasaki, Japan.

Bright fluorescence from nitrogen-vacancy (NV) centres in diamond is critical for many applications. The NV centre is a fluorescent defect in the diamond lattice that has gathered interest in the science community in the last decades. Its applications range from optically detected sensing using ensembles of NV centres to nanometer scale fluorescent markers for fluorescence microscopy. The examples mentioned take advantage of the photostable, near-infrared emission from a large number of NV centres and the study of techniques to enhance their fluorescence is constantly needed. Our study, both experimental and theoretical, focuses on the effect of magnetic field on the fluorescence of the NV centre. Our results demonstrate a non-monotonic trend in the emission that shows an enhancement of the fluorescence at high magnetic field values. With the addition of a phenomenological model we further investigate the photophysical behaviour of the NV centre and explore the implications in magnetic field-driven applications.

3:45 PM C1.02

Variation of Nitrogen Vacancy Center Concentrations in CVD Diamond Epitaxial Lateral Overgrown on Tungsten and HTHP Diamond Substrate Zongchen Liu, Jiao Fu, Zhangcheng Liu, Feng Wen, Wei Wang, Shuwei Fan and Hongxing Wang; Institute of Wide Bandgap Semiconductors, Xi'an Jiaotong University, Xi'an, China.

The negative charged nitrogen vacancy center (NV) in diamond is a promising light source for quantum applications, including quantum information processing and high sensitive detectors, such as magnetometer, thermometer and gyroscopes. The concentration of NV is one of the key factors that influences sensitivity of detector. For instance, the gyroscopes sensitivity can be greatly improved by increasing the NV concentration. Therefore, it is significant to find an approach to increase the NV center concentration in diamond. Here, CVD synthetic system is chosen to grow epitaxial lateral overgrowth (ELO) diamond layer on type Ib HTHP diamond substrate (no NV centers exist), whose surface is patterned by tungsten and, therefore, a higher NV center concentration around the tungsten metal even the HTHP substrate underneath the tungsten is gotten. Both higher NV⁰ and NV⁻ center concentrations are found in ELO diamond layer above the tungsten metal. We find that both NV⁰ and NV⁻ concentrations are increased above the tungsten, and the concentration of NV is much higher than that of NV0 barely exist beneath the tungsten. The higher NV center concentration is caused by the higher vacancy concentration, which could be increased by the existence of tungsten metal in CVD growth process. The details will be presented in the conference.

4:00 PM *C1.03

Engineering Coherent Defects in Diamond <u>Nathalie de Leon</u>; Electrical Engineering, Princeton University, Princeton, New Jersey, United States.

Engineering coherent systems is a central goal of quantum science and quantum information processing. Point defects in diamond known as color centers are a promising physical platform. As atom-like systems, they can exhibit excellent spin coherence and can be manipulated with light. As solid-state defects, they can be produced at high densities and incorporated into scalable devices. Diamond is a uniquely excellent host: it has a large

band gap, can be synthesized with sub-ppb impurity concentrations, and can be isotopically purified to eliminate magnetic noise from nuclear spins. Specifically, the nitrogen vacancy (NV) center has been used to demonstrate basic building blocks of quantum networks and quantum computers, and has been demonstrated to be a highly sensitive, noninvasive magnetic probe capable of resolving the magnetic field of a single electron spin with nanometer spatial resolution. However, realizing the full potential of these systems requires the ability to both understand and manipulate diamond as a material. I will present two recent results that demonstrate how carefully tailoring the diamond host can open new opportunities in quantum science. First, currently-known color centers either exhibit long spin coherence times or efficient, coherent optical transitions, but not both. We have developed new methods to control the diamond Fermi level in order to stabilize a new color center, the neutral charge state of the silicon vacancy (SiV) center. This center exhibits both the excellent optical properties of the negatively charged SiV center and the long spin coherence times of the NV center, making it a promising candidate for applications as a single atom quantum memory for long distance quantum communication. Second, color centers placed close to the diamond surface can have strong interactions with molecules and materials external to the diamond, which makes them promising for nanoscale sensing and imaging. However, uncontrolled surface termination and contamination can degrade the color center properties and give rise to noise that obscures the signal of interest. I will describe our recent efforts to stabilize shallow NV centers within 5 nm of the surface using new surface processing and termination techniques. Specifically, we are able to demonstrate reversible and reproducible control over the top layer of atoms. These highly coherent, shallow NV centers will provide a platform for sensing and imaging down to the scale of single atoms. References [1] B. C. Rose, D. Huang, Z. Zhang, A.M. Tyryshkin, S. Sangtawesin, S. Srinivasan, L. Loudin, M. L. Markham, A. M. Edmonds, D. J. Twitchen, S. A. Lyon, and N. P. de Leon, "Observation of an environmentally insensitive solid state spin defect in diamond," arXiv:1706.01555 [condmat.mtrl-sci] (2017) [2] B. C. Rose, G. Thiering, A.M. Tyryshkin, A. M. Edmonds, M. L. Markham, A. Gali, S. A. Lyon, and N. P. de Leon, "Strongly Anisotropic Spin Relaxation in the Neutral Silicon Vacancy Center in Diamond," arXiv:1710.03196 [quant-ph] (2017)

4:30 PM C1.04

Engineering Spin Defects in <100> and <111> Oriented, Isotopically Controlled Diamond Layers Christian Osterkamp, Priyadharshini Balasubramanian, Thomas Unden, Boris Naydenov and Fedor Jelezko; Institute for Quantum Optics and Center for Integrated Quantum Science and Technology, Ulm University, Ulm, Germany.

The negatively charged nitrogen-vacancy center (NV) in diamond is one of the most promising candidates for the realization of a quantum sensor. The fluorescence of a single NV can be detected and its electron spin can be polarized, read-out and manipulated at ambient conditions. The creation of NVs on demand is an important step towards quantum magnetic- and electric field sensors1. We engineer 15NVs by delta doping during a plasma enhanced chemical vapor deposition (PECVD) process² and we are able to produce an isotopically pure diamond environment by changing the ratio of ¹²C/¹³C atoms in the growth chamber. By polarizing the ¹³C spins in the vicinity of an NV center, a nano nuclear spin ensemble can be created, which is important for the further development of nuclear magnetic resonance spectroscopy3. This effect can even be improved if the NV center is embedded in a 12C environment which is spin free, therefore preserves the coherence of the electron spin and increases the sensitivity⁴. A high nitrogen incorporation rate combined with the controllability of the NV axis alignment makes <111> oriented diamond an interesting material for the creation of ensembles. Shallow ensembles can be used for NV magnetometry and the detection of very small electric and magnetic fields⁵. In our experiments we are able to show perfect NV alignment along the <111> diamond axis with a NV concentration of 20 ppb. Our new modified vacuum system enables us to produce ultrapure diamond with the potential to control the 15NV creation in 12C enriched diamond layers for both orientations <100> and <111>. The study of nitrogen to NV conversion as well as the intrinsic nitrogen content of our isotopically controlled layers are further goals of our research. References 1 C. Müller et al., Nat. Comm., 5 4703 (2014) 2 C. Osterkamp et al., Appl. Phys. Lett. 106, 113109 (2015) ³ T. Unden et al. in preparation (2018) ⁴ H. Watanabe et al., Diamond & Related Materials 20 (2011) 980-982 5 H. Ishiwata et al., Appl. Phys. Lett. 111, 043103 (2017)

^{*} Invited Paper

K2: Nanodiamonds II Session Chair: Shery Chang Monday Afternoon, May 21, 2018 Room: Agassiz/Fremont

3:30 PM *K2.01

Not all Nanodiamonds are Created Equal—The Optical Properties of Nanodiamonds and Applications in Biology Philipp Reineck^{1,2}, Desmond Lau¹, Lindsay Parker⁹, Emma Wilson^{1,2}, Nicholas Nunn³, Leevan Trindade⁵, Cholaphan Deeleepojananan⁷, Marco Capelli^{1,2}, Shery Chang⁸, Takeshi Ohshima⁴, David A. Simpson⁵, Andrew D. Greentree^{1,2}, Brant C. Gibson^{1,2}, Petr Cigler⁶, Olga Shenderova³ and Vadym N. Mochalin⁷; ¹Physics, RMIT University, Melbourne, Victoria, Australia; ²ARC Centre of Excellence for Nanoscale BioPhotonics, Melbourne, Victoria, Australia; ³Adamas Nanotchnologies, Raleigh, North Carolina, United States; ⁴National Institutes for Quantum and Radiological Science and Technology, Takasaki, Japan; ⁵University of Melbourne, Melbourne, Victoria, Australia; ⁶The Czech Academy of Sciences, Prague, Czechia; ⁷Missouri University of Science and Technology, Rolla, Missouri, United States; ⁸Arizona State University, Flagstaff, Arizona, United States; ⁹Macquarie University, Sydney, New South Wales, Australia.

Nanodiamonds have unique physical, chemical and optical properties, making them highly attractive for applications from neuroimaging to tribology. Yet, depending on the synthesis and processing conditions, many properties of different types of nanodiamonds are fundamentally different. This presentation will discuss recent progress in the fabrication and characterisation of both high-pressure high-temperature nanodiamonds and detonation nanodiamonds with a focus on the particles' fluorescence properties.¹⁻³ Several applications of fluorescent nanodiamonds in biology will also be presented. 4,5 1. Reineck et al, Effect of Surface Chemistry on the Fluorescence of Detonation Nanodiamonds, ACS Nano, 2017, 11,10924. 2. Reineck et al, Bright and photostable nitrogen-vacancy fluorescence from unprocessed detonation nanodiamond, Nanoscale, 2017, 9, 497. 3. Reineck et al, Visible to near-IR uorescence from single-digit detonation nanodiamonds: excitation wavelength and pH dependence, Sci. Rep., 2018, 8, 2478. 4. Reineck / Gibson, Near-Infrared Fluorescent Nanomaterials for Bioimaging and Sensing, Adv. Opt. Mater., 2016, 5, 1600446. 5. Reineck et al, Brightness and Photostability of Emerging Red and Near-IR Fluorescent Nanomaterials for Bioimaging, Adv. Opt. Mater., 2016, 4, 1549.

4:00 PM K2.02

Nanodiamond–Gold Core Shell Nanoparticles for Bio-Imaging Applications Yu-Chung Lin^{1, 2}, Elena Perevedentseva^{1, 5}, Zhe-Rui Lin¹, Christoph E. Nebel³, Giorgio Speranza⁴, Artashes Karmenyan¹ and <u>Chia-Liang Cheng</u>¹; ¹Department of Physics, National Dong Hwa University, Hualien, Taiwan; ²Institute of Physics, Academia SInica, Taipei, Taiwan; ³Fraunhofer Institute for Applied Solid State Physics, Trento, Italy; ⁴Plasma Advanced Materials and Surface Engineering CMM – FBK, Trento, Italy; ⁵Lebedev Institute of Physics, Russian Academy of Science, Moscow, Russian Federation.

It has been a new trend to develop multifunctional nanoparticle (NP) for bio-medical applications. The methods to integrate diverse functionalities are developed. One of the promising approaches is synthesis of hybrid NP with synergetic properties. This paper discusses multifunctional applications of core-shell nanodiamond-gold (ND@Au) nanoparticles and demonstrates number of imaging modalities using them. Development of the nanoparticles (NP) for bio and medical applications is in high degree motivated by perspectives of their multifunctional uses. Therefore, theranostic applications can include simultaneously such functionalities as drug delivery or other medical treatments with multimodal imaging, and are now widely developed on the base of nanoparticles of various properties [1]. One of the approaches to obtain the NP with pre-established necessary properties is creation of hybrid structures for a combination of complementary facilities or obtaining synergistic properties [2]. In the present work we consider diamond-based nanostructures formed by SiV-

doped nanodiamond (ND) cores coated with a gold shell [3]. This NP combines abilities and advantages of fluorescent ND and of the metal with plasmonic properties. The silicon impurities in a diamond lattice form Sivacancies or SiV color centers. ND enriched with SiV is considered a promising NP for bio-imaging due to its intense and narrow fluorescence peak located in the near-infrared range, the most convenient for imaging of biological systems. The gold shell of NP provides additional opportunities for bio-imaging [4] and treatment based, in particular, on localized plasmon surface resonance (SPR) and thermal properties of gold. Additionally, both ND and Au nanostructures have been demonstrated as bio-compatible; their surface properties allow functionalization with different methods and different kinds of molecules thus they can be used in drug delivery studies and applications. In the present work we discuss multifunctional applications of ND@Au NP and demonstrate their applications for multimodal imaging, based on the optical properties both ND and gold, via mapping of distribution of spectral and fluorescence lifetime intensities. A number of imaging and sensing modalities for multifunctional ND@Au nanoparticles are demonstrated and add to more facilities for ND@Au bio applications. Particularly, ND@Au nanoparticles can be a convenient agent for applications like photothermal imaging and treatment [7] and for studies involving photoacoustic effect. Reference: [1] G. Bao, S. Mitragotri, S. Tong. Multifunctional Nanoparticles for Drug Delivery and Molecular Imaging. Annu. Rev. Biomed. Eng. 2013, 15, 253-282. [2] B. Shi, X. Du, J. Chen, L. Fu, M. Morsch, A. Lee, Y. Liu, N. Cole, R. Chung, Multifunctional Hybrid Nanoparticles for Traceable Drug Delivery and Intracellular Microenvironment-Controlled Multistage Drug-Release in Neurons, Small 13(20), 2017 [3] L. Minati, C.L. Cheng, Y.C. Lin, J. Hees, G. Lewes-Malandrakis, C.E. Nebel, F. Benetti, C. Migliaresi, G. Speranza, Synthesis of novel nanodiamonds-gold core shell nanoparticles, Diamond & Related Materials 53 (2015) 23-28 [4] I. I. Vlasov, A. A. Shiryaev, T. Rendler, S. Steinert, S.-Y. Lee, D. Antonov, M. Vörös, F. Jelezko, A. V. Fisenko, L. F. Semjonova, J. Biskupek, U.Kaiser, O. I. Lebedev, I. Sildos, P. R. Hemmer, V I. Konov, A. Gali, J. Wrachtrup. Molecular-sized fluorescent nanodiamonds. Nature Nanotechnology 9, 54-58 (2014)

4:15 PM K2.03

Amplified Sensitivity of Nitrogen-Vacancy Spins in Nanodiamonds Using All-Optical Charge Readout <u>David A. Hopper^{1,2}</u>, Richard Grote², Samuel M. Parks² and Lee Bassett²; ¹Physics, University of Pennsylvania, Philadelphia, Pennsylvania, United States; ²Quantum Engineering Laboratory, Electrical and Systems Engineering, University of Pennsylvania, Philadelphia, Pennsylvania, United States.

Nanodiamonds containing nitrogen-vacancy (NV) centers offer a versatile platform for sensing applications spanning from nanomagnetism to in-vivo monitoring of cellular processes. In many cases, however, weak optical signals and poor contrast demand long acquisition times that prevent the measurement of environmental dynamics. Here, we demonstrate the ability to perform fast, high-contrast optical measurements of charge distributions in ensembles of NV centers in nanodiamonds and use the technique to improve the spin readout signal-to-noise ratio through spinto-charge conversion [Hopper et al. arXiv:quant-ph/1709.00465 (2017)]. A study of 38 nanodiamonds with sizes ranging between 20-70 nm, each hosting a small ensemble of NV centers, uncovers complex, multipletimescale dynamics due to radiative and non-radiative ionization and recombination processes. Nonetheless, the NV-containing nanodiamonds universally exhibit charge-dependent photoluminescence contrasts and the potential for enhanced spin readout using spin-to-charge conversion. We use the technique to speed up a T1 relaxometry measurement by a factor of five. These results provide a straightforward method for improving stateof-the-art nanoscale quantum sensors by improving upon current spin readout techniques and contributing additional measurement capabilities. Increased readout fidelity translates to improved sensitivity for nanoscale NMR and electrochemical sensing. NV mediated NMR requires NVs near diamond surfaces and small duty cycle measurements in which the electron and target nuclear spins are allowed to interact. Improving the spin readout SNR will allow for new physical regimes to be probed. In addition to the spin readout, high-contrast charge readout of NVs in nanodiamonds enables fast, direct measurements of electrochemical potential variations. This measurement modality has the capability to allow for pure optical measurements of in-vivo biological signals such as neuron action potentials.

^{*} Invited Paper

4:30 PM K2.04

Developing Nanodiamond Based Spin Relaxometry for Medical and Microbiological Applications Yori Ong, Aryan Morita and Romana Schirhagl; University of Groningen, Groningen, Netherlands.

The optical and magnetic properties of the nitrogen-vacancy (NV) center make fluorescent nanodiamonds (FNDs) highly interesting for applications in biology. Due to their infinite photostability, they can be used as probes that can be tracked for extended durations. Another challenging application is the use of FNDs to extract magnetic information from the environment. In cell biology, magnetic noise originates from free radical molecules that play a key role in normal metabolism, ageing and cell death.Successful development of nanodiamond based magnetic sensing in biology would provide access to information on a time and length scale that is currently unavailable in the life sciences. The endeavor requires a multidisciplinary effort, ranging from physical characterization to biocompatibility studies and instrument development. To this aim, we have performed studies on the physical and chemical properties of nanodiamonds, as well as their interaction with various cell types. Single-center FNDs have been shown to possess angular geometries, which is expected to relate to their interaction with cell walls and intracellular pathways^{1,2}. While they show good biocompatibility with many model systems, such as macrophages, HeLa cells and yeast cells3,4, uptake efficiency among different cell types varies greatly. With respect to bacteria, nanodiamonds in high concentrations show a slight antibacterial effect, dependent on genus and strain, and their respective behavior in colloidal suspension. In accordance with experiments on smaller detonation nanodiamonds, the antibacterial effect of FNDs has been linked to their surface chemistry⁵. The FNDs suitability as a biosensor in microbial research was assessed by evaluating bacterial viability and metabolism under varying levels of exposure to the particles. Furthermore, the clinically relevant biofilm mode of bacterial growth was considered. At the current level of investigation, interfacing or internalization of FNDs for magnetic sensing purposes show no significant adverse effects on the biological systems under study, providing a positive outlook on the development of new applications. References [1] Ong, S.Y. et al. (2017), Phys. Chem. Chem. Phys., 19, 10748-10752 [2] Chu, Z. et al. (2014) Sci. Rep., 4, 4495 [3] Hemelaar, S. et al. (2017), Sci. Rep., 7, 720 [4] Van der Laan, K.J. et al. (2017), Sci. Rep., 7, 5862 [5] Wehling, J. et al. (2014), ACSNano, 8:6, 6475–6483

4:45 PM K2.05

Purification and Characterization of Nanodiamond by Surface Enhanced Raman Scattering Yonhua Tzeng, Yen-Shiang Mao and YenChen Lin; Electrical Engineering, National Cheng Kung University, Tainan, Taiwan.

Surfaces of nanodiamond of 2-5 nm in sizes possess a wide variety of functional groups and non-diamond carbon phases. For many applications, it is desirable to begin further processing of nanodiamond from a purer and mostly sp3 bonded diamond phase. Therefore, removal of non sp3 bonded carbon phases and functional groups on surfaces of nanodiamond becomes necessary. In this work, nanodiamond is subjected to wet chemical etching, thermal treatment in hydrogen, and dry etching by hydrogen plasma. Raman scattering is a convenient means of characterizing diamond and nanocarbon phases. By the use of surface enhanced Raman scattering (SERS) substrates made of combined graphene and silver nanostructures which have been shown to exhibit Raman scattering signal enhancement factor as high as 10E14 times for R6G molecules, as received nanodiamond and purified nanodiamond are characterized to reveal the evolution of nanodiamond towards a purer diamond phase after nanodiamond surfaces are etched by selected wet chemical, thermal, and plasma etching. Effects of post-etching diamond CVD processes on the purified nanodiamond will also be discussed.

EP1: Innovation and Entrepreneurship Session I Session Chair: Jeffrey T. Glass Monday Afternoon, May 21, 2018 Room: Humphreys

5:00 PM *EP1.01

P-Type Diamond Epitaxial Layers Growth for Power Electronics, Ready for Industry with DiamFab <u>Gauthier Chicot</u>^{1,2}, David Eon^{1,2}, Etienne Gheeraert^{1,2} and Julien Pernot^{1,2}; ¹DiamFab, Grenoble, France; ²Univ. Grenoble Alpes, Grenoble INP, Institut Néel, Grenoble, France.

Nowadays power electronics is everywhere, inside your smartphone, your car and in every nodes of the energy distribution grid. Highest efficiency, reliability and space saving are required for such devices. Silicon has been widely used in power electronics but shows now its limitation to follow the trend imposed by power electronics industry. Like so, wide bandgap semiconductors will gradually replace silicon. Among them, diamond thanks to superlative properties is considered as the ultimate semiconductor. As part of all the industrial processes needed to fabricate a diamond device, the epitaxial layer growth is one of the most critical since most of the electrical performances will depend of the quality of these active layers. Push by mechanical application and jewelry, substrates are commercially available and the device fabrication process relies on traditional process. Conversely, the diamond epitaxy step is highly specific and requires strong experiences in diamond growth. By working on epitaxial diamond growth for more than 20 years, we have acquired a unique know how. We are able to grow diamond layers with an accurate doping and thickness control. This allows us to cover a wide range of boron doping level going from almost insulating material (1014 cm⁻³) to metallic conduction diamond (> 5x10²⁰ cm⁻³). This allows managing the stack of different layers necessary to fabricate unipolar devices. Thanks to a well-controlled growth rate original doping profile are also possible. For instance, very abrupt transition from low doping to metallic boron concentration has been demonstrated for diamond delta doped layer fabrication [1]. But to take all the best of diamond material a particular intention has to been paid to the design. Thanks to our experience in diamond devices fabrication we are able to determine the best layer design to reach the best performances [2]. Finally, as a guarantee of quality, diamond Schottky diodes that are the state of the art were fabricated on our diamond epitaxial layers [3]. For all these reasons we are creating DiamFab company to provide electronic grade diamond epitaxial layers to anyone who intends to fabricate diamond power devices. References [1] Chicot el al., J. Appl. Phys. 116, 083702 (2014). [2] Chicot et al., Diam. Relat. Mater., 69, 68 (2016). [3] Traore et al., Appl. Phys. Lett., 104, 052105 (2014).

^{*} Invited Paper

PS1: Poster Session I Monday Afternoon, May 21, 2018 5:30 PM - 7:30 PM Room: Doyle/Rees

PS1.01

Chemical and Structural Properties of RF (N₂) Plasma Treated Diamond (100) Surfaces Mohammed Attrash, Mohan Kumar Kuntumalla and Alon Hoffman; Schulich Faculty of Chemistry, Technion – Israel Institute of Technology, Haifa, Israel.

Diamond surface crystal structure quality and chemical composition play a vital role for many applications, such as activation and properties of NV centers emission in the near surface of diamond. Nitrogen terminated diamond (N-terminated diamond) can be fabricated by exposing diamond to N, radio frequency (RF) plasma. In this work, we investigate the interaction of N, RF plasma onto single crystal diamond (100). To minimize surface damage the plasma conditions were optimized such as flow rate, and grounded mesh electrode placed at the plasma source exit, to prevent interaction between ions and the diamond surface. Optical emission spectroscopy was used to characterize the species present in the plasma. Thermal stability and surface structure of N-terminated diamond were investigated in-situ by using x-ray photoelectron spectroscopy and low energy electron diffraction. HCN and N₂ desorption from N-terminated diamond was investigated by using temperature-programed desorption (TPD). Optical emission spectroscopy showed that increasing flow rate can decrease the ratio of charged-to-neutral nitrogen particles. TPD spectra showed first order reaction, N2 spectra had two desorption peaks with maximum at 180 C and 610 C, while the CN spectra had desorption peak at 600 C.

PS1.02

Characterization of Single Crystal Diamond Grown on Different Off-Cut Angles from the (100) Crystallographic Plane Ayan Bhattacharya¹, Ramon Diaz¹, Aaron Hardy² and Timothy A. Grotjohn^{1,2}; ¹Electrical and Computer Engineering, Michigan State University, East Lansing, Michigan, United States; ²Fraunhofer USA Center for Coatings and Diamond Technologies, East Lansing, Michigan, United States.

Single crystal diamond (SCD), that is grown by chemical vapor deposition (CVD) technique, is a material of great scientific and technological interest due to its exceptional mechanical, thermal, optical and electronic properties. However, applications of SCD for electronic devices ask for minimal nitrogen impurities to be present in the substrate, otherwise charge trapping, charge transport and electric field breakdown properties will be affected. This is one of the prime criterion for growth of SCD for high energy particle detector application[1]. But such a growth process in a very low nitrogen environment comes with its cost, which can be undesirable surface morphologies and dislocation defects that reduce the quality of the grown layer and therefore making it less suitable for any detector and electronics applications. Also the growth rates become slow for SCD in low nitrogen impurity environment and adds to an additional challenge to grow thick layers (more than 100 µm). An alternative way is to grow diamond on a surface with an off-cut from the (100) crystallographic plane to reduce defects originating from the top surface[2] during deposition. In this work, we have looked into growth strategies on 2.5° , 5° and 10° offcut substrates from the (100) crystal plane and along the [100] direction. Both bevel cut substrates and parallel plate substrates with offcuts were used. All depositions were conducted in a 2.45 GHz a microwave plasma assisted CVD reactor[3] using hydrogen and methane feedgas mixtures. Typical durations for depositions were 2.5, 12 and 24 hours. The end-product grown layer had thickness ranging from 30 μm to 250 µm. The major focus of these study was to examine the grown in terms of terrace and step formation and the spatial distribution of steps and terraces on the overall surface with respect to offcut angle and growth duration. Any appearance of pyramidal features, the evolution of the top growth surface with time, and the growth rate variation versus the position of the top surface as the top surface evolves with time were studied in

detail. We have also looked into grown layers with the offcut being along [110] direction. The quality of the grown layers (in terms of nitrogen impurities) have also been studied by photoluminescence. An overall relationship between offcut angle, step heights and terrace width distribution, and impurity distribution will be presented. The authors would like to acknowledge the fund on NSF grant 1628958. 1) A. Bhattacharya, T. A. Grotjohn and A. Stolz, Degradation of single crystal diamond detectors in swift heavy ion beams, Diamond and Related Materials (2016) 70, 124-131. 2) O. Maida, H. Miyatake, T. Teraji, T. Ito, Characterization of substrate off-angle effects for high-quality homoepitaxial CVD diamond films, Diamond and Related Materials (2008) 17, 435-439. 3) J. Lu, Y. Gu, T.A. Grotjohn, T. Schuelke, J. Asmussen, Experimentally defining the safe and efficient, high pressure microwave plasma assisted CVD operating regime for single crystal diamond synthesis. Diamond and Related Materials (2013) 37, 17–28.

PS1.03

Cubic Boron Nitride on Semiconducting Diamond and Its Application to Neutron Detection <u>Jesse Brown</u>, Yu Yang, Joseph Shammas, Franz Koeck and Robert J. Nemanich; Physics, Arizona State University, Tempe, Arizona, United States.

We have successfully deposited cubic boron nitride (c-BN) on nitrogen doped, polycrystalline diamond and boron doped, single crystal diamond, using plasma enhanced chemical vapor deposition (PECVD) employing fluorine chemistry. In situ X-ray photo spectroscopy (XPS) was utilized to characterize the sp³ bonding structure of c-BN. Cubic boron nitride is the zinc-blende analog to diamond, which is now being considered as an ideal material for radiation detection. TEM measurements demonstrate local epitaxy for c-BN on polycrystalline diamond. Boron containing materials are particularly relevant to detecting neutrons because of their large cross section to neutron capture and alpha particle emission. Moreover, diamond and c-BN have similar lattice constant and optical band gap which enables an integrated detector design. We present an approach to deposit c-BN on the p-type layer of a PIN diamond device in order to create a neutron detector. With the similar structural and outstanding thermal properties of c-BN and diamond, these devices can be used in a variety of applications, including high temperature environments.

PS1.04

Increasing the Area of Single Crystal Diamond Plates Grown by a Combination of Mosaic Technique and Lateral Growth Ramon D. Diaz¹, Aaron Hardy² and Timothy A. Grotjohn^{1, 2}; ¹Electrical and Computer Engineering, Michigan State University, East Lansing, Michigan, United States; ²Fraunhofer USA Center for Coatings and Diamond Technologies, East Lansing, Michigan, United States.

The electrical, mechanical, and thermal properties of diamond make it a promising material for new generation electronic devices. One of the challenges is the need for fabricating larger size Single Crystal Diamond (SCD) wafers with minimal defects. In this study, SCD plates were successfully grown based on the mosaic technique combined with conditions known for enhanced lateral growth. In earlier works [1] the plates were tiled together using substrates created by a process consisting of ion implantation followed by growth and electrochemical separation. The approach in this work is different in that a starting substrate is cut in two, then each of the two pieces are grown larger laterally followed by a growth that stitches them back together with a larger area. A series of steps were taken to ensure good crystallographical orientation, starting with laser cutting the tiles from the same 3.5mmx3.5mmx1.4mm HPHT (High Pressure High Temperature) seed. The original area of the two substrates was increased by growing a layer of diamond via MPACVD (Microwave Plasma-Assisted Chemical Vapor Deposition) using a pocket configuration which favors lateral growth [2][3]. The grown tiles are polished and kept in aligned contact by their base with a polycrystalline diamond plate used as reference on the holder during growth. This step produced a continuous layer diamond across the two pieces. The grown together substrate was then re-polished and re-grown multiple times by MPACVD process until the contact (stitched region) interface disappeared, effectively merging the tiles back into a single crystal. Off-plane variations of the initial cut and their effect were explored. The defect densities from the top $500-600 \mu m$ grown layer from each step were analyzed by etch pit counting and birefringence measurements. This process shows the feasibility of combining known lateral growth techniques for expanding the area of

^{*} Invited Paper

SCD wafers. The authors would like to acknowledge funding for this work from MIT Lincoln Laboratory. [1] Yamada, H., A. Chayahara, Y. Mokuno, Y. Kato, and S. Shikata. "A 2-in. Mosaic Wafer Made of a Single-Crystal Diamond." *Applied Physics Letters* 104, no. 10 (2014): 102110. https://doi.org/10.1063/1.4868720. [2] S. Nad, A. Charris, and J. Asmussen. "MPACVD Growth of Single Crystalline Diamond Substrates with PCD Rimless and Expanding Surfaces." Applied Physics Letters 109, no. 16 (2016): 162103. https://doi.org/10.1063/1.4965025. [3] A. Charris, Toward the Rapid Growth of High-Quality, Polycrystalline Rimless, and Large Area Single Crystal Diamond Substrates. Michigan State University, 2017.

P\$1.05

Relation of Activated Gas Phase Chemistry and Substrate Temperature on the Microstructural Properties of Diamond Films Deposited from CH₄/NH₃/H₂ Gas Mixture Sergey Elfimchev, Yulia Stotsky, Rozalia Akhvlediani and Alon Hoffman; Schulich Faculty of Chemistry, Technion-Israel Institute of Technology, Haifa, Israel.

In this work we investigated some aspects of diamond growth from ammonia, methane and hydrogen precursors by hot filament CVD (HFCVD) method. The samples were deposited from different NH₃/CH₄/ H₂ gas mixtures, pressures and different surface temperatures. The obtained polycrystalline diamond films were investigated using HRSEM, Raman and XPS spectroscopies. The structural evolution from ultra nanocrystalline diamond (UNCD) films to sub-microcrystalline structure was associated with increasing the ammonia concentration and substrate temperature. According to the SIMS measurements, it was found that nitrogen concentrations strongly depend on NH₂/CH₄ ratio and substrate temperature. The impact of total pressure and filament temperature during the diamond deposition on film morphology and composition is also discussed in this paper. We investigated the gas phase composition in-situ during the diamond deposition process by mass spectrometry. We examined the gas phase chemistry using deuterium gas (D₂) and deuterated methane (CD₄). Based on the obtained results the diamond growth mechanism from ammonia precursor in the HFCVD was proposed. It is suggested that by controlling the deposition parameters from NH₂/CH₄/H₂ gas mixtures the diamond grain size and nitrogen concentration can be effectively tuned.

PS1.06

Improving the CVD Growth Duration and Quality of Thick Heavily Boron Doped Diamond Epitaxial Layers Aaron Hardy¹, Robert B. Rechenberg¹, Michael Becker¹, Timothy A. Grotjohn^{1, 2} and Thomas Schuelke^{1, 2}; ¹Center for Coatings and Diamond Technologies, Fraunhofer USA, East Lansing, Michigan, United States; ²Electrical and Computer Engineering, Michigan State University, East Lansing, Michigan, United States.

As a material for high power electronic devices, single crystal diamond has the potential to outperform other materials in terms of power density and heat dissipation. In order for these devices to be realized, high quality, heavily boron doped epitaxial layers (p⁺ layers) must be synthesized faster and more reliably. This study presents working solutions to a number of problems commonly reported in the synthesis of p+ diamond via microwave plasma assisted chemical vapor deposition (MPACVD), including low growth rate, non-epitaxial crystallite formation, and premature reactor shutdown due to soot formation. In this study, a 2.45GHz tunable cavity type MPACVD reactor is used. Depositions are done on a thermally floating sample stage, because the higher power required to reach deposition temperature with an actively cooled sample stage results in unnecessary heating of the quartz dome. The high concentration of dopant gas (>2000ppm B/C) needed to get sufficient boron incorporation in the diamond $(10^{19} - 10^{20} \text{ cm}^{-3})$ often causes unwanted soot formation around the plasma. Eventually this soot coats the quartz dome to the point that the plasma becomes unstable and the process must be terminated. Unnecessary heating of the system expedites this process. Therefore, in order to get long deposition times, it is best to use the minimum power required to operate the plasma such that the substrate reaches the process temperature. In order to achieve adequate plasma power density at relatively low forward power (1.3 – 1.5 kW), process pressure was increased from 240 to 300 Torr. Other techniques employed include the addition of CO₂ to the process gasses in order to etch away crystallites which may pop off of the sample holder and land on the diamond during deposition [1]. Additionally, switching from (100) oriented substrates to

those cut at an angle of 3° off-axis from the (100) plane in the [010] direction results in higher boron incorporation into the epitaxial layer, and smoother surface morphology after deposition. These key improvements result in higher conductivity p* layers, and up to 400% increase in run time. Previously, unwanted shutdowns due to soot formation regularly occurred after less than 12 hours of deposition, and the p* layers produced had room temperature resistivities between 1×10^{-2} and 9×10^{-2} Ωcm as measured by four-point probe. In comparison, the revised process has successfully been sustained for 47 hours, resulting in a 300 μm thick p* layer with a room temperature resistivity of 2.2×10^{-2} Ωcm . Acknowledgement: The authors would like to thank the US Dept. of Energy ARPA-E grant no. DE-AR0000455 for funding. **References** [1] Teraji, Tokuyuki., 2015, Journal of Applied Physics 118, 115304, p.1-8

PS1.07

Morphological and Surface Properties of Diamond Thin Films Deposited at Low Substrate Temperatures Mohan Kumar Kuntumalla, Miriam Fischer, Rozalia Akhvlediani and Alon Hoffman; Schulich Faculty of Chemistry, Technion - Israel Institute of Technology, Haifa, Israel.

In this work, polycrystalline diamond thin films were deposited on Si (100) substrates by using CO₂ (with and without) gas in the CH₄/H₂ gas mixture by hot-filament CVD in the 200 to 720 °C back substrate -surface temperature range by force-cooling. An exhaustive temperature calibration has been carried out with outmost care to measure the films' top-surface temperature during the diamond deposition. It was found that for silicon substrate (thickness 500 μm) the temperature difference (ΔT)between the back substrate and the top film surface was in the range 200 to 450 °C. Scanning electron microscopy was used to study the surface morphology of the grown films. Raman spectroscopy was used to determine the diamond and non-diamond phases whereas XPS and HREELS were utilized to investigate the diamond films' surface. It was observed that the substrate top-surface temperature plays a crucial role on the diamond nucleation and growth process. A low substrate temperature results in formation of diamond films having nanometer sized granular features in comparison to the commonly anticipated columnar growth at higher temperatures. The growth-rate is largely reduced with temperature decrease due to low mobility of active species on the evolving surface. Our results are important for the quest to deposit diamond films at low substrate temperatures for electronic and other applications.

PS1.08

The Interface and Mechanical Properties of CVD Single-Crystal Diamond by Multilayer Epitaxial Growth Yun Zhao, Chengming Li, Kang An, Xiongbo Yan, Lifu Hei, Jinlong Liu, Liangxian Chen, Junjun Wei and Fanxiu Lu; Institute for Advanced Materials and Technology, University of Science and Technology Beijing, Beijing, China.

The single-crystal diamond homoepitaxial layers with multilayer are synthesized by changing nitrogen input on the substrate of high pressure high temperature (HPHT) Ib synthetic diamond substrate using microwave plasma chemical vapor deposition (MPCVD) method. The fluorescence and phosphorescence characteristics of the cross section of the CVD layers grown on the HPHT seed crystal were investigated by DiamondViewTM instrument. The stress and impurity distribution at the interface were measured by Raman and infrared spectroscopy. The characteristics of internal and surface defects of the HPHT Ib seed crystals were observed by X-ray topography. The formation of dislocations at the interface and the inheritance of dislocations between the seed and the CVD layer are discussed. The dislocation density of the HPHT Ib seed and CVD homoepitaxical layers after each growth were gained by X-ray rocking curves measurement, and it is found that the dislocation density increases almost 4 times after double homoepitaxial growth. The hardness and fracture toughness of different CVD layers are measured by indentation test. The relationship between the change of dislocation density and the mechanical properties are investigated after multiple growth of single crystal diamond. The hardness and fracture toughness of multilayer growth layer are increase trend. It can be concluded that single crystal diamond with the multiple stratification characteristics will improve the wear resistance of single crystal diamond cutter.

PS1.09

Selective Growth of Phosphorous Doped Single Crystalline Diamond on (100) Oriented Diamond Substrates Robert B. Rechenberg¹, Michael Becker¹, Aaron Hardy¹, Matthias Muehle¹, Stephen Zajac², Timothy A. Grotjohn^{2, 1} and Thomas Schuelke^{1, 2}; ¹Center for Coatings and Diamond Technologies, Fraunhofer USA, East Lansing, Michigan, United States; ²Electrical and Computer Engineering, Michigan State University, East Lansing, Michigan, United States.

Diamond has unique properties which make it an excellent candidate for high frequency and high-power electronics. Its thermal conductivity, high breakdown voltage, and wide bandgap enable applications with higher power densities and greater efficiency. To enable those diamond properties for applications such has high power diodes, a high-quality diamond growth is needed and a fabrication process needs to be developed. One processing step that is desired for electronic device fabrication is a selective growth of n-type diamond on undoped or p-type doped diamond. Such n-type regions can serve as the n-type regions in pn and pin diodes and as heavily doped regions for contacts to n-type (100) surface diamond. It is common to fabricate diamond electronic structures on the (100) surface. So far, the fabrication of n-type diamond with high doping concentrations on (100) surfaces has proven difficult due to several factors. Phosphorous for example has a higher incorporation efficiency into the (111) lattice orientation and can reach a doping level of 10²⁰ cm⁻¹ versus 10¹⁸ cm⁻¹ for (100) [1]. Whereas boron doped diamond has been successfully grown with high doping concentrations of 10²⁰ cm⁻¹ on (100) substrates [2]. The very limited availability of (111) oriented diamond substrates and the more difficult processing does not make it a good candidate as a substrate. Thus, a process was developed to selectively grow highly phosphorous doped diamond on boron doped diamond using (100) oriented substrates. The selective doping process consist of starting with a boron doped or undoped diamond layer on a substrate. In order to achieve high phosphorus doping concentrations, a process was developed to etch either (111) or (113) facets into SCD. A thin layer of nickel was deposited and patterned using a lift-off process. Depending on the feature dimensions, patterning was either done using e-beam or conventional photolithography. Afterwards, the Ni coated substrate was heated to about 900C in a hydrogen atmosphere for 1-2 hours which then formed pyramidlike etching structures with open facets. Afterwards, phosphorous doped diamond was deposited and resulted in selective growth onto the (111) surfaces but not the (100) surfaces. To characterize the diode regions, contacts were deposited onto the boron and phosphorous doped diamond surfaces and the I-V curves were measured. The I-V curves show pn junction characteristics. References [1] Kato, H., Makino, T., Yamasaki, S., & Okushi, H. (2007). N-Type Diamond Growth By Phosphorus Doping on (001) -Oriented Surface. Journal of Physics D: Applied Physics [2] Demlow, S. N., Rechenberg, R., & Grotjohn, T. (2014). The effect of substrate temperature and growth rate on the doping efficiency of single crystal boron doped diamond. Diamond and Related Materials

PS1.10

XANES and EXAFS Study of P Doped Diamond Shinichi Shikata¹, Koji Yamaguchi¹, Akihiko Fujiwara¹, Yusuke Tamenori², Kazuki Tsuruta², Takatoshi Yamada³, Shannon S. Nicley⁵, Ken Haenen⁵ and Satoshi Koizumi⁴; ¹School of Science and Technology, Kwansei Gakuin University, Sanda, Japan; ²Japan Synchrotron Radiation Research Institute, Sayo, Japan; ³AIST, Tsukuba, Japan; ⁴NIMS, Tsukuba, Japan; ⁵Hasselt University, Diepenbeek, Belgium.

n type drift layer with high electron mobility and highly doped contact layer is highly expected for the power device application of diamond for energy saving electric systems. However, n type P doped diamond is still under development looking for high concentration and high mobility [1]. The structural analysis of P atom in diamond cell in conjunction with hydrogen and dangling bond is important to realize higher doping concentration and high mobility [2,3]. In the previous report [4], the structural analysis using X-ray absorption fine structure (XAFS) by synchrotron radiation (SR) X-ray was carried out for the P doped (111) diamond films, and the results were compared with those from simulations. XAFS measurements were carried out at the BL27SU beamline of SPring-8 SR facility. The P positions in the interstitial sites additional to the substitutional site were confirmed by both X-ray Absorption Near Edge Structure (XANES) and Extended X-ray Absorption Fine Structure (EXAFS) measurement. In this paper, various

types of samples grown in three research institutes are measured and analysed by both XANES and EXAFS. The information of samples is as follows;a) (111) 2.0um, P conc.:3E20, H2:unknown, (AIST) 2009b) (111) 2.78um, P conc.:4.1E19, H2: 3.2E18, (NIMS) 2014c) (111) 6.5um, P conc.:3.5E19, H2: 4E19, (NIMS) 2015d) (111) 25um, P/C conc.(reactor phase):5000ppm, H2: unknown, (Hasselt Univ.) 2016 By the XANES measurement, following results were observed. 1) b) and c) showed higher intensities for 2148eV peak compared to specimen a); the peak corresponds to substitutional site. 2) A peak near 2147eV is divided into two peaks for specimen b) and c). Higher intensity of the right-hand peak is observed for sample c) which contains high H2 concentration. This corresponds to substitutional site with H compared with the model simulation using conventional software named "Athena". 3) Due to the lower doping concentration, the signal of d) spectrum was very weak. It showed very different one from a) b) and c) with very broad peak at 2147eV and peak around 2050eV disappeared. It showed presumably combined peak of substitutional site, substitutional site with H and partly interstitial site with H. EXAFS measurements are carried out for a) b) and c). The position of the first and second peak observed for b) and c) were both closer to the substitutional site of the simulated results, compared to sample a). The dopant site associated with the neighbouring structure including H and dangling bonds might depend highly on the growth conditions as well as the machine design. Ref. [1]S.Koizumi et al., Chap.9 Physics and Applications of CVD Diamond (2008) [2]M.Katagiri et al., APL85(04)6365 [3]H.Okazaki et al., APL 98(11)082107 [4]S.Shikata et al., Applied Physics Letters, 110 (2017) 072106

PS1.11

Single-Crystal Diamond Growth with Nitrogen and Oxygen Alternate Addition by Microwave Plasma CVD Yan-Feng Wang, Xiaohui Chang, Wei Wang, Dan Zhao, Jiao Fu, Zongchen Liu, Guoqing Shao, Zhangcheng Liu, Shuwei Fan, Renan Bu, Jingwen Zhang and Hongxing Wang; Xi'an Jiaotong University, Xi'an, China.

Diamond film collects all the outstanding properties of mechanics, electronics, heat, and optics etc. together making it have potential applications in the fields of wide range optical transparent window material, super-hard coating tolls, and electron devices. 1,2 The microwave plasma chemical vapour deposition (MPCVD) method is one of the most promising technologies for producing single-crystal diamond. In order to obtain high quality diamond, oxygen is often added into MPCVD chamber, because oxygen can etch graphite on diamond surface during diamond growth. However, the rate of diamond growth with oxygen addition is lower than arc-jet methods, etc. In order to obtain high-rate diamond growth, nitrogen is often added into MPCVD chamber. However, this method can deteriorate the diamond quality because of the nitrogen doping. Therefore, in order to obtain diamond with high quality and highrate, nitrogen and oxygen are alternately added into MPCVD chamber during diamond growth. In this work, investigation of nitrogen and oxygen alternate addition during diamond growth by MPCVD has been carried out. HPHT (001) Ib single crystal diamonds in dimension of 3×3×0.5 mm³ were used as substrates. The growth of diamond films was carried out in a conventional 5-kW, 2.54-GHz MPCVD system (AX-6500, Sekitechnotron) using gas mixtures of hydrogen, methane, oxygen and nitrogen. The growth conditions were summarized in Table 1. The way of nitrogen and oxygen alternate addition was shown in figure 1. References 1.J. lsberg, J.Hammersberg et al. 'High carrier mobility in single-crystal plasma-deposited diamond' DOI:10.1126/science.1074374, Science. 2.R.F. Davis, Z. Sitar et al. 'Carter Critical evaluation of the status of the areas for future research regarding the wide band gap semiconductors diamond, gallium nitride and silicon carbide' DOI:10.1016/0921-5107(88)90032-3, Materials Science and Engineering:B.

PS1.12

Epitaxial Growth of Ir Film on MgO (001) Substrate at Low Temperature by Sputtering Kongting Wei, Kang Wang, Fei Li, Jiao Fu, Guoqing Shao, Yan-Feng Wang, Shengli Wu and Hongxing Wang; Institute of Wide Bandgap Semiconductors, Xi'an Jiaotong University, Xi'an, China.

Ir can be used for the metal electrode material, and as a buffer layer to improve the crystalline quality of the oxide film, and Ir is the unique substrate material for heteroepitaxial growth of large area single crystal diamond. Ir film has been deposited epitaxially on various oxide

substrates. The previously reported epitaxial deposition of Ir film on MgO(001) substrate mainly uses magnetron sputtering or electron beam evaporation or pulsed laser deposition at high temperatures, usually greater than 650 °C.An extended structure zone diagram including energetic deposition proposed by André Anders proves the possibility of epitaxial metal thin films at lower temperatures. In this article, we describe a new deposition method that can be used to prepare a good epitaxial Ir film at a lower deposition temperature on MgO(001) substrate. Using a bias RF sputtering deposition method, a seed layer was first formed on a MgO (001) substrate material, and then an epitaxial metal Ir film was prepared using conventional sputtering deposition from room temperature to 600 °C. The Ir(001) preferred orientation was studied by XRD, the epitaxial orientation was confirmed by EBSD, the orientation of Ir is parallel to the substrate orientation, meets the relationship Ir(001)// MgO(001), and the surface morphology was studied by atomic force microscopy. The details will be presented on the conference.

PS1.13 CANCELLED

Etching Rates of Nitrogen-Incorporated Ultrananocrystalline Diamond Thin Film in Hydrogen and Oxygen Plasmas Measured by an Interferometry Method

PS1.14

Ion-Induced Surface Pattern Formation on Diamond-Like Carbon Films <u>Hans Hofsaess</u>¹, Omar Bobes¹ and Kun Zhang²; ¹Faculty of Physics, 2nd Institute of Physics, Göttingen, Germany; ²Physics, Sichuan University, Chendgu, China.

Ion induced surface pattern formation is a well-studied subject since several decades. The major mechanisms leading to periodic surface nanopatterns were curvature dependent ion erosion (Bradley-Harper Theory) and curvature dependent mass redistribution (Carter-Vishnyakov effect). During the last years several novel mechanisms and theoretical models have been developed and proposed, including the role of surface impurities or surfactants, the role of ion implantation, phase separation, hydrodynamic models and the generation and relaxation of stress. For several reasons diamond-like amorphous carbon thin films appear as an ideal model case to investigate these recently proposed effects. The films are by definition amorphous, so that crystal structure effects are absent, and exhibit an almost atomically flat ultra-smooth surface. Depending on the DLC film preparation conditions, the initial existing compressive film stress can be tuned. Carbon ions are readily available as mass selected ions beams and allow the investigation of pattern formation due to implantation of self ions. Irradiation with e.g. B+, N+ or F+ ions allows the study of pattern formation for amorphous binary compounds. Moreover, there is a strong tendency of DLC to accommodate significant amounts of implanted noble gas ions in form of small bubbles exhibiting liquid noble gas density at extremely high pressure. Therefore, ion implantation of noble gas ions also appears as a significant contribution to the evolution of surface patterns. In this contribution, we present experiments on ion induced pattern formation on tetrahedral amorphous carbon films on Si substrates using ion irradiation of different ion species at energies ranging from few hundred eV up to several 10 keV. We compare our results with the predictions of different theoretical models using parameters determined from Monte Carlo simulations of ion solid interaction.

PS1.15

Synthesis of Hydrophobic Micro and Nanostructures Based on Metal Oxides, Carbon Nanotubes and Polymer Wastes for the Separation of Oil-Water Mixtures Brenda I. Gomez, Maximo Oliva, Cynthia E. Ibarra Torres, Thelma Serrano, Yolanda Peña, Oxana V. Kharissova and Boris Kharisov; Universidad Autónoma de Nuevo León, Monterrey, Mexico.

Petroleum hydrocarbons are the most important and used energy resources in the world; however, they are the main environmental pollutants. One of the biggest petroleum pollution is caused by petroleum spills [1-2]. Spills occur mainly in the open sea due to accidents or improper practices during the exploration and extraction of the material, which affect the entire ecosystem when the event occurs [3]. For the remediation of the places where spills occur, various classic techniques are used, such as containment barriers, oil skimmers, chemical dispersants, controlled burning, manual cleaning, etc. [4] which serve to remove most of the oil spills and clean the contaminated place. However, novel alternatives to solve this problem are in progress due to the fast development of nanotechnology. The use of several oxides as adsorbent materials for hydrocarbons has certain advantages over conventional materials, such as, for example, larger surface area or greater adsorption capacity [5]. In addition, they can be modified chemically or physically to be oleophilic and hydrophobic, require less storage space and can be reusable. Materials on their basis can be used in the remediation of oil spills, wastewater treatment, separation of hydrocarbon mixtures and different types of oil in water [6]. In this work, the use of different composites based on metal oxides, mainly of iron and nickel, in combination with carbon nanotubes and silica nanoparticles, is proposed. For the oil adsorption, the composites were synthesized by solid-state reactions and self-combustion of precursors. We selected these materials, since they have been proved to be easy coupled in batch type reactors; also, due to their magnetic properties, some of these composites allow their easy removal from the water. The capacity of adsorption was determined for hematite composites / nickel oxide and maghemite / carbon nanotubes. Both systems were characterized by techniques such as SEM and XRD and adsorption tests, performed using crude oil. In addition, absorbent materials were prepared from polymer wastes (recycled polyethylene terephthalate, PET, from bottles) using salt templates and hydrophobized using carbon nanotubes and silica nanoparticles. Their application for oil removal showed enhanced capacity, efficiency and reusability. References [1] Novaki, L.P., Moraes, E. O., Goncalves, A.B. et al (2016). Solvatochromic and Solubility Parameters of Solvents: Equivalence of the Scales and Application to Probe the Solubilization of Asphaltenes. Energy & Fuels, 30(6), 4644-4655 [2] Rogel, E., Miao, T., Vien, J., & Roye, M. (2015). Comparing asphaltenes: Deposit versus crude oil. Fuel, 147, 155-160. [3] (2005). Manual sobre la contaminación ocasionada por hidrocarburos. IMO, Sección IV, 236 pp. [4] Silva, R.F. et al. (2014). Applications of Biosurfactants in the Petroleum Industry and the Remediation of Oil Spills. International Journal of Molecular Sciences, 15, 12523-12542. [5] Yang, J., Song, H., Yan, X., Tang, H., & Li, C. (2014). Superhydrophilic and superoleophobic chitosan-based nanocomposite coatings for oil/water separation. *Science+Business Media, 21*, 1851-1857. [6] Saber, O., Mohmed, N., & Arafat, S. (2015). Conversion of iron oxide nanosheets to advanced magnetic nanocomposites for oil spill removal. RSC Advances, 5, 72863-72871.

PS1.16

Needle-Like Structure of Ultrananocrystalline Diamond Films for UV Detector Applications Chi-Wen Liu¹, Yin-Tung Albert Sun¹ and Chii-Ruey Lin²; ¹Graduate Institute of Manufacturing Technology, National Taipei University of Technology, Taipei, Taiwan; ²Department of Mechanical Engineering and Institute of Precision Mechatronics Engineering, Hsinchu, Taiwan.

This research work focused on the fabrication of nitrogen-incorporated ultra-nanocrystalline diamond (N-UNCD) films and their characteristics including surface morphology, microstructure, electrical property and photoconductivity aiming at applications in UV detector detection. Needle-like N-UNCD were synthesized using a home-made microwave plasma jet chemical vapor deposition (MPJCVD) system with CH4/H2/ Ar/N2 as gaseous precursors, and then Au interdigital electrodes were deposited onto the surface of the as-grown N-UNCD films by the sputtering technique. Annealing procedures were conducted at various temperatures to obtain Ohmic contact of N-UNCD/Au structure. It was found that the N-UNCD/Au structure annealed at 500 °C exhibited a good ohmic contact characteristic and high photodetection efficiency as well as rapid response to UV irradiation in air ambient with the high photo-dark current ratio about 3.1 orders of magnitudes. The achievement of our current study indeed opens up a prospect of photodetector applications of needle-like N-UNCD films in the future.

PS1.17

Magnetic Properties of Thermal and Microwave-Plasma CVD Deposited Fe-Filled Carbon Nanotubes—Comparative Study for Magnetic Devices Sekhar C. Ray; Physics, University of South Africa, Johannesburg, South Africa.

Fe-filled Carbon nanotube (CNT) arrays are fabricated using thermal and microwave-plasma chemical vapour deposition (CVD) methods. SEM and TEM characterizations showed that the well-aligned CNTs were 80-500 μm in length, with outer (inner) diameter of 20-50 (10-20) nm. The CNTs grown by thermal CVD shows higher magnetization than those grown by microwave-plasma CVD, which may be ascribed to the formation of metal-oxides in the CNT structures. Out of all samples, thermal CVD grown Fe-filled CNTs showed maximum magnetic coercivity ≈ 732 Oe and this value is higher than that of bulk iron. X-ray diffraction and Raman spectroscopy results are correlated with magnetic behaviours. The high coercivity and the structure of single-domain Fe-nanoparticles isolated by antiferromagnetic CNTs make it a promising candidate for high-density magnetic recording media.¹

PS1.18

Magnetic Properties of Metal-Free Nitrogen-Doped Graphene Quantum Dots Muhammad Shehzad Sultan¹, Vladimir I. Makarov¹, Frank Mendoza¹, Wojciech M. Jadwisienczak², Danilo Barrionuevo¹, Muhammad Sajjad⁴, Brad Weiner³ and Gerardo Morell¹; ¹Physics, University of Puerto Rico, San Juan, Puerto Rico, United States; ²School of Electrical Engineering and Computer Science, Ohio University, Athens, Ohio, United States; ³Chemistry, University of Puerto Rico, San Juan, Puerto Rico, United States; ⁴Physics and Astronomy, Western Kentucky University, Bowling Green, Kentucky, United States.

Metal-free nitrogen doped graphene quantum dots (N-GQD) were synthesized and, for the first time to our knowledge, their magnetic properties were investigated. The results indicate that N-GQDs exhibit superparamagnetic behavior. The specific size, shape and zigzag edge structure of N-GQDs were taken into account to explain the origin of the observed magnetism. The magnetization dependence led to estimating the N-GQD material magnetic permeability for different ambient temperatures. From the zero-field-cooled (ZFC) and field-cooled (FC) magnetization measurements, carried out at 50 Oe magnetic field strength, we estimated the blocking temperature $T_{\rm B}$ to be around 300 K. On the basis of the experimental data analysis, the magnetic permeability, number of correlated spins per single N-GQD, and number density of superparamagnetic N-GQD per gram of material were estimated. The excellent magnetic properties together with optical properties manifested by N-GQDs have the potential to lead to high performance biomedical and data recording devices.

PS1.19

(Late News) The Effects of Gamma Radiation on Carbon Nanotube Structure and Field Emission Performance <u>Tasso von Windheim</u>; Electrical and Computer Engineering, Duke University, Durham, North Carolina, United States.

Solid-state technology dominates the consumer electronics market because of the low cost associated with large-scale integration. However, there are numerous applications in which solid-state devices are unreliable or do not provide adequate performance, particularly applications with military systems operating in high radiation environments. In these applications, vacuum microelectronic devices present an attractive alternative. Despite the performance advantages, the use of vacuum electronics has been limited because there is no versatile and reliable microscale platform that enables integration of large numbers of vacuum circuit elements on a single substrate. To address this need, RTI International in collaboration with Duke University has been developing a Microelectromechanical systems (MEMS) platform with integrated carbon nanotube (CNT) field emitters that enables integration of high-performance microelectronic vacuum components into functional circuits on a single silicon substrate. In this study, we investigate the effects of radiation on the field emission properties of CNTs. CNTs were deposited on silicon substrates using microwave plasma enhanced chemical vapor deposition. The samples were exposed to gamma radiation doses of 0.5 Mrad(Si), 1 Mrad(Si), and 3 Mrad(Si). Typical lifetime exposure level for a spacecraft is around 0.3-0.5 Mrad(Si). Scanning electron microscopy revealed no changes in the film morphology and no damage was visible on the surfaces of the AMWCNTs. Raman spectroscopy was used to investigate the change in defect density caused by irradiation, and revealed that gamma irradiation at all dose levels caused a reduction in defect density as indicated by a reduced ratio of the D to G peak height observed in the Raman spectra. The decrease in D/G ratio for the 0.5 Mrad(Si), 1 Mrad(Si), and 3 Mrad(Si) was 7%, 11%, and 2% respectively.

Due to relationship between defect density and field emission, the field emission performance was affected by irradiation. Turn-on field ($\rm E_{to}$) increased by 1 V/micron for the sample exposed to 1 Mrad(Si) gamma radiation and increased by 5 V/micron for the sample exposed to 3 Mrad(Si) gamma radiation. There was no observed change in $\rm E_{to}$ for the sample exposed to 0.5 Mrad(Si) gamma radiation. So while defect density was affected at all radiation dose levels, significant changes in field emission performance was only observed for samples dosed at 1 Mrad(Si) and 3 Mrad(Si), which are much greater than the expected lifetime exposure levels for a typical spacecraft. In addition, analysis of the field emission performance using the Fowler-Nordheim model indicates that the decrease in defect density reduces self-heating. This is further evidence that CNTs are indeed a potential candidate for use in vacuum microelectronics in high-radiation environments such as outer space.

PS1.20

A Betavoltaic Microcell Based on Semiconducting Single-Walled Carbon Nanotube Arrays/Si Heterojunctions <u>Jinwen Zhang</u>; Institute of Microelectronics, Peking University, Beijing, China.

This paper reports a novel betavoltaic (BV) microcell based on semiconducting single-walled carbon nanotube (s-SWCNT) arrays/Si heterojunctions. There are three new features. Firstly, the new BV microcell is constructed by the aligned arrays of p-type s-SWCNTs onto n-type silicon forming p-n heterojunctions as energy conversion, rather

than Schottky junctions such as SWCNTs thin film/Si [1] and Au/s-SWCNT/Ti [2] in our previous works. Secondly, the s-SWCNT arrays/Si p-n heterojunctions display better rectification characteristics than the foregoing two Schottky junctions with a leakage current (I₀) as low as 1.5pA and an ideality factor (n) of 1.83. Thirdly, the new BV microcell reveals higher performance than our previous ones. Under 7.8mCi/cm² ^{63}Ni irradiation, $V_{\rm OC}\!=\!62mV,$ $J_{SC}\!=\!3.8\mu A/cm^2,$ fill factor (FF) of 33.4% and energy conversion efficiency (η) of 9.8% were achieved.BV microcells are attractive candidates for micro-power sources owning to their high energy density, long lifetime and durability in extreme environments. Several semiconducting materials have been studied such as GaN and GaAs [3, 4]. However, most microcells are limited to the low current density and energy conversion efficiency. Our group first proposed and demonstrated that SWCNTs could be considered as a perfect material for BV microcells. Given that the p-n junction exhibits better rectification property than Schottky junction [5], we present the design, fabrication and performance of a novel BV microcell based on s-SWCNT arrays/Si p-n heterojunctions in this paper.In conversion part, the aligned arrays of p-type s-SWCNTs contact with the n-type silicon substrate (2-4 Ω cm) to form p-n heterojunctions. The front and back electrodes of Au forming ohmic contact with s-SWCNTs and Si respectively are employed to transport carriers to external circuit. In source part, ⁶³Ni thin film on a nickel foil continuously emits β particles for energy supply. Under radiation, electron-hole pairs (EHPs) excited and generated in the depletion region can be separated and swept by the build-in electric field into external circuit to produce electrical current.Cr/Au and Ti were sputtered and patterned on silicon substrate with 300nm thick silicon dioxide (SiO₂) layer in sequence. Cr/Au was sputtered on the back side of silicon substrate and sintered at 320°C. Whole wafer was diced to 6980μm×6980μm dies as samples. The semiconducting-enriched (99%) SWCNT solution of NanoIntegris was diluted with deionized water down to 0.1µg/ml. After switching on the AC voltage source (Vpp=5V and f=5MHz), a drop of the suspension was dripped onto a sample and blown off after 60s. Then a staircase-increased negative drain bias from 0V to -20V was applied to selectively burn off metal-SWCNTs with 15V on the back gate. Next, the Ti and uncovered SiO, layer were etched by BHF so that the parallel and aligned s-SWCNTs adhered tightly to Si substrate by capillary force (Fig. 4). 7.8mCi/cm² ⁶³Ni film was electroplated on a nickel foil. In our testing system, the conversion part was aligned to the source part face to face with 1mm gap. I-V curve in dark of the device proves good rectifying characteristics. By fitting it with the ideal diode equation $I=I_0[\exp(qV/nkT)-1]$, the leakage current was deduced to 1.5pA with n=1.83, indicating that s-SWCNT arrays/Si p-n heterojunction has better characteristics than SWCNTs based Schottky junctions. Under radiation, the I-V curve moves downwards revealing $V^{}_{\rm oc}$ of 62mV and $J^{}_{sc}$ of 3.8 $\mu A/$ cm^2 and the P-V curve shows FF of 33.4% and η of 9.8%. It proves that s-SWCNT arrays/Si heterojunctions have huge potential for application in betavoltaic microcells. References [1] Anonymous Reference No. 1. [2] Anonymous Reference No. 2. [3] M. Lu, et al., Energy Conversion and Management, vol. 52, pp. 1955-1958, 2011. [4] C. W. Liang, et al., Nano Lett., vol. 8, pp. 1809-1812, 2008. [5] Y. B. Liu, et al., vol. 70, Appl. Radiat. Isotopes., pp. 438-441, 2012

PS1.21

Synthesis and Extensive Characterization of Mesoporous Graphene-Based Nanomaterials to Establish Structure-Activity Relationship Sanju Gupta, Brendan Evans and Romney Meek; Western Kentucky University, Bowling Green, Kentucky, United States.

Graphene-based nanomaterials including few-layer graphene oxide, reduced graphene oxide, three-dimensional scaffolds of graphene and graphene-carbon nanotube aerogels and their nitrogenated counterparts are synthesized using environmentally friendly methods. These nanomaterials are extensively characterized in terms of surface morphology and structure using electron microscopy, optical and vibrational techniques which provided microscopic structure and unprecedented physical properties useful for electrochemical energy storage and water desalination. Raman spectroscopy (RS) provided lattice dynamical structural characterization at nanoscale revealing collective atomic/molecular motions and localized vibrations. We analyzed Raman spectra in terms of prominent band position and intensity ratio that help to quantify microscopic stress/strain, structural disorder, number defect density, respectively.

Photoluminescence excitation (room temperature luminescence with varying excitation wavelength) spectra revealed the electronic band

structure such that distinct localized pi electrons were found in-between the band gap for the functionalized graphene. In addition, we have used advanced electrochemical microscopy technique to investigate dynamic physicochemical processes occurring at the surface of these exotic graphene-family nanomaterials and at the solid/liquid (electrode/ electrolyte) interface. We discuss our experimental findings in terms of the multiple roles of oxygen and nitrogen functional groups and establish (micro)structure-property-activity relationship, relevant for alternative energy, catalysis, sensing and water treatment, to name a few.

PS1.22

Graphene/TiO₂ Composites for Infrared Radiation Shielding <u>Artur Malolepszy</u>, Marta Mazurkiewicz-Pawlicka and Leszek Stobinski; Faculty of Chemical and Process Engineering, Warsaw University of Technology, Warsaw, Poland.

Inorganic nanocrystals attract great interest due to their unique physical properties and immense potential for applications. In particular, nanocrystals offer exciting opportunities for technologies on the interfaces between chemistry, physics, biology, and medicine. The aim of our research was investigation of the influence of titanium dioxide / graphene composites on the infrared radiation shielding. Depending on the content of TiO, the composites exhibits different IR shielding efficiency. Graphene oxide (GO) were prepared via modified Hummer's method. Titanium dioxide nanoparticles were deposited on the surface of graphene flakes by the hydrothermal method. Isopropyl alcohol solution of Titanium(IV) isopropoxide precursor was initially mixed with water and then heated in the microwave pressure Teflon reactor at the temperature around 220 C and under the pressure of 55 bars for 30 min. The obtained composites were characterized by: i) X-ray diffraction (XRD) - to determine the average size of crystallites and the phase composition, ii) electron microscopy (SEM/STEM) - for imaging the metal oxide distribution on the surface of graphene flakes, iii) thermo-gravimetric analysis (TGA) - for determining the thermal stability and percentage content of graphene and metal oxides. Absorption of IR radiation were measured using FT-IR spectrometer in the range of $400 - 7000 \text{ cm}^{-1}$.

PS1.23

Nanocrystalline Graphene Film Growth on Non-Metallic Substrates by Hot Filament Chemical Vapor Deposition Sandra M. Rodriguez¹, Alvaro Instan¹, Frank Mendoza¹, Ram Katiyar¹, Brad Weiner² and Gerardo Morell¹; ¹Physics, University of Puerto Rico at Rio Piedras, San Juan, Puerto Rico, United States; ²Chemistry, University of Puerto Rico at Rio Piedras, San Juan, Puerto Rico, United States.

Direct synthesis of nanocrystalline graphene by hot filament CVD on nonmetallic materials is reported. Samples were deposited in a low pressure atmosphere of methane with an average substrate temperature of $904^{\circ}\mathrm{C}$ followed by convection cooling to room temperature. The structural properties of the carbon films were analysed using Raman Spectroscopy and Atomic Force Microscopy (AFM). Raman mapping showed that the ratio of the 2D/G peaks intensity is in the range of 0.41 to 0.68 over 90% of the scanned surface area of $150 \times 100 \ \mu\mathrm{m}^2$. The average value of the 2D peak FWHM was $60\mathrm{cm}^{-1}$ and the average ratio of D/G peak intensity was 1.3, indicating that the carbon films are composed of nanometer-scale crystallites. AFM in tapping mode was used to estimate the number of layers of the grown graphene films. The potential applications in electrical devices to measure biological responses are also discussed.

PS1.24

Synthesis and Characterization of Various Graphene Foams Leszek Stobinski, Artur Malolepszy, Marta Mazurkiewicz-Pawlicka, Tomasz Kotkowski, Robert Cherbanski and Eugeniusz Molga; Faculty of Chemical and Process Engineering, Warsaw University of Technology, Warsaw, Poland.

Graphene foams (GF) exhibit a high sorption capacity due to their high porosity and very low density, generally less than 0.01 g/cm³. Due to their diverse chemical nature, they may exhibit specific sorption activity against various gases. These properties can be used in the process of selective removal of individual gases from their mixtures. Graphene foams, due to their high sorption capacity, can be applied as storage tanks for various gases. In future GF can be widely used in the removal of various types of gaseous and liquid pollutants. Due to their high electrical conductivity GF

can be also used as an excellent carrier for electrocatalysts in various types of fuel cells as well as an excellent carrier for various types of metal and metal oxide nanoparticles used in catalytic chemical processes. GF due to its high porosity is also an excellent heat insulator resistant to high temperatures and an aggressive chemical environment. The authors of this presentation will present the synthesis process of various types of pure and doped graphene foams and characterize their physicochemical properties. The starting material for these synthesis is graphene oxide and reduced graphene oxide.

PS1.25

Surface Enhanced Raman Scattering Based Molecular Sensors Enabled by Graphene Quantum Dots Templated Growth of Silver Nanoparticle Arrays Yonhua Tzeng, JiunChi Lai and YingRen Chen; Electrical Engineering, National Cheng Kung University, Tainan, Taiwan.

Plasmonic coupling induced enhancement in Raman scattering signal strength from laser illuminated molecules has been proven to be an effective means of detecting, indentifying, and quantify essential molecules for biomedical, environmental, and industrial applications. The enhancement factor of the stronger Raman scattering signal strength emitted from the same amount of molecules is achieved by local strong electric field at so-called hot-spots induced by plasmonic charge separation of closely spaced neighboring metal nanoparticles such as gold, silver and copper. The key issues for excellent SERS sensors are the creation of highdensity hot spots, the higher electric fields at hot spots, the placement of molecules at hot spots, the suppression of fluoresence background signal, and the collection of Raman scattering signal with a high signal to noise ratio. In this paper, novel SERS molecular sensors with Raman enhancement factors on the order of 10E14 for R6G and 10E9 for Adenine will be presented and discussed. The excellent performance of such SERS molecular sensors has been made possible by the combination of fluorescence quenching by graphene and preferred closely spaced and irregular shaped silver nanopartilees grown between graphene nanoislands/ quantum dots. Practical applications of such high performance molecular sensors will be discussed.

PS1.26

Tribological Properties on Carbon Nanotubes Containing Copper-Diamond Composite Films Deposited by Cold Spray Technique Nobuhisa Ata¹, Nana Okimura¹, Ryota Ohtani², Hiroshi Uetsuka², Naoto Ohtake¹ and Hiroki Akasaka¹; ¹Mechanical Engineering, Tokyo Institute of Technology, Tokyo, Japan; ²Asahi Diamond Industrial Co., Ltd, Chiba, Japan.

The metal-based diamond composites have been used as whetstones because the diamond has the high hardness. On these metal-diamond whetstones, there are problems that high processing torque and worn out quickly when they process the hard metals are required. Thus, adding lubricant into metaldiamond composite can reduce energy consumption due to lower processing torque and increasing their tool life due to lower amount of wear. Carbon nanotube (CNT) have not only solid lubrication property but also high thermal conductivity. Whetstones are heated for high temperature by frictional heat during the grinding hard materials. Since, lubricants are also required high thermal conductivity to exhaust heat, CNTs are suitable as lubricants.Cold spray (CS) is one of the process to prepare "mm" thick coatings from plastic material particles, directly. In addition, metal based composite films with high hardness materials can be obtained by a simple process using a mixture of plastic metal and high hardness particles. Thus, CS is able to fabricate metal-diamond composites without dies required conventional methods such as metallurgical processes, etc. In this study, CNTs containing copper-diamond composite films deposited by CS technique from mixture of copper, diamond particles and CNTs, and investigated the effect of CNT addition on tribological properties for the reduction of energy consumptions at materials processing. Copper particles with a mean diameter of 20 mm (HWQ 20: Fukuda Metal Foil & Powder Co., Ltd) were used as the matrix of the film. Diamond particles (IRM5, Tomei Diamond Co. Ltd.) with a mean diameter of 4-8 mm were used. Vapor growth carbon nanofiber (VGNF: Showa Denko Co., Ltd.) which mean diameter is 80 nm and I_G/I_D is 20 was used as CNT. Cu, diamond particles and CNTs were mixed by a coffee mill for 10 s. The concentration of the diamond particles and CNTs mixture was fixed at 2.4 and 0.5 wt.%. A1050 aluminum plates were used as substrates. The surface of the aluminum plate was processed by a sand blaster with white alumina particles (#46, Fuji

Manufacturing Co., Ltd.) to increase the surface area for improving the deposition efficiency. An advanced cold gas system (Medicoat Co., Ltd.) based on a DIMET 412-K cold spray system (TEIN TC Co., Ltd.) was used as the CS system. Acceleration gas was air with 0.64 MPa in pressure 723 K in temperature. The particle feed rate was maintained at 10 g/min. The distance between the end of the nozzle and the substrate surface was maintained at 12 mm. The scan speed and number of scan were 50 mm/s and 20, respectively. The copper-diamond film was also fabricated same conditions as the composites films for comparison. The friction coefficient of the samples were measured by Ball on Disc (BoD) test. The fabricated composite films surface were processed by milling machine and obtained flat space for the BoD test. SUJ2 ball with 6 mm diameter was conducted to BoD test. The load and the speed of rotation were maintained at 1 N, and 400 rpm. The maximum number of the rotations was 50000. The CNTs contained copper-diamond composite film with 500 mm in thickness was fabricated by the CS technique. As the results of BoD test, the mean friction coefficient of the CNT contained copper-diamond composite film was 0.56 and that of the copper-diamond composite film without CNT was 0.88. Thus, CNTs addition into the copper-diamond composite film can reduce the friction coefficient by approximately 36 %. Sliding marks on the SUJ2 ball was observed by laser microscopy to estimate the wear rates. Specific wear rates for the CNTs containing film and the non CNTs containing film were 3.3×10⁻⁵ and 3.7×10⁻⁵ mm³/N m, respectively. There were no significant reduction in grinding efficiency by the CNTs addition into the copperdiamond composite films. These results suggested that adding CNTs into the copper-diamond composite film can reduce the processing torque without reduction of grinding ability.

PS1.27

Graphene Synthesis Using Passivation of Porous Silicon by Carbon Atoms Kurbangali Tnyshtykbayev², Ardak Ainabayev², Oleg Kononenko⁴, Zamart Ramazanova², Ramiz Zulkharnay² and Zinetula Z. Insepov², ¹,¹³; ¹Nuclear Engineering, Purdue University, West Lafayette, Indiana, United States; ²Nazarbayev University, Astana, Kazakhstan; ³Condensed Matter Physics, National Research Nuclear University, Moscow, Russian Federation; ⁴IPTM, Moscow, Russian Federation.

Experimental data on the synthesis of graphene (Gr) and graphene oxide (GO) by acetylene passivation of porous silicon (PS) using a CVD method are presented. Various regimes of low-temperature synthesis of carbon nanocomposites Gr and GO on a PS surface are shown. By selecting a carbonization mode of the PS surface, it was possible to form spatially-oriented graphene and graphene-like structures in the temperature range of $T=350-500\,^{\circ}\mathrm{C}$. Various carbon nanocomposites on the surface of PS were obtained using the Raman and IR spectroscopy the formation, at three synthesis temperatures of 1050, 500, and 350 $^{\circ}\mathrm{C}$. Passivation of PS by carbon atoms open the way for fabrication of spatially oriented graphene with an architecture that repeats the PS topology. The proposed method allows synthesis of a large area Gr sheets with an extremely high surface area, which will significantly widen the scope of its application. Additionally, possibility of passivation of a PS surface, which has a high chemical resistance due to the presence of graphene layers, is discussed.

PS1.29

P-Type Surface Conductivity of Diamond/MoO₃ with an Al₂O₃ Interfacial Layer Yu Yang, Xingye Wang, Yichen Yao and Robert J. Nemanich; Arizona State University, Tempe, Arizona, United States.

P-type surface conductivity of diamond can be readily achieved by surface transfer doping. It provides an alternative strategy to enable high power and high frequency diamond FET operation. Recent studies have reported surface transfer doping on diamond using various high work function activation compounds, such as MoO₂, V₂O₅, Al₂O₅ etc. For materials with high electron affinity (Ea>4.3 eV), their lowest unoccupied molecular orbitals are below the valence band maximum of H-terminated diamond, such that electron transfer from the diamond to the oxides is energetically favorable. While a higher work function of the oxide leads to a higher sheet hole concentration at the diamond surface, the hole mobility is found to decrease which limits high frequency operation. It is thought that the charge transfer negative ions distributed inhomogenously near the interface give rise to Coulomb scattering in the hole accumulation layer. The lack of controllability of the induced hole concentration and the reduced mobility limits the application of surface transfer doping in diamond electronic devices. In this work, the hole concentration and

mobility is modulated by affecting the distance between the hole accumulation layer in diamond and the negative ions in the oxide layers. The surface conductive diamond was achieved with MoO, as an acceptor layer and Al₂O₂ as an interfacial layer. The Al₂O₂ layers were deposited by plasma enhanced atomic layer deposition (PEALD) on hydrogenterminated, undoped diamond (100) surfaces, immediately followed by a remote hydrogen plasma treatment. A molybdenum layer was deposited using electron beam deposition (MBD), and the MoO₃ oxidation state was achieved with an oxygen plasma treatment. Using Hall measurements, the diamond sheet resistance, hole concentration and mobility were characterized with Al₂O₃ interfacial layers of different thickness. The changes of the band bending and the hole accumulation layer were monitored using in situ X-ray photoelectron spectroscopy (XPS) where the binding energy of the diamond C 1s core level was correlated with electrical measurements. The valence band offsets are found to be 2.7±0.2 and 3.1±0.2 eV for Al₂O₂/H-terminated diamond and MoO₂/diamond, respectively. With MoO₃ deposited on Al₂O₃, the Al 2p core level shifted to lower binding energies, indicating that the original negative charges close to diamond surface transfer into the MoO, layer. The increase of Al₂O₃ interfacial layer thickness adds to the distance between the hole accumulation layer and the negative ions, and apparently reduces the ionic scattering leading to an increase of hole mobility. Conversely, the hole concentration decreases which may be ascribed to a lower efficiency of surface transfer doping. By combining two oxides (Al₂O₃ and MoO₃ in this case), the hole mobility and concentration were modulated by altering the thickness of the interfacial layer. This work provides a strategy to achieve a surface conductive diamond surface with optimized hole concentration and mobility. This research is supported by a grant from MIT-Lincoln Laboratories and National Science Foundation (DMR – 1710551).

A1: Diamond Growth I Session Chairs: Martin Kuball and Matthias Schreck Tuesday Morning, May 22, 2018 Room: Humphreys

* Invited Paper

8:30 AM *A1.01

Controlling the Stability of Diamond's Surface Conductivity Travis C. Wade¹, M.W. Geis¹, C.H. Wuoriol¹, T.H. Fedynsyshyn¹, M.E. Plaut¹, S.A. Vitale¹, J.O. Varghese¹, Timothy A. Grotjohn², Robert J. Nemanich³ and Mark A. Hollis¹; ¹MIT Lincoln Laboratory, Lexington, Massachusetts, United States; ²Electrical and Computer Engineering, Michigan State University, East Lansing, Michigan, United States; ³Arizona State University, Tempe, Arizona, United States.

The H-terminated surface of diamond when activated with NO, forms a highly conductive surface layer. This activated surface was used to make field effect transistors (FETs) with drain currents > 1 A/cm². However, the conductance of NO₂-activation diamond decreases over time. Previous reports have suggested that during NO, exposure (NO,-activation), NO,- forms on the diamond surface and generates positive carriers (holes) in the diamond, making the diamond surface conductive. Evaporation of NO, from the diamond is reported to be responsible for the loss of conductance. We report here on x-ray-photoelectron-spectroscopy (XPS) surface characterization of single crystal diamonds and on infrared absorption of diamond powder. After activation, XPS showed the presence of N atoms on the diamond surface, but infrared absorption found no evidence of the nitrite ion (NO₂-), but instead the nitrate ion (NO₂). Two wet chemistry techniques determined the concentration of NO₃ - per milligram of diamond powder. With the powder's surface area measured by the BET technique, the surface NO₃-concentration was measured to be between $6.2x10^{13}$ and $8.2x10^{13}$ cm⁻². This is in the same range as the carrier densities, $3x10^{13}$ to $9x10^{13}$ cm⁻², determined by Hall mobility and surface conductivity measurements of single crystal diamonds. Using similar techniques, the concentration NO₂ was determined to be below the detection limit, < 10¹² cm⁻². NO₂ activation was observed to decrease the surface concentration of H, presumably by reacting with NO₂. How this affects the surface conductance is unknown. Other oxidizing chemistries (O,, WO, and V₂O₂), also chemically react with the H-terminated diamond surface with comparable effect. Contrary to an evaporation theory, both the surface conductance and the surface H atoms are stable (+/- 10% over a week) when stored in dry nitrogen, with or without NO₂-activation. However, surface conductance and the concentrations of NO₃- and H decrease when exposed to laboratory air over a period of hours to days. Infrared absorption measurements showed the reduction of surface NO₃ and H atoms during laboratory air exposure, but gave no indication of what reactions are responsible for their loss in laboratory air. Stabilizing diamond's NO₂activated conductance will not be obtained by reducing the evaporation of NO₂, but by protecting the diamond surface from atmospheric contaminants. This presentation will discuss surface activation as well as possible encapsulation techniques. This material is based upon work supported by the Assistant Secretary of Defense for Research and Engineering under Air Force Contract No. FA8721-05-C-0002 and/or FA8702-15-D-0001. Any opinions, findings, conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the Assistant Secretary of Defense for Research and Engineering.

9:00 AM A1.02

Evaluation of Heteroepitaxial Diamond Grown on 1 Inch A-Plane Sapphire Substrate Seongwoo Kim, Yuki Kawamata, Kenjiro Ikejiri, Kokichi Fujita, Daiki Fujii, Kengo Nishiguchi and Yutaka Kimura; Diamond Development, Adamant Namiki Precision Jewel Co., Ltd., Tokyo, Japan.

Increasing the diameter and crystallinity of the diamond substrate is important for research and development activities of diamond semiconductors. Heteroepitaxial diamond growth method has a high potential to obtain a large-size diamond substrate compared to other growth methods of that. Due to the difference of the thermal expansion co-efficient between diamond and basal (such as MgO) substrate material,

crack generation and random delamination are generated in the diamond film during the cooling process from the diamond growth temperature to RT. Therefore, it is very difficult to enlarge the diameter of the substrate. We have reported growth method in detail, where fabrication of the freestanding heteroepitaxial diamond substrate via microneedles prevent the crack generation and the delamination¹⁾. Microneedles play a role as the buffer or shock absorption, and a large-size diamond substrate of 10mm × 10mm was achieved. Production of large-size diamond crystals is challenging because of two reasons: (1) the diameter of the commercially available MgO substrate is limited up to 1-inch. (2) its cost increases exponentially increases with the diameter of the MgO substrate. Although the hexagonal crystal structure of sapphire is different from a cubic structure of diamond and iridium, the sapphire substrate is another candidate because of its chemical stability, size, and cost-effectiveness. Therefore, heteroepitaxial diamond growth on the sapphire substrate instead of MgO substrate was demonstrated. The conventional process for heteroepitaxial diamond growth on the MgO substrate was applied to the A-plane sapphire substrate size of with 10mm × 10mm. The grown crystal was grown without any cracks and breakage. The thickness of the diamond crystal was $800\mu m$. The as-grown diamond crystal was characterized by X-ray diffraction (XRD) and Raman spectroscopy. The diamond {111} X-ray pole figure shows fourfold symmetry with four sharp peaks at $\psi \approx$ 55°, indicating that the freestanding diamond has a single-crystal structure without polycrystalline inclusions or misoriented blocks. Furthermore, as a result of X-Ray Rocking Curve measurements, the crystallinity was improved for both (400) and (311) compared to the case that of the MgO substrate. Heteroepitaxial diamond growth on the 1-inch A-plane sapphire substrate was successfully demonstrated in the same manner.A comprehensive comparison between the diamond growth methods on MgO substrate and on the sapphire substrate will be discussed.

9:15 AM A1.03

Room-Temperature Direct Bonding of Diamond to Aluminum <u>Jianbo</u> <u>Liang</u>¹, Shoji Yamajo¹, Martin Kuball² and Naoteru Shigekawa¹; ¹Osaka City University, Osaka, Japan; ²University of Bristol, Bristol, United Kingdom.

Power electrinics semiconductor devices such as metal oxide semiconductor field effect transistor (MOSFET), bipolar junction transistor (BJT), insulated gate bipolar transistor (IGBT), high electron mobility transistor (HEMT) etc. are extensively used in home electronics, telecommunication, transport, electric grid and numerous other applications. The high operating power density in such devices commonly produces an increase in temperature near the active region, which would degrade device performance and reliability. Therefore, the suppression of the temperature rise is particularly important to increases the operational power density. Diamodn has the highest thermal conductivity among materials and is an ideal materials to suppress the rise in the device temperature. The application of diamond substrate to the fabrication of the devices is attracting much attention due to its superior heat transfer property, compared to that of Si, sapphire or SiC substrates. The integration of GaN-based devices and diamond by direct growth and wafer bonding has recently been demonstrated, resulting in improved thermal management and three-fold increase in areal power density.^{2,3} For any device, an effecitive thermal boundary resistance exists between the device and the heat sink, which is a significant thermal barrier for heat transfer from the devices to the heat sink. The device was directly mounted onto the heat sink by solder bonding.4 However, the thermal conductivity of the solder materials such as AgSn and AuSn are very low in comparison with that of diamond. In this work, we bonded polycrystalline diamond substrates to aluminum at room temperature by surface activated bonding (SAB)5 and investigated the structural porperties and the bonded strenght of the interfaces. A straight line can be clearly observed at the center of STEM images of the diamond/Al bonded interfaces, which corresponds to the bonded interface. No structural deficits or hollow spaces as cracks were observed at the interface. The obtained results indicate that SAB method is applicable for bonding diamond with dissmilar materials at room temperature. Reference 1. L.S. Pan and D. R. Kania, Diamond: Electronic Properties and Applications (Springer, London, 1995). 2. H. Sun, R. B. Simon, J. W. Pomeroy, D. Francis, F. Faili, D. J. Twitchen, and M. Kuball, Appl. phys. Lett. 106, 111906 (2015). 3. D. Francis, F. Faili, D. Babic, F. Ejeckam, A. Nurmikko, and H. Maris, Diamond Relat. Mater. 19(2), 1525 (2012). 4. M. Kuznetsov, F. Hakimi, R. Sprague, and A. Mooradian, IEEE Photonics Technol. Lett. 9, 1063 (1997). 5. J. Liang, S. Masuya, M. Kasu, and N. Shigekawa, App. Phys. Lett. 110, 111603 (2017).

9:30 AM A1.04

Diamond Nanofeathers <u>Hongjun Zeng</u>, Nicolaie Moldovan and Grace Catausan; Advanced Diamond Technologies, Inc, Romeoville, Illinois, United States.

Due to excellent mechanical, chemical and biological stability and the widest potential window among natural materials, diamond is an ideal material choice in the applications of sensors, supercapacitors, catalysts, or solar energy conversion etc. Large surface area of a diamond electrode could significantly elevate the device's performances such as high sensitivity in a sensor, high power density in a supercapacitor, high efficiency on a catalyst interface, or higher conversion rate in a solar cell. However, realizing a large surface area of diamond is more challenging than fabricating a large surface area of any other carbon allotrope such as activated carbon or graphene. Herein we report a special diamond structure, so-called diamond nanofeather which is highly porous with large surface area. The nanostructure is realized by removing the relatively more instable sp² carbon content from the grain boundaries of doped NCD (nanocrystalline diamond) or UNCD® (ultrananocrystalline diamond) via a prioritized electrochemical etching process and by leaving the pure sp³ carbon structure. Since NCD and UNCD have very high density of the grain boundary compared to other polycrystalline diamonds, after the sp² carbon is removed from the grain boundaries, a large number of nanovacancies and hence large surface area is generated. Further electrochemical or other oxidation of sp3 through these vacancies can adjust the size of the nano pores to meet an optimized diamond-pore ratio to meet surface area requirement for specific applications. The nanofeather structure also helps us revisit the mechanism of NCD and UNCD deposition from a new perspective

9:45 AM A1.05 DISCUSSION TIME

10:00 AM BREAK

10:30 AM *A1.06

Thick Homoepitaxial Layer Growth—A New Approach to Obtain Desired Substrates <u>Tokuyuki Teraji</u>; National Institute for Materials Science, Tsukuba, Japan.

Selection of suitable diamond substrates is important for fabricating highperformance devices. However, single crystal substrates being suitable for each device is sometimes difficult to obtain. Alternative way to prepare these substrates is to grow thick diamond layer with desired function on the generally-used substrates. The thicker the homoepitaxial layer is, the smaller an effect of original substrate becomes [1]. Then, the growth technique of functionalized (super high purity, isotopically-enriched, and so on) diamond layer becomes considerably important. We recently found the growth conditions being optimized for long-duration homoepitaxial diamond growth; formation of non-epitaxial crystallites is effectively suppressed by adding oxygen in the source gas [2]. This growth condition is suitable to accomplish the purpose mentioned above. In this talk, some examples that indicate advantage of the thick layer deposition will be presented. References T. Teraji, T. Yamamoto, K. Watanabe, Y. Koide, J. Isoya, S. Onoda, T. Ohshima, L.J. Rogers, F. Jelezko, P. Neumann, J. Wrachtrup, S. Koizumi, Homoepitaxial diamond film growth: high purity, High crystalline quality, isotopic enrichment, and single color center formation, Phys. Stat. Sol. (a) 212, 2365 (2015). T. Teraji, High-quality and high-purity homoepitaxial diamond (100) film growth under high oxygen concentration condition, J. Appl. Phys. 118, 115304 (2015).

11:00 AM A1.07

Improvement of CVD Diamond on Chemical Mechanical Polished Diamond Substrate Daiki Fujii¹, Satoshi Masuya², Koki Oyama¹, Hidetoshi Takeda¹, Yutaka Kimura¹, Seongwoo Kim¹ and Makoto Kasu²; ¹Adamant Namiki Precision Jewel Co., Ltd., Adachi-ku, Japan; ²Department of Electrical and Electronic Engineering, Saga University, Saga City, Japan.

Diamond can be used for wide range of applications, not only for jewelry. Diamond has a lot of attentions to be an ultimate semiconductor material because of its physical properties like as the wide band gap and high carrier mobility. The ultimate semiconductor devices, such as high frequency device and high-power transistor, are also expected application by these physical properties^{1,2)}. In semiconductor device fabrications,

damage- and roughness-free substrate surface is essential. For this purpose, in conventional semiconductors, chemical mechanical polishing (CMP) is widely used. However, in diamond there are very few reports of CMP. In this work, we perform CMP on single-crystal diamond substrates and investigate its effect on the surface properties. The samples were HPHT single crystal diamond (001) (~4 x 4 x 0.5 mm³). First, all the surfaces were mechanical polished (MP) to adjust the thickness, the off angle, the roughness. Next, CMP was performed on the diamonds with different processing time; 0 h (MP only), 5 h, 50 h and 100 h. The surface roughness before DC plasma CVD growth was less than 0.2 nm. Finally, the diamond films were homoepitaxially grown on the MP and CMPtreated diamond substrates for 10 hours by DC-plasma CVD method.In synchrotron radiation X-ray section topography observation, we have found different contrast between the substrate subsurface with and without CMP. Further, we confirmed pits on the CVD diamond surface grown on the substrate without CPM. However, as the CMP procession time increased, the pit density decreased. Eventually, on the surface for 100 h CMP, no pits were observed. From these results, we concluded that the subsurface damage leads to pit formation in homoepitaxial growth, and CMP on the substrate is effective to prevent pit formation in CVD diamond growth. 1) H. Aida et al., Appl. Phys. Express 9, (2016) 035504. 2) M. Kasu et al., Appl. Phys. Express 5, (2012) 025701.

11:15 AM A1.08

Homoepitaxial Growth of High Quality Single Crystal Diamond by Introducing a Buffer Layer <u>Guoqing Shao</u>, Renan Bu, Zhangcheng Liu, Wei Wang and Hongxing Wang; Xi'an Jiaotong University, Xi'an, China.

Due to its excellent properties such as wide band-gap, high carrier mobility, low dielectric constant, high radiation hardness and high thermal conductivity, diamond is considered as a feasible candidate for next generation semiconductor material. The high quality thick diamond substrate has relied on synthesis under the high pressure high temperature (HPHT) and microwave plasma chemical vapor deposition (MPCVD) techniques. These diamond growth methods were mostly driven by highly promising application in high-power, high-frequency electronic device, quantum technologies, optical windows, jewellery and mechanical tools. The effects of growth temperature, gas composition, substrate holder and pressure are very important in the growth of diamond morphology, quality and specific electronic properties. However the presence of extended defects and mismatch strain are still a major issue in diamond growth. The buffer layer was introduced during the deposition can improve the quality of epitaxial film by releasing the mismatch stress and preventing threading dislocations extends to the epitaxial layer. In this work, the introduction of preferred orientation polycrystalline diamond (POPCD) buffer layer were grown on HPHT (100) single-crystal diamond (SCD) substrate, and then the SCD layer were grown on POPCD buffer layer by the choice of suitable growth parameters in MPCVD reactor. The structure and morphology of POPCD buffer layer and SCD epitaxial layer were revealed using scanning electron microscopy (SEM) and high resolution transmission electron microscopy (HRTEM). The quality and residual stress of diamond was investigated by X-ray diffraction and Raman spectroscope. To measure the dislocation densities of HPHT substrate and epitaxial diamond layer, the etch pits were formed by H/O plasma in MPCVD reactor. We have also revealed the growth scheme of diamond to illustrate how continues diamond films were grown on POPCD buffer layer. Our reports provide a novel strong feasibility for applying this concept in heteroepitaxial growth of diamond. The details will be presented at the conference.

11:30 AM A1.09

Bulk Crystal Growth for Realization of Inch-Size Single-Crystal Diamond Wafers Hideaki Yamada, Akiyoshi Chayahara, Shinya Ohmagari, Yuki Asahara and Yoshiaki Mokuno; ADPARC, AIST, Osaka, Japan.

For realization of several future applications of diamond, it is indispensable to establish technique to fabricate inch-size wafers. In addition to growth and processing techniques for such a large size wafers, preparation of the seed wafers with the same size is perquisite condition. Recently, inch size bulk crystals were achieved by using the HPHT method [1]. In the case of microwave plasma (MWP) CVD, deposition area could be 2inch diameter for frequency 2.45GHz, and 4-6inch size could be grown by up-scaling the conventional apparatus with lower

frequency. One may be able to obtain several inch size wafers in principle. We reported a 2inch size mosaic diamond wafer by using MWPCVD [2], but the size of the constituent mono-crystal diamond plates is still limited to half inch [3]. We studies techniques to prepare bulk crystal by using MWPCVD and obtained several millimeter size bulk crystal. Observation of the cross section of several millimeters thick crystal shows that quality saturate into a certain level for a certain thickness by introducing Oxygen into the source gas mixture and adopting pulse mode discharge. We have achieved to continue the growth for several hundred hours, and to reduce interfaces among the growths, from which defects are generated. [1] A. Katrusha, ICDCM 2017. [2] H. Yamada, et al., Appl. Phys. Lett. 104 (2014) 102110. [3] Y. Mokuno, et al., Diamond and Related Materials 18 (2009) 1258.

11:45 AM A1.10

High Pressure Synthesis of Boron Nitride Crystals and Impurity Controls for Their Functionalizations <u>Takashi Taniguchi</u>; High Pressure Group, National Institute for Materials Science, Tsukuba, Japan.

Hexagonal boron nitride BN (hBN) is chemically and thermally stable, and has been widely used as an electrical insulator and heat-resistant materials. Cubic BN (cBN), which is a high-density phase, is an ultra-hard material second only to diamond. The color of the cBN crystals were always amber or brownish so far which is attributed by carbon andoxygen impurity. After some struggles for high pressure and high temperature (HPHT) solution growth, we could find suitable solvent of Ba-BN system which gave us almost colorless cBN with band-edge nature[1]. At the same time, high purity hBN crystals were recovered in the same HPHT capsule and their attractive potential as a wide-band gap material was realized. It is emphasized that hBN crystals exhibits superior properties as ultra violet light emitter[2] as well as a substrate of graphene devices[3]. On the other hand, cubic boron nitride and diamond heterojunction crystals can be obtained under HPHT and their interface structure was realized[4]. While the current subject is to realize how the major impurities such as carbon and oxygen affect the properties of hBN and cBN, some progerss for the realization for the application of 2D substrates and photonic materials have been achieved. Also, controlling of boron and nitrogen isotope ratio (10B, 11B and 15N) in hBN and cBN crystals can be now carried out by methatheisis reaction under HPHT. In this paper, our recent studies on hBN and cBN single crystals growth under HP with respect to impurity / isotope controles and their functionalizations will be reported. [1] T.Taniguchi, K.Watanabe, J.Cryst.Growth, 303,525 (2007). [2] K. Watanabe, T. Taniguchi, A. Niiyama, K. Miya, M. Taniguchi, Nature Photonics 3, 591(2009). [3] C.R. Dean, A.F. Young, I. Meric, C. Lee, W. Lei, S. Sorgenfrei, K Watanabe, T. Taniguchi, P. Kim, K.L.Shepard, J. Hone, Nature Nanotechnology, 5,722 (2010). [4]C.L.Chen, Z.C.Wang, T. Kato, N.Shibata, T.Taniguchi, Y.Ikuhara, Nature Commun. 6, 6327 (2015).

> G2: Graphene and 2-D Materials II Session Chair: Jimmy Davidson Tuesday Morning, May 22, 2018 Room: Agassiz/Fremont

8:30 AM G2.01

Novel Approach to Synthesize High Quantum Yield Nitrogen-Doped Graphene Quantum Dots Muhammad Shehzad Sultan¹, Vladimir I. Makarov¹, Frank Mendoza¹, Wojciech M. Jadwisienczak², Muhammad Sajjad³, Brad Weiner⁴ and Gerardo Morell¹; ¹Physics, University of Puerto Rico, San Juan, Puerto Rico, United States; ²School of Electrical Engineering and Computer Science, Ohio University, Athens, Ohio, United States; ³Physics and Astronomy, Western Kentucky University, Bowling Green, Kentucky, United States; ⁴Chemistry, University of Puerto Rico, San Juan, Puerto Rico, United States.

The graphene quantum dots (GQDs), a zero-dimensional graphene quantum structure, have triggered an intense research worldwide. GQDs possess unique optical, chemical and physical properties as compared to conventional quantum dots (QDs), such as low toxicity, biocompatibility, optical stability, chemical inertness, high photostability and good water-solubility and therefore hold great application potential in biomedical,

optoelectronics and energy storage devices. The doping of GQDs with heteroatoms is one of the most effective ways to tune their photoluminescence emission and to increase quantum yield. In this study, we developed a novel approach to synthesize high-quality Nitrogen-doped graphene quantum dots (N-GQDs) with high quantum yield, via irradiation of s-triazene in a solution with benzene by using pulsed laser. The TEM, HRTEM, XPS, Energy Dispersive Spectroscopy (EDS), Raman spectroscopy, FTIR, XRD were carried out to observe the morphology, size distribution, crystalline structure and to prove successful doping of GQDs with nitrogen atoms. To observe optical properties of as synthesized N-GQDs, the UV-vis and Photoluminescence measurements were carried out. The as-synthesized N-GQDs exhibit high quality crystalline structure of graphene with an average size of about 3.7 nm. A high quantum yield was exhibited by the obtained N-GQDs as compare to the pristine GQDs. The obtained N-GQDs with oxygen-rich functional groups exhibit a strong emission at 456 nm. These outcomes result in an ample opportunity for the biomedical and optoelectronic applications.

8:45 AM G2.02

Large-Area Membranes and Mesoporous Capacitive Deionization Electrodes from Graphene—A Breakthrough in Water Desalination Sanju Gupta; Western Kentucky University, Bowling Green, Kentucky, United States.

The development of scalable cost-effective robust and resistant to biofouling membranes would have a profound and direct impact on current commercial and emerging markets for water detoxification. In this work, we developed large-area nanofiltration membranes using (1) shear aligned discotic nematic phase of graphene oxide and (2) holey graphene with narrow hole size distribution via controlled metal catalytic oxidation. We also prepared interconnected network of mesoporous graphene-based 'hybrid'electrodes with carbon nanotubes to achieve optimal desalination during capacitive deionization (CDI) of brackish water, attributed to higher specific surface area, electrical conductivity, good wettability of water, environmentally safe, efficient pathways for ion and electron transportation, as potential successor of current filtration membranes. The pressure driven transport data on highly ordered (nanoslit and nanopore), continuous, thin films of multi-layered graphene oxide and holey graphene is expected to demonstrate faster transport of ions for salt water, higher retention for charged and uncharged organic probe molecules with hydrated radii above 5Å as well as modest retention of mono- and di-valent ionic species in salts for ~150 nm thick membranes. The highly ordered graphene nanosheets and hierarchical porous (mesoscale and nanoscale) graphene in the plane of the membrane make organized, molecule-hugging cylindrical and spherical channels, respectively, thus enhance the permeability and hydrodynamic conductivity. The results illustrate that both the meso and nanoscale pores are favorable for enhancing CDI performance by buffering ions to reduce the diffusion distance from external electrolyte to the interior surfaces and enlarging surface area. The findings demonstrated the feasibility of the graphene-based membranes and electrodes compared to currently existing conventional polymeric and ceramic-based technology. Supported by KY NSF EPSCoR Grant.

9:00 AM G2.03

Nanoporous Graphene Film Fabrication with Cluster Ion Beams Zinetula Z. Insepov^{2, 1, 3}, Ardak Ainabayev², Aidyn Shaikhov², Abat Zhuldassov², Zamart Ramazanova², Sean Kirkpatrick⁴ and Michael Walsh⁴; ¹Nuclear Engineering, Purdue University, West Lafayette, Indiana, United States; ²Nazarbayev University, Astana, Kazakhstan; ³Condensed Matter Physics, National Research Nuclear University, Moscow, Russian Federation; ⁴Exogenesis Corporation, Billerica, Massachusetts, United States.

Density-functional theory (DFT) was used for studying the number of layers of a several layer graphene (SLG). Large-scale parallel molecular dynamics (MD) simulations (Lammps) were employed for studying interactions of accelerated cluster ions with ultrathin films, such as graphene, MoS₂, HOPG, and BN. Graphene was grown by CVD on a Ni (111) surface from acetylene and transferred to a Si surface. Graphene oxide was synthesized using a Hummers' method. Argon gas cluster ions were produced by nAccel accelerator, with total energy of $E=30~{\rm KeV}$, average cluster ion sizes between 100 and 2000 Ar atoms, and cluster ion fluences ranging from 1×10^8 to $1\times10^{14}~{\rm cm}^{-2}$ (Exogenesis Corp, MA USA). The Raman spectroscopy (Horiba) study of the irradiated samples was

conducted by a 632 nm laser wavelengths and 100x objective with a laser spot size of ~1 μm, 2 mW power and atomic force microscopy (AIST NT) measurements were carried out in a tapping mode by SUPERSHARPSILICONTM AFM probes for high resolution. Our DFT simulation results were validated with the Raman spectra of several layers graphene (SLG). The MD simulations showed formation of nanometersized holes in the 2D films irradiated with Ar (n = 100-1000) gas clusters, with acceleration energies of E = 1 - 50 eV/atom. The MD results showed formation of uniform holes, with the diameters of 10-20 nm, on the ultrathin films, and were used for optimization of the experimental fabrication of holes in 2D-films. The computer simulation results were confirmed with irradiation experiments, where Ar gas cluster ions were used to bombard graphene, graphene oxide, HOPG, MoS, and BN 2Dfilms, with total cluster ion energy of 30 KeV. Uniformly distributed holes, several nanometers in diameters, were experimentally observed by atomic force microscope (AFM) for the first time. Raman spectra were experimentally observed on the irradiated vs non-irradiated samples and calculated using a density-functional theory (DFT) method.

9:15 AM G2.04

(Late News) Defect Mediated Transport in the Reverse I-V
Characteristics of Diamond-Based PIN Diodes Mehdi Saremi¹,
Raghuraj Hathwar¹, Maitreya Dutta², Franz A. Koeck³, Robert J.
Nemanich³, Srabanti Chowdhury² and Stephen M. Goodnick¹;
¹Department of Electrical Engineering, Arizona State University, Tempe,
Arizona, United States; ²Department of Electrical Engineering, University
of California, Davis, Davis, California, United States; ³Department of
Physics, Arizona State University, Tempe, Arizona, United States.

Diamond is one of the most promising candidates for high power and high temperature applications, due to its large bandgap and high thermal conductivity. In particular, the large bandgap of diamond compared to other materials such as GaN and SiC, should result in commensurately higher breakdown voltages when the limiting current mechanism is band to band impact ionization, with breakdown fields expected on the order of 10 MV/cm. However, as a result of the growth and fabrication process of diamond-based devices in this emerging technology, structural defects such as threading dislocations (TDs) may degrade the electrical properties of such devices, by providing a leakage path for current leading to unwanted excess current. Understanding and control of such defects are important for improving device technology particularly the reverse breakdown characteristics. Here, we have analyzed the voltage and temperature dependent behavior of the reverse bias current-voltage characteristics in diamond PIN diodes, grown by microwave plasma enhanced chemical vapor deposition (MPCVD) on high pressure high temperature (HPHT) single crystal diamond substrates. Over a range of temperatures from 323 K to 423 K, the reverse current can be described by combination of hopping conduction at low bias and Poole-Frenkel emission (PFE) at higher bias, which is hypothesized to occur through TDs assuming typical values of the TD density found in epitaxial grown material.

9:30 AM G2.05

(Late News) Single Crystal Diamond Deposition by Toroidal Plasma CVD Amanda Charris, William Holber, Robert Basnett, Andrew Basnett, Doug Hansel and Adam Brown; Plasmability, LLC, Austin, Texas, United States.

As an alternative to microwave plasma reactors typically used for chemical vapor deposition (CVD) of diamond material, we have been investigating for several years the use of a toroidal plasma reactor. The toroidal reactor operates with a 400 kHz power source operating at up to 11 kW of coupled power. The plasma shape is a line source, so that it is ideal for growth of single crystal diamond on multiple seeds places along the line of plasma. Samples can be retracted during the growth process, so that the growing surface can be kept at all times at an optimum distance relative to the plasma core. A wide range of process parameters have been investigated, with the aim of optimizing growth rate, growth quality and chamber cleanliness. Work will be described that improves the quality of the bulk diamond material by eliminating growth defects and minimizing polycrystalline material at the edges. Single crystal diamonds are grown on 5 mm x 5 mm and 7 mm x 7 mm CVD (100)-oriented diamond seeds. The growth rate for high quality single crystal diamond was in the range of 30 mm/hr to 70 mm/hr. The local growth environment at the seed is

controlled by adjusting (1) chamber configuration and (2) gas flow injection. This work is carried out in part under support from Army Research Laboratory SBIR Contract W911QX-16-P-0222.

9:45 AM G2.06

(Late News) 5 µm PIN Diamond Neutron Detector with Low Gamma Background Jason Holmes¹, Franz Koeck¹, Raghuraj Hathwar¹, Mehdi Saremi¹, Jesse Brown¹, Brianna S. Eller¹, Manpuneet Kaur¹, Anna Zaniewski¹, Holly Johnson¹, Ben Fox¹, Robert J. Nemanich¹, Ricardo Alarcon¹, Stephen M. Goodnick¹, Maitreya Dutta², Mohamadali Malakoutian² and Srabanti Chowdhury²; ¹Arizona State University, Tempe, Arizona, United States; ²University of California, Davis, Davis, California, United States.

Diamond detectors are known for having high radiation hardness compared to other semiconductors, minimal dark current at room temperatures, and fast response times. A PIN diamond with a thin I-layer, 5 microns, is being developed as a highly compact thermal neutron detector which does not require an external bias. The detectors are single crystal diamond with I-layer and N-type layer grown via plasma enhanced CVD (PECVD) on a p-type substrate. By incorporating a Boron growth over the PIN diamond, thermal neutrons interact with Boron-10 due to its high cross section and convert into a Li-7 and alpha particle (1.47 MeV), the alpha then being detected in the diamond. A common background in neutron environments are gamma-rays. By operating in a pulse mode and due to the extremely thin I-layer, the detector becomes insensitive to gamma-rays. Studies so far using a Po-210 alpha source will be discussed. Testing at Oak Ridge National Laboratory is planned for Summer 2018. We acknowledge funding from ARPA-E (Grant DE-AR0000453).

10:00 AM BREAK

E2: Characterization II Session Chair: Yasuo Koide Tuesday Morning, May 22, 2018 Room: Agassiz/Fremont

10:30 AM E2.01

New Insights into Identification of Carbon (C 1s) Peak in X-Ray Photoelectron Spectroscopy Analysis of Crystalline and Polycrystalline Diamond and Associated Effects from Argon Atoms Insertion in the Lattice Jean-François Veyan², Elida de Obaldia².³, Jesus J. Alcantar-Peña² ⁴, Jorge Montes-Gutierrez².⁴, María J. Arellano-Jimenez⁵, Zaibing Guo⁶, Husam N. Alshareef⁶, Miguel J. Yacaman⁶ and Orlando Auciello¹; ¹Materials Science and Engineering and Bioengineering, The University of Texas at Dallas, Richardson, Texas, United States; ²Materials Science and Engineering, University of Texas at Dallas, Richardson, Texas, United States; ³Facultad de Ciencias y Tecnología, Universidad Tecnológica de Panamá, Panama City, Panama; ⁴Departamento de Física, Universidad de Sonora, Hermosillo, Mexico; ⁵Department of Physics and Astronomy, University of Texas at San Antonio, San Antonio, Texas, United States; ⁶Materials Science and Engineering, King Abdullah University of Science and Technology, Thuwal, Saudi Arabia.

The C 1s peak's electron binding energy in X-ray photoelectron spectroscopy (XPS) analysis of crystalline diamond and diamond films has been extensively investigated. The splitting of the C 1s peak is usually reported as the combination of C 1s/sp³ and C 1s/sp² bonding in the sample. In this work, we present a systematic study of polycrystalline diamond films and single crystal diamond (SCD) via XPS analysis, with special focus on clarifying critical analysis issues related to the identification of the C 1s/sp3 and C 1s/sp2 positions in the XPS electron biding energy coordinate, which appear confusing from prior work in the literature, potentially due to problems related to sample charging. The systematic XPS analysis of polycrystalline diamond films and SCD described here, demonstrated that charging of the insulating diamond surface, resulting from X-ray induced electron emission, introduce substantial uncertainty to unmistakably identify the position of the C 1s peaks related to the sp³ and sp2C atoms bonding of diamond and graphite, respectively. In addition, the work reported here demonstrated that the use of Ar⁺ ion bombardment,

generally done to clean the samples surface, introduces another uncertainty in the identification of C 1s XPS peak in the low binding energy position, as the sp2 C atoms bonding of graphite, as generally reported in the literature in relation to XPS analysis of diamond surfaces cleaned by Ar⁺ ion bombardment. The detailed XPS analysis of Ar⁺ ion bombarded diamond samples shows that the C 1s identified as correlated with the sp² C atoms bonding of graphite, is, in reality, the result of Ar atom incorporation in the diamond lattice formed by sp³ C atoms bonding. In fact, the low energy C 1s peak identified in this paper as $C_{D\text{-}Ar}$ peak, corresponds to C atoms associated to the Ar atoms inserted n the diamond lattice during Ar+ ion bombardment surface cleaning. The correlation of the C_{D-Ar} peak and the Ar+ beam energy as well as the concentration of Ar atoms inserted in the diamond lattice is discussed. The incorporation of Ar atoms in the diamond samples have been confirmed via complementary high resolution transmission electron microscopy (HRTEM) and nanoscale resolution Rutherford backscattering (RBS) analysis, the latter showing that the Ar atoms are inserted through a 2 nm layer depth from the surface.

10:45 AM E2.02

Spectroscopic Characterisation of Single Point Defects in Single-Crystal Boron Doped CVD Diamond Phil L. Diggle^{1, 2}, Ben L. Green^{1, 2}, Matthew L. Markham³, Daniel J. Twitchen³ and Mark E. Newton^{1, 2}; ¹University of Warwick, Coventry, United Kingdom; ²Diamond Science and Technology Centre for Doctoral Training, Coventry, United Kingdom; ³Element Six (UK) Ltd., Didcot, United Kingdom.

Chemical vapour deposition (CVD) synthesis enables the growth of single crystal diamond with controlled incorporation of dopants, impurities and defects in the crystal. The progress toward precise control of diamond growth, driven in part by research in quantum communication and sensing[1], has enabled the routine study of point defects at the single defect level. Many of these defects possess quantum properties which are potentially exploitable in applications such as remote temperature measurement^[2], quantum communication^[3,4] and magnetic sensing^[5]. This study reveals that in boron-doped (~1.3 ppm) CVD diamond, previously unreported single point defects can be found with Debye-Waller factors as high as 0.65 at room temperature. Under 532 nm excitation, we observe two classes of zero phonon line – one centred at 580 nm, and another at 612 nm. A high degree of optical polarisation is seen in the excitation and emission of both classes of defects: analysis shows this is consistent with the defects having a dipole moment parallel to [001]. Furthermore, these defects are prone to spectral shift and splitting, which is likely due to a Stark shift from changes in the local environment. Our results highlight that further work is required on point defects in CVD grown diamond, especially in the presence of boron doping. It is apparent that a greater understanding of the incorporation of boron into CVD diamond is needed; both for its role (if any) in newly-identified defects, and as a charge stability tool for other defects^[6,7]. **References** [1] M.L. Markham, et al., Diam. Relat. Mater. 20 (2011) 134-139. doi:10.1016/j. diamond.2010.11.016. [2] J.-W. Fan, et al., ACS Photonics. (2018) doi:10.1021/acsphotonics.7b01465. [3] J.N. Becker, et al., Nat. Commun. 7 (2016) 13512. doi:10.1038/ncomms13512. [4] M.-A. Lemonde et al. arXiv (2018) arXiv:1801.01904 [5] L. Rondin, et al., Rep Prog Phys. 77 (2014) 56503. doi:10.1088/0034-4885/77/5/056503. [6] B.L. Green, et al, Phys. Rev. Lett. 119 (2017) 96402. doi:10.1103/PhysRevLett.119.096402. [7] B. C. Rose, et al., arXiv (2017) arXiv:1706.01555

11:00 AM E2.03

Superconductivity in Thin- and Micro-Structured Boron-Doped Diamond <u>Taisuke Kageura</u>¹, Masakuni Hideko¹, Ikuto Tsuyuzaki¹, Shotaro Amano¹, Yoshihiko Takano², Minoru Tachiki², Syuuichi Ooi² and Hiroshi Kawarada¹; ¹Waseda University, Tokyo, Japan; ²NIMS, Tsukuba, Japan.

Superconducting electronics is expected to be applied to quantum computing, high sensitive magnetic field sensing and microwave filter, searching for superconducting materials suitable for application is an urgent research topic. A low temperature superconductor made of a non-oxide material is desirable for a high-performance, complexed and integrated superconducting devices. Superconducting boron-doped diamond is excellent materials for these superconducting devices because it is mechanically, thermally and electrochemically stable, does not deteriorate over time in performance, and the superconducting temperature (Tc) can be controlled by doping concentration[1]. However, there are few reports for evaluating the superconducting properties from the viewpoint

of manufacturing a superconducting devices. So in this study, we report the superconducting properties of thin and micro-structured boron-doped diamond with Tc=10K. First we investigated the doped layer thickness dependence of Tc. The superconducting boron-doped layer were synthesized by custom-built wave-guide type MPCVD system. In this study, two types of gas introduction method at the start of synthesis were examined. The first method (conventional method) is to simultaneously supply methane and tri-methyl-boron (TMB) gas at the start of synthesis. The second method (new method) is to decompose TMB by plasma in advance and supply methane thereafter. The latter intends to improve boron incorporation efficiency in the early stage of synthesis. The sample synthesized by conventional method shows that Tc(offset) is 10K above 500nm, but it decreases as the doped layer becomes thinner below 500nm region. On the other hand, the sample synthesized by new method shows that Tc(offset) is 10K even if the thickness become 100nm, and the sample with 42nm thickness has Tc(offset)=7.8K. The obtained results are compared with the theoretical equation about thickness dependence of Tc generally applied to superconducting materials such as Nb [2]. The experimental value coincided with the theoretical equation up to about 100 nm, however the experimental value tended to be lower than the theoretical formula as the film thickness became thinner. This is in good agreement with the film thickness which the boron concentration saturates (conventional: -200 nm, new: 100 nm). So it is said that the new method is effective for improving boron incorporation efficiency in the early stage of growth. And this results also demonstrated that Tc = 10 K can be obtained up to a thickness of 100 nm or more even if the growth thickness is below critical film thickness (about 200-300 nm) at which crystal defects are introduced due to lattice mismatch between the substrate and the doped layer. Then we evaluated the line width dependence of Tc using thin (160nm) film. Superconducting diamond line (1-100µm) were formed by oxygen plasma selective etching of the superconducting diamond film with Tc(onset) and Tc(offset) =9.5K, 9K. Here, Tc (onset) / Tc(offset) are defined as the temperature which the normalized resistivity R (T) / R (300 K) reaches 90%/10%, respectively. Experimental results revealed that both Tc (onset) and Tc (offset) did not decrease even if the line width was $1~\mu m$. This means that diamond has high workability in fabricating a fine superconducting device. One interesting point is that 1 µm sample shows s strange two-step transition, the first transition occurred at 9.5K and the R (T) / R (300 K) becomes below 1%, then second transition occurred at 7.5K and the resistivity becomes zero. The origin of this two-step transition were under investigation, we estimated that a part where the superconductivity locally weakened like Josephson junction was formed due to the influence of microfabrication. [1] Kawano. A, Kawarada. H, et al. Physical Review B 82, 085318 (2010). [2] Wolf, S. A.,et.al. Journal of Vacuum Science and Technology 13, 145-147 (1976).

11:15 AM E2.04

Conductive-Probe AFM Characterization of Atomically Well-Defined Diamond (111) Surfaces Norio Tokuda, Ryo Yoshida, Masatsugu Nagai, Taira Tabakoya, Tsubasa Matsumoto and Takao Inokuma; Kanazawa University, Kanazawa, Japan.

Recently, we realized inversion channel diamond MOSFETs with normally-off operation using ALD-Al₂O₃ films as the gate oxide, which were deposited on OH-terminated diamond (111) surfaces [1]. However, the field-effect mobility was much lower than an ideal one because of interface states in the oxide/diamond interface. The origin of the interface states is not yet well understood. An atomic force microscope (AFM) with a conductive probe such as Kelvin probe force microscope (KFM) and conducting AFM(CAFM) is a powerful tool for electrical characterization on a nano-scale [2-4]. In the case of SiO₃/Si interfaces, interface states exist around the steps of the Si surfaces [3,4]. In this study, we investigated the distribution of the interface states by the nano-scale characterization of the local current and potential on OH-terminated diamond (111) surfaces with step-terrace structures using KFM and CAFM. A lightly boron-doped p-type single-crystalline diamond (111) substrate was used. Atomically well-defined diamond (111) surfaces with step-terrace structures were selectively formed on trench sidewalls of the substrate via anisotropic etching processes of solid solution reaction of carbon to nickel and hydrogen-plasma treatment. OH-terminated diamond (111) surfaces were formed by water-vapor annealing. The OH-terminated diamond (111) surfaces were characterized by KFM using a PtSi probe and CAFM using a Pt-coated probe. KFM and CAFM results showed high interface states density at the single- and bunching-step regions on the diamond (111)

surfaces. This result indicates that the steps on the diamond surfaces cause a degradations of current transport properties. Therefore, the formation of step-free diamond (111) surfaces will be a key method in realizing diamond power devices with outstanding performance. References [1] T. Matsumoto, H. Kato, K. Oyama, T. Makino, M. Ogura, D. Takeuchi, T. Inokuma, N. Tokuda, S. Yamasaki, Sci. Rep. 6, 31585 (2016). [2] B. Rezek, C.E. Nebel, Diamond Relat. Mater. 14, 466 (2005). [3] R. Hasunuma, A. Ando, K. Miki, Y. Nishioka, Appl. Surf. Sci. 162, 547 (2000). [4] M. Murata, N. Tokuda, D. Hojo, K. Yamabe, Thin Solid Films 414,56 (2002).

B2: Diamond Electronics II Session Chair: James Butler Tuesday Afternoon, May 22, 2018 Room: Humphreys

* Invited Paper

1:30 PM B2.01

Diamond FinFET Technology for Future Power/RF Electronics Biqin Huang, Xiwei Bai, Stephen Lam and Kenneth Tsang; HRL Laboratories LLC, Malibu, California, United States.

Diamond fin field-effect transistor (diamond FinFET) without a conventional hydrogen-terminated channel will be discussed. Leveraging fin and hole accumulation by metal-oxide-semiconductor (MOS) structures, we are able to effectively control the channel conduction. Enhancement mode devices with 100-nm—wide fins were designed and fabricated to ensure that the channel pinched off at zero gate bias. Device characteristics at various temperatures will be discussed. With improved ohmic contact, larger than 300mA/mm current density were measured at 150°C, orders of magnitude more than current density at room temperature. The potential application of diamond FinFET technology in future power/RF electronics will be discussed.

1:45 PM B2.02

Breakdown Mechanism of C-H Diamond MOSFETs Compared with Other Wide Bandgap Materials <u>Hiroshi Kawarada</u>; Waseda University, Tokyo, Japan.

Breakdown voltage V_R is now more than 2000 V[1] in hydrogen terminated (C-H) diamond MOSFETs with 2 dimensional hole gas (2DHG) as channel and drift layer. In general, the maximum breakdown voltage relates with gate-drain distance L_{GD} , i.e., drift layer length in lateral FETs. At present, V_R/L_{GD} (a criteria for evaluating high voltage FET) is ~ 1 MV/cm in the C-H diamond FET [2] and is almost equivalent to those of SiC and AlGaN/GaN lateral FETs (0.8-1.2 MV/cm) and is higher than that of the oxygen-terminated (C-O) diamond MESFET [3] using lightly boron doped layer (~ 0.5 MV/cm). How V_0 is defined in the C-H diamond MOSFET and other wide bandgap FETs is an important issue, because the $V_{\rm g}/L_{\rm GD}$ around 1 MV/cm is far less than critical breakdown fields $(E_{\rm c})$ of SiC (>3.0 MV/cm), GaN (>3.0MV/cm), and diamond (>5.0 MV/cm). Even in the large grain poly crystalline diamond, the $V_{\rm B}/L_{\rm GD}$ in the MOSFET with $V_R \sim 1800 \text{V}$ [4] shows $\sim 1 \text{MV/cm}$. The grain boundaries or defects are not a substantial reason for breakdown in these cases. We have recently found that breakdown frequently occurs at the drain side of drift layer, not its gate side. This phenomenon is very similar to those observed in AlGaN/GaN lateral FETs [5]. In our study, the electric field distribution has been estimated by a device simulation based on the negative charge sheet at Al₂O₃/C-H diamond interface [2], which reproduces experimental I-V characteristics of 2DHG FET very well [2]. Only by the negative charge sheet, however, the electric field is located near the gate side of drift layer. Adding positively charged layer under the undoped homoepitaxial layer (channel and drift layer), we could reproduce a highly localized electric field peak which exceeds 5.0 MV/cm near the drain side (the opposite side) of drift layer. The electric field peak explains breakdown phenomenon quite well. The positive charged layer might originate from the ionization of nitrogen donor of type Ib substrate (nitrogen density >10¹⁸ cm⁻³) under the undoped homo-epitaxial layer (500 nm in thickness) through hole injection and recombination. The positively charged layer is useful for charge compensation to realize the

homogeneous electric field (super junction) for ideal power device if the positive charged layer exactly neutralizes the negative charge sheet at the interface. Well controlled nitrogen doped layer is quite necessarily to design high power diamond devices. [1] Y. Kitabayashi, H. Kawarada et al., IEEE Electron Device Lett., **38**, 3, 363 - 366 (2017). [2] H. Kawarada et al., Scientific Reports, **7**, 42368/1-8, (2017). [3] H. Umezawa, T. Matsumoto and S. Shikata: IEEE Electron Device Lett. **35**, 1112 (2014). [4] M. Syamsul, H. Kawarada et al., Applied Physics Letters. **109**, 203504/1-4 (2016). [5] M. Kuzuhara, IEEE Trans. Electron Devices, **62**, 405 (2015).

2:00 PM *B2.03

Complex Charge Transfer in Chameleon Diamonds: A Model of the Color Change Process James E. Butler¹, Keal Byrne¹, Wuyi Wang² and Jeffrey E. Post¹; ¹Department of Mineral Sciences, Smithsonian Institution, Washington, DC, United States; ²Gemological Institute of America, Carlsbad, California, United States.

A group of natural diamonds change their color tint from green to yellow based on their exposure to light and heat. These diamonds also emit long live phosphorescence after uv excitation. We have observed optical response of these diamonds to optical and thermal excitation and developed a model to explain the observed phenomena. A principle element of the model is the proposal of an acceptor state which should be observable in the near IR. Subsequently, we have observed the near IR absorption to this acceptor state supporting our model of charge transfer processes in these diamonds.

2:30 PM B2.04

Logic Circuits Using Depletion/Enhancement-Modes H-Diamond MOSFETs Yasuo Koide^{1, 2}, Jiangwei Liu³, Masataka Imura³ and Meiyong Liao³; ¹Research Network and Facility Services Division, NIMS, Tsukuba, Japan; ²Amano-Koide Collaborative Research Lab., NIMS, Tsukuba, Japan; ³Research Center for Functional Materials, NIMS, Tsukuba, Japan.

Diamond is a candidate material for next-generation power electronics and integrated circuit (IC) which operate under extreme environment. In order to use an advantage of high-density hole channel of hydrogenated diamond (H-diamond) surface, we have developed the high-k stack gate dielectrics for H-diamond MOSFETs, such as HfO₂/HfO₂, LaAlO₂/Al₂O₃ Ta₂O₂/Al₂O₃, and ZrO₂/Al₂O₃, TiO₂/Al₂O₃, and AlN/Al₂O₃ prepared by a combination of sputter-deposition (SD) and atomic layer deposition (ALD) techniques [1-5] and Y₂O₃ [6] prepared by e-beam deposition. In addition, we have developed both of normally-on and off modes MOSFETs, which usually named by D and E modes, respectively, by using the stack gate structure and controlling the threshold voltage (V_x) [7]. The D/E-modes control provided demonstration of the inverter circuit [8]. After improving the electrical properties of H-diamond MOSFETs and fabricating D/Emodes H-diamond MOSFETs controllably, next challenge is to combine these devices to be logic circuits. In this paper, the H-diamond NOT and NOR logic circuits composed of D-mode ALD-Al₂O₂/H-diamond and E-mode SD-LaAlO₃/ALD-Al₂O₃/H-diamond MOSFETs were fabricated. Electrical properties of the MOSFETs and logic circuits were characterized. By controlling V_T value from the FET fabrication process, the H-diamond NOT and NOR logic circuits composed of D- and E-modes MOSFETs were fabricated. The Al₂O₂/H-diamond and LaAlO₂/Al₂O₂/Hdiamond MOSFETs operated with D-mode and E-mode characteristics, respectively. Distinct inversion characteristics were observed for the H-diamond MOSFET NOT logic circuit. The gain maximum increased from 1.2 to 26.1 with input voltages of $V_{\scriptscriptstyle DD}$ changing from –5.0 to –25.0 V. The H-diamond NOR logic circuit showed the clear NOR gate characteristics. References 1. "Low on-resistance diamond field effect transistor with high-k ZrO, as dielectric," J.W. Liu, M.Y. Liao, M. Imura, A. Tanaka, H. Iwai, and Y. Koide, Sci. Reports, 4, 6395 (2014). 2. "High-k ZrO₂/Al₂O₃ bilayer on hydrogenated diamond: Band configuration, breakdown field, and electrical properties of field-effect transistors," J.W. Liu, M.Y. Liao, M. Imura, and Y. Koide, J. Appl. Phys., 120, 124504 (2016). 3. "Structural properties and transfer characteristics of sputter deposition AlN and atomic layer deposition Al₂O₂ bilayer gate materials for H-terminated diamond field effect transistors," R.G. Banal, M. Imura, J.W. Liu, and Y. Koide, J. Appl. Phys., 120, 115307 (2016). 4. "Effect of off-cut angle of hydrogen-terminated diamond(111) substrate on the quality of AlN towards high-density AlN/diamond(111) interface hole channel" M. Imura, R. Banal, M.Y. Liao, J. Liu, T. Aizawa, A. Tanaka, H. Iwai, T. Mano, Y. Koide J. Appl. Phys. 121, 025702 (2017). 5. "Deposition of TiO₂/Al₂O₃ bilayer on hydrogenated diamond for electronic devices: Capacitors, field-effect transistors, and logic inverters," J. W. Liu, M. Y. Liao, M. Imura, R. G. Banal, and Y. Koide, J. Appl. Phys. 121, 224502 (2017). 6. "Enhancement-mode hydrogenated diamond metal-oxidesemiconductor-field-effect transistors with Y₂O₂ oxide insulator grown by electron beam evaporator," Appl. Phys. Lett. 110, 203502 (2017). 7. "Control of normally on/off characteristics in hydrogenated diamond metal-insulator -semiconductor field-effect transistors," J.W. Liu, M.Y. Liao, M. Imura, T. Matsumoto, N. Shibata, Y. Ikuhara, and Y. Koide, J. Appl. Phys. 118, 115704 (2015). 8. "Diamond logic inverter with enhancement-mode metal-insulator-semiconductor field effect transistor," J.W. Liu, M.Y. Liao, M. Imura, E. Watanabe, H. Oosato, Y. Koide, Appl. Phys. Lett. 105, 082110 (2014).

2:45 PM B2.05

Gate Oxide Electrical Stability of Diamond P-Type MOS Capacitors Oluwasayo I. Loto^{1, 2}, Mathieu Florentin^{1, 2}, Marie-Laure Hicks³, Alexander Pakpour-Tabrizi³, David Eon^{1, 2}, Julien Pernot^{1, 2}, Richard Jackman³ and Etienne Gheeraert^{1, 2, 4}; ¹CNRS, Inst NEEL, Grenoble, France; ²Univ. Grenoble Alpes, Inst NEEL, Grenoble, France; ³Diamond Electronics Group, London Center for Nanotechnology, University College London, London, United Kingdom; ⁴Graduate School of Pure and Applied Sciences, University of Tsukuba, Tsukuba, Japan.

The wide band gap, high breakdown field and thermal conductivity of semiconducting diamond has generated interest in its use for high voltage and high frequency power electronics. Several diamond-based field-effecttransistors (FETs) have already revealed good $\rm I_{on}/I_{Off} \, ratio^1$ and high blocking voltage capability (~2kV)2 in a wide range of operating temperatures (up to 400°C)³. The promising potential of these diamond devices will necessitate the study of other design considerations such as complexity, cost, power density and reliability. Reliability issues relating to widely investigated GaN and SiC power devices are obtainable in literatures. For devices which need a metal-oxide- semiconductor interface, an obvious reliability concern is the stability of the gate stack. We have fabricated diamond MOSCAPs using Al₂O₃ gate oxide to study the response of flatband voltage to bias stress. It has been previously reported that near interface and oxide charges could have significant effect on the performance and the stability of MOS devices. A recent work4 has demonstrated the positive effects caused by the post-oxidation annealing on the distribution of interface traps and on the Fermi level pinning at the oxide/diamond interface. Hence, diamond MOSCAPS fabricated for this study were annealed. In this talk, the effect of gate voltage-induced-stress on the stability of the flatband voltage ($V_{\scriptscriptstyle FB}$) will be shown for p-type diamond metal-oxide-semiconductor capacitors (MOSCAPs). The flatband voltage $(V_{_{FB}})$, which is directly connected to the threshold voltage⁵ gives information on the quality of the diamond/semiconductor interface and helps to evaluate the reliability of MOS-based devices. Two types of timedependent stress tests have been performed and the result obtained will be discussed. References 1. H.Umezawa, T.Matsumoto, & S.I.Shikata, S. I. (2014). Diamond metal-semiconductor field-effect transistor with breakdown voltage over 1.5 kV. IEEE Electron Device Letters, 35(11), 1112-1114. 2. Y.Kitabayashi, T.Kudo, H.Tsuboi, T.Yamada, D.Xu, M. Shibata,... & A.Hiraiwa, « Normally-Off C-H Diamond MOSFETs With Partial C-O Channel Achieving 2-kV Breakdown Voltage ». IEEE Electron Device Letters, 38(3), 363-366. (2017). 3. H. Kawarada, T. Yamada, D. Xu, H. Tsuboi, Y. Kitabayashi, D. Matsumura, A. Hiraiwa, (2017). Durability-enhanced two-dimensional hole gas of CH diamond surface for complementary power inverter applications. Scientific Reports, 7, 42368. 4. T.T. Pham, M. Gautierez, C. Masante, N. Rouger, D. Eon, E. Gheeraert, D. Araujo, and J. Pernot Diamond/Al₂O₃ interface for gate control diamond transistors. Submitted at Journal of Applied Physics 5. T.Teraji, Y.Garino, Y.Koide, & T.Ito. Low-leakage p-type diamond Schottky diodes prepared using vacuum ultraviolet light/ozone treatment (2009).

3:00 PM BREAK.

K3: Nanodiamonds III Session Chair: Yonhua Tzeng Tuesday Afternoon, May 22, 2018 Room: Agassiz/Fremont

1:30 PM *K3.01

Production and Characterization of Customized Diamond
Nanoparticles <u>Laia Gines</u>¹, Soumen Mandal¹, David Morgan², Ryan
Lewis³, Paola Borri³, Georgia F. Wood⁴, Julie V. Macpherson⁴ and Oliver
A. Williams¹; School of Physics and Astronomy, Cardiff University,
Cardiff, United Kingdom; ²Cardiff Catalysis Institute, School of
Chemistry, Cardiff University, Cardiff, United Kingdom; ³School of
Biosciences, Cardiff University, Cardiff, United Kingdom; ⁴Department of
Chemistry, University of Warwick, Coventry, United Kingdom.

The wide range of applications have made diamond nanoparticles an interesting material. Due to diamond bio-compatibility¹ and non-cytotoxicity², diamond nanoparticles have been used in biomedical applications such as optical bio-imaging³ and drug delivery⁴. Diamond nanoparticles have also been extensively used as seeds, or nucleation centres, for synthetic diamond growth³. Furthermore, single photon sources (SPS) based on defects in diamond, known as colour centres, have also attracted an increasingly interest for quantum computation applications. In particular, the SiV colour centre has been highlighted as a promising photon source due to its interesting optical properties at room temperature⁶. Another well-known colour centre, the NV, stands out for its spin manipulation possibilities, making this colour centre attractive as a

^{*} Invited Paper

magnetic field sensor and for magnetic imaging⁷. Although there is a large variety of commercial diamond nanoparticles available, the demanding applications require the use of high quality diamond nanoparticles. Most of the commercial particles contain non-neglectable amount of metals, which is detrimental for magnetic-related applications. Besides, nondiamond carbon is also present in commercial diamond nanoparticles, leading to unwanted particles aggregation8. For all the reasons above, it is highly important to be able to customize the diamond nanoparticles, and produce them according to their ultimate application. The most versatile method for diamond nanoparticles production, is the use of milling techniques^{9,10} to crush bulk diamond. High quality diamond nanoparticles can be obtained crushing high quality commercial bulk samples, such as electronic grade single crystal diamond, or crushing CVD grown diamond films. For instance, diamond nanoparticles with SiV colour centres (with silicon incorporated either from a solid source or from a gas phase) can be created milling down diamond films grown onto passive substrates. Nevertheless, the particles need to be subjected to deep cleaning methods and centrifugation processes to remove the contaminants produced during the milling and to obtain the desired nanoparticles size distribution. The choice of the appropriate milling material is also critically determined by the acceptable contaminants in the ultimate application. Nanoparticles obtained with both tempered steel and silicon nitride materials have been extensively studied by XPS measurements, Raman and TEM measurements. Diamond nanoparticles' production (including diamond nanoparticles with SiV centres) and different characterization methods will be discussed. 1 L. Tang, C. Tsai, W.W. Gerberich, L. Kruckeberg, and D.R. Kania, Biomaterials 16, 483 (1995). 2 A.M. Schrand, H. Huang, C. Carlson, J.J. Schlager, E. Osawa, S.M. Hussain, and L. Dai, J. Phys. Chem. B 111, 2 (2006). 3 Y.Y. Hui, C. Cheng, and H. Chang, J. Phys. D. Appl. Phys. 43, 374021 (2010). 4 V.N. Mochalin, O. Shenderova, D. Ho, and Y. Gogotsi, Nat. Nanotechnol. 7, 11 (2012). 5 O.A. Williams, O. Douhéret, M. Daenen, K. Haenen, E. Osawa, and M. Takahashi, Chem. Phys. Lett. 445, 255 (2007). 6 J.N. Becker, J. Görlitz, C. Arend, M. Markham, and C. Becher, Nat. Commun. 7, 13512 (2016). 7 G. Balasubramanian, I.Y. Chan, R. Kolesov, M. Al-Hmoud, J. Tisler, C. Shin, C. Kim, A. Wojcik, P.R. Hemmer, A. Krueger, T. Hanke, A. Leitenstorfer, R. Bratschitsch, F. Jelezko, and J. Wrachtrup, Nature 455, 648 (2008). 8 O.A. Williams, J. Hees, C. Dieker, W. Jäger, L. Kirste, and C.E. Nebel, ACS Nano 4, 4824 (2010). 9 E. Neu, C. Arend, E. Gross, F. Guldner, C. Hepp, D. Steinmetz, E. Zscherpel, S. Ghodbane, H. Sternschulte, D. Steinmüller-Nethl, Y. Liang, A. Krueger, and C. Becher, Appl. Phys. Lett. 98, 1 (2011). 10 S. Heyer, W. Janssen, S. Turner, Y.G. Lu, W.S. Yeap, J. Verbeeck, K. Haenen, and A. Krueger, ACS Nano 8, 5757 (2014).

2:00 PM K3.02

Mapping N-V Centers in Individual Nanodiamond Particles Using High-Energy Resolution Electron Energy Loss Spectroscopy in a STEM Shery Chang¹, Katia March¹, Olga Shenderova² and Christian Dwyer¹; ¹Arizona State University, Tempe, Arizona, United States; ²Adamas Nanotechnologies, Inc., Raleigh, North Carolina, United States.

Nitrogen-vacancy centers (N-V) in nanocrystalline diamonds have been studied extensively for their interesting photoluminescence properties. Negatively-charged N-V centers (N-V) can emit visible light that is readily detectable at room temperature. Observation of N-V centers, particularly the optically active N-V, has becoming routine using optical fluorescence microscopy. Combined with AFM, the locations of the N-V as well as the particle size can be correlated. Other spectroscopy techniques including Raman and electron paramagnetic resonance (EPR) are also valuable in measuring different types of color centers in diamond. While much effort in the community has been geared toward better control of the surface structure and chemistry as well as the placement of the N-V centers, the questions remain as to what the effect of the host structure and the surface environment to the activation/deactivation of the N-V centers. To answer this question, a technique that is capable of detecting not only the presence of the N-V centers but also its location at near atomicresolution so that the relationship of the N-V center and its surrounding host structure can be elucidated. We have previously shown that core-loss electron energy loss spectroscopy (EELS) in the carbon K-edge has the pre-edge features that are associated with the presence of point defects. Using a combination of theoretical EELS spectra calculations, we were able to quantify the amount of point defects in the sample. While this is very useful, the life-time broadening in the core-loss spectra means that different types of N-V center may not be directly differentiated. Here we

demonstrate that we can now directly measure the signals arising from the N-V centers in individual nanoparticles. The measurement is based on high-energy resolution EELS in a scanning transmission electron microscope (STEM). Using the low-loss region of EELS, we can measure the electronic transitions within the nanodiamond bandgap to detect N-V centers. We used a STEM operated at low voltages (40-60 kV) with both high spatial (~0.1 nm) and high-energy (16 meV) resolutions. Such capability allows us to observe the inter-band transition at high spatial resolution without damaging to the structures of particles. Our initial results were demonstrated on ~70 nm nanodiamond particles synthesized by the HPHT methods. We found that a small fraction of particles within the sample show transitions corresponding to the presence of N-V centers. Interestingly, a relatively large fraction of particles do not exhibit these transition states. The reason, we then confirmed is due to the lack of nitrogen in the particle. These observations highlight the importance of dopant control for nanodiamond particles.

2:15 PM K3.03

Aspects of Particulate Nanodiamond as Applied to Host Systems for Enhancement of Their Properties Ehsan Languri², Farzin Marshali², Wayne Johnson², Lino Costa³, Kathleen Lansford³, Blake Branson¹, Dave Kerns¹ and Jimmy L. Davidson¹; ¹International Femtoscience Inc., Nashville, Tennessee, United States; ²Tennessee Technological University, Cookeville, Tennessee, United States; ³University of Tennessee Space Institute, Tullahoma, Tennessee, United States.

Particulate nanodiamond, aka detonation nanodiamond, has been explored for its utility as an additive to liquid and solid systems to enhance properties of said systems, such as improved thermal conductivity. Our work has shown that to achieve these improvements it is essential that the nanodiamond from its as-received condition must be cleaned, totally deaggregated, then functionalized for the appropriate host material if the benefits of particulate nanodiamond in a host system are to be realized. It is insufficient to simply break up, mill or otherwise mechanically treat the nanodiamond and then add it to the system; this does not achieve the dispersed, de-aggregated truly segregate nominal ~ 5 nanometer particle size and distribution that is essential to keep the FND permanently dispersed and enhance the host properties at small concentrations. We have prepared different functionalized nanodiamond, FND, samples, where the functional group is specifically intended to have its terminal chemical bonding couple with the host matrix such that the desired improvement in the host bulk, such as thermal conductivity or strength will be achieved. Because the effective diameter of molecular influence in the host matrix can be much greater than the 5 nanometers of the "core" nanodiamond, concentrations of FND in the range of ppm can result in double digit improvements of, for example, thermal conductivity or flexural strength. Examples of these FND applications will be presented. Furthermore, it is imperative that the nanodiamond particles' surface be chemically processed such that they are functionalized, that is the appropriate chemical (functional) groups are attached to the diamond nanoparticles which are compatible with and appropriately interact at the molecular level with the host system such as water, oils or polymers and related types of matrices. In this paper we will present examples of how appropriately functionalized nanodiamond combined with practical, in-use bulk materials, at proportions well below 1% of the host can significantly enhance the thermal and strength properties of liquid and solids (polymer) host systems and present and discuss the advantages derived from such treatments. For example, many cooling loop systems, be they for electronics or engines, are water based. We have examined an FND formulation and evaluated it in a precisely controlled and monitored water cooling loop which allows the direct determination of the heat transfer coefficient, a more practical parameter than thermal conductivity, and an improvement of over 20% was observed at FND concentration of 500pm. An appropriate FND formulation to enhance the polymers used in composites was applied and improvement in the flexural strength was observed. These evaluations and others, along with a detailed discussion of how FND can achieve these striking effects at very low additive concentrations, will be presented.

2:30 PM K3.04

Active Sites Determination and De-Aggregation of Detonation Nanodiamond Particles Sundas Iqbal; School of Material Science and Engineering, NPU China, Xian, China.

Acid-base titration method has been applied to detonation nanodiamond (DND) powder to determine active site functional groups attached on DND surfaces for de-aggregation of DND powders using different functionalization techniques. The UV/Ozone treated DND containing carboxylic group on its surface, tri-ethylene-tetra-amine (TETA) having ammine functionality, and ball milling treatment using ammonium bicarbonate as de-agglomerating and functionalizing media for ammine group attachment, are the functionalization techniques applied to DND powder. Functionalization and morphology of DND samples were characterized by XRD, SEM, RBS, ERDA and FTIR, while for measuring de-agglomeration and particle size distribution we performed dispersion tests and DLS of DND in three different solvent media. During investigation, the amount of carboxylic acidin the UV/Ozone functionalized DND sample has been observed to be 0.3 and 0.25 mM (milli moles) on the surface of DND powder with mole percentage of 1.28 and 2.86 % in the forward and backward titrations respectively. Similarly, the amount of ammine group (NH2)in TETA and ball-milling treatment has been observed to be 4 and 5 mM with mole percentages of 0.35 and 0.49 % respectively. Results showed that ball-milling treatment provides superior results and de-aggregation of DND powders.

2:45 PM K3.05

Top-Down Route to NV Fluorescent Nanodiamonds Using Detonation Vadym N. Mochalin¹, Ibrahim Abdullahi¹, Martin Langenderfer⁴, Nicholas Nunn³, Marco Torelli³, Catherine Johnson⁴, William Fahrenholtz² and Olga Shenderova³; ¹Chemistry, Missouri University of Science and Technology, Rolla, Missouri, United States; ²Materials Science and Engineering, Missouri University of Science and Technology, Rolla, Missouri, United States; ³Adamas Nanotechnologies, Raleigh, North Carolina, United States; ⁴Mining and Explosives Engineering, Missouri University of Science and Technology, Rolla, Missouri, United States.

Detonation is a technique for manufacturing 5 nm diameter nanodiamonds, which are known as detonation nanodiamonds. Although these nanodiamonds are believed to have numerous NV centers, only a small fraction of them are optically active. As a result, the vast majority of detonation nanodiamond particles remain non-fluorescent for reasons that are not completely clear. On the other hand, bright and stable optically active NV fluorescent centers can be produced by irradiating microcrystalline diamonds that are synthesized by high pressure, high temperature (HPHT) processes. Reduction of size of these microcrystalline diamonds by milling yields brightly fluorescent NV nanodiamonds with the sizes down to 10 nm. However, milling of microcrystalline diamonds is a long and expensive process. Here, we investigate the possibility of producing smaller NV fluorescent nanodiamonds by mixing commercially available NV microdiamonds with explosives and detonating the mixture in the conditions typical for detonation nanodiamond synthesis. In our experiments fractions of microdiamonds with average sizes 150um, 40um and 15um had been used. After purification of the detonation product from metal impurities and non-diamond carbon, we observed significant fragmentation of the starting micron-sized particles including fractions of particles below 1 micron. The behavior of NV fluorescent HPHT diamonds in detonation environments depending on their initial size and the potential to use detonation as a cheaper and faster way to reduce the size of NV fluorescent HPHT diamonds below 100 nm will be discussed.

3:00 PM BREAK

C2: Diamond Photonics/Spintronics II Session Chair: Philipp Reineck Tuesday Afternoon, May 22, 2018 Room: Humphreys

3:30 PM C2.01

Nanoplasmon Enhanced Diamond UV Photodetector Xiaohui Chang, Yan-Feng Wang, Zhangcheng Liu, Jiao Fu, Xiaofan Zhang, Shuwei Fan, Renan Bu, Jingwen Zhang, Wei Wang and Hongxing Wang; School of Electronics and Information Engineering, Wide Band Gap Semiconductors, Xi'an, China.

Diamond is a promising material for UV photodetector due to its outstanding properties, such as wide band gap (5.5eV), high thermal conductivity (22W/cm•K), high carrier mobility (electron:4500 cm²/V•s, hole:3800cm²/V •s) and high saturation velocity (1.5X10⁷cm/s). However, as the indirect bandgap semiconductor, diamond leads to significantly lower absorption coefficients compared to direct wide bandgap materials. Therefore, the responsivity of diamond-based UV photodetectors is difficult to achieve expected value. Nanoplasmonic structures can achieve enhancement of localized field through coupling of incident light and metal nanoparticles. Therefore, increase of responsivity is expected by enhancing optical absorption of incident photons within the semiconductor region near each nanoparticle. And enhancement effect increases with metal nanoparticles closer to the semiconductor layer. Palladium has usually been suggested as an alternative plasmonic material in the UV regions of the spectrum. In this work, a diamond UV photodetector with embedded Pd nanoparticles was fabricated on a CVD single crystal diamond film which was grown on Ib-type HPHT diamond substrate. Firstly, after growth of single crystal diamond film, the surface was changed from hydrogen termination to oxygen termination through boiling the sample in acid mixture. Secondly, about 10nm Pd film was deposited on the half of this sample surface by electron beam deposition technique. Thirdly, the sample was annealed in the N₂ ambience at 800°C for 1h. Finally, Ti/Au interdigital electrodes were patterned on Pd area and no Pd area respectively by electron beam evaporation technique. The morphologies of Pd nanoparticles and the quality of diamond film were investigated. The sample's photoelectric properties also have been evaluated. The detail will be presented in the conference.

3:45 PM C2.02

Hybrid Photovoltaics—Covalent Functionalization of Boron-Doped Diamond via Sonogashira Cross-Coupling Jorne Raymakers^{1, 2}, Hana Krysova³, Anna Artemenko⁴, Shannon S. Nicley^{1, 2}, Jan Čermák⁴, Pieter Verstappen¹, Daria Miliaieva⁴, Sam Gielen¹, Alexander Kromka⁴, Ladislav Kavan³, Bohuslav Rezek^{4, 5}, Wouter Maes^{1, 2} and Ken Haenen^{1, 2}; ¹Institute for Materials Research (IMO), Hasselt University, Diepenbeek, Belgium; ²IMOMEC, IMEC vzw, Diepenbeek, Belgium; ³J. Heyrovsky Institute of Physical Chemistry, CAS, Prague, Czechia; ⁴Institute of Physics, CAS, Prague, Czechia; ⁵Faculty of Electrical Engineering, Czech Technical University in Prague, Prague, Czechia.

As recently shown, boron-doped nanocrystalline diamond electrodes are promising alternative photocathode materials for p-type dye-sensitized solar cells (DSSCs) [1]. Their excellent stability, good transparency in the visible and near-infrared region and high hole mobility gives them a clear advantage over the currently employed NiO electrodes. Additionally, the possibility to covalently functionalize diamond surfaces can provide a more stable interface between the electrode and the light-absorbing material, resulting in a more robust device [2]. Unfortunately, the generated currents for p-type diamond based DSSCs are still several orders of magnitude lower as compared to state-of-the-art NiO photocathodes. These low currents can mainly be ascribed to a poor surface coverage, which is partly related to the relative chemical inertness of the diamond surface. Recently, we have expanded the scope of diamond surface chemistry via careful investigation of the Sonogashira crosscoupling, with special emphasis on both the surface coverage and possible remaining contamination [3]. Here, the previously optimized two-step functionalization approach, consisting of the diazonium grafting and subsequent Sonogashira cross-coupling, is employed to attach a novel

alkyne functionalized dithienopyrrole-benzothiadiazole light-harvesting small molecule to heavily B-doped nanocrystalline diamond films. 150 nm thick films were grown on Si p-type conductive substrates by microwave plasma enhanced CVD, while similar films were grown on a Au mirror on Corning Eagle glass allowing subsequent FTIR analysis. The chemical reaction conditions were optimized to maximize the surface coverage, as it was observed from X-ray photoelectron spectroscopy and photoelectrochemical measurements that this is directly related to the generated currents. Additionally, the Sonogashira reaction was compared with functionalization of azidophenyl terminated diamond films via the well-known Cu(I) catalyzed azide-alkyne cycloaddition (CuAAC) to determine the influence of the linker on charge separation and thus the photocurrent generation. References [1] H. Krysova, L. Kavan, Z. Vlčková Zivcová, W.S. Yeap, P. Verstappen, W. Maes, K. Haenen, F. Gao, C.E. Nebel, RSC Advances 5 (2015), 81069-81077. [2] W.S. Yeap, X. Liu, D. Bevk, A. Pasquarelli, L. Lutsen, M. Fahlman, W. Maes, K. Haenen, ACS Applied Materials & Interfaces 6/13 (2014), 10322-10329. [3] J. Raymakers, A. Artemenko, S.S. Nicley, P. Štenclová, A. Kromka, K. Haenen, W. Maes, B. Rezek, Journal of Physical Chemistry 121 (2017), 23446-23454

4:00 PM C2.03

Responsivity Improvement of Diamond UV Photodetector with Novel Device Design Zhangcheng Liu¹, Dan Zhao¹, Jin-Ping Ao², Xiaohui Chang¹, Yan-Feng Wang¹, Jiao Fu¹, Minghui Zhang¹ and Hongxing Wang¹; ¹Xi'an Jiaotong University, Xi'an, China; ²Tokushima University, Tokushima, Japan.

Deep ultraviolet (DUV) photo detection is becoming increasingly important for its variable applications such as flame detection, engine monitoring, chemical sensing, and intersatellite communications. Among many wide bandgap semiconductor materials used to fabricate DUV photodetectors, diamond is a promising candidate owning to its high carrier mobility, high thermal conductivity (22 W/cm), low dielectric constant (~5.7) and high radiation hardness. The researches of diamond DUV photodetector are mainly focused on film quality and device design. For device design, there exists a trend to fabricate three dimensional structure to optimize the electric field distribution in the bulk. J. Forneris et al and Kang Liu et al applied groove-shaped electrode structure with metal electrodes parallel to diamond strips to enhance the device performance. In this work, DUV photo detectors with electrodes perpendicular to diamond strips were fabricated with a much easier process, in which quasi one dimensional carrier transport might be realized. A thin layer of diamond epitaxial layer was firstly grown on the high-pressure high-temperature single crystal diamond substrate. Then, ICP etching method was used to fabricate diamond strips on the surface. Sequentially, a second thinner diamond film was grown on the processed sample. Before and after the second growth step, photodetectors were fabricated on the sample with interdigitated electrodes vertical to the diamond strips. Compared with the planar structure detectors, the novel detectors own higher responsivity. The details will be presented at the conference.

4:15 PM C2.04

High-Temperature Solar Cells Based on Black Diamond Technology Daniele M. Trucchi¹, Alessandro Bellucci¹, Marco Girolami¹, Matteo Mastellone¹, Stefano Orlando¹, Andrea Orsini³, Riccardo Polini², Stefano Salvatori³ and Veronica Valentini¹; ¹Institute for Structure of Matter, National Research Council of Italy, Monterotondo (RM), Italy; ²Department of Chemical Sciences and Technologies, Univ di Roma Tor Vergata, Roma, Italy; ³Faculty of Engineering, Università degli Studi Niccolò Cusano, Rome, Italy.

High-temperature solar cells are possible by exploiting the Photon-enhanced Thermionic Emission (PETE) concept, which represents a novel and very attractive mechanism for the exploitation of solar radiation, especially if concentrated, and characterized by promisingly high conversion efficiency (>50%). PETE converters rely on the concept that engineered semiconductor photocathodes can provide a very efficient electron emission, induced by hot-electrons produced by photons with sufficient energy, combined to a thermionic emission, sustained by the high temperatures induced by every other thermalization process. Surface nanotexturing combined to surface-hydrogenation, aimed at achieving negative electron affinity conditions and a work function as low as 1.7 eV

with a nitrogen-doping of the emitting-layer, are here proposed as a radically new and potential effective PETE cathode completely based on chemical-vapour-deposited (CVD) diamond, able to operate up to temperatures of 800 °C. CVD diamond is transparent to solar radiation due to its wide bandgap, consequently black diamond technology was developed for drastically increase its absorption coefficient and photogeneration capability under sunlight irradiation¹. The final p/i/n structure merges the technologies of surface texturing by fs-laser2, boronimplantation for formation of buried p-type layer3, and laser-induced graphitic microchannels4, to form an innovative defect-engineered black diamond cathode for the conversion of concentrated solar radiation operating at high temperature. Results under high-flux solar simulator will be reported and discussed by demonstrating for the first time the PETE effect at temperatures from 350 to 550 °C. References 1. P. Calvani, A. Bellucci, M. Girolami, S. Orlando, V. Valentini, R. Polini, and D. M. Trucchi, "Black Diamond for Solar Energy Conversion", Carbon 105 (2016) 401-407. 2. A. Bellucci, P. Calvani, M. Girolami, S. Orlando, R. Polini, and D. M. Trucchi, "Optimization of Black Diamond Films for Solar Energy Conversion", Applied Surface Science 380 (2016) 8-11. 3. A. Bellucci, P. Calvani, M. Girolami, V. Carcelen, S. Michaelson, A. Hoffman, and D. M. Trucchi, "Buried Boron-Doped Layer for CVD Diamond Photo-Thermionic Cathodes", IEEE Transactions on Nanotechnology 15 (2016) 862-866. 4. M. Girolami, L. Criante, F. Di Fonzo, S. Lo Turco, A. Mezzetti, A. Notargiacomo, M. Pea, A. Bellucci, P. Calvani, V. Valentini, and D. M. Trucchi, "Graphite distributed electrodes for diamond-based photon-enhanced thermionic emission solar cells", Carbon 111 (2017) 48-53.

4:30 PM C2.05

Layer Structure and Characterization of a Diamond Based Thermionic Electron Emitter Sergey Elfimchey, Yulia Stotsky, Rozalia Akhvlediani and Alon Hoffman; Schulich Faculty of Chemistry, Technion-Israel Institute of Technology, Haifa, Israel.

The purpose of this work is the construction of a diamond based electrode for thermionic electron emission (TEE). Nitrogen-doped polycrystalline diamond films were deposited on molybdenum substrates by a hot filament CVD (HFCVD) method. We investigated diamond films with different morphology and nitrogen content produced by variation of the NH₂/CH₄/ H, gas mixture ratio, substrate temperature and gas pressure. It was found that deposition conditions have a strong effect on the effective electrode work function and TEE yields. To improve the electrical contact and the electron transport between molybdenum substrates and nitrogen-doped diamond films we used ultra-nanocrystalline diamond (UNCD) interlayer. The optimal deposition conditions for UNCD interlayer were studied in this work. In addition, we examined different molybdenum surface pretreatments to improve TEE yields. The produced diamond electrodes were investigated by HRSEM, Raman, XPS, UPS spectroscopies and 4PP measurements. The TEE curves were measured at ultra-high vacuum conditions in the 300-600°C temperature as a function voltage.

4:45 PM C2.06

Fabrication of Compact Microlens Arrays <u>Tian-Fei Zhu</u>, Jiao Fu, Zongchen Liu, Zhangcheng Liu, Yan-Feng Wang, Guoqing Shao, Dan Zhao, Xiaohui Chang, Shaopeng Zhang, Yan Liang, Juan Wang and Hongxing Wang; Xi'an Jiaotong University, Xi'an, China.

Fabrication of microlens arrays with high fill factor in chemical vapor deposited single crystal diamond is described. An optimized photoresist reflow and inductively coupled plasma etching processes are used to realize compact diamond microlens arrays. First, photoresist pillars developed by photolithography are reflowed on hotplate. The effects of reflow temperature, time and photoresist thickness on occupancy ratio of photoresist spherical segments are investigated. Second, via dry etching, compact diamond microlens arrays are fabricated by transferring the spherical segment pattern into substrate. Eventually, the compact diamond microlens arrays demonstrate uniform arrangement and good optical performance with projection experiment

F1: Carbon Nanotubes Session Chair: Eleftheria Roumeli Tuesday Afternoon, May 22, 2018 Room: Agassiz/Fremont

3:30 PM F1.01

Covalent Bond Formation Between Collapsed Carbon Nanotubes Using ReaxFF Molecular Dynamics Simulations Benjamin Jensen¹, Jae-Woo Kim², Godfrey Sauti¹, Kristopher Wise¹, Emilie Siochi¹, Liang Dong³ and Haydn Wadley³; ¹NASA Langley Research Center, Hampton, Virginia, United States; ²National Institute of Aerospace, Hampton, Virginia, United States; ³University of Virginia, Charlottesville, Virginia, United States.

Despite the well-known high mechanical properties of individual carbon nanotubes (CNTs), bulk CNT formats have much lower properties because the weak van der Waals interactions between tubes permit nanotube sliding under load. Covalent bonds are more robust linkages enabling more effective load transfer across the tubes. To maximize the mechanical properties, these inter-CNT bonds must be distributed throughout the material at a density sufficient to prevent tube sliding and telescoping, while minimizing disruption of the CNT structure. This work used molecular dynamics simulations on simplified CNT structures to identify conditions that promote formation of covalent bonds, and to analyze the structures that result from the bond formation process. Reactive molecular dynamics simulations using the ReaxFF_{C-2013} parameter set were used to model atomic interactions and bond formation between 8 nm diameter double-walled CNTs with collapsed 'dog bone' cross sections. For each system, the pressure was increased along isotherms ranging from 1000 to 3500 deg C. Snapshots of the system were quenched to room temperature and zero pressure to permit analysis of equilibrium structural features. The results indicate that high temperature and pressure can induce direct covalent bonding between adjacent CNTs and that the high curvature regions of the collapsed tubes react at lower pressures than planar regions.

3:45 PM F1.02

High Strength Macroscale Carbon Nanotube Structures by Covalent Bond Formation Jae-Woo Kim¹, Godfrey Sauti², Benjamin Jensen², Kristopher Wise², Emilie Siochi², Liang Dong³ and Haydn Wadley³; ¹NASA LaRC/NIA, Hampton, Virginia, United States; ²NASA LaRC, Hampton, Virginia, United States; ³University of Virginia, Charlottesville, Virginia, United States.

Carbon nanotubes (CNTs) have been shown to possess excellent mechanical properties at the nanoscale level. However, after scale-up to formats such as CNT sheets and yarns, the mechanical properties are significantly lower due to poor load transfer within tubes (wall to wall), between tubes in bundles, and between bundles of tubes. Based on guidance from computational modeling, research was conducted to create covalent bonds between CNTs in an effort to enhance load transfer in CNT based structural materials. In this study, spark plasma sintering (SPS) was used to obtain pressure and temperature conditions likely to produce tubeto-tube bonding in macroscale CNT sheets. Various temperatures (1000 $\sim 1500~^{\circ}\text{C})$ and processing pressures (0 $\sim 60~\text{MPa})$ were applied to CNT sheets under pulsed dc electric current, where the sheets were compressed between two graphite dies in an argon atmosphere. Raman spectroscopy, x-ray diffraction, and microscopic data revealed the formation of cubic diamond and nano-diamond domains, depending on the conditions applied in the SPS experiments. These results indicate that local conditions at the CNT/CNT junctions are capable of inducing covalent bond formation. Mechanical testing of SPS-processed CNT sheets suggests that these bonds contribute to enhanced mechanical properties, especially in densely packed and highly aligned CNT sheets.

4:00 PM F1.03

Study of Carbon Nanotube Absorption by Fractal Analysis of Catalyst Nanoparticles and Carbon Nanotubes <u>Adam Pander</u>^{1, 2}, Fumiya Nagamine¹, Sachio Hayashi¹, Keisuke Takano³, Makoto Nakajima⁴, Akimitsu Hatta^{1, 2} and Hiroshi Furuta^{1, 2}; ¹Kochi University of Technology, Kami, Japan; ²Center for Nanotechnology, Kami, Japan; ³Shnishu University, Nagano, Japan; ⁴Osaka University, Osaka, Japan.

A dependence between a light absorption of carbon nanotube (CNT) forest and its properties, such as height, density, and alignment, is generally well known. In CNT forest, local regions of higher and lower density of CNTs exist and are the main reason behind the variation of the total absorption throughout the forest. Local fluctuations of the formation process of catalyst nanoparticles result in the variation of the agglomeration of a catalyst material and give rise to a diverse distribution of catalyst sizes and densities throughout the individual sample. Under certain conditions, the formation of small catalyst particles occurs, implying a smaller distance between each grain, which results in a higher area density. On the contrary, the agglomeration of bigger nanoparticles requires more of the catalyst material, therefore, the distance between individual particles increases. The size and density of catalyst particles are closely correlated to the diameter and density of CNTs within the forest1. With the increase of the density of CNT forest, higher vertical alignment is usually observed, which leads to the increase of the light absorption. Despite this fact, the self-correlation between the distribution of low and high-density areas in CNT forest, and the local absorption of light remains unknown. For that reason, in order to quantify the degree of CNT forest density and resultant absorbance, a scale dependent parameter named fractal dimension was utilized. Fractals are complex geometric designs that repeat themselves or their statistical properties on many scales, and because of that are "self-similar". Also in CNT field, fractal geometry was used for the quantitative description of the morphology of CNTs and derived materials 2. This approach was adapted before to study the conductance and thermoelectric properties of CNTs prepared by the vaporization of graphite by the plasma discharge, and also to investigate the adsorption of polyacrylic acid on a modified CNT surface. Furthermore, the self-similar organization of CNTs, in which SWNTs form into bundles, bundles into wires, and then into fibers ³, was also studied. The main purpose of this work is to investigate the self-correlation and self-similar organization between size and density of catalyst nanoparticles and CNTs, and the local light absorption. In the presented study, the fractal dimension is measured and the fractality is determined. The density of CNTs was characterized by scanning electron microscopy (SEM), while the size and density of catalyst particles were investigated by atomic force microscope (AFM). The distribution of the area-dependent light absorption was measured using area mapping by micro-Raman spectroscopy and the FT-IR microscope. The study of the self-correlation and the variation between areas with different density offers a new and important explanation of the diversity of optical properties CNT forest. This work was supported by KAKENHI No. 17K06205, 24560050, and the joint research project of Inst. of Laser Eng., Osaka University (2016B1-FURUTA, 2017B1-FURUTA). 1. Pander, A., Hatta, A. & Furuta, H., Appl. Surf. Sci. 371, 425-435 (2016). 2. Smajda, R., Kukovecz, Á., Kónya, Z. & Kiricsi, I., Carbon N. Y. 45, 1176-1184 (2007). 3. Sun, C.-H., Li, F., Ying, Z., Liu, C. & Cheng, H.-M., Phys. Rev. B 69, 33404 (2004).

> EP2: Innovation and Entrepreneurship Session II Session Chair: Jeffrey T. Glass Tuesday Afternoon, May 22, 2018 Room: Humphreys

5:00 PM *EP2.01

Diamond High Power Devices Commercialization Manpuneet
Benipal, Anna Zaniewski and Robert J. Nemanich; Advent Diamond Inc,
Scottsdale, Arizona, United States.

Advent Diamond is mobilizing years of research based at Arizona State University to make single crystal diamondbased electronics. Recent breakthroughs in growing phosphorous-doped layers and Ohmic contacts

^{*} Invited Paper

enable the commercial development of high-power diamond diodes. These and future diamond-based electronic components can enable the more efficient operation of motors, power grids, electric vehicles, charging stations, geothermal/oil and gas drilling, power processing units for space applications and radiation detectors. Because of its wide bandgap, high breakdown field and high mobility, diamond electronics can handle high power, and are robust to both high temperatures and the high radiation environment encountered in space, making them ideal for applications in harsh environments or where cooling would be impractical. Advent is the recipient of an NSF Small Business Innovation and Research grant for the development of freewheeling diodes.

PS2: Poster Session II Tuesday Afternoon, May 22, 2018 5:30 PM - 7:30 PM Room: Doyle/Rees

PS2.01

Nitrogen and Hydrogen Distribution in Delta-Doped Diamond Films Mohammed Attrash, Mohan Kumar Kuntumalla and <u>Alon Hoffman;</u> Schulich Faculty of Chemistry, Technion – Israel Institute of Technology, Haifa, Israel.

Nitrogen delta-doped diamond layers can be fabricated by exposing diamond substrate to N, radio frequency (RF) plasma, followed by growing a diamond capping layer. Recently it was found that on preferential retention of hydrogen is occurs within the volume where the nitrogen atoms are localized. Here we investigate the hydrogen and nitrogen distribution and bonding in nitrogen delta-doped diamond layers produced by thin method. The influence of grain boundaries on nitrogen distribution in polycrystalline diamond films was studied. Delta layers were fabricated on single crystal (100) diamond substrate (i.e., no grain boundaries) and polycrystalline diamond thin films with different grainboundary densities onto the front and back surfaces of the films. X-ray photoelectron spectroscopy (XPS) was used to study the nitrogen chemical states present on the N₂ RF plasma treated diamond surface in-situ. To measure the nitrogen concentration and hydrogen TOF-SIMS was used. The concentration of nitrogen is of order of 10¹⁹ cm⁻³ in nitrogen deltadoped layers. The depth profile (FWHM) of nitrogen distribution in diamond films of front surface (R_{rms} =34.6 nm) and back surface (R_{rms} =6.9 nm) are 32.3 nm and 24.6 nm respectively. From our study it is concluded that the source of hydrogen is from the capping layer deposition process.

PS2.02

Grain-Orientation Dependence of Anodic Corrosion Effects in Boron-Doped Diamond Francesca Celine I. Catalan¹, Norihiko Hayazawa¹, Yasuyuki Yokota¹, Raymond A. Wong¹, Takeshi Watanabe², Yasuaki Einaga³, ⁴ and Yousoo Kim¹; ¹Surface and Interface Science Laboratory, RIKEN, Wako, Japan; ²Department of Electrical Engineering and Electronics, Aoyama Gakuin University, Sagamihara, Japan; ³Department of Chemistry, Keio University, Hiyoshi, Japan; ⁴ACCEL, Japan Science and Technology Agency, Tokyo, Japan.

Recently, boron-doped diamond (BDD) has been extensively studied for its exceptional properties and potential as an efficient electrode. Previous reports, however, have shown that significant degradation in surface morphology¹, and electrochemical performance and stability² can occur in BDD films especially in high current density anodic treatments. Here, we describe the chronological progression of corrosive degradation in highlydoped polycrystalline BDD film electrodes under high current density anodic conditions. The morphological and chemical properties of BDD were systematically analyzed as the electrolysis time is increased up to 12 hours. High-resolution, grain-dependent anodic corrosion was observed for the first time in polycrystalline BDD using polarized Raman spectroscopy and Raman spectral imaging. As confirmed by X-ray Photoelectron Spectroscopy (XPS), high-resolution Raman analysis suggests that degradation starts via the graphitization of sp³ carbon on the BDD surface by methyl radicals (CH $_3$). Further reactions with hydroxyl (OH) creates carbonyl groups specifically on (100)-grain facets and (111)grain edges. Raman spectral imaging showed that as the sp³ carbon atoms are etched from the surface, sub-surface boron dopants are exposed and subsequently detached from the lattice as the BDD undergoes longer anodic treatment. The observed grain-orientation dependence of the corrosion process in BDD offers new and important insights into the chemical interactions occurring on the BDD surface during electrochemical treatments, allowing for future works to be geared towards minimizing degradation without sacrificing BDD electrode efficiency. [1] Kashiwada, T., et al, *ACS Appl. Mater. Interfaces* **8** (2016), 28299-28305. [2] Chaplin, B., et al, *Electrochim. Acta* **89** (2013), 122-131. [3] Catalan, FCI, et al, *J. Phys. Chem. C* **121** (2017), 26742-26750.

PS2.03

Decoration of Growth Sector Boundaries with Single Nitrogen Vacancy Centres in As-Grown Single Crystal HPHT Synthetic Diamond Phil L. Diggle^{1, 3}, Ulrika F. D'Haenens-Johansson², Ben L. Green^{1, 3}, Chris M. Welbourn¹, Wuyi Wang², Andrey Katrusha⁴ and Mark E. Newton^{1, 3}; ¹University of Warwick, Coventry, United Kingdom; ²Gemological Institute of America, New York City, New York, United States; ³Diamond Science and Technology, Coventry, United Kingdom; ⁴New Diamond Technology, St. Petersburg, Russian Federation.

It is well known that the morphology of diamonds produced by high pressure high temperature (HPHT) synthesis in a metal solvent is in a large part determined by the relative development of the {100} and {111} faces, however relatively large {113}, {110}, and {115} growth sectors can be found in material grown with nitrogen "getters" in order to produce relatively pure diamond. It is also understood that the morphology of synthetic diamond crystals grown from metal-carbon systems changes from cube to octahedron with increasing temperature and that the orientation of the seed initiation surface can also significantly affect the morphology of the grown crystals. Furthermore, the major impurities, nitrogen and boron, are heterogeneously incorporated in the different growth sectors. New Diamond Technology (NDT) have demonstrated that large (lateral dimension > 10 mm), relatively pure (nitrogen and boron impurities less than 0.1 ppm in some growth sectors), low birefringence HPHT diamonds can be routinely produced. We report on confocal photoluminescence microscopy studies of plates cut from HPHT diamonds grown by NDT. We show that the growth sector boundaries between {001} and either {113} or {111} sectors are decorated with negatively charged, nitrogen vacancy (NV-) defects. Furthermore we observe a dependence on the distribution based on the type of interface, such that single NVs can be seen at the {001}/{111} interface and ensembles only at the $\{001\}/\{113\}$ interface. The bulk $\{001\}$ growth sector contains essentially no NV defects and there is no decoration involved for an interface involving the {011} growth sector. The optical and spin properties of these NV- defects have been studied by optically detected magnetic resonance (ODMR) spectroscopy, and these data will be reported. ODMR studies reveal that there is no preferential orientation of the incorporated NV- defects. The production and distribution of NVdefects at growth sector boundaries will be discussed. Furthermore, we identify that negatively charged silicon vacancy (SiV-) and the 1.4 eV nickel centres are present in both {111} and {113} growth sectors in agreement with previously presented results1. The presence and distribution of these defects will be discussed. References 1. W. Wang, U. F. S. D'Haenens-Johansson, L. Loudin, and P. Johnson, 'Distributions of the [Si- V-] Defect in Diamonds', SBDD XXI Hasselt (2016)

PS2.04

Phosphorus-Doped Diamond Films by Red Phosphorus Evaporation Sergey Elfimchev¹, Anatoly Bekkerman¹, Yulia Stotsky¹, Tatyana Kravchuk² and Alon Hoffman¹; ¹Schulich Faculty of Chemistry, Technion-Israel Institute of Technology, Haifa, Israel; ²Solid State Institute, Technion-Israel Institute of Technology, Haifa, Israel.

A novel simple method for phosphorus-doped diamond films deposition by hot filament CVD (HFCVD) method is presented in this work. Red phosphorus was thermally evaporated and transferred inside the HFCVD chamber by hydrogen flow. The amount of phosphorus was controlled by the source temperature and additional pre-heating of gas mixture. The creation of phosphine gas was detected by mass spectrometry measurements. High concentrations of phosphorus incorporated into polycrystalline and single crystal diamonds was detected by SIMS measurements. The deposited samples were also investigated by high resolution SEM, Raman and photoluminescence spectroscopies. The

method described in this work for phosphorus-doped diamond deposition is controllable, safe and cheap.

PS2.05

Application of a Boron Doped Diamond Electrode Stack for Remediation of Single and Mixed Perfluoroalkyl Compounds in Water Mary Ensch^{1, 2}, Cory Rusinek², Michael Becker² and Thomas Schuelke^{2, 3}; ¹Chemical Engineering and Materials Science, Michigan State University, Haslett, Michigan, United States; ²Fraunhofer USA Center for Coatings and Diamond Technologies, East Lansing, Michigan, United States; ³Electrical and Computer Engineering, Michigan State University, East Lansing, Michigan, United States.

Perfluoroalkyl compounds (PFAS) are persistent in the environment and are toxic to humans, making them a priority in wastewater treatment. Two of the main compounds detected around the world are the two eight carbon chain compounds, perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS) [1,2]. These compounds are man-made and were manufactured in various consumer and industrial products allowing for their release in the air, water, and soil throughout the early 1950s. The EPA has since set a drinking water advisory level of 70 parts per trillion (ppt) for PFOA and PFOS, both individually and combined [3]. Replacements for these compounds have been shorter chain PFAS such as the four carbon chain compounds, perfluorobutanoic acid (PFBA) and perfluorobutane sulfonate (PFBS). However, these shorter chain compounds are still non-biodegradable and are accumulating in the environment. Some of the most recent reports even have reported PFBA and PFBS at higher levels then PFOA and PFOS at this time. Boron doped diamond (BDD) has proven to be an excellent electrode material in early investigations of pollution remediation. BDD has a high over potential for oxygen generation which allows for the creation of hydroxyl radicals and has shown the ability to degrade some of the PFAS compounds in the research literature [1,2]. To better understand the process requirements to degrade these compounds, more studies need to be done with varying current densities and different mixtures of initial components. In doing so, a better understanding of required current densities will be obtained and the knowledge of if the current needs to be increased significantly for shorter chain compounds. Insight into the required current densities for various compounds will lead to the ability to optimize a process based on the initial concentrations of the components in a given water sample. This study aims to investigate the effects of current density on the degradation of individual compounds and mixtures using a BDD electrode stack comprised of two double side coated anodes and three double sided cathodes. The working electrode surface area is roughly 110 cm². In such, five current densities, 5-70 mA/cm², were evaluated for PFOA, PFOS, PFBS, PFBA individually and for varying mixtures of the four. Studies were analyzed with a fluoride ion selective electrode (ISE) to find optimized process parameters. The ISE works by measuring a membrane potential for a specific analyte, such as F-, and provides a logarithmic relationship between the measured potential and the concentration of the ion in solution. This allows for the determination of the amount of free Fions as the degradation of the PFAS proceeds. From these studies, reaction order and each compound's reaction rate, at each current density, was determined. pH and temperature changes throughout each run were also monitored. Throughout the 8-hour degradation process, the pH for each solution has been between 5-11 and increased as time progressed. The temperature in the lowest current density study increased to an average of 30 °C whereas the highest current density study reached final temperatures of up to 70 °C. Early results indicate that higher current densities have a significant advantage in the early stages of the degradation process, providing over a 50% increase in the F- concentration obtained in the first hour when comparing the lowest and highest current densities for PFOA. Higher current also showed a benefit when degrading PFOS in the first hour, although PFOS has shown to be more difficult to degrade than PFOA. [1] Schaefer, C. E., et al. Chemical Engineering Journal 317 (2017) 424-432 [2] Qiongfang, Z., et al. Electrochimica Acta 77 (2012) 17-22 [3] Environmental Protection Agency. Web. Basic Information About Per- and Polyfluoroalkyl Substances (PFAS) (2018)

PS2 06

A Theoretical Study of Negative Electron Affinity from Aluminium on the Diamond Surface Michael James^{1, 2}, Paul W. May¹ and Neil Allan¹; ¹University of Bristol, Bristol, United Kingdom; ²Bristol Centre for Functional Nanomaterials, Bristol, United Kingdom.

The ability to form a negative electron affinity (NEA) surface on diamond is of interest for electron emission applications. These surfaces form when atoms electropositive relative to carbon terminate the surface. Simple H-termination gives NEA but hydrogen desorption occurs at elevated temperatures (>700 °C).1 This makes H-terminated diamond unsuitable for high-temperature applications. Previous work has studied NEA surfaces generated by termination from group I, II and transition metals (TMs).²⁻⁴ Larger group I metals do not have thermal stability but smaller group I and II metals, such as Li and Mg, show promise. Computational work on various first-row TMs including Cu, Ni, Ti and Zn, suggest carbideforming TMs give larger NEA and experimental work has shown <10 Å thin films of various TMs can give NEA.Density functional theory calculations were performed to simulate the addition of up to 1 ML of aluminium on the diamond surface. Aluminium is a carbide-forming metal that bonds sufficiently well to be used for Ohmic and Schottky contacts,5 and here we show it is of interest for NEA applications as well. For all metals on diamond, different bonding sites can affect electron affinity (EA), sometimes showing positive EA. Here we show sites can be differentiated by ionic or covalent behaviour. For certain Al coverages, for example 0.25 ML coverage on the (100) O-terminated surface, we find adsorption energies were observed to be larger than for H-termination whilst maintaining a NEA. References 1. W. F. Paxton, M. Howell, W. P. Kang and J. L. Davidson, J. Vac. Sci. Technol. B, Nanotechnol. Microelectron. Mater. Process. Meas. Phenom. 30, (2012), 21202. 2. K. M. O'Donnell, T. L. Martin, N. A. Fox and D. Cherns, Phys. Rev. B. 82, (2010), 115303. 3. K. M. O'Donnell, T. L. Martin and N. L. Allan, Chem. Mater. 27, (2015), 1306–1315. 4. A. K. Tiwari, J. P. Goss, P. R. Briddon, a. B. Horsfall, N. G. Wright, R. Jones and M. J. Rayson, EPL Europhysics Lett. 108, (2014), 46005. 5. D. A. Evans, O. R. Roberts, A. R. Vearey-Roberts, D. P. Langstaff, D. J. Twitchen and M. Schwitters, Appl. Phys. Lett. 91, (2007), 132114.

PS2.07

Electron Spectroscopy Investigation of Nitride Polycrystalline Diamond Surfaces by RF (N₂) Plasma Mohan Kumar Kuntumalla, Mohammed Attrash, Fengnan Li and Alon Hoffman; Schulich Faculty of Chemistry, Technion - Israel Institute of Technology, Haifa, Israel.

In this work, we demonstrate a simple technique to induce adsorption of nitrogen onto diamond surfaces with low levels of damage. This was achieved by exposing the surfaces to N₂ RF plasma. Nitridation process has been carried out for different N, plasma exposure time (10, 20 and 30 minutes) and pressure ($3x10^{-3}$ and $7x10^{-3}$ torr) at plasma power of 36 W. A neutralizer was placed at the exit of the plasma source to decrease the density of the high energetic nitrogen species in the plasma arriving at the surface. The structural, chemical and electronic properties of nitrided (asprocessed, annealed and hydrogenated) diamond surfaces were evaluated by using HREELS, XPS and UPS, respectively. It is observed that the structural quality of diamond surfaces can be optimized by using a neutralizer during the nitridation process. XPS and HREELS measurements confirmed the nitrogen incorporation into the diamond surfaces. The HREELS analysis of nitrided diamond surfaces suggest that the plasma processing pressure and the exposure time play a crucial role in controlling surface structural quality. The presence of the optical phonon (300 meV), which is a signature feature of well-defined and defects-free diamond surface; confirm the structural quality of nitrided surfaces. Hydrogenation of annealed surfaces revealed that the surface structural recovery is possible with low level of structural damage.

PS2.08

Evaluation of Highly B Doped HPHT Diamond Crystallinity by Electron Backscatter Diffraction Akio Matsushita, Koji Ashida, Daichi Dojima, Kazunori Koide, Yuki Tsuchida, Tadaaki Kaneko and Shinichi Shikata; Physics, Kwansei Gakuin University, Sanda, Japan.

Low resistivity p+ substrate is essential to realize high power diamond device for energy saving power electronics. Highly B doped p+ high pressure and high temperature (HPHT) substrate is receiving much

attention as a seed crystal for low resistivity p+ substrate growth, because of the small difference of lattice mismatch and doping concentration, compared with an insulating HPHT seed. However, due to small quantities in the current market, the commercially available p+ HPHT substrate is still under the development associated with B incorporation during HPHT growth. According to the recent reports [1,2], four [111] growth sectors can be clearly seen in the p+ substrate by RAMAN mapping and X ray topography measurements, with the large variations of crystalline quality and B incorporation [3]. In this paper, we focus on the crystallinity of the growth sectors and report on the evaluation of crystal orientation by electron backscatter diffraction (EBSD) method. Three commercially available B doped p+ HPHT (001) single crystal substrates (3.0mm \times 3.0mm) having a doping concentration from 5 to 9×10^{19} cm⁻³ were used. High performance EBSD system (Zeiss MERLINTM) is used to perform quantitative crystallographic analysis to investigate growth sector boundaries. The incident electron beam of 20 keV was introduced into the specimen tilted 70 degree from horizontal axis. In this configuration, the maximum penetration depth of electron is about 900nm, however, the electrons scattered from the diamond layer down to 70nm below the surface are estimated to contribute to Kikuchi pattern information. By the EBSD method, the lattice distortion can be captured by analyzing the pattern change as tensor of normal, shearing strain and lattice rotation. Four boundaries regions of four growth sectors for p+ HPHT substrate were measured and observed following imperfect crystallographic disorders. First, 40 µm² area across the boundary between the [-1-11] and [1-11] growth sector was measured. It was found the [001] axis of [1-11] growth sector was rotated from [001] to [-1-10] direction compared to [-1-11] growth sector for 0.02degree. The same measurements were carried out along the sector boundary, and same phenomena were found in several more places. No tendency of rotation was observed along the boundary, and the highest rotation angle of 0.05 degree was measured. These rotations of the crystal axis were observed for all four sector boundaries. For other boundary, the [001] axis of [-111] growth sector was rotated from [001] to [-1-10] compared to [-1-11] sector, the [001] axis of [1-11] growth sector was rotated from [001] to [-110] compared to [111] sector, and the [001] axis of [-111] growth sector was rotated from [001] to [1-10] compared to [111] sector. This fact shows "grain boundaries" are formed for p+ HPHT substrate. The measurements were carried out for another two substrates, and the same phenomena were found for the second substrate. Third substrate was found to be perfect single crystal without any grain boundaries. In our previous study, the SBDs fabricated on boundaries have relatively lower reverse-bias voltages compared to devices fabricated on no dislocation regions, seemingly have poor crystal qualities.[4] These phenomena presumably due to the "grain boundaries" observed by EBSD in this paper. Since the p+ crystal growth by HPHT method, the crystallinity might be affected by fluctuations of temperature and pressure, associated with the presence of B, the optimization of growth conditions presumed to be difficult compared to the conventional insulating substrate. The improvement of p+ crystal growth technology is highly desired to advance toward the seed crystal of p+ substrates for power device applications. References [1] S.Shikata., Diam.Relat.Mat, 65(2016)168 [2] K.Srimongkon, et al., ibid, 63(2016)21 [3] S.Shikata, et al., Diam.Relat.Mat, 73(2017)241 [4] N. Akashi, et al., Mat.Sci.For, (2018) accepted for publication Dec.9 2017

PS2.09

Diamond High-Power Switching Transistor—Design, Device Structure Analysis and Demonstration Matthias Muehle¹, Timothy A. Grotjohn^{2, 1}, Tom Zimmermann^{2, 1}, Yuelin Wu², Michael Becker¹ and Thomas Schuelke^{1, 2}; ¹Fraunhofer USA, East Lansing, Michigan, United States; ²Electrical and Computer Engineering, Michigan State University, East Lansing, Michigan, United States.

Single crystalline diamond (SCD) based electronics has the potential to significantly impact and change the state of the art technology for high-power electronics. In recent years, significant development has made in the fabrication of diamond electronic devices, which are capable of operating in high-power regimes. These include the demonstration of high voltage Schottky diodes with breakdown voltages exceeding 1.7 kV with high current densities [1], as well as the demonstration of various field effect transistor design concepts for high breakdown voltage [2]. The next step in technology development is the implementation of design ideas for the realization of a high-power switching transistors that have both high forward current and high reverse bias breakdown voltages. As realization

of such a high-power switching transistor we propose a mixed verticallateral transistor structure. A combination of vertical and lateral features allows the design of a transistor which is capable of withstanding high voltages while also being able to conduct high currents. The high current is achieved by a lateral current path with the current controlled by a gate structure, and the high voltage region being a larger area p- vertical region. The vertical component contributes the capability of high current conduction with only little losses (minimized R_{on}) in the way of operating a vertical diode in forward direction [3]. First, we report on the controlled growth and material properties of the individual layers (p⁺, p⁻ and n). Temperature-dependent (room temperature up to 400C) Hall effect measurements, I-V and C-V characteristics are used to determine mobility, majority carrier concentration and lifetime, and compensation concentration. Various Ohmic and Schottky contact metal configurations are evaluated. It is demonstrated that annealed Ti/Pt/Au Ohmic contacts result in the lowest contact resistance while providing good adhesion. It is shown that the formation of good Schottky contacts is the most critical and problematic to achieve. It is demonstrated that Mo/Au Schottky contacts provide good compromise of adhesion and electric performance. We present the proposed semiconductor processing steps for the realization of the vertical-lateral high-power transistor based on the individual films and contacts studied. We will also present different design variations of doping concentration and sizes to achieve various currents and breakdown voltages. The target breakdown voltages are in the range of 1-2 kV. Finally, preliminary data of transistors fabricated will be reported. References [1] Diamond Schottky Didoes, abstract submitted to this conference. [2] Umezawa et al., IEEE Electron Device Letters 35 (2014) 1112-1114 [3] Dutta et al., IEEE Electron Device Letters 38 (2017) 600

PS2.10

Diamond Brewster Windows for Microwave Transmission <u>Theo A. Scherer</u>, Gaetano Aiello and Dirk Strauss; IAM-AWP, Karlsruhe Institute of Technology KIT, Eggenstein-Leopoldshafen, Germany.

The diamond Brewster window consists of a diamond disk brazed to two copper waveguides at the Brewster angle of 67.2°. Being 140 mm the maximum available diameter in the market for a CVD diamond disk, the aperture of the waveguide has an upper limit of 50 mm. Because of the high heat loads occurring in gyrotrons that operate in the MW power range, a greater aperture of the waveguide is required, and this leads to the need of very large diameter disks. The current target is an aperture of 63.5 mm, leading to the need of a 180 mm minimum diameter for the diamond disk in the Brewster configuration. State of the art microwave plasma reactors are not capable of growing diamond disks of such size. In collaboration with the industrial partner Diamond Materials GmbH (Freiburg, Germany), experiments with unconventional method were thus first carried out in a small scale: to join diamond fragments by overgrowing the joint gap with diamond to obtain large diamond disks. The hot filament method was used and a dedicated reactor was designed, assembled and tested. The hot tungsten filament was placed right in the center of the joint gap having the shape of a V-groove. In this work, the results of such experiments shall be reported and discussed. In addition, the results of the very first attempts of growing directly large area diamond disks with a diameter of 180 mm in a microwave plasma reactor shall be presented. A first setup of reflectometry and polarization diagnostics for future high power multi-frequency transmission windows with low reflection coefficient will be discussed.

PS2.11

Fabrication of Diamond Composites with Rare Earth Fluoride Nanoparticles for X-Ray Scintillators Vadim S. Sedov¹, Sergei Kuznetsov¹, Maria Mayakova¹, Andrew A. Khomich¹, Victor Ralchenko^{2, 1} and Pavel Fedorov¹; ¹General Physics Institute of Russian Academy of Sciences (GPI RAS), Moscow, Russian Federation; ²Harbin Institute of Technology, Harbin, China.

Diamond nanophotonics is an area of modern materials science of an increasing interest. Extensive efforts in this field are focused at the impurity-related color centers in diamond (e.g., nitrogen-vacancy (NV), silicon-vacancy (SiV), germanium-vacancy (GeV), chromium- or nickel-related defects, etc.), that typically show narrow (a few nanometers at room temperature) zero-phonon lines (ZPL) in photoluminescence (PL) spectra and short PL decay times on the order of nanoseconds. These color

centers can be easily produced by ion implantation in diamond or by chemical vapor deposition (CVD) with in-situ doping. The known PL properties of the impurity-vacancy centers are quite suitable for engineering single photon emitters, the search for new and better PL sources in a diamond matrix, which could extend their spectral range and increase their decay time and efficiency, is still an ongoing activity. The goal of such studies is to extend the capabilities of diamond-based PL materials. More specifically, RE in form of nanoparticles (or atoms) with bright X-ray fluorescence can complement X-ray-transparent and steadfast diamond matrix to fabricate scintillators with tunable high-performance and relatively low absorption of the incident X-ray beam. Recently, Magyar et al. [1] reported the first such attempt to introduce RE atoms into a diamond lattice by doping diamond single crystals with Eu(III) tri-(2,6pyridine dicarboxylic acid) complex. In our first studies [2], we used europium fluoride (EuF3) nanoparticles to fabricate "Diamond-EuF3" composite films. Chemical vapor deposition (CVD) technique was used for the synthesis of diamond to grow the composite material, which showed bright PL at 612 nm. In this work, our method was applied for the preparation of the multilayered composites with different RE fluorides (Eu, Ce, Er, Dy, Ho etc.) in our search to find the best combination of materials for X-ray scintillation applications. Both PL and X-ray fluorescence performance of best "Diamond-RE" composites will be reported. The study was funded by a grant from the RFBR #16-29-11784.1. A. Magyar, W. Hu, T. Shanley, M.E. Flatté, E. Hu, I. Aharonovich, "Synthesis of luminescent europium defects in diamond" Nat. Comm, vol.5, pp3523, 2014.2. V.S. Sedov, S.V. Kuznetsov, V.G. Ralchenko, M.N. Mayakova, V.S. Krivobok, S.S. Savin, A.K. Martyanov, I.D. Romanishkin, A.A. Khomich, P.P. Fedorov, V.I. Konov "Diamond-EuF, nanocomposites with bright orange photoluminescence", // Diamond and Related Materials. 2017. v.72. p.47-52.

PS2.12

Influence of the Surface Oxidation to the Spin Coherence of Properties of Single NV Centers in Nano-Diamonds Ryuta Tsukahara¹, Masazumi Fujiwara³, Yoshihiko Sera², Hideki Hashimoto² and Shinichi Shikata¹; ¹Physics, Kwansei Gakuin University, Sanda-shi, Japan; ²Chemistry, Kwansei Gakuin University, Sanda-shi, Japan; ³Osaka City University, Sumiyoshi-ku, Japan.

The NV center in diamond is a point defect showing excellent stable emission, that allows optical readout and manipulation of the spin states, and entanglement between photons and the spin qubits. [1,2] Thus, the applications such as quantum information processing, nanoscale, and magnetometry are receiving much attention. Also for the diamonds of single nm size attracts attention in biology and medical. For the measurement of NV center, the coherence time of the electron spin is very important. Especially for nano-diamond, spin coherence time (T₂) is limited to a few microseconds. For nano-diamond, the distance between the NV center and the surface is small, the spin property of the NV centers are more sensitive to the surface chemistry compared to the bulk diamond. To achieve longer lifetime of coherence in bulk diamond, surface oxidation has been studied and it was found to be effective for NV center, by the factor of 1.7 compared to H terminated surface. [3] The surface oxidation is also expected to be effective for the nano-diamond, thus we have tried to apply this method to nano-diamond particles diluted in water. In this study, a commercially available nano-diamond sample (Microdiamant, MSY0-0.05, Type Ib, HPHT, Median: 25 nm) was used. This sample was centrifuged, and then supernatant water was carefully separated by a micropipette to isolate the precipitate. In order to examine quantitative understanding of the relationship between the surface condition of nano-diamond and the coherence time, four kinds of samples were prepared; a) As received, b) chemical oxidation at 90°C for 6hours, c) annealed in air at 550°C for 1 hour, and d) annealed in air at 550°C for 2 hours. Spin coherence time (T₂) was measured for NV center using optically detected magnetic resonance (ODMR) and spin echo method for 20 samples of each four treatments. We employed Rabi and spin echo sequences in the pulsed ODMR measurements. Raman spectroscopy was used for the confirmation of graphite layer (sp2) removal. X-ray electron spectroscopy (XPS) and Fourier transform infrared spectroscopy (FTIR) were also carried out to examine the oxygen-termination of the nanodiamond surface. From the measurements of Raman, XPS and FTIR, it was confirmed that annealing at 550 ° C in air removed the graphite layer on the surface of the nano-diamond, resulted in oxygen terminated surface. Spin coherence time T, was measured and the surface condition of the

nano-diamond were analyzed. The observed average coherent time T₂ for the four treatments were a) $\sim 1.8 \mu s$, b) $\sim 1.4 \mu s$, c) $\sim 2.5 \mu s$ and d) $\sim 2.6 \mu s$. This fact showed that the spin coherence time was extended by annealing the nano-diamonds in air at 550°C. The longest T₂ was 6.1 µs for the sample which was annealed in air at 550°C for 2 hours. This is about 1.3 times higher than that of un-treated sample. Unfortunately, the positive effect for longer lifetime was not observed for the chemical oxidation. In summary, we have conducted the statistical evaluation of the spin coherence time of single NV centers in 25 nm-sized nano-diamond in relation to the surface condition. The longer spin coherence time of the NV center in the nano-diamond was obtained by the annealing in air at 550°C for 2 hours. The longest spin coherence time of 6.1 μs was obtained; however, further improvement is required for the biotechnology application. References [1] F.Jelezko, T.Gaebel, I.Popa, A.Gruber, and J.Wrachtrup, Phys.Rev. Lett. 2004, 92, 076401. [2] E.Togan, Y.Chu, A. S. Trifonov, L.Jiang, J.Maze, L.Childress, M.V.G.Dutt, A.S.Sorensen, P.R.Hemmer, A. S.Zibrov, and M. D.Lukin, Nature (London) 2010, 466, 730. [3] K. Ohashi, T. Rosskopf, H. Watanabe, M. Loretz, Y. Tao, R. Hauert, S. Tomizawa, T. Ishikawa, J. Ishi-Hayase, S. Shikata, C. L. Degen, anod K. M. Itoh. Nano Lett. 13 (2013) pp.4733-4738

PS2.13

Thermionic Emission Studies of Diamond Gary Wan¹, Alex Croot^{1, 2}, Mattia Cattelan² and Neil Fox^{1, 2}; ¹Physics, University of Bristol, Bristol, United Kingdom; ²Chemistry, University of Bristol, Bristol, United Kingdom.

Thermionic emission offers a potential way for direct conversion of thermal to electric energy in a thermionic converter, where thermally excited electrons from a cathode are emitted and passed through a vacuum gap to a colder anode. According to the Richardson-Dushman equation, the main barrier for high emission current is the material work function. With suitable doping and termination, diamond is known to exhibit a low work function. Various studies on thermionic emission of diamond have been done before, but typically only the current density's temperature dependence is recorded. This is in part due to the instability of the diamond surface at high temperatures. Energy-filtered electron emission microscopy and electron spectroscopy techniques available in the Bristol NanoESCA facility were used to study diamond samples with various dopants. Samples were kept at elevated temperatures for thermionic emission, while different light sources were used for photoemission. Work function was determined for the diamond with and without a hydrogen termination at various temperatures by observing the electron emission distribution in the low energy region. X-ray photoemission spectra of the C1s peak was used to monitor charging and band bending of the sample.

PS2.14

Investigation of Corrosion Resistance on the Si Doped Diamond-Like Carbon Films Using SPR Phenomenon Jumpei Nishikawa, Naoki Sugihara, Naoto Ohtake and Hiroki Akasaka; Tokyo Institute of Technology, Meguro-ku, Japan.

To investigate the doping effect of silicon of the corrosion resistance of diamond-like carbon films, the silicon doped films etched by nitric acid were evaluated using a surface plasmon resonance (SPR) device with a multilayer structure consisting of an DLC layer on metal. Si doped DLC films with different concentration was deposited by pulsed plasma chemical vapor deposition method. A flow cell for the introduction of nitric acid solution was placed on the DLC layer of the multilayer structure. A nitric acid solution was used in the etching tests. The Kretschmann configuration was used for SPR measurement, and the SPR angle was determined as the angle with minimum reflectivity. The SPR angle decreased with increasing duration of nitric acid injection into the flow cell, indicating that the film was corroded by the nitric acid. The thickness of the films was calculated from the SPR angle. The rates of decrease in the thickness were decreased with Si concentration incresing. Although the Si doping is good effect for nitric acid solution, the corrosion rates against sodium hydroxide were incresed with increasing of Si doping concentration. These results indicate that the Si doping is good for the chemical resistance to nitric acid solution, not good for sodium hydroxide.

PS2.15

New Approaches for the Fabrication of Free Standing TiO₂ NWs/CNTs and SiNWs/CNTs Anodes, Appropriate for High Performance Lithium-Ion Batteries Juan C. Arango¹, Francisco Márquez¹, Carmen Morant², Abniel Machín³ and Sergio Pinilla²; ¹The School of Natural Sciences and Technology, Universidad del Turabo, Caguas, Puerto Rico; ²Applied Physics, Autonomous University of Madrid, Madrid, Spain; ³School of Professional Studies, Ana G. Méndez University System, Orlando, Florida, United States.

Titanium dioxide nanowires (TiO2NWs), are considered as one of the most promising candidates to be used as anode material for lithium-ion batteries (LIBs). TiO₂ is abundant, has low work potential, its synthesis process has a high yield, and is a non-toxic and non-corrosive solid. Due to these characteristics, this material is positioned as a promising environmentally friendly material towards the future of clean energy storage and transportation, through batteries. However, bare TiO2NWs does not possess high capacity (ca.330mAhg-1), which restricts the good characteristics of this material when compared with the batteries currently manufactured with graphite anodes (ca.372mAhg⁻¹). Silicon, with a specific capacity of ~4200mAh/g, is considered as the most promising candidate to be used as anode material for lithium-ion batteries (LIBs). Among its good features, silicon is abundant, has low work potential, and has a high theoretical specific capacity, which is more than ten times higher than that of graphite (commercial anode). However, silicon undergoes an enormous change in volume (ca.275%) during the lithiation/ de-lithiation processes, resulting in a disintegration of the material, and subsequent electrical disconnection. To mitigate these common issues within each described material, the use of single walled Carbon nanotubes (CNTs) rises as a possible solution among the scientific community; CNTs are outstanding nanostructures, widely studied for their potential use in LIBs anodes, due to their conductive, resistance and mechanical properties. In this research, TiO2NWs were synthetized through a hydrothermal approach. Likewise, Silicon nanowires (SiNWs) were synthetized through a Metal Assisted Chemical Etching (MACE) approach. Reaching high crystallinity and purity nanostructures suitable for LIBs. After that, commercial CNTs (RhenofitCNT-4) were added in various percentages to obtain mixtures of TiO2NWs/CNTs and SiNWs/ CNTs. These mixtures were carried using mixtures of different solvents, preventing the formation of clusters throughout the electrode surface. Finally, each mixture was left to dry overnight, to reach a homogenous deposition of the mixtures over the intended current collector (Cu film). The resulting electrodes show high flexibility and resistance due to the CNTs presence, reaching membrane-like structures usable as free-standing anodes suitable for LIBs. In addition, the physical framework formed by the CNTs around the TiO, NWs or SiNWs will help accommodate the mechanical strain caused during the lithiation-delithiation process in LIBs, while improving the life lasting of the materials, increasing the capacity of the hybrid structure. The characterization of the materials was carried out by scanning electron microscopy (SEM), Raman spectroscopy and energy dispersive X-ray spectroscopy (EDX). The electrochemical characterization of the synthesized materials was made through charge/ discharge profiles and cyclic voltammetry.

PS2.16

Sonication Enhanced ODMR Response of NV Colour Center Nanodiamond Powders Mateusz Ficek¹, Maciej Glowacki¹, Krystian Sycz², Mariusz Mrózek², Adam Wojciechowski², Wojciech Gawlik² and Robert Bogdanowicz¹; ¹Gdansk University of Technology, Gdansk, Poland; ²Institute of Physics, Jagiellonian University, Krakow, Poland.

The color center may couple an electron to become negatively charged (SiV- or NV-) with spin S=1. The nonzero spin is responsible for paramagnetic properties of the center. The vacancies may be created in diamond powders or in a bulk CVD or HTHP diamond. Due to photoconversion between the charged and neutral forms of the center and its light-power dependence, the NV centers are very attractive for applications in various quantum devices. Nitrogen-vacancy (NV) centers are the most widely studied crystallographic defect in the diamond lattice since their presence causes strong and stable fluorescence. The negative charge state of the defect (NV-) is especially desired because of its potential for quantum information processing. In this study, fluorescent suspensions of diamond particles have been produced by microbeadassisted ultrasonic disintegration of commercially obtained

diamond powder containing NV color centers1. The sample characterization was be based on optical (absorption, emission, Raman) and microwave spectroscopy with high spatial (confocal, ~µm3) and spectral (sub-Hz) resolution. The measurements were performed using a technique of optical detection of magnetic resonance (ODMR) improved by development of the microwave hole burning technique². Moreover, thin layer of the diamond particles has been deposited on a silicon substrate and examined using scanning electron microscopy (SEM). The development and applications of quantum technologies and nonlinear spectroscopy for studies of color centers in diamonds is very fast. Thanks to the stable crystallographic and electron structure of diamond, NV-color centers exhibit very stable electronic spectra, resistant to various perturbations. Their excellent optical and spin properties (paramagnetism) allow one to use different resonance and spintronic methods of and enable precision metrology. Acknowledgements This work was supported by the research grant NATO SPS G5147 and by Polish National Science Centre (NCN) under the Grants No. 2016/22/E/ST7/00102 and 2016/21/B/ ST7/01430 and by the Polish National Centre for Research and Development (NCBiR) under the project Techmatstrateg Diamsec 347324. The DS funds of Faculty of Electronics, Telecommunications and Informatics of the Gdansk University of Technology are also acknowledged.

PS2.17

Characterization of Frictional Properties of Carbon Onions with Different Molecular Morphology Atsushi Hirata¹, Yuta Nishimura² and Yuko Aono¹; ¹Department of Mechanical Engineering, Tokyo Institute of Technology, Tokyo, Japan; ²Department of Mechanical Sciences and Engineering, Tokyo Institute of Technology, Tokyo, Japan.

Carbon onion is an attractive nanoparticle as solid lubricant and abrasive in the realm of mechanical engineering. A large amount of carbon onion nanoparticles can be produced by annealing of nanodiamond or carbon black at temperatures above 1800 degree C. In this process, the molecular morphology of carbon onion largely depends on annealing temperature and the shape of the particles varies from sphere, polyhedron to hollow polyhedron with the temperature. The current study is designed to characterize frictional properties of carbon onions with different particular shape. Carbon onions are prepared by annealing of nanodiamond at 2000, 2400 and 2800 degree C. The morphology of carbon onions are observed by transmission electron microscopy and are confirmed as the spherical, polyhedron and hollow polyhedron nanoparticles. Raman spectroscopy and dynamic light scattering (DLS) are also employed to analyse the chemical microstructures and cohesive property, respectively. Friction measurement is carried out using a ball-on-disc friction tester to examine how the difference of morphology affects the tribological properties of carbon onions. Balls of high carbon chromium bearing steel (SUJ-2) and silicon wafer are used for sliding materials. For illuminating the frictional properties of the carbon onions, the minimum coefficients of friction and the lubrication lives that are sliding distances when friction coefficients increase and then reach 0.1 are measured in vacuum.Raman spectroscopy and DLC reveal that the crystallinity and dispersion of polyhedral carbon onion produced at 2400 degree C are the highest among the carbon onions. The results of friction measurement show that the minimum coefficients of friction for the carbon onions produced at 2000 and 2800 degree C are lower than that of the carbon onion at 2400 degree C, and the lubrication lives of the carbon onions at 2000 and 2800 degree are longer.

PS2.18

Soft X-Ray Irradiation Effect on the Hydrogenated Si Containing DLC Films Kazuhiro Kanda¹, Shotaro Tanaka¹, Ryo Imai¹, Shuto Suzuki¹, Masahito Niibe¹, Takayuki Hasegawa² and Tsuneo Suzuki³; ¹Laboratory of Advanced Science and Technology for Industry, University of Hyogo, Kamigori, Japan; ²Synchrotron Analysis L.L.C., Kamigori, Ako, Hyogo, Japan; ³School of Nuclear System Safety Engineering, Nagaoka University of Technology, Kamitomioka, Nagaoka, Niigata, Japan.

Hydrogenated diamond-like carbon (H-DLC) films, which contain hydrogen more than 40 %, are expected as solid lubricant for artificial satellite, because they maintain low friction in vacuum. On the other hand, astronautic materials used in low Earth orbit (LEO), where artificial satellites revolve, are required the durable against space environments, which are different from those on the earth, such as, soft X-ray, atomic oxygen, high vacuum and so on. It was founded that none doped H-DLC films were etched by

atomic oxygen irradiation, but H-DLC films, which included several percent of Si, had durable against atomic oxygen. In this study, irradiation effect on hydrogenated Si-containing DLC (Si-DLC) films by the exposure to soft X-ray, which was another space environment, was investigated. DLC films are generally durable against X-ray exposure. However, desorption of hydrogen in H-DLC films caused by soft X-ray exposure was reported. Therefore, clarification of the effect of soft X-ray exposure on hydrogenated Si-DLC films is important to ensure that hydrogenated Si-DLC films are used in LEO, safely. To investigate the soft X-ray irradiation effect on the hydrogenated Si-DLC films, we exposed such films to synchrotron radiation (SR) in the soft X-ray region and measured several film properties. Hydrogenated Si-DLC films were deposited on Si wafer using an amplitudemodulated radio-frequency plasma-enhanced chemical vapour deposition method. The soft X-ray irradiation of hydrogenated Si-DLC films was carried out at BL06 in the NewSUBARU synchrotron facility of the University of Hyogo. The SR at the BL06 sample stage had a continuous spectrum from the infrared to soft X-ray region less than 1 keV. The SR dose [mA · h] was derived as the product of the ring current [mA] and the exposure time [h]. Elemental compositions of the hydrogenated Si-DLC films were determined by the combination of elastic recoil detection analysis and Rutherford backscattering spectrometry. The atomic ratios of hydrogen, silicon and carbon of the hydrogenated Si-DLC film before the SR irradiation were \approx 0.4, \approx 0.2 and \approx 0.4, respectively. These atomic ratios were not changed after SR exposure. To discuss on variations in the local structure of the hydrogenated Si-DLC film due to the exposure to SR, C-K edge and Si-K edge NEXAFS spectra were measured at BL09A and at BL05A in NewSUBARU, respectively. In the C K-edge NEXAFS spectra of hydrogenated Si-DLC films before SR irradiation, the σ^* peak was narrower than that of typical DLC film and shifted to lower energy of about 5 eV, due to influence of peak of 289 eV derived $\sigma^*(C-Si)$ peak. In addition, the peak intensity of $1s \rightarrow \pi^*$ transition at 285.4 eV was weak. After SR exposure, the π^* peak intensity increased by the irradiation of 15 mA·h of the soft X-rays dose. On the other hand, the σ^* peak became broader and shifted to higher energy. Therefore, C-C bond increased at surface of hydrogenated Si-DLC films by SR exposure. In the Si-K edge NEXAFS spectra of hydrogenated Si-DLC films, the peak of 1846.8 eV derived from SiO, drastically increased by the irradiation of SR until 800 mA h dose. However, the intensity of this peak decreased suddenly by the irradiation beyond 1500 mA·h dose. On the other hand, the intensity of the energy range about 1843-1846 eV increased. The absorption in this energy range was derived from deoxidization of SiO₂. These results mean that the Si oxidization layer was formed by SR exposure, but the formed Si oxidization layer was deoxidized by the additional exposure to SR. From these experimental results, durable surface layer against X-ray exposure was formed at the surface of hydrogenated Si-DLC films by the irradiation of soft X-ray. These change are considered to be caused by the desorption of hydrogen from the hydrogenated Si-DLC films. Hydrogen atoms are desorbed by excitation due to soft X-ray irradiation, and the remaining carbon atoms bond to each other.

PS2.19

Biosensor Applications of the Composites of Carbon Nanoforests and Nanoparticles of Strontium Aluminate Doped with Rare-Earth Elements Patsy Y. Arquieta and Oxana V. Kharissova; Universidad Autónoma de Nuevo León, Monterrey, Mexico.

Currently, new biosensors with fluorescent properties on the basis of carbon allotropes are in development for possible applications in biotechnology area. In this work, the carbon forest-like nanocomposites with strontium aluminate SrA112O19 doped with rare-earth elements possessing fluorescent properties were prepared via spray pyrolysis. The synthesis was carried out in dry nitrogen atmosphere in the temperature range from 780 to 850°C. The used metal oxides were those of samarium (Sm), europium (Eu), neodymium (Nd), lanthanum (La), cerium (Ce), as well as some their combinations. The deposition of carbon nanotubes was carried out on the optical fibers surface to reach an uniform growth of forest-like carbon nanotubes, adding to its surface the nanoparticles of metal oxides. The formed products were characterized by XRD, Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), FTIR Spectroscopy, Raman Spectroscopy and UV/visible spectroscopies. Synthesis, properties, structural peculiarities, and applications of nanoforests and related nanostructures are discussed, revealing better results with use of lanthanum oxide. The deposited strontium aluminate nanoparticles were found to be in the range of sizes of 20-60 nm being uniformly distributed on the surface of MWCNTs. Possible applications

for the formed MWCNTs-supported luminescent composites as persistently luminescent biosensor materials are proposed.

PS2,20

Nanodiamond Surface Modification and Technology Development of a High Dose Non-Steroidal Anti-Inflammatory Drug Contained in a Medical Patch Whungwhoe Kim¹, C.K. Rhee² and A.P. Puzyr³; ¹Nuclear Material, Korea Atomic Energy Research Institute, Taejon, Korea (the Republic of); ²Korea Atomic Energy Research Institute, Taejon, Korea (the Republic of); ³Institute of Biophysics, Russian Academy of Sciences, Siberian Branch, Krasnoyarsk, Russian Federation.

Detonation nanodiamond (ND) is receiving much attention due to its remarkable mechanical, electrical and thermal properties, facile surface tailoring, narrow size distribution of primary particles and high biocompatibility. The rich surface chemistry of ND, the absence of toxic impurities and small size make ND a promising material for biomedical and technical applications. In this research, potential of ND in drug delivery has been demonstrated many times for a variety of compounds. The most important advantages of ND as a drug delivery vehicle stem from its strong physical adsorption controlled by the surface functional groups as well as a wide surface area of ND that is ideal for conjugating various biochemical substances. A high loading capacity of non-steroidal anti-inflammatory drugs (NSAID) was discussed with using lower concentration of ND.

PS2.21

Cu-DLC Composite Fabricated by Cold Spray Technique Nana Okimura, Nobuhisa Ata, Naoto Ohtake and Hiroki Akasaka; Tokyo Institute of Technology, Tokyo, Japan.

Diamond-Like Carbon (DLC) film is consisted of sp³ and sp² hybridized bonding carbon and hydrogen atoms, has high hardness and low friction coefficient. Therefore, DLC films are applied industrial fields for low energy consumption. On the other hand, DLC films can be obtained only as coating. In general, a few µm thick DLC films can only be deposited on the substrate with plasma chemical vapor deposition (CVD) method or physical vapor deposition (PVD) with plasma. Hence we can only obtain the DLC in the shape of thin film. In this research, we focused on cold spray (CS) technique to obtain the DLC contained metal composite. Thick metal films such as "mm" can be formed by colliding solid and plastic deformation of metal particles to substrate at a supersonic velocity by the conventional CS process. We propose that the DLC contained metal composite film with "mm" thick were deposited from the DLC coated metal particles by the cold spray (CS) technique to obtain the low frictional metal thick coating. DLC films was deposited on copper particles with a mean diameter of 20 µm (Cu-HWQ-20: Fukuda Metal Foil & Powder Co., Ltd.) with pulse plasma CVD process. Copper particles was placed on stainless tray of a negative electrode at the vacuum chamber. Acetylene gas was introduced to the vacuum chamber to 3.0 Pa. The applied voltage was maintained at -3.0 kVp with 14.4 kHz, and DLC film was deposited 30 min on cupper particles. These DLC coated copper particles used as source materials, the Cu-DLC composite film was fabricated by CS process. Advanced cold gas system (Medicoat Co., Ltd.) based on a DIMET 412-K cold spray system (TEIN TC Co., Ltd.) was used as the CS system. A1050 aluminum plates were used as substrates. The surface of aluminum plate was processed by a sand blaster with white alumina (#46, Fuji Manufacturing Co., Ltd.) to increase the surface area for improvement the deposition efficiency. Acceleration gas was air with 0.6 MPa and 723 K. The particle feed rate was 10 g/min. The distance from the nozzle to the substrate surface was 12 mm. The nozzle was scanned 10 times by 50 mm/s. The Cu film was fabricated by the same way from copper particles for comparison. To estimate the introduction DLC into the Cu matrix, the obtained film was analyzed by Raman spectroscopy. Surface of the Cu-DLC composite film was milled to obtain smooth surface, and tribological properties were evaluated by Ball on Disk (Bod) test using SUJ2 balls with 6 mm diameter. Load and rotation speed were 1 N and 400 rpm, respectively. At 20 min in deposition duration, the average of thickness in the obtained Cu-DLC composite film was 130 µm. Raman spectrum of the Cu-DLC composite film showed the broaden peaks of Graphitic and Disorder bands same as DLC films at around 1350 and 1590 cm⁻¹, respectively. Hence, these results indicated that the DLC contained copper composite thick film was deposited from DLC coated copper particles by CS technique. Average of friction coefficient between

the Cu-DLC composite film and SUJ2 ball was 0.41 while that of Cu film was 0.57. Therefore, friction coefficient of the Cu-DLC composite film was 30% lower than that of Cu film. Additionally, the observation of the film surface after BoD test, width of wear tracks on the Cu-DLC composite and Cu films were 450 and 650 mm, respectively. These results indicated the lubricant characteristic was brought to Cu film by introduction of DLC.

PS2.22 CANCELLED

Synthesis and Characterization of Highly Biocompatible Graphene Oxide Quantum Dots

PS2.23

Electronic and Magnetic Properties of Si-NPs-Doped Multiwall Carbon Nanotubes <u>Sekhar C. Ray;</u> Physics, University of South Africa, Johannesburg, South Africa.

The electronic structure and magnetic properties of silicon nanoparticles doped multiwall carbon nanotubes (MWCNTs) were investigated using C 1s and Si 2p-2s X-ray photoelectron spectroscopy (XPS), ultraviolet photoemission spectroscopy (UPS), Raman spectroscopy and SQUID-measured magnetization M-H loops. The result of XPS indicates that silyl radical based strong covalent bonds were formed in MWCNTs surface, which induced local structural relaxations and enhanced sp^3 -hybridization. Work function and valence band maximum were estimated from UPS measurements. Magnetic studies show that the magnetization is decrease due to conversion of sp^2 to sp^3 -hybridization along with the formation of Si-C and Si-O bonding in MWCNTs. The decrease in magnetization in MWCNTs on Si-NPs doping is another routes to tailor the magnetic properties of MWCNTs materials for magnetic device applications.

PS2.24

Graphene-Carbon Nanotube Aerogel as Organic Thermoelectrochemical Energy Harvesters—Hydrothermal Synthesis and Structural Characterization <u>Sanju Gupta</u> and Romney Meek; Western Kentucky University, Bowling Green, Kentucky, United States.

We prepared three-dimensional scaffolds comprising different ratios of graphene oxide nanosheets and carbon nanotubes (GA-CNT, hereon) macro-assemblies. The resulting hydrothermally synthesized hydrogels are freeze-dried and thermally reduced to yield graphene and graphene-carbon nanotube aerogels with ultralow densities and tunable mesoscopic pore sizes. These 'all carbon' aerogels prepared as monolithic solids from suspensions of few-layer graphene oxide nanosheets and small diameter multiwalled carbon nanotubes in which organic wet chemistry is used to cross-link the individual sheets and with carbon nanotubes. In contrast to methods that utilize physical cross-links between graphene oxide nanosheets, this approach with polymeric linkers and organic functionalization provides covalent carbon bonding among the graphene sheets and molecular attachment with carbon nanotubes, respectively, thus facilitating rapid and facile electron transport. As a result they are expected to exhibit improved electrical conductivities, moderate thermal conductivity, highly interconnected multiplexed topology with large internal surface areas thus promoting enhanced surface ion adsorption which makes these mesoporous materials viable candidates for use in harvesting thermos-electrochemical energy and energy storage technologies. We used complementary analytical characterization techniques including electron microscopy and tomography, x-ray diffraction, electrical property, Raman spectroscopy and Raman mapping in order to establish microstructure-processing-property correlations.

PS2.25

Graphene-Mediated Surface Enhanced Raman Spectroscopy (G-SERS) and Detection of Biomolecules Sanju Gupta and Alexander Banaszak; Western Kentucky University, Bowling Green, Kentucky, United States.

In this work, we prepare graphene-mediated surface-enhanced Raman scattering (G-SERS) platforms comprising few-layer graphene nanosheets decorated gold nanoparticle for biomedical and bio-nanotechnology. Raman scattering is surface-sensitive and nondestructive inelastic light scattering vibrational spectroscopy technique. SERS, a specialized form of Raman spectroscopy, is useful for rapid and precise identification of small biomolecules and industrially relevant chemical dyes at ultralow

concentration. This phenomenon is due to enhanced Raman signals by several orders of magnitude on the SERS-active surfaces. While the key point of SERS technology is the metal nanoparticles, which generates localized surface plasmon resonances in response to laser exposure and the resulting electromagnetic enhancement, controlling interparticle gap, the diameter of the metal nanoparticles and their ratio on graphene supports offer an advance toward sensitive G-SERS platforms via localized hybridization at graphene-metal interface. We have used low temperature thermal reduction technique to produce few-layer functionalized graphene supports and wet chemistry for size tunable gold nanoparticles as costeffective facile synthesis approaches for G-SERS platforms. Simple and high-throughput arrays (so-called 'biochip') are developed by decorating graphene nanosheets with gold nanoparticles as well as sandwiching gold nanoparticle and few-layer graphene for cascaded signal amplification to differentiate nucleotide bases (adenine; A, thymine; T, cytosine; C, guanine; G) and to detect beta-carotene and malachite green chemical dye.

PS2 26

Ultrathin 2D Film Irradiation by Highly Charged Ions Zinetula Z. Insepov^{2, 1, 3}, Ardak Ainabayev², Abat Zhuldassov², Aidyn Shaikhov² and Mititaka Terasawa⁴; ¹Nuclear Engineering, Purdue University, West Lafayette, Indiana, United States; ²Nazarbayev University, Astana, Kazakhstan; ³Condensed Matter, National Research Nuclear University, Moscow, Russian Federation; ⁴LASTI, Himeiji, Japan.

Irradiation of 2D ultrathin films by highly charged ions (HCI) offers a shallow modification of theirs surfaces with ability to control density and size of defects by changing fluence of ions and kinetic/potential energies of ions. It is known that in order to get any structural changes in material irradiated by HCI, the 2D films should have as low as possible surface conductivity, in order to realize a Coulomb explosion mechanism of crater formation. By controlling potential and kinetic energy values one can sputter material from topmost surface of irradiated samples, for example, kinetic energy can be regulated to very small amount of few keV, that restrict ions further penetration into more deeper region of irradiated samples, while whole potential energy relaxes onto surface in nanometer volume. That allows avoiding formation of defects in deeper layers, which makes potentially HCI perfect instrument for nanostructuring, cleaning and surface modification for thin films such as graphene, BN or MoS, However mechanism of defect formation during HCI interaction with 2D ultrathin films is not clear and it requires further investigations [1]. In general HCI is very good for formation few nanometer in diameter defects low conductivity materials. For carbon materials such as HOPG, carbon nanomembranes (CMN), and graphene it shown that high potential energy (charge state ~ q >25) and low kinetic energy (several keV) required to form induced friction type defect in graphene, hillocks in HOPG and nanopores in CNM [2-4]. In this study we used HCI to create defects and investigate their formation in 3D ultrathin films such as graphene, graphene oxide, BN and MoS, using Raman spectroscopy, AFM and electron microscopy. In addition Density Functional Theory (DFT) and large scale molecular dynamics (MD) simulations were used to investigate interactions between HCI and ultrathin films. References: [1] Gruber, E., Wilhelm, R.A., Pétuya, R. et al. (2016) Ultrafast electronic response of graphene to a strong and localized electric field. Nat. Commun. 384, 7, 13948. [2] Hopster, J. et al. (2014), Damage in graphene due to electronic excitation induced by highly charged ions, 2D Mater., 1 011011. [3] Aumayr, F., Facsko, S., El-Said, A., Trautmann, C., and Schleberger, M. (2011), Single ion induced surface nanostructures: a comparison between slow highly charged and swift heavy ions, J. Phys.: Condens. Matter, 23, 393001. [4] Ritter, R., Wilhelm, R.A., Stöger-Pollach, M. et al (2013), Fabrication of nanopores in 1 nm thick carbon nanomembranes with slow highly charged ions. Appl. Phys. Lett., 102, 063112.

PS2.27

A Novel Graphene-Photosensitizer Complex for Cancer Phototherapy Siheng Su¹ and Jingjing Qiu²; ¹Mechanical Engineering, California State University Fullerton, Fullerton, California, United States; ²Mechanical Engineering, Texas Tech University, Lubbock, Texas, United States.

Based on its tow dimensional geometry, graphene can penetrate biological barriers for targeted therapy. However, compared to metal nanoparticles, graphene has limited photo to thermal conversion efficiency because of relative low near infrared light (NIR) absorption. Herein, through conjugation with photosensitizers, photo to thermal conversion ability of

graphene is enhanced, effectively suppressing brain tumour growth. Specifically, to simulate the heating process inside biological tissue with graphene under NIR irradiation, graphene oxide was embedded in hydrogel and its temperature evolution under irradiation was experimentally and theoretically studied before *in vitro* and *in vivo* studies. Both results showed that increasing absorption ability through photosensitizer conjugation was a practical way to improve photo to thermal effects. Moreover, the conjugated graphene-photosensitizer was demonstrated to kill around 80% of U87-MG cancer cells *in vitro* and supress tumor growth *in vivo*.

PS2 28

Effect of High-Pressure Wet-Jet Milling Process on Reducing the Electrical Percolation Threshold of CNT Polymer Composite Film Using CNT Dispersion Anas A. Almowarai¹, Yoshiyuki Show², Tetsu Endo² and Tsubasa Yoshikawa²; ¹Science and Technology, Tokai University, Hiratsuka, Japan; ²Electrical and Electronic Engineering, Tokai University, Hiratsuka, Japan.

Carbon Nanotubes (CNTs) have superior mechanical, electrical and thermal properties. One of the applications of CNTs is a filler in polymeric matrix. Composites of Carbon nanotube in polymeric matrices have favorable electrical properties, which make them useful for applications such as photovoltaic devices and flat-panel displays. However, CNTs have a strong tendency to bundle together and that limits their applications. Therefore, appropriate dispersion process must be applied to use CNT in polymer composites to increase the electrical conductivity at lower CNT concentrations. In this study, an experiment was conducted to study the electrical percolation of CNT added into a polymer matrix. The study aims to show the percolation threshold can be lowered by exfoliating the CNT aggregation and dispersing individual CNTs in the suspension by using high-pressure wet-jet milling process. A commercially available thin MWCNTs produced via catalytic chemical vapor deposition and surfactant were mixed in distilled water at the concentration of 0.5wt.% and were pre-dispersed by ultrasonic homogenizer for 1min. The pre-dispersed CNT fluid was dispersed using high-pressure wet-jet milling (WJM) process at an injection pressure of 50MPa up to the processing of 100times. The CNT/PTFE composite film was formed at various CNT concentration from the CNT dispersion and the PTFE dispersion. SEM and TEM images of the CNT suspension before and after applying high- pressure wet-jet mill were taken to see the effect of the high-pressure wet-jet mill in the CNT size. The conductivity of the CNT/PTFE composite film was measured by four-point probe method to show the effect of the highpressure wet-jet mill on the percolation threshold of the CNT/PTFE composite film. Results of the study will be discussed at the poster session.

PS2.29

Fabrication of Nanocarbon by In-Liquid Plasma and Its Application Tetsu Endo¹, Yoshiyuki Show¹, Tsubasa Yoshikawa¹ and Anas A. Almowarai²; ¹Electrical and Electronic Engineering, Tokai University, Hiratuka, Japan; ²Science and Technology, Tokai University, Hiratsuka, Japan.

Nanocarbons are used as a supporting material of platinum (Pt) catalyst for polymer electrolyte fuel cell (PEFC). Acetylene black, which is a kind of nanocarbon, is a popular supporting material, because it has high surface area and electrical conductivity. The acetylene black is generally formed from vapor phase. In this study, the nanocarbon is synthesized from liquid phase by using the in-liquid plasma method. Moreover, the synthesized nanocarbon is applied to the supporting material of Pt catalyst. A glass beaker with the volume of 50 ml was used as vessel in which the in-liquid plasma is generated. The ethanol or 2-propanol of 40 ml was filled into the beaker as a source substance of the nanocarbon. A pair of carbon rod (glassy carbon) was placed as electrodes in it. The distance of the electrodes was 1 mm. The unipolar pulse voltage of 4 kV was applied between them and generated plasma in the ethanol or 2-propanol. The generated plasma decomposes the ethanol and the 2-propanol molecules and synthesizes nanocarbon in it. The nanocarbon synthesized by the inliquid plasma method showed aggregates of carbon particles with the diameter of less than 100 nm. When the 2-propanol was used as a source substance, the synthesized rate of the nanocarbon was 1.2mg/min, which was higher than that of the ethanol by 4 times. The nanocarbon synthesized by the in-liquid plasma was applied to a supporting material of platinum (Pt) catalyst for polymer electrolyte fuel cell (PEFC). The Pt

particles as a catalyst was formed on the synthesized nanocarbon by the in-liquid plasma method, which was used Pt electrodes, [1]. The fuel cell was assembled with the Pt/nanocarbon catalyst synthesized by the in-liquid plasma method. The fuel cell showed the open voltage of 0.9V and the maximum power of 65mW/cm². The above results indicate that the nanocarbon synthesized by the in-liquid plasma works as the supporting material of the Pt catalyst for the fuel cell. Reference [1] Y. Show and Y. Ueno, Nanomaterials 2017, 7, 31; doi:10.3390/nano7020031

PS2.30

Fabrication of Pt/CNT Catalyst for Fuel Cell Using CNT Dispersion <u>Tsubasa Yoshikawa</u>¹, Yoshiyuki Show¹, Tetsu Endo¹ and Anas A. Almowarai²; ¹Electrical and Electronic Engineering, Tokai University, Hiratsuka, Japan; ²Science and Technology, Tokai University, Hiratsuka, Japan.

Carbon nanotube (CNT) has high aspect ratio and high specific surface area. Therefore, the CNT is one of candidate supporting materials for platinum (Pt) catalyst in fuel cell, because the Pt catalyst requires to be supported on carbon material with the high surface area supports nanoscale particles. The commercial CNTs are generally an agglomerate with the size of up to 1mm. Therefore, the effective surface area of the CNT agglomerate is not high comparing with that of individual CNT. It indicates that the CNT agglomerate needs to exfoliate into individual CNTs in order to apply the CNT into the supporting material for platinum (Pt) catalyst in fuel cell. In this paper, the CNT agglomerates are exfoliated by the wet-jet milling method. Our previous study showed the wet-jet milling method forms individual CNTs from the agglomerates and disperses them in the water [1]. This CNT dispersion fluid is stable without sedimentation for a long duration of time. This CNT dispersion fluid is an ideal material as the supporting material of Pt catalyst. The formation of the Pt catalyst on the CNT by the in-liquid plasma was carried out by the following procedure. A glass beaker with the volume of 50 mL was used as vessel in which the in-liquid plasma was generated. A CNT dispersion of 40 mL was filled into the beaker. Two pieces of Pt wire were added as an electrode in the CNT dispersion. The distance of the Pt electrodes was 1 mm. A unipolar pulse voltage of 2.8 kV was applied between them and the plasma was formed in the carbon black dispersion. The generated plasma spatters the surface of the Pt electrodes and disperses Pt particles in the carbon black dispersion. Therefore, Pt particles with the weight of 12mg were formed on the surface of the CNT in the dispersion by the plasma processing time of 20 min. Polymer electrolyte membrane fuel cells (PEMFC) were fabricated with the Pt catalyst, which were formed on the CNT surface using the in-liquid plasma method. The fuel cell fabricated with the CNT, which was not applied in the in-liquid plasma, showed no output voltage, because no Pt catalyst existed on the surface of the CNT. The fuel cells fabricated with the Pt particles, which were formed on the CNT surface by the in-liquid plasma method, showed 0.97 V in open voltage. A maximum output power of 3.8W was observed for this fuel cell. The above results indicate that the CNT supports the Pt particles and the Pt on the CNT works as a catalyst for the fuel cell. Reference [1] A. Almowarai, Y. Ueno and Y. Show, Journal of Nanomaterials Volume 2015, Article ID 315017; http://dx.doi. org/10.1155/2015/315017 [2] Y. Show, A. Hirai, A. Almowarai, Y. Ueno, Thin Solid Films 596 (2015) 198-200 [3] Y. Show and Y. Ueno, Nanomaterials 2017, 7, 31; doi:10.3390/nano7020031

B3: Diamond Electronics III Session Chair: Julien Pernot Wednesday Morning, May 23, 2018 Room: Doyle/Rees

8:30 AM B3.01

Enhancement of Electronic Properties of Low-Boron Diamond Epilayers After Ar/O₂ Etching on HPHT (100) Substrates Cyrille C. Barbay¹, Christine Mer-Calfati¹, Nicolas Tranchant¹, Samuel Saada¹, Jean-Charles Arnault¹, Ingrid Stenger⁴, Verena Zürbig³, Lutz Kirste³, Loto Oluwasayo² and David Eon²; ¹CEA LIST, Gif-sur-Yvette, France; ²Institut Néel, Grenoble, France; ³Fraunhofer Institute for Applied Solid State Physics IAF, Freiburg, Germany; ⁴Groupe d'Etude de la Matière Condensée, Versailles, France.

Single crystal diamond behaves excellent skills for power electronic devices like its high breakdown voltage and the elevated mobilities for electrons and holes [1]. The main challenge remains the improvement of the crystalline quality which requires to lower the density of dislocations. Dislocations can originate from the defective subsurface of the HPHT diamond substrates after the mechanical polishing [2]. After threading into the epilayers, these dislocations emerge as pyramidal hillocks [3] affecting the devices electronic performance [4]. The removal of this defective layer is thus primordial to better control the crystalline quality of diamond epilayers. We investigated the effect of the Ar/O₂ etching of HPHT (100) Ib Sumitomo substrates on the electronic properties of low boron doped diamond epilayers. This pre-treatment was first optimized tuning the RFpower, the pressure and the O₂ content to measure the etching rate and the RMS roughness. Optimized conditions led to fast etching rates up to 9 μm/h and to smooth surfaces (RMS roughness from 3.5 to 2 nm on 40x40 μm² areas). We performed a comparative study on two half-etched HPHT (100) substrates of different suppliers (Sumitomo Ib and New Diamond Technology IIa). After removal of 10 mm, the efficiency of the Ar/O₂ etching was tested by evaluating the crystalline quality using Raman Spectroscopy and High Resolution X-ray Diffraction. The crystalline quality of low boron doped (1016 at/cm3) epilayers, 2 µm thick, grown simultaneously on the two etched substrates was compared to non-treated substrates by Raman and Cathodoluminescence. The SEM voltage contrast allowed to highlight the threading dislocations on non-pretreated areas and their removal after etching. Electronic properties investigated by Hall measurements show a clear improvement in term of carrier mobility and resistivity (from 3 to 1.7 W.m) on pre-etched area of Ib Sumitomo substrates. This project has received funding from the European Union's Horizon 2020 Program under Grant Agreement no. 640947 (Greendiamond). [1] M. Schreck et al., MRS Bulletin 39 (2014) 504; [2] I.Friel et al., Diamond and Related Materials 18 (2009) 808; [3] A. Tallaire et al., Diamond and Related Materials 17 (2008) 60; [4] M. Suzuki et al., Physical Status Solidi a 210 (2013) 2035;

8:45 AM B3.02

Carrier Transport Mechanism of Diamond p*-n Junction by Using Schottky-pn Junction Structure Toshiharu Makino^{1,2}, Naoto Ozawa², Aboulaye Traore¹, Hiromitsu Kato¹, Masahiko Ogura¹, Yukako Kato¹ and Satoshi Yamasaki¹,²; ¹National Institute of Advanced Industrial Science and Technology, Tsukuba, Japan; ²Graduate School of Pure and Applied Science, University of Tsukuba, Tsukuba, Japan.

Diamond has excellent physical and electrical properties such as high breakdown voltage, high thermal conductivity, and high carrier mobility. Based on these properties, diamond is expected to be a promising semiconductor for electronic and optoelectronic applications. On the other hand, diamond p-n and p-i-n junctions, which are the base structures of the above mentioned applications, have some issues such as the deep dopant levels of impurities (0.36 eV for B acceptor and 0.56 eV for P donor). These deep dopant levels degrade the device properties due to the low free-carrier density at room temperature, and thus the high resistivity of the p- and n-type layers. In order to overcome this issue, we have previously fabricated diamond p^{+} -i-n junction, where both the heavily B- and P-doped p^{+} - and n+layers, show hopping conduction [1]. We achieved a high current density

of 15000 A/cm² at the forward bias of 35 V at room temperature. Moreover, a high rectification ratio of about 10^8 at ± 10 V was reported. However, the carrier transport mechanism from p⁺- or n⁺-layer (hopping conduction) to i-layer (band conduction) has still not been understood. In this study, we have investigated carrier transport properties from hopping conduction-(p⁺-) to band conduction- (n-) layers by using Schottky-pn diode (SPND) structure [2]. The diamond SPND is composed by a fully depleted n-layer (P concentration: 0.2~1x10¹⁷ cm⁻³, thickness: 330 nm) sandwiched between a highly doped p⁺-layer (B concentration: 5x10²⁰ cm⁻³, thickness: >5 mm) and a Schottky metal. In other words, the SPND is consisted of two parts, that is, p*-n junction and n-type Schottky junction, and these junctions are connected tandemly. The SPND is a unipolar device with holes as carriers. The series resistance of n-layer can be neglected for a forward bias voltage higher than the flat band voltage because of a strong internal electric field in the n-layer, and thus hole current by drift conduction. Therefore, we can focus on the conduction properties of the p⁺-layer and hole injection mechanisms from p⁺- to n-layers by measuring the current-voltage properties. We have measured the temperature dependence of the currentvoltage properties for the SPND in order to investigate the hopping-level in the p⁺-layer, which is difficult to measure experimentally from the general Hall Effect measurement. We successfully estimated it as ~60 meV above the valence band. This means that we can decrease the ionization energy of holes by using heavily doped hopping layer, and the carrier injection from p⁺- to n-layers can easily occur even at room temperature. Thus, heavily impurity-doped hopping layer is useful for diamond devices with highcurrent operation. This work was partially supported by SIP (NEDO). References [1] K. Oyama et al., Appl. Phys. Lett. 94, 152109 (2009). [2] T. Makino, et al., Appl. Phys. Lett. 94, 262101 (2009).

9:00 AM B3.03

A High Current Regime for Schottky-PIN Diamond Diodes Raghuraj Hathwar¹, Maitreya Dutta², Franz A. Koeck¹, Mehdi Saremi¹, Srabanti Chowdhury², Stephen M. Goodnick¹ and Robert J. Nemanich¹; ¹Arizona State University, Tempe, Arizona, United States; ²University of California, Davis, Davis, California, United States.

Diamond Schottky-PIN diodes with undoped intrinsic drift layers show very high current densities (>500A/cm²) at low forward current voltage. We present temperature dependent I-V results of Schottky-PIN diodes fabricated on (100) p-type diamond substrates. Examination of the current density vs voltage shows an initial exponential dependence that is followed by a transition to a V2 dependence. As the voltage is increased further the I-V reaches the linear I vs V dependence expected for ohmic limitation by the contact layers. The V² regime has been predicted for space charge limited current in Schottky diodes and has been described by the Mott-Gurney relation. The high current densities indicate an operational mode that is between that of a vacuum diode and a semiconductor Schottky barrier. In a vacuum diode space charge limited current exhibits a V^{3/2} dependence as described by Child's law. In a space charge limited Schottky diode, the space charge limited current transport is further limited by phonon scattering resulting in the V2 dependence described by the Mott-Gurney relation. A simple model is presented which displays the transitions of the three different regimes (exponential thermionic emission, V² space charge limited Mott-Gurney, and linear ohmic limited current). Temperature dependent results of the same diamond diode are presented and described in terms of the model. Perhaps the most stunning aspect of this effect is that the diode specific on-resistance (R_{one}) decreases as the voltage increases. The results indicate a new mode of operation of diamond power devices where the high carrier mobilities and low phonon scattering support current injection and drift such that the resistance is reduced as the current is increased. This research is supported through the ARPA-E SWITCHES program and the NASA HOTTECH program

9:15 AM B3.04

Charge Transport Characteristic in Diamond pn Diodes Takehiro Shimaoka¹, Satoshi Koizumi¹ and Tanaka Manobu²; ¹National Institute for Materials Science, Tsukuba, Japan; ²High Energy Accerelator Research Organization, Tsukuba, Japan.

Diamond *pn* diodes have been studied to be utilized as deep ultra violet (DUV) light emitting diode and DUV sensor as well as to the basic research on power electronics applications. We expect the diode can also be applied for radiation detector under harsh environment such as nuclear reactor, accelerator etc. The advantages of *pn* diodes are operation without

external bias voltage (photovoltaic) and a possibility to adjust sensitivity against radiations in the variation of energy range by doping profile design. In this study, we report transport characteristics of charges in diamond pn diodes created by alpha-particles injection. We formed pseudo-vertical diamond pn diodes. Homoepitaxial p^+ , n^- , and n^+ - layers were grown on {111} HPHT type Ib single-crystal diamond substrate by MWPCVD. Mesa structure was formed by reactive ion etching to isolate each diode. Ohmic contacts were formed by EB deposition of Ti/Mo/Au both on the mesa structure of n^+ -layer and on the p^+ -layer. A fully depleted layer thickness and a built-in bias estimated by C-V characteristics were respectively 1.2 µm and 4.7 V. We measured alphaparticle induced charge distribution of the diamond pn diode. Alphaparticles from 241 Am radioactive source were injected from the n^+ contact. Since the energy of alpha-particles is 5.486 MeV and their extinction range in diamond are 13µm, alpha-particles pass through the depletion layer of the diamond pn diode. The diode showed induced charge distribution by alpha-particles injection without external bias voltage (photovoltaic operation). We also evaluated bias dependency of charge collection efficiency (CCE) in the depletion layer. Thirty volts of reverse bias voltage was required for a CCE saturation. Expected electric field strength in the depletion layer was 0.3 MV/cm. Typically, diamond radiation detectors are made of several hundred microns of thick CVD diamond. They show a saturation of CCE at several kV/cm to 10kV/cm of electric field strength. Our sample requires more than ten times of higher electric field strength for the saturation than that of thick diamond detectors. In the case depletion layer thickness is smaller than extinction range of alpha-particles, electric field strength through depletion layer supposed be decreased by transiently increase of space charge density caused by electron-hole pair creation. Regarding as the CCE greater than 30 V of reverse bias voltage, charge multiplication was not observed up to 120 V, which is expected to be 1 MV/cm of field strength. We will further study charge multiplication process under higher field strength to extract impact ionization coefficient in diamond pn junction.

9:30 AM B3.05

Fabrication and Testing of High Power Diamond Schottky Diodes up to 1800V Stephen Zajac¹, Aaron Hardy², Timothy P. Hogan¹ and Timothy A. Grotjohn¹; ¹Michigan State University, East Lansing, Michigan, United States; ²Fraunhofer USA, East Lansing, Michigan, United States.

Diamond as a semiconductor material offers the promise of electronic devices with higher reverse breakdown voltages, higher operating powers, and higher operating temperatures when compared to silicon based electronics. These improved metrics are possible because of the unique characteristics of diamond such as a high breakdown electric field strength, high hole and electron mobility, and high thermal conductivity. A challenge has been growing epitaxial diamond material in the quantities and qualities needed for high power devices. Much of the past research has focused on understanding the material properties of diamond, and has succeeded in fabricating devices with high reverse voltages and high current densities, but the large area, practical devices have been elusive. More recent research has focused on looking at what is necessary to create these practical devices [1,2]. This talk will focus on the research undertaken to fabricate practical diamond Schottky diodes, and the incremental progress made increasing both the breakdown voltage and the forward current density. The starting point for this work are the results obtained in [3], which achieved a 240V breakdown voltage, and a 300 A/ cm² forward current density with 200 um diameter contacts. In order to achieve higher breakdown voltage, the doping concentration of the lightly boron doped layer is reduced by lowering the diborane level in the feed gas mixture. At the same time, 2% oxygen is added and the deposition temperature is raised to 900°C to maintain high quality epitaxial diamond growth. Vertical Schottky diodes are fabricated with p+ doped substrates to measure the properties of the grown diamond. Comparison is made between growing the p- drift layer on the p+ substrate, and a new technique in which the p- drift layer is grown first, followed by growing the p+ substrate. The diamond seed is then removed through laser cutting and polishing to reveal the p-drift layer. One end result on a 4 mm by 4 mm substrate with 200 µm diameter contacts and a boron doping level of 1 x 1016 cm-3 in the drift layer as measured by CV had a measured breakdown voltage of 1.8 kV, and a forward current density of 300 A cm⁻². Acknowledgement: The authors would like to thank the US Dept. of Energy ARPA-E grant no. DE-AR0000455 for funding. References: [1] H. Umezawa, et al., 1Ω On-Resistance Diamond Vertical-Schottky Barrier

Diode Operated at 250 °C, Applied Physics Express 6 (2013), http://dx.doi.org/10.7567/APEX.6.011302 [2] V.D. Blank, et al., Power high-voltage and fast response Schottky barrier diamond diodes, Diamond Relat. Mater. (2015), http://dx.doi.org/10.1016/j.diamond.2015.01005 [3] S. Nicley, et al., "Fabrication and characterization of a corner architecture Schottky barrier diode structure" physica status solidi (a) Vol. 212 (11), (2015) Pages 2410-2417, DOI: 10.1002/pssa.201532220

9:45 AM B3.06

Fabrication of Dual-Termination-Planar Diamond Schottky Barrier Diode Dan Zhao, Zhangcheng Liu, Juan Wang, Jiao Fu, Yan-Feng Wang, Guoqing Shao, Jingwen Zhang, Shuwei Fan, Wei Wang and Hongxing Wang; Xi'an Jiaotong University, Xi'an, China.

Diamond is a promising semiconductor for future high-power and hightemperature applications due to its excellent properties, such as high breakdown field, wide band-gap and high carrier mobility, etc. Schottky barrier diodes (SBDs) with low forward voltage drop at forward bias and low leakage current at reverse bias are required. However, there is a tradeoff between the forward voltage drop and the reverse leakage current in the design of conventional SBDs. Therefore, new design concepts are necessary to improve the device performances. It was found that the barrier height of metal contact on oxygen-terminated diamond is lower than that of metal contact on fluorine-terminated diamond. Thus, combining oxygen termination with fluorine termination to fabricate dualtermination SBDs may obtain special properties. In this research, the electrical properties of dual-termination-planar (DTP) Schottky barrier diodes (SBDs) on diamond have been investigated, in which oxygenterminated area and fluorine-terminated area were obtained by ultraviolet ozone and ICP treatments. These DTP-SBDs obtained the same forward voltage drop as the SBD fabricated on oxygen-terminated diamond and the leakage current comparable to those of SBDs fabricated on fluorineterminated diamond. The results will be presented on the conference.

10:00 AM BREAK

G3: Graphene and 2-D Materials III Session Chair: Mario Hofmann Wednesday Morning, May 23, 2018 Room: Agassiz/Fremont

8:30 AM G3.01

Defect Engineering in SnS₂ and MoS₂ for Solar Fuels with Enhanced Conversion Efficiency and Product Selectivity <u>Li-Chyong Chen</u>; National Taiwan University, Taipei, Taiwan.

Photocatalytic CO, conversion to hydrocarbon fuels, the solar fuels, making possible simultaneous solar energy harvesting and CO₂ reduction, is considered a killing two birds with one stone approach to solving the energy and environmental problems. However, the development of solar fuels has been hampered by the low conversion efficiency and lack of product selectivity of the photocatalysts. Here, we present defect engineering (interstitial, substitutional, and vacancy) in chalcogenides as a viable method towards promising photocatalysts for CO, reduction reaction (CO2RR). Specifically, two cases will be illustrated: the carbondoped SnS, (abbreviated as SnS,-C hereafter) nanosheets and single to few-layer MoS, with controlled vacancies by plasma treatment. For the first case, the SnS₂-C nanosheets with a typical layer thickness of ~40 nm were synthesized using an L-cysteine-based hydrothermal process. Compared with undoped SnS2, the interstitial carbon doping induced microstrain in the SnS, lattice, resulting in different photophysical properties. Density functional theory calculations were performed for the formation energy, along with the CO₂ adsorption and dissociation on differently configured SnS₂-C for CO2RR. Experimentally, the SnS₂-C exhibited a highly effective photocatalytic activity in gas phase with a photochemical quantum efficiency exceeding 0.7 % under visible light, which is ~250 times higher than that of its undoped counterpart, and also a world-record high value reported for inorganic catalyst. For the second case, the MoS₂ single to few layers were prepared by vapor transport deposition, followed by hydrogen plasma post-treatment. With increasing

hydrogen plasma treatment time, we observed a trend of blue-shift in the A_{1g} peak and red-shift in E_{2g} peak in their Raman spectra, implying creation of sulfur vacancies, of which the resultant stoichiometry ratio of Mo/S was further investigated by X-ray photoelectron spectroscopy. In addition, scanning tunneling microscopic images clearly supported that there were missing atoms in the MoS $_2$ layers after hydrogen plasma treatment. Productivity and selectivity of CO2RR were found to be strongly dependent with the different Mo/S ratios of the MoS $_2$ single to few layers. The role and interplay of the defects and the hosting materials as well as their effects on CO2RR will be discussed in this presentation.

8:45 AM G3.02

Solution Giant Gate Graphene FET (G3FET) pH Sensor Syamsul Mohd¹, Shaili Falina¹, Masataka Hasegawa², Yoshinori Koga² and Hiroshi Kawarada¹; ¹Waseda University, Shinjuku, Japan; ²National Institute of Advanced Industrial Science and Technology (AIST), Tsukuba, Japan.

The use of pristine exfoliated bilayer graphene grown by chemical vapor deposition to fabricate Giant Gate Graphene FET (G3FET) as a pH sensor will be reported. Several reports exist on graphene as pH sensors [1,2] and its increase sensitivity with defects induced methods [3,4]. In this project, we implement a mild anodic oxidation method to retain the pristine graphene condition and non-encapsulated device as pH sensor. Initially the project starts with two different substrates of bilayer graphene on p-type Si and quartz. A large dimension of 10 mm gate width (W_c) and 6 mm source drain length $(L_{\rm SD})$ was fabricated hence the designated name for the FET device. G3FET shows a typical graphene FET output characteristics within the range of 0 V to -0.2 V of gate-drain voltage at pH of 7. Transfer characteristics of the G3FET for a constant drain-source voltage of -0.15 V for wide range Carmody Buffer Solutions from pH 2 to pH 12 were used to analyze pH sensitivity. The pH sensitivity measured were ± 12 mV/ pH. Additionally, graphene surface was electrochemically oxidized using cyclic voltammetry method immersed in Carmody Buffer Solution pH 7 with potential region from 0.8 V to 1.3 V. A slight improvement of sensitivity increases up to ±22 mV/pH was attained. Further, we will demonstrate a device without encapsulation by replacing the source and drain with titanium metallization. Via this implementation, we took the advantage of naturally occurring oxide on titanium replacing the encapsulation, hence provide an unsophisticated fabrication process approach. References: 1. P.K. Ang, et. Al, 'Solution-gated epitaxial graphene as pH sensor', J. Am. Chem. Soc. 130 (2008) 14392-14393. 2. J. Ristein et.al., 'Characteristics of solution gated field effect transistors on the basis of epitaxial graphene on silicon carbide', J. Phys. D: Appl. Phys. 43 (2010) 345303. 3. C.X. Lim et. Al., 'Direct voltammetric detection of DNA and pH sensing on epitaxial graphene: an insight into the role of oxygenated defects', Anal. Chem. 82 (2010) 7387-7393. 4. Mikhail Yu. et. al, 'Monitoring of epitaxial graphene anodization', Electrochimica Acta. 238, (2017) 91-98.

9:00 AM G3.03

Enhanced Stability and N-Doping Effect of Al₂O₃ Passive Layer on MoS₂ Field-Effect Transistor-Based DNA Biosensor Tung Pham¹, Jhoann Lopez¹, Mei Yang², Youngwoo Rheem¹ and Ashok Mulchandani¹; ¹University of California, Riverside, Riverside, California, United States; ²College of Bioengineering, Chongqing University, Chongqing, China.

Molybdenum disulfide (MoS₂), a member of 2D-semiconducting material family, features a direct band gap of 1.9 V in its monolayer structure. This tunable band gap suggests MoS, is a suitable conducting channel for fieldeffect transistors (FETs). In addition, the highly sensitive surface potential of MoS2, which can be controlled by surface chemical and biological modification, allows the feasibility of FET applications in biological sensors. In order to fabricate a MoS2-based FET biosensor, the immobilization of biological receptors can be done directly on MoS, using hydrophobic interaction, thiolation, silanization, and disulfide linkage etc., in which the device requires incubating in wet condition. In this work, we report for the first time the degradation of CVD-MoS, FET-based sensor in the presence of water, which possibly causes the false response in detection. We conclude the degradation is not due to the properties of MoS, altered during the exposure to water but the physical delamination of the material from the substrate, which in turn reduces the gating effect in FET transfer mode. We are able to alleviate the problem by coating the device with a 35 nm thick aluminum oxide (Al₂O₃) layer using atomic layer deposition (ALD). This passive oxide layer not only acted as a

protective layer against device degradation but also enhanced the n-doping effect in the FET. This permited multiple methods of detection, including using FET characteristic curve and chemiresistive I-V measurement at zero gate voltage (V $_{\rm g}=0$ V). Additionally, the ${\rm Al}_2{\rm O}_3$ layer provided available sites for functionalization with deoxyribonucleic acid (DNA) probes via silanization using (3-aminopropyl)triethoxysilane (APTES) and glutaraldehyde for biosensing. The functionalized ${\rm Al}_2{\rm O}_3$ coated MoS $_2$ FET-based DNA biosensor was applied to detect mutations in human TP53 gene, a biomarker for a variety of cancers. The fabricated devices demonstrated high selectivity between the complimentary and noncomplimentary DNAs, high sensitivity and excellent limit of detection at concentration as low as femto molar.

9:15 AM G3.04

Hydrogen Terminated Diamond Field Effect Transistors with Two-Dimensional (2D) h-BN as Gate Dielectric Jinlong Liu^{1,2}, Kiran K. Kovi³, Shikai Deng¹, Ralu Divan¹, Liliana Stan¹, Suzanne Miller¹, Jianqiang Lin¹ and <u>Anirudha Sumant</u>¹; ¹Argonne National Laboratory, Lemont, Illinois, United States; ²University of Science and Technology Beijing, Beijing, China; ³Euclid TechLabs, Bolingbrook, Illinois, United States.

Hydrogen terminated diamond field effect transistors (FETs) have attained great attention due to their interesting properties and improved performance. In this work, we report H- terminated diamond FETs using two-dimensional (2D) h-BN as the gate dielectric. The FETs with h-BN and h-BN/Al₂O₃ as gate dielectrics are fabricated and the device characteristics are compared. We show that incorporation of 2D h-BN with conventional Al₂O₃ dielectric layer show improved device performance at much lower total dielectric layer thickness and is promising for the development of hydrogen terminated diamond based MISFETs.

9:30 AM G3.05

Carbon Addition in Metal Chalcogenides for Enhanced Performance of the Anode in Lithium-Ion Battery Kuei-Hsien Chen^{1, 2}, Li-Chyong Chen², Venu Gopal¹ and Zeru Syum¹; ¹Institute of Atomic and Molecular Sciences, Academia Sinica, Taipei, Taiwan; ²Center for Condensed Matter Sciences, National Taiwan University, Taipei, Taiwan.

Metal chalcogenide is an attractive candidate for the anode material in lithium ion battery (LIB). However, the performance and lifetime still need to be improved. Two-dimensional SnS2 grown on nitrogen doped carbon nanotubes (CNTs) becomes an approach to enhance specific capacity and cycling performance of Li-ion batteries. Herein, CNTs were prepared via a microwave plasma-enhanced chemical vapor deposition (MPECVD) and followed by the low-temperature solvothermal route to deposit uniform tin disulfide (SnS2) nanosheet on the surace. The resultant hybrid thin film we prepared as the core-shell assembly of SnS2 nanosheet composite around CNTs exhibited a superior electrochemical performance of 1250 mAh/g after 120 cycles compare to the pristine SnS2 of 981mAh/g after 100 cycles. Detailed characterization has been carried out to elucidate the unique architectures of the self-supported SnS2/CNTs and its superior electrochemical performance. Meanwhile, carbon addition to Cu2ZnSnS4 (CZTS) has been demonstrated to be effective in high rate anode material with high capacity. Carbon coated CZTS electrodes were produced via a hydrothermal process with carbon precursor. The CZTS@C anode delivered a capacity of 830 mAh/g at a current density of 100 mA/g, which is much better in stability compared to the pristine CZTS without carbon addition. The excellent performance could be attributed to the high electrical conductivity of the uniform carbon coating. Detailed Raman and HR-TEM analysis have been performed to understand the role of carbon addition

9:45 AM G3.06 DISCUSSION TIME

10:00 AM BREAK

A2: Diamond Growth II Session Chair: Ken Haenen Wednesday Morning, May 23, 2018 Room: Doyle/Rees

10:30 AM A2.01

Growth and Characterization of Heavily Phosphorus Doped N-Type Diamond Thin Films <u>Satoshi Koizumi</u>, Tokuyuki Teraji and Takehiro Shimaoka; NIMS, Tsukuba, Japan.

Establishment of low resistivity n-type diamond formation technique is a great task and important for the electrical device applications. This is caused from deep donor level in the present n-type diamond doped with phosphorus. Hopping conductivity dominated in heavily P-doped sample is a hopeful solution against this problem. For this, we need to dope the concentration exceeding 1019 cm-3. In such heavily doping regimes we face to the problem of significant drop of incorporation efficiency of P atoms from gas phase into diamond layers by CVD doping. We are solving the problem by modifications of CVD machine especially focusing on the gas flow [1]. Additionally we also revealed the influence of substrate off angle on the incorporation efficiency. Finally, we obtained the doping efficiency of P atoms into diamond with more than 5% using low off angled substrates even in heavy doping regime [2]. Recently one of the authors succeeded to grow thick {111} CVD layer on conventional HPHT diamond substrate. The surface of the thick layer exhibits clear step-terrace structure with excellent homogeneity of the morphology. In the present research we deposited heavily P doped diamond on as grown {111} CVD thick layers and characterized the crystalline nature and the electrical properties. {111} thick CVD layers were grown NIMS type microwave plasma CVD system [3] on HTHP Ib diamond (2×2×0.5mm³) with the thickness of 70 µm. Then P-doped diamond thin films were grown on the thick layer using similar NIMS type microwave plasma CVD system designed specially to achieve high doping efficiency of P atoms. The growth conditions for P-doping are, CH₄/H₃: 0.05 %, PH₃/CH₄: 1%, total gas pressure: 100 Torr at a substrate temperature 900~910 °C. The electrical properties have been characterized by Van der Pauw Hall measurements under AC magnetic field over wide temperature range (RT~873K). Impurity concentrations have been measured by SIMS depth profile analysis. The off angle of {111} thick layer was estimated within 0.5~1° from growth late and doping efficiency of P-doped layer comparing with those of our data obtained from off angle experiments [2]. The off angle of diamond substrate, \sim 2.5 $^{\circ}$ shifted to lower off angle after thick layer growth. This is in good agreement with the situation observed in conventional diamond growth that exhibits low off angle area formation parallel to the edge and that expands the width with growth thickness increases. In this case, low off angle area grew wide enough to cover most of sample surface after 70 mm of diamond growth. P concentration of heavy doped layer was 8.7×10¹⁹ cm⁻³. Hall measurement results showed clear n-type behaviour at high temperature regime and hopping conductivity dominated at lower than 550K. Resistivity was about 47 Ωcm at RT and that is the lowest value ever obtained at P/C: 1% condition of CVD doping. It is confirmed that high crystalline perfection of thick CVD layer clearly helps to obtain high quality P-doped layer. [1] R. Ohtani, T. Yamamoto, S. D. Janssens, S. Yamasaki and S. Koizumi, Appl. Phys. Lett., 105, 232106-1~3 (2014). [2] T. Yamamoto, S.D. Janssens, R. Ohtani, D. Takeuchi and S. Koizumi, Appl. Phys. Lett., 109, 182102-1~5 (2016). [3] T. Teraji, T. Yamamoto, K. Watanabe, Y. Koide, J. Isoya, S. Onoda, T. Ohshima, L. J. Rogers, F. Jelezko, P. Neumann, J. Wrachtrup, and S. Koizumi, Phys. Status Solidi A 212 (11), 2365-2384 (2015).

10:45 AM A2.02

CVD-Diamond Coatings with Low Electrical Resistance by Silicon Incorporation Markus Prieske¹ and Frank Vollertsen^{1, 2}; ¹BIAS - Institute of Applied Beam Technology, Bremen, Germany; ²University of Bremen, Bremen, Germany.

Diamond is an electrical insulator with a resistance higher than $10^{10}\,\Omega.$ For applications as in semiconductor technologies or to avoid static electricity in components induced by friction, diamond coatings with a lower electrical resistance are necessary. The aim of this study was to achieve a homogenous distribution of incorporated silicon in polycrystalline CVD-diamond. The polycrystalline diamond coatings were deposited by a laser-based plasma

chemical vapour deposition (CVD) process without a chamber at atmospheric pressure. The in situ silicon incorporation was realised by the supply of a solid silicon carbide precursor into the plasma flame during the CVD process. To obtain a more homogenous distribution of evaporated silicon, the influence on the plasma flame of different plasma nozzle geometries were simulated by flow simulations in ANSYS. The crystal structure of the diamond films was verified by Raman spectroscopy and scanning electron microscopy. The resistance of the diamond coating is modified by the silicon incorporation. Deposition without a plasma nozzle led to a variation of the resistance from $k\Omega$ at the edges to higher than 60 M Ω in the centre of the coating. A sheet resistance of 2.43 M Ω \pm 1.98 M Ω was measured by a 4 point probe van der Pauw resistivity measurement on a single diamond crystal in the centre of the coating. Photoluminescence measurements were done with an excitation wavelength of 248 nm on an area of 18 mm². A sharp peak at 738 nm proofs the existence of silicon vacancy centres and thereby a doping of diamond crystals. A plasma nozzle in form of a pipe with a conical upper part enabled the deposition of a CVD-diamond coating with a homogenous distribution of silicon. The deposited diamond coating has a homogenous resistance in the $k\Omega$ range on the entire surface.

11:00 AM A2.03

Growth in D₂/CH₄/B₂H₆ Microwave Plasma-Assisted CVD Process Michal Sobaszek¹, Robert Bogdanowicz¹, Miroslaw Sawczak², Mateusz Ficek¹, Galina Grigorian³, Piotr Caban⁴, Aleksander Herman⁵ and Adam Cenian²; ¹Metrology and Optoelectronics, Gdansk University of

Enhanced Efficiency of Boron Doping of Diamond Thin Films by

Technology, Gdansk, Poland; ²Institute of Fluid Flow Machinery, Gdansk, Poland; ³St. Petersburg State University, St. Petersburg, Russian Federation; ⁴Institute of Electronic Materials Technology, Warsaw, Poland; ⁵Inorganic Chemistry, Gdansk University of Technology, Gdansk, Poland.

The Microwave Plasma-Assisted Chemical Vapour Deposition (MWPACVD) of diamond thin films was investigated in this work. The influence of plasma composition, mainly deuterium addition, on boron doping level was studied using optical emission spectroscopy (OES), laserinduced breakdown spectroscopy (LIBS), Raman spectra, spectroscopic ellipsometry, and Hall mobility measurements. The main species of microwave plasma such as C₂, CH, BH, D, H, and H₂ were detected and some plasma parameters, like characteristic temperatures, were investigated and discussed on the basis of plasma emission spectra. For the plasma containing deuterium an increased dissociation of B₂H₄ and intense boronradicals' production was confirmed. In consequence, a higher doping level of diamond films was observed by means of laser-induced breakdown spectroscopy (LIBS). Deuterium modifies the mechanism of boron incorporation into thin films leading to increased boron concentration. Lower concentrations of sp^2 phases and CH defects have been noticed in the films deposited in the plasma with a deuterium than with hydrogen addition. A strong correlation of BH, density with doping level was found. The BH, molecules appear as the most important doping species. Also, a correlation between calculated BH concentration and the boron-incorporation (content) level in diamond lattice was confirmed. Moreover, all BH_x (x = 0–3) species can bind to radical sites on the diamond {100} surface to form stable complexes. The increase in the carrier concentration and the decrease in the Hall mobility for the BDD samples grown in deuterium was registered. This work was supported by the Polish National Science Centre (NCN) under the Grant No. DEC-2017/01/X/ST7/02045, National Centre for Research and Development (NCBR) TECHMATSTRATEG1/347324/12/NCBR/2017 and by NATO SPS G5147. The DS funds of Faculty of Electronics, Telecommunications and Informatics of the Gdansk University of Technology are also acknowledged.

11:15 AM A2.04

Single Crystal and Polycrystalline Diamonds with Ge-V Color Centers Produced in Microwave Plasma by In Situ Doping from Germane GeH₄ Gas Vadim S. Sedov¹, Artem K. Martyanov¹, Andrey P. Bolshakov¹, Andrew A. Khomich¹, Oleg Kudryavtsev¹, Vladimir S. Krivobok², Sergei Nikolaev², Kirill Boldyrev⁴ and Victor Ralchenko^{3, 1}; ¹General Physics Institute of Russian Academy of Sciences (GPI RAS), Moscow, Russian Federation; ²Lebedev Physical Institute of the Russian Academy of Sciences (LPI RAS), Moscow, Russian Federation; ³Harbin Institute of Technology, Harbin, China; ⁴Institute of Spectroscopy RAS, Moscow, Russian Federation.

Color centers in synthetic diamond, having pronounced photoluminescence (PL), attract considerable attention in view of applications in quantum-information technologies (single-photon sources), biomedicine (optical biomarkers), and magnetic sensors [1]. Currently, new PL sources in diamond and diamond composites [2] are actively searched to extend the spectral range and properties of existing color centers. Recently, the "Germanium-Vacancy" (Ge-V) color center with bright PL at wavelength of ~602 nm was reported in a number of papers [3-6]. In this work we synthesized both polycrystalline and single-crystal diamond films using microwave plasma CVD technique without use of solid Ge as the dopant source. To this aim small additions of germane (GeH₄) gas to standard CH₄-H, mixtures were used to form Ge-Vcolor centers in diamond with PL spectra (T = 5 - 300 K) showing intensive peak at the wavelength of 602 nm with full width at half maximum (FWHM) ranging from ~5 nm (R.T) to 1.75 nm (5 K). The thickness and the growth rate of diamond films were measured in-situ using laser interferometry technique. The increasing the GeH, concentration leads to decrease in the growth rates of diamond: from 3 mm/hour without GeH, addition to 1 mm/hour for 10% GeH₄/CH₄. The detailed dependence of the PL intensity on GeH₄/CH₄ concentration (1:1000 ÷ 1:1) and growth temperature (750-1000 °C) will be reported. This work was supported by the Russian Science Foundation, grant No. 14-12-01403-P.1. Aharonovich, I., Neu, E. "Diamond nanophotonics". Adv. Opt. Mater. 2 (2014) 911-928. 2. Sedov, V. S., et al. "Diamond-EuF3 nanocomposites with bright orange photoluminescence." Diamond Relat. Mater. 72 (2017): 47-52. 3. Ralchenko, V. G., et al. "Observation of the Ge-vacancy color center in microcrystalline diamond films." Bulletin of the Lebedev Physics Institute", 42 (2015) 165-168. 4. Iwasaki T., et al. "Germanium-vacancy single color centers in diamond", Sci. Rep. 5, 12882 (2015). 5. Palyanov Y. N., et al. "Germanium: a new catalyst for diamond synthesis and a new optically active impurity in diamond", Sci. Rep. 5, 14789 (2015). 6. Ekimov, E.A., et al. "Germanium-vacancy color center in isotopically enriched diamonds synthesized at high pressures." JETP letters 102, (2015): 701-706.

11:30 AM A2.05

Photoluminescence Studied of Arsenic Related Defects in CVD IIa Diamond After (100) Orientation Ion-Implanted Kaiyue Wang^{1, 2}, Yufei Zhang², Zongchen Liu¹ and Hongxing Wang¹; ¹Xi'an Jiaotong University, Xi'an, China; ²Taiyuan University of Science and Technology, Taiyuan, China.

Due to its wide band gap, high thermal conductivity, high carrier mobility and high breakdown field, diamond has been attracting much interest as the potential material of high-frequency high-power electronic device [1-3]. The device requires a high concentration of acceptors or donors, which could be obtained by the methods of doping. There are a great number of works on the doping of diamond. For n-type diamond, the nitrogen is the most common observed donor impurity and usually exists in natural diamond [4]. However, the quite-deep donor level (Ed (N)=1.7 eV) is formed between the intermediate of diamond forbidden band, which makes it useless for semiconductor devices at room temperature [5]. As the same group V elements, arsenic is the shallower doping and expected as a more promising substitutional donor candidate [6]. The theoretical works showed that the substitutional arsenic provided the shallower donating level than phosphorus, at 0.4 eV below the bottom of the conduction band [7, 8]. Netherless, there is less common involvement of the arsenic related defects in diamond as far. In this work, the arsenic related defects of diamond were investigated by technology of low temperature photoluminescence, and some new results were presented and discussed. A slice of ultrapure colourless diamond was involved which was synthesized by MPCVD. The (100) surface orientation of the sample

was implanted with arsenic ions at 300K, with an energy of 130keV and a dose of 10¹³ ions cm⁻², and then annealed at temperatures of 400C, 800C and 1500C. The Photoluminescence examination was performed by Horiba micro-Raman spectrometers fitted with Linkam liquid nitrogen cooled stages at an excitation wavelength of 532 nm and recorded as a function of temperature in the 100~250K range. **Reference** [1] M. Schwander, K. Partes, Diam. Relat. Mater. 20 (2011) 1287. [2] K. Wang, J. W. Steeds, Z. Li, Appl. Phys. Lett. 110 (2017) 152101. [3] M.-A. Pinault-Thaury, T. Tillocher, N. Habka, Mater. Sci. Eng. B 176 (2011) 1401-1408. [4] K. Wang, J. W. Steeds, Z. Li, Mater. Character. 94 (2014) 14-18. [5] M. Kasu, M. Kubovic, Jap. J. Appl. Phys. 49 (2010) 110209. [6] A. P. L. Batista, F. R. Ornellas, Surface Science 641 (2015) 159-165. [7] S. J. Sque, Phys. Rev. Lett. 92 (2004) 017402. [8] J. P. Goss, P. R. Briddon, R. J. Eyre, Phys. Rev. B 74 (2006) 245217.

11:45 AM A2.06

Pre-Treatment Effects for P-Doped Diamond (100) Growth Using Pulsed Mode Deposition Xingye Wang, Franz A. Koeck and Robert J. Nemanich; Arizona State University, Tempe, Arizona, United States.

There is growing worldwide recognition that CVD diamond materials will impact future high-power electronics for applications that include power system distribution, electric vehicles and motor control. Nitrogen, a common impurity in natural diamond, has a covalent bond length (0.74Å) that is similar to diamond (0.77Å), but due to a lattice distortion, N incorporation exhibits a deep donor level at ~1.7 eV below the conduction band minimum. Phosphorus has been considered as another candidate for n-type doping, which has shown a donor level of ~0.6 eV experimentally. Phosphorus doping was initially achieved by incorporation during CVD growth on (111) diamond surfaces. Nevertheless, for practical applications, the growth of phosphorus-doped diamond on (100) surfaces is necessary. In this research, a pulse mode deposition method is employed to grow phosphorus-doped diamond layers on (100) single crystal diamond substrates. Different pre-treatment conditions including the usage of O/H plasma, H-plasma cleaning time and low CH₄ plasma time were employed in the growth. The results indicate that with the employment of an initial O/H plasma or H/CH, plasma, resulted in a higher P-doped diamond growth rate. In addition, a phosphorus doping concentration of ~1×10¹⁹ atoms/cm³ was achieved. Acknowledgement: this research supported by a grant from MWE Ltd.

> D1: Bio/Chem Applications of Diamond I Session Chair: Robert Hamers Wednesday Morning, May 23, 2018 Room: Agassiz/Fremont

10:30 AM D1.01

Improving the Lifetime of Ultrananocrystalline Diamond Microelectrodes for Dopamine Detection—Minimizing Surface Fouling via Electrochemical Cycling Prabhu U. Arumugam^{1, 2}, An-Yi Chang², Gaurab Dutta² and Shabnam Siddiqui¹; ¹Center for Biomedical Engineering Rehabilitation Science, Louisiana Tech University, Ruston, Louisiana, United States; ²Institute for Micromanufacturing, Louisiana Tech University, Ruston, Louisiana, United States.

Monitoring of extracellular neurochemicals on a chronic basis is critical to the understanding and treatment of several brain disorders. Deep Brain Stimulation (DBS) has emerged as a viable alternative treatment method to standard medications and therapeutic intervention. To maximize clinical benefits and reduce side-effects, DBS relies on a closed-loop neurochemical feedback approach to guide modulation of the stimulation parameters required to achieve optimal results. Currently, electrochemical techniques using carbon fiber microelectrodes (CFM) are routinely applied to monitor changes in the levels of key neurochemicals in the brain. But CFM require frequent chemical etching after use to clean their fouled surfaces and to maintain high sensitivity. This limits their useful lifetime to only few hours in many cases. In this research, we developed 250-µm boron-doped ultrananocrystalline diamond (BDUNCD) microelectrodes to detect neurochemicals chronically with minimal loss of electrode sensitivity and electron-transfer kinetics. Specifically, we studied the

surface biofouling due to dopamine (DA) oxidation and their by-products and the surface chemical fouling due to the application of anodic and cathodic potentials. We integrated the microelectrodes with microfluidics to study the fouling characteristics of BDUNCD microelectrodes for the first time using amperometry and fast scan cyclic voltammetry and then develop an *in vivo* compatible electrochemical cycling-based surface cleaning method. A new understanding of the BDUNCD microelectrodes' properties for chronic DA measurements will be discussed applying voltammetry, scanning electron microscopy, silver deposition imaging and electrochemical impedance spectroscopy techniques.

10:45 AM D1.02

Antibody Modified Boron-Doped Diamond for Ultrasensitive and Label-Free Detection Influenza Virus Robert Bogdanowicz¹, Dawid Nidzworski².5, Katarzyna Siuzdak³, Pawel Niedzialkowski⁴, Michal Sobaszek¹, Paulina Weiher⁵, Miroslaw Sawczak³, Elzbieta Wnuk².5, W.A. Goddard III⁶, Andres Jaramillo-Botero⁶ and Tadeusz Ossowski⁴; ¹Faculty of Electronics, Telecommunications and Informatics, Gdansk University of Technology, Gdansk, Poland; ²SensDx Ltd, Warsaw, Poland; ³Polish Academy of Sciences, Szewalski Institute of Fluid-Flow Machinery, Gdansk, Poland; ⁴Faculty of Chemistry, University of Gdansk, Gdansk, Poland; ⁵Institute of Biotechnology and Molecular Medicine, Gdansk, Poland; ⁶California Institute of Technology, Pasadena, California, United States.

According to the World Health Organization (WHO), almost 2 billion people each year are infected worldwide with flu-like pathogens including influenza. In this study, we describe a novel universal diamond biosensor, which enables the specific detection of the virus at ultralow concentrations, even before any clinical symptoms arise^{1,2}. The Si/BDD electrodes were synthesized in an MW PA CVD system (Seki Technotron AX5400S, Japan) on p-type Si wafers with (100) orientation. All samples were doped using diborane (B₂H₆) dopant precursor; [B]/[C] ratio was 10 000 ppm in the plasma resulting in an acceptor concentration of 3×10²¹ cm⁻³. The BDD surface was electrochemically functionalized using 4-aminobenzoic acid self-assembled monolayer (SAM), and anti-M1 antibodies were captured onto the SAM. The absorption of the M1 protein onto anti-M1 sites of the electrode change its electrochemical impedance spectra, which is then related to the viral concentration. The multifunctionalization of the BDD electrode surface by antibodies, by electrografting with diazonium salts, creates a novel approach for detection of influenza virus on an excellent detection level. We achieved a limit of detection of 1 fg/ml in saliva buffer for the M1 biomarker, which corresponds to ~10 viruses per sample in 5 mins. The limit of detection $LOD = 3 \times SD/slope$ equals 0.7 fg. Furthermore, the universality of the assay was confirmed by analyzing different strains of influenza A virus. Modification of BDD electrodes and subsequent attachment of antibodies was confirmed by X-ray photoemission spectroscopy (XPS). The antibody-carboxyl interaction induces stereoselectivity of the system leading to a high packing density of antibodies and electronic performance of sensor are confirmed by density functional theory (DFT) studies. The electrode stability and interference experiments revealed that the shape of the impedance spectra of the modified electrode is preserved after 24h of incubation in PBS buffer. Acknowledgements This work was supported by the Polish National Science Center (NCN) under Grant No. 2014/14/M/ ST5/00715 and 2016/21/B/ST7/01430. The DS funds of the Faculty of Electronics, Telecommunications, and Informatics and the Faculty of Chemistry at the Gdansk University of Technology are also acknowledged. R. Bogdanowicz wants to thank Prof. W. A. Goddard for the invitation and hosting in California Institute of Technology. The Fulbright Commission is acknowledged for financial support of this fellowship. References T. Matsubara, et al.. PNAS 113, vol. 32 (2016): p. 8981-8984.D. Nidzworski, et al., Sci. Rep. vol. 7(1) (2017), , 7(1), p.15707.

11:00 AM D1.03

Technological Development for Commercialization of Transformational New Generation of Dental Implants Coated with Biocompatible/Corrosion Resistant Ultrananocrystalline Diamond (UNCD) Coating Karam Kang¹, Jesus J. Alcantar-Peña¹, Daniel Olmedo³, Sergio Renou³, Deborah Tasat⁴.⁵ and <u>Orlando Auciello¹.²;</u> ¹Materials Science and Engineering and Bioengineering, The University of Texas at Dallas, Richardson, Texas, United States; ²LLC Company, Original Biomedical Implants, Richardson, Texas, United States; ³Department of Oral Pathology, School of Dentistry, Universidad Nacional de Bueno Aires, Buenos Aires, Argentina; ⁴Department of Histology and Embryology, School of Dentistry, Universidad Nacional de Buenos Aires, Buenos Aires, Argentina; ⁴School of Science and Technology, National University of San Martin, San Martin, Argentina.

Pure titanium and titanium alloys are widely used in commercial dental implants and other orthopedics. However, these materials can suffer electrochemical corrosion when implanted in the human body, inducing the release of metallic ions/particles from the titanium surface, causing tissue inflammation and implant failure. A novel ultrananocrystalline diamond (UNCD) coating developed and patented by one of the authors (OA) and colleagues exhibit outstanding mechanical, bionert/ biocompatible properties and strong resistance to chemical attack by body fluids, as shown previously for UNCD-coated Si microchip implanted in the eye to restore sight to people blinded by retina degeneration. This presentation focus on describing the R&D performed to develop the technological approach to produce Ti-based dental implants coated with UNCD films using an industrial-type MPCVD large volume coating of dental implants for low cost fabrication. UNCD coatings were produced by flowing an Ar/CH₄/H₂ gas mixture previously developed to produce the patented UNCD coatings currently in the market in UNCD coated mechanical pumps seals and bearings. Dental implants (up to 10) were positioned vertically on a circular holder uniformly distributed in a circular pattern, similar to the one designed to hold up to 500 dental implants (~ 8 mm log, 2-3 mm diameter) around a 200 mm diameter holder with circular concentric patterns. A uniform plasma was produced around all implants, which were coated, in a single coating process, with extremely uniform UNCD films (~ 0.2 - 0.3 µm thick), as shown by complementary Raman and SEM analysis. Corrosion studies indicated that UNCD-coated dental implants were not corroded as bare Ti-alloy implants. Histological evaluation of UNCD-coated Ti-alloy samples obtained using an in vivo experimental animal model bone implantation showed extremely good biocompatibility of the coating and oseointegration in the animal bone and NO particles ejection, as it occurs with current commercial Ti-alloy based dental implants. In addition, UNCD-coated cp-Ti and Ti-6Al-4V alloys exposed to artificial saliva (pH 6.5) showed no corrosion. In summary, the R&D to be discussed in this presentation shows the pathway for an industrial-scale process to produce UNCD-coated dental implants for insertion into the market in a foreseeable future.

11:15 AM D1.04

Science and Technology of Tailored Hydrophilic to Super-Hydrophobic/Corrosion-Abrasion Resistant/Biocompatible Micron to Ultrananocrystalline Diamond Coating Surfaces Ana G. Montaño^{1, 2}, Jesus J. Alcantar-Peña², Pablo Tirado^{4, 2}, Alejandro Abraham³, Elida de Obaldia^{2, 5} and Orlando Auciello^{2, 3}; ¹Departamento de Investigación en Polímeros y Materiales, Universidad de Sonora, Hermosillo, Mexico; ²Department of Materials Science and Engineering, University of Texas at Dallas, Dallas, Texas, United States; ³Department of Bioengineering, University of Texas at Dallas, Dallas, Texas, United States; ⁴Departamento de Investigación en Física, Universidad de Sonora, Hermosillo, Mexico; ⁵Facultad de Ciencia y Tecnología, Universidad Tecnológica de Panamá, Panamá, Panama

Polycrystalline diamond films with different grain size, ranging from ultrananocrystalline diamond (UNCD/3-5 nm) to nanocrystalline diamond (NCD/10-100s nm) to microcrystalline diamond (MCD/ \geq 1 micron), were grown by the Hot Filament Chemical Vapor Deposition (HFCVD) technique and characterized by Raman spectroscopy, Atomic Force Microscopy (AFM), X-ray Photoelectron Spectroscopy (XPS), and Water Contact Angle (WCA) analysis. The formation of polycrystalline diamond films with grain structures from MCD to UNCD were confirmed by Raman analysis, which showed the characteristic peaks of these films. The difference in grain size was confirmed by calculations based on TEM

diffraction patterns. The wettability properties of all polycrystalline diamond films investigated were tailored from as-grown semi-hydrophobic to hydrophilic surfaces by oxygen plasma treatment, and to highlyhydrophobic by CF, plasma treatment of flat surfaces, and via synergistic tailoring of surface chemistry and morphology at the micro and nanoscale to super-hydrophobic. Oxygen plasma (30 seconds) treatment of Hydrogen-terminated surface of UNCD film, resulted in -O and -OH surface termination, as revealed by XPS analysis, inducing a hydrophilic state, characterized by a water contact angle value close to 0 degrees. On the other hand, a (60 seconds) CF₄- based plasma treatment of the UNCD flat surface produced a highly hydrophobic behavior, for which the hydrophobicity results from the formation of carbon-monofluoride species on the surface, as revealed by XPS analysis. AFM imaging of the plasmatreated UNCD films revealed no significant change in the surface roughness after either O2 or CF4 treatments when they were compared with the surface of the as-grown UNCD films. Nevertheless, as previously stated, a surface-chemistry alteration was confirmed by XPS, which is directly responsible for the change in the wettability behavior. On the other hand, a small increase of the WCA was observed as function of grain size from UNCD to MCD films. This effect provided the bases for exploring an additional process to increase even further the hydrophobicity of polycrystalline diamond film surfaces. Thus, subsequent research discussed in this abstract, revealed that the synergistic complementary effect of 3-D structured + plasma-induced fluorinated surface termination of UNCD, NCD and MCD films resulted in super-hydrophobic surfaces that revealed WCA up to 170 degrees, thus producing the highest superhydrophobic surface of diamond material demonstrated today.

11:30 AM D1.05

Diamond Scaffolds for Bioengineering Applications <u>Aaqil Rifai</u>¹, Elena Pirogova¹, Andrew D. Greentree² and Kate Fox^{1,3}; ¹School of Electrical and Biomedical Engineering, RMIT University, Melbourne, Victoria, Australia; ²ARC Centre of Excellence for Nanoscale BioPhotonics, School of Science, RMIT University, Melbourne, Victoria, Australia; ³Center for Additive Manufacturing, RMIT University, Melbourne, Victoria, Australia.

With increasing frequency of hard tissue replacement and fracture treatment, novel materials are required to provide long term solution. Orthopaedic implants are commonly fabricated using metallic scaffolds, where the biological aspects are a by-product of the metallic interface surrounding the bone or soft tissue. Although metals have proved to be effective, it is important that we investigate alternative biomaterials. Due to the in vivo bio-compatibility of carbon, bio-interfacing carbon scaffolds are examined. Nanodiamond is an ideal bio-interfacing material as it provides a platform for bioactivity and osseointegration. To understand the new material, a comprehensive characterisation and interface assessment is undertaken. Additively manufactured titanium samples are first developed using selective laser melting to form three-dimensional scaffolds. The titanium is coated using optimised nanodiamond. Nanodiamond coated selective laser melted titanium scaffolds are examined in a range of biomedical settings to map diamond incorporation with the cellular biocompatibility and bacterial attachment to the scaffold. As a result, we have altered the nanodiamond material interface to enhance the bioactivity of the implantable scaffold. This facile method of nanodiamond synthesis represents a promising platform for an array of superior bio-interfacial implants.

D2: Bio/Chem Applications of Diamond II Session Chair: Orlando Auciello Wednesday Afternoon, May 23, 2018 Room: Doyle/Rees

1:30 PM D2.01

Influence of Embedded Plasmonic Ag Nanoparticles on Photocatalytic Response of Diamond Thin Films Robert J. Hamers, Shuo Li and Jason Bandy; Chemistry, University of Wisconsin-Madison, Madison, Wisconsin, United States.

H-terminated diamond is well known to be an excellent electron emitter when excited with above-band light due to diamond's unusual property of negative electron affinity. One drawback to diamond is the fact that excitation of electrons to the conduction band requires short-wavelength excitation, <225 nm wavelength, to exceed diamond's 5.5 eV bandgap. One approach to overcoming this large bandgap is to use diamond-metal heterostructures, as metal with lower work functions can provide a pathway to excitation from the metal valence band to the diamond conduction band. Recently we have investigated another approach to enhancing diamond's optical properties by embedding Ag nanoparticles into the diamond film. Ag has a lower work function that diamond, while the plasmonic properties of Ag can enhance the optical response and concentrate the electric field. Here we show that incorporation of <50 nm Ag nanoparticles into diamond flms significantly increases the photoelectric emission and photocatalytic activity of diamondWe have measured the optical response Ag-embedded diamond films and also measured the electron emission into vacuum and into water. In both cases, electron emission is clearly observed at subbandgap wavelengths, and the incorporation of Ag nanoparticles leads to a significant enhancement in photocatalytic yield for the photo-induced reduction of N₂ to NH₃. This reaction has an activation barrier of 3.1 eV, and therefore the ability to produce NH, from N, in water cannot be accomplished by traditional electrochemical schemes, but requires electrons in water with sufficiently high energy to produce solvated electrons and the solvated hydrogen atom H. Since incorporation of Ag into the film is also accompanied by an increase in graphitic carbon, there are several possible pathways to enhancement of electron yield. In this talk we will discuss some of the possible mechanisms of enhancement and discuss the prospects for enhancement of diamond's photocatalytic properties using embedded plasmonic nanoparticles.

1:45 PM D2.02

Photochemistry with Diamond <u>Jonathon Barkl</u>, Anna Zaniewski and Robert J. Nemanich; Physics, Arizona State University, Glendale, Arizona, United States.

This project examines the feasibility of conducting photochemistry in the visible light spectrum by utilizing thin diamond films in an aqueous solution. As a semiconductor, diamond is unique due to its wide band gap of 5.5 eV. The unique band structure enables hydrogen terminated diamond to have a negative electron affinity, meaning the conduction band edge is at a higher energy than the resting energy of an electron in vacuum. This property enables the emission of electrons from the surface of the diamond into the aqueous solution, creating an "energy reservoir" that can be used to initiate energy intensive chemical reactions. Previous studies have demonstrated this ability and the subsequent use of the aqueous electrons to induce the reduction of nitrogen to ammonium through utilizing light in the ultraviolet spectrum. This project explores the properties of the diamond films, substrates, and experimental setups in order to conduct the same reduction reaction while using light in the visible spectrum by lowering the effective work function of the diamond such that the energy required to emit electrons is achievable with visible light.

^{*} Invited Paper

2:00 PM D2.03

Effects of Deposition Parameters on the Surface Morphology and Wear Performance of Monolayer CVD Diamond Grinding Tools Xiaotian Shen and Fanghong Sun; School of Mechanical Engineering, Shanghaijiaotong University, Shanghai, China.

The hot filament chemical vapor deposition technique (HFCVD) is used to fabricate monolayer grinding tools with high-quality diamond grains. The high pressure and high temperature (HPHT) diamond seeds are dispersed on substrates uniformly by a spin coater machine. Then simultaneous growth of CVD diamonds on both seeds and substrates is employed which anchor the seeds on substrates. An investigation is undertaken to explore the effects of deposition parameters on the growth behavior and wear performance of diamond grains. The seeds are grown with varying amounts of deposition parameters including carbon concentration, reactive pressure, substrate temperature and bias intensity. The morphology and quality of as-grown diamond grains are characterized by the field emission scanning electron microscopy (FESEM), X-ray diffraction (XRD), and Raman spectroscopy. In order to evaluate the grinding performance of the diamond grains, the wear and failure mechanism of grains are tested by grinding of SiC ceramics. The grinding forces are measured and the morphologies of diamond grains as well as SiC ceramics are observed. The results indicate that the growth of nano-diamonds induces the formation of spherical polycrystalline grains, while the growth of large micron-diamonds leads to the formation of cube-octahedral single crystalline grains. Furthermore, surface defects of original seeds are eliminated under a set of optimized parameters, and thus high-quality single crystalline grains with smooth diamond planes can be fabricated. The grinding results demonstrate that the main wear mechanisms of the grains are attritious wear and fracture, while pullouts of grains can be hardly observed. Besides, the wear rates of single crystalline grains are significantly lower than that of the polycrystalline grains.

2:15 PM D2.04

Diamond Detectors for Uses in a Proton Therapy Beam Holly Johnson^{1, 2}, Jason Holmes¹, Franz Koeck¹, Raghuraj Hathwar¹, Anna Zaniewski¹ Ricardo Alarcon¹, Stephen M. Goodnick¹ and Robert J. Nemanich¹; ¹Arizona State University, Tempe, Arizona, United States; ²NASA Space Grant, Tempe, Arizona, United States.

Proton beam therapy is a form of cancer treatment that allows us to target and treat cancerous cells using high-energy protons. The protons pass through the skin and tissue and release a majority of their energy at a specific depth; this peak of energy deposition is known as a "Bragg Peak". In order to only target tumors and avoid affecting healthy cells, it is necessary to have precise knowledge of the beam's energy and position. However, current silicon-based detectors are damaged by the radiation and need to be replaced often, require frequent calibration, and are susceptible to noise due to its small band gap of 1.14 eV. Diamond has a band gap of 5.45 eV, meaning it is less susceptible to thermal noise and is more robust to radiation damage than silicon. In this project, we demonstrate a diamond-based proton detector. We acknowledge ARPA-E (Grant DE-AR0000453), National Science Foundation (DMR - 1710551), NASA Space Grant, and the ASU-Mayo Clinic Seed Grant.

2:30 PM D2.05

Controlling Directional Liquid Motion on Diamond and Diamond/β-SiC Composite Films for Microfluidic Devices Tao Wang; Shenzhen Institutes of Advanced Technology, Chinese Academy of Sciences, Shenzhen, China.

Control of wetting and directed liquid transport is key for microfluidic devices and biosensors. Here, we report a superhydrophobic diamond film and diamond/SiC gradient films synthesized by using a hot filament chemical vapor deposition (HFCVD) technique and its application as robust and chemically inert means to actuate water and hazardous liquids. The wettability of the diamond film was influenced by the topography (roughness). By designing cauliflower like structures combined with mirco-/nanodiamond, superhydrophobicity was achieved. For the gradient surface, the diamond/ β -SiC ratio of the composite films gradually changed from pure diamond to β -SiC. Water was transported via the gradient from the hydrophobic (hydrogen-terminated diamond) to hydrophilic side (hydroxyl-terminated β -SiC) of the gradient surface. The driving distance and velocity of water was influenced by the surface roughness. The droplet

moved a longer distance with a maximum velocity on the nanogradient surface. As diamond and β -SiC are chemically inert, the gradient surfaces can be used to drive hazardous liquids and reactive mixtures, which was signified by the actuation of hydrochloric acid and sodium hydroxide solution. In addition, the length of the gradient is tunable to the anticipated application. We envision that the superhydrophobic diamond and diamond/ β -SiC gradient surfaces have high potential as actuator for water transport in microfluidic devices, DNA sensor and implants which induce guided cell growth.

2:45 PM D2.06

Tribological Behaviors of the Diamond Films Sliding Against the T800/X850 CFRP Laminates Xinchang Wang, Chengchuan Wang, Xiaotian Shen and Fanghong Sun; School of Mechanical Engineering, Shanghai Jiao Tong University, Shanghai, China.

The machining of carbon fiber reinforced plastics (CFRP) laminates and CFRP-metal stacks adopting the diamond coated cutting tools has become a research hotspot in the engineering domain. During the drilling process of the CFRP, the frictional resistance and hole quality are intimately associated with the tribological behaviors of the drill material, but tribological behaviors of the varieties of extensively-adopted diamond films sliding against the CFRP have rarely been studied. In the present investigation, five typical types of diamond films are deposited on some WC-Co balls, including the micro-crystalline diamond (MCD), nano-crystalline diamond (NCD), boron-doped MCD (BDMCD), boron-doped NCD (BDNCD), along with the double-layer boron-doped micro-crystalline and nano-crystalline composite diamond (BDMC-NCCD). The diamond coated balls are submitted to the tribological tests, sliding against the cross sections of T800/ X850 CFRP laminates. The influences of the normal load F_{*} , sliding velocity v and the ratio of growth durations of the NCD to the BDMCD for the BDMC-NCCD film (N/B, No.1-1.5/10.5, No.2-3/9, No.3-4/7.5, No.4-5/6) are clarified. By respectively controlling growth durations, the thicknesses of all the diamond films are in the range of 12-14 µm. Wellshaped micro-sized diamond grains can be clearly observed on surfaces of the MCD and BDMCD films, while surfaces of the NCD and BDNCD films are both covered by densely-accumulated nano-sized grains, forming cauliflower-like nodules. Surface roughness R₂ values of the NCD, BDNCD and BDMC-NCCD films are lower than those of the MCD and BDMCD films. The MCD film has the highest diamond purity. Either the boron incorporation or the nanocrystallization induces the increment of the nondiamond. Therefore, the nano hardness values of the NCD, BDMCD and BDNCD films are lower than that of the MCD film. Most of the BDMC-NCCD films are also harder than NCD and BDNCD films, due to the influence of exposed micro-sized grains or the "reinforcement effect" of the underlying BDMCD layer. Due to the combined effect of the micro-sized grains and the boron incorporation, the BDMCD and BDMC-NCCD films perform the best adhesion. Owing to the grain refinement, the less fiber adhering to the film surface, and the increment of the non-diamond phase performing lubricating effect, the coefficient of the friction (COF) values of the nano-sized diamond surfaces are lower than those of the micro-sized diamond surfaces. The COF value of the BDMC-NCCD film with the lowest N/B is close to that of the BDMCD film, while the that of the BDMC-NCCD film with the highest N/B is at the same level as that of the NCD film. Besides, the SA-COF of any film will increase with F_n , while reduce with increasing v.The gradual wear of the diamond coated ball sliding against the CFRP is mainly associated with the hardness of the diamond film, therefore, the MCD film shows the smallest wear rate I_d , then the BDMCD one, then the BDMC-NCCD ones, while the NCD and BDNCD films show the largest I_d . The I_d will increase with either the F_n or the v. The film delamination acts as another wear mechanism after the longduration and higher-load sliding test, which often occurs in the drilling test, dependent on the film-substrate adhesion. The threshold F_{α} for inducing the delamination of the NCD and BDNCD films is about 14 N, and that for the MCD film is about 25 N, and that for the BDMCD and BDMC-NCCD films is about 35 N. Under the same F_n that can induce delamination of any diamond film (≥35 N), the required test durations for BDMCD and BDMC-NCCD films are longer than those for NCD and BDNCD films. Generally speaking, the better film-substrate adhesion can effectively prohibit the premature film delamination, and can delay it (prolong the lifetime of the diamond coated ball) under the condition of the frictional wear when the F_n even exceeds the threshold load.

3:00 PM BREAK

H1: Nanocarbons Session Chairs: Rodney Ruoff and Anirudha Sumant Wednesday Afternoon, May 23, 2018 Room: Agassiz/Fremont

* Invited Paper

1:30 PM *H1.01

Nanocarbon Fillers in Polymer Materials—Towards Tailoring the Composite Mechanical Performance Eleftheria Roumeli and Chiara Daraio; California Institute of Technology, Pasadena, California, United States.

Carbon materials with one or more dimensions in nanoscale exhibit, among others, astonishing mechanical, thermal and electrical properties, combined with extremely high surface areas¹. Properties such as Young's modulus in the order of 1 TPa², strength between 50-150 GPa³, metallic to semiconducting electrical performance⁴ and thermal conductivity up to 3500 W mK^{-13,5} are the main reasons behind the constant interest of the scientific community across many disciplines for these different forms of carbon. The properties and performance of these materials are governed by their structure, physical dimensions and geometry. In this review I will focus on the utilization of different forms of nanocarbons to tune the mechanical properties of polymer materials, highlighting the factors that govern the incorporation of nanocarbon fillers in the polymer matrix. Polymers represent a significant class of materials due to their versatility, variety and availability from both natural and synthetic processes⁶. Their numerous applications ranging from structural materials, adhesives, pharmaceuticals and biomedical materials impact significantly both our society and economy. Composite materials in which the main component is polymer and a foreign material is introduced as a filler, represent an efficient and elegant way to tune the performance or even induce new properties to the host polymer material. Here, we will focus on the mechanical properties of polymers and how they are influenced by the incorporation of synthetic nanocarbon fillers. We will categorize these effects by filler geometry and hosting polymer type (synthetic/natural) and explore which parameters control the mechanical reinforcement. The nanocarbon forms we will discuss are nanodiamonds, carbon nanotubes and graphene. Their geometry, surface area and surface chemistry are the key factors to control their dispersion, adhesion, alignment and confinement in a polymer matrix, which ultimately define the level of mechanical reinforcement⁷⁻¹¹. Another equally critical factor to achieve control over the mechanical properties of a polymer composite is the fabrication method itself¹². Modern additive manufacturing technologies have provided alternative methods to design materials that exhibit unique, unprecedented, engineered properties. Utilizing technologies such as stereolithography and two-photon polymerization to produce architected polymer materials has shown that we can attain previously unexplored performance by design^{13,14}. These architected polymer materials provide a new exiting platform to study phenomena occurring upon nanofiller incorporation. In this perspective, the host material's geometry is also a decisive factor for the performance of the composite and by tuning it to approach dimensions close to the ones of the filler, we can utilize filler confinement and alignment to unravel otherwise unattainable mechanical reinforcement levels¹². 1. Gogotsi, Y. et al. Carbon Nanomaterials (Taylor & Francis, 2013). 2. Zheng, X. et al. Science (80-.). 344, 1373-1377 (2014). 3. De Volder, M. F. L. et al. Science (80-.). 535, 535-539 (2013). 4. Odom, T. W., et al. Nature 391, 62-64 (1998). 5. Balandin, A. A. Nat. Mater. 10, 569-581 (2011). 6. Painter, P. C. et al. Essentials of Polymer Science and Engineering. (DEStech Publications, Inc, 2008). 7. Coleman, J. N., et al. Carbon N. Y. 44, 1624-1652 (2006). 8. Mochalin, V. N. et al. Diam. Relat. Mater. 58, 161-171 (2015). 9. Papageorgiou, D. G., et al. Prog. Mater. Sci. 90, 75-127 (2017). 10. Roumeli, E. et al. J. Phys. Chem. B 118, 11341-11352 (2014). 11. Roumeli, E. et al. Macromol. Mater. Eng. **300**, 611–626 (2015). 12. Krödel, S., et al. Submitted Manuscript (2018). 13. Montemayor, L., et al. MRS Bull. 40, 1122-1129 (2015). 14. Maleque, M. et al. Materials Selection and Design. (Springer Singapore, 2013).

2:00 PM H1.02

Synthesis, Structure and Sorption Capacity of Three-Dimensional Carbon Networks Carolina V. Rojas¹, Neida Santacruz³, Gerardo Morell² and Brad Weiner¹; ¹Department of Chemistry, Universidad of Puerto Rico, San Juan, Puerto Rico; ²Department of Physics, Universidad of Puerto Rico, San Juan, Puerto Rico; ³Department of Physical Sciences, Universidad of Puerto Rico, San Juan, Puerto Rico.

We synthesized three-dimensional (3D) carbon structures by Chemical Vapor Deposition (CVD) and characterized their structural properties. We employed the 3D pore system of zeolites (NaY) as solid template structures to assist in the synthesis of 3D carbon network structures. The carbon source consisted of radicals formed from the thermal decomposition of acetone, in a relatively facile and fast chemical deposition process. The 3D carbon network structures were characterized with X-ray diffraction, Raman spectroscopy, UV-Vis absorption, X-ray photoelectron spectroscopy, field emission scanning electron microscopy, and transmission electron microscopy. The results show that the 3D carbon network consists of micro-nano architectures composed of graphene, graphene oxide, and carbon nanotubes. We also measured the CO, gas storage capability of the 3D carbon networks at ambient conditions, showing that these are functional carbon materials with high sorption capacity. These 3D carbon architectures show promise for industrial applications

2:15 PM H1.03

Novel Carbon Nanotori-Based Additives for Lubricants with Superior Extreme Pressure Properties Oxana V. Kharissova¹, Patsy Y. Arquieta¹, Laura P. Parás² and Demofilo Maldonado-Cortes²; ¹Universidad Autónoma de Nuevo León, Monterrey, Mexico; ²Chemistry, Universidad de Monterrey, Monterrey, Mexico.

The variety of structural forms and a wide range of physical and chemical properties have caused development of a new scientific branch: the electronics based on carbon nanostructures. At the present time, carbon nanostructures are the main functional material for the development of electronic devices of broad applications. One of the most perspective forms of carbon nanostructures for practical application is the nanotori. A torus-like structure, called carbon nanotori, attracts special attention among the family of carbon nanomaterials. The broad prospects for potential applications of nanotori in various scientific and technological fields are caused an interest to carbon nanotori. In this work, new waterbased lubricants were prepared by their dispersing in distilled water at varying concentrations. Formed nanolubricants showed excellent stability even without the use of dispersing agents. The load-carrying capacity of nanolubricants under extreme pressures was determined with a T-02 fourball tribotester, according to the ITeE-PIB Polish test method for testing lubricants under scuffing conditions. In this test, a linearly increasing load is applied until the lubricant film disappears. Carbon nanotori were able to improve the load-carrying capacity by up to 920-950% and the load for scuffing initiation increased from 850 N to 1820 N. The results found in this study demonstrate the potential of carbon nanotori as extreme pressure additives for water based lubricants employed for metal-forming processes.

2:30 PM H1.04

Onion-Like Carbon Driven Superlubricity at Macroscale with Two-Dimensional Materials-Nanoparticles Ensembles Diana Berman, Badri Narayanan, Mathew J. Cherukara, Subramanian Sunkaranarayanan, Ali Erdemir, Alexander Zenovev and Anirudha Sumant; Argonne National Laboratory, Lemont, Illinois, United States.

In our previous studies we have demonstrated that the new superlubricity mechanism at macroscale by combined uses of graphene mixed with nanodiamonds sliding against diamond-like carbon (DLC) [1]. In particular, we showed that superlow friction regime (the coefficient of friction is 0.004) is observed when graphene patches wrap around the nanodiamonds and form nanoscrolls with reduced contact area sliding against an incommensurate DLC surface. In the present study, we show that other two dimensional (2D) layered materials such as molybdenum disulfide (MoS $_2$) is also capable of demonstrating superlubricity through unique tribochemical reaction with carbon leading to formation of onion-like carbon (OLC) at the tribological interface. We have observed that beyond some initial run-in period, the friction comes down to some un-

measurable levels and maintains in that state for a very long period of time, despite the fact that introduced 2D film of MoS, is only a few nanometer thick. Our detailed experimental and theoretical investigations suggest that formation of OLCs is possible through tribochemical reaction with these 2D materials that could occur at the tribological contact due to high contact pressure. These OLCs behaves in a similar way described earlier in our previous studies [1], providing reduced contact area and incommensurability with respect to the sliding DLC surface leading to superlubricity. We will discuss the detailed mechanism and highlight the similarities and differences with the previously demonstrated superlubricity involving graphene-nanodamond ensembles. This new discovery broadens the fundamental understanding of frictional behavior of 2D materials beyond graphene and opens a wide range of possibilities for implementing them in mechanical and tribological applications involving static, sliding, and rotating contacts. References: 1. Macroscale Superlubricity Enabled by Graphene Nanoscroll Formation D. Berman, S. A. Deshmukh, S. Sankaranarayanan, A. Erdemir, and Anirudha. V. Sumant Science, 348, 1118-1122, (2015). 2. Operando Tribochemical Formation of Onion-Like-Carbon Leads to Macroscale Superlubricity Diana Berman, Badri Narayanan, Mathew J. Cherukara, Subramanian Sankaranarayanan, Ali Erdemir, Alexander Zinovev, and Anirudha V. Sumant Nature Communications, (Accepted, 2018)

2:45 PM H1.05

(LATE NEWS) Structural, Electronic and Optical Properties of Nano-Structural BNC₃ Alloys Andrew V. Chizmeshya; School of Molecular Sciences, Arizona State University, Tempe, Arizona, United States.

Density functional theory is used to elucidate the fundamental properties of a hypothetical BNC, nano-alloy comprised entirely of covalently linked near-tetrahedral "B-N-C₂" units. The unique structure and composition is analogous to that of recently synthesized hybrid III-V/IV alloys, such as AlPSi, which adopts a silicon-like structure. The C1c1 ground state structure of BNC₃ is predicted to be dynamically stable and almost perfectly cubic, with a lattice parameter only slightly larger (~1%) than that of diamond. Its calculated bulk modulus is ~13% smaller than that of diamond, but slightly larger than that of cubic BN. Thermodynamically BNC, is found to be stable relative to elemental reference states, but metastable relative to disproportionation into cubic BN and carbon (graphite and/or diamond) in analogy with its AlPSi, analog. Orientational disordering of the "B-N-C₃" raises the lattice energy by only +52 meV/ atom, while a pseudo-random BNC, alloy is found to be considerably less stable (+233 meV/atom). The direct and indirect band gaps of the ordered ground state are predicted to be $E_a^{DIR} = 6.38 \text{ eV}$ and $E_a^{IND} = 4.72 \text{ eV}$, about 5% and 8% smaller than their diamond counterparts, respectively. This leads to a moderate increase in the absorption coefficient compared to diamond over the 6-10 eV range, and a ~0.4 eV red shift in the absorption edge. Infrared and Raman spectra obtained using density functional perturbation theory reveal a rich vibrational structure which can be interpreted in terms of concerted intra- and inter-tetrahedral B, N and C displacements. The formation of BNC, from molecular sources is also briefly discussed.

3:00 PM H1.07

Nanodiamond, Graphene and Carbon Nanotube Enabled High-Capacity and Long Cycling-Life Silicon Based Lithium-Ion Battery Anodes Yonhua Tzeng, WeiChih Huang and MinSiang Huang; Electrical Engineering, National Cheng Kung University, Tainan, Taiwan.

Modern needs by electric vehicles and mobile devices for high-capacity and long-cycling-life lithium ion battery rely on innovative battery electrodes to be invented. The most commonly applied graphite anode provides only about 400 mAh/g charge storage capacity. Silicon, when fully charged, provides about 4000 mAh/g theoretical charge storage capacity, which is about 10 times of that of its graphite counterpart. However, cycling between a fully charged and discharged silicon anode causes the volume of silicon to expand and shrink by as much as about 400%, which may lead to pulverization of silicon and the loss of electrical contacts between breakoff silicon and the current collector of a battery. The increased silicon surface of pulverized silicon reacts with the electrolyte to form solid-electrolyte interphase and caused the limited supply of electrolyte and lithium to be depleted prematurely leading to severe shorteneing of battery lifetime. Coating of nanodiamond with novel grain-boundary engineered nanostructures, vertically grown nanocarbon/

graphene walls, carbon nanotubes and carbon fibers on silicon provides means of increasing physical integrity, chemical inertness, electrical conductivity and multiple-path connectivity of silicon in the anode. In this paper, we will present several excellent means of maintaining charge storage function of silicon anode after a large number of cycles between charged and discharged lithium ion battery. High capacity silicon anode with more than 2000 mAh/g and capable of cycling for more than 100 times almost without any noticeable degradation has been achieved. Materials and electrical characterisics, electrochemical properties, and mass production issues will be discussed.

C3: Diamond Photonics/Spintronics III Session Chair: Tokuyuki Teraji Wednesday Afternoon, May 23, 2018 Room: Doyle/Rees

3:30 PM C3.02

Submicron Single Crystal Diamond Thin Film for Photonics and Quantum Sensing Applications Kumaravelu Ganesan¹, Alastair D. Stacey¹, Athavan Nadarajah¹, Patrick Salter² and Steven Prawer¹; ¹School of Physics, University of Melbourne, Parkville, Victoria, Australia; ²Department of Engineering Science, University of Oxford, Oxford, United Kingdom.

Integrated photonic quantum information processing and sensing devices are of growing interest for a number of applications such as highperformance nonlinear optics, optomechanics, sensing and metrology. To enable applications beyond traditional classical optics, innovative materials are necessary to provide the tools for chip-based quantum information processing. Diamond is a particularly attractive candidate material because of a unique combination of desirable material properties, including a high refractive index, exceptionally high thermal conductivity and high Young's modulus. High quality, single crystalline membranes with low-level impurities and low absorption loss and of acceptable size are a prerequisite to meet the demanding requirements targeting large photonic circuits and sensing devices. In the present work, we addressed two key challenges in order to bring laboratory demonstrations closer to real-world applications; (1) high quality diamond membranes and (2) methods for processing diamond at larger scales. We demonstrate a set of techniques for the fabrication of sub-micron single crystal diamond membrane windows (SCDMW). from single crystal type-IIa diamond seeds. The membrane sizes are up to 3x3 mm and the thickness ranges from 100nm -1mm and are supported by diamond scaffolds for ease of handling and further processing. The optical and mechanical quality of these membranes were tested and proved to be suitable for photonics and quantum sensing applications. We also successfully engineered good quality NV centers using femtosecond laser writing [3] as well as nitrogen implantation techniques. Several characterizations including Raman, photo luminescence and mechanical resonance were carried out to assess the quality of the fabricated diamond membranes. A nanophotonic device, optical ring resonator, was fabricated on one of the membranes. Confocal microscopy and ODMR measurement were performed on the NV centers engineered by a combination of femtosecond laser irradiation and thermal annealing. The fabrication techniques are scalable and offering great prospects as a platform for integrated photonics, quantum information processing and sensing devices. References [1] Piracha, A. H., Ganesan, K et. al. Scalable fabrication of high-quality, ultra-thin single crystal diamond membrane windows. Nanoscale, 8(12) (2016). [2] Piracha, A.H., Rath, P., Ganesan, K, et al Scalable Fabrication of Integrated Nanophotonic Circuits on Arrays of Thin Single Crystal Diamond Membrane Windows Nano Letters, 16 (5), pp. 3341-3347 (2016) [3] Chen, Y-C et al Laser writing of coherent colour centres in diamond, Nature Photonics, 11 (2), pp. 77 (2017)

3:45 PM C3.03

Progress Towards a Nitrogen-Vacancy Color Center Laser Jan Jeske¹, Desmond Lau¹, Xavier Vidal², Liam McGuinness³, Philipp Reineck¹, David A. Simpson⁴, Brett Johnson⁴, Marcus W. Doherty⁵, Sarath R. Nair², Andrew Wood², Ondrej Kitzler², Rich Mildren², Jeffrey McCallum⁴, Fedor Jelezko³, Shinobu Onoda⁶, Takeshi Ohshima⁶, Thomas Volz², Jared H. Cole¹, Brant C. Gibson¹ and <u>Andrew D. Greentree¹</u>; ¹RMIT University, Melbourne, Victoria, Australia; ²Macquarie University, Sydney, New South Wales, Australia; ³Universitat Ulm, Ulm, Germany; ⁴University of Melbourne, Melbourne, Victoria, Australia; ⁵Australian National University, Canberra, Australian Capital Territory, Australia; ⁶National Institutes for Quantum and Radiological Science and Technology, Takasaki, Japan.

The negatively charged nitrogen-vacancy color center is an astounding diamond defect that is finding applications including quantum sensing, quantum cryptography, and quantum information processing [1]. Since at

least 1986, it has also been proposed as laser medium [2]. The original motivation was to explore nitrogen-vacancy lasers as a tunable laser source across its broad phonon sidebands from 637nm to around 800nm, however no such laser has yet been realized. More recently, it has been shown that a nitrogen-vacancy laser would provide an alternative approach to readout for a nitrogen-vacancy magnetometer [3]. Because the readout would be coherent, rather than the conventional incoherent readout for optically detected magnetic resonance, enhanced sensitivities are predicted. We recently showed stimulated emission from nitrogen-vacancy ensembles [4]. Here we report our progress towards realizing a nitrogenvacancy laser showing results from nitrogen-vacancy ensembles in freespace and fibre cavities. References [1] I. Aharonovich, A. D. Greentree and S. Prawer, Nature Photonics 5, 397 (2011) [2] S. C. Rand, in Tunable Solid State Lasers II. Vol. 52 of Springer Series in Optical Sciences 276 (Springer, 1986) [3] J. Jeske, J. H. Cole and A. D. Greentree, New Journal of Physics 8, 013015 (2016) [4] J. Jeske et al., Nature Communications 8:14000 (2017)

4:00 PM C3.04

Synthesis of Plasmonic Ag Nanostructures Embedded in Diamond Films with Tunable Optical Properties Shuo Li, Jason Bandy and Robert J. Hamers; Chemistry, University of Wisconsin-Madison, Madison, Wisconsin, United States.

Diamond is well known for its good mechanical robustness and extreme chemical stability. While diamond often adopts different colors due to defects and/or specific impurities introduced during growth, the integration of plasmonic nanoparticle into diamond provide an alternative approach to alter diamond's color. Nanosized transition metals such as Ag and Au exhibit size- and shape-dependent plasmonic features in the visible region of the spectrum. Here we demonstrate that Ag nanoparticles can be embedded within diamond films by a three-step process: (1) a very thin film of Ag is deposited onto a diamond surface, (2) the Ag film is dewetted into individual nanoparticles, and (3) a second diamond film is grown to encapsulate the Ag nanoparticles. By growing the diamond-Agdiamond films on quartz substrates, the plasmonic properties of the nanoparticle films can be characterized via transmission optical spectroscopy, as well as visually through the distinct coloration of the diamond films. Cross-sectional SEM of fractured diamond-Ag-diamond structures clearly shows individual nanoparticles with sizes ranging from ~10 nm to 100 nm depending on experimental conditions. UV-vis spectra clearly show the plasmonic peaks in good correspondence of SEM. TEM analyses show the boundary between the diamond-Ag nanostructures, which has some voids but generally shows columnar growth of diamond extending radially from the Ag nanoparticle cores. Electromagnetic simulation results of the diamond-Ag composites also suggest possible air voids and graphite phases in the structure. The plasma-assisted Ag NPs embedded in diamond is a unique way to impart tunable optical properties to diamond thin films.

4:15 PM C3.05

Design and Fabrication of a Diamond Immersion Metalens for Quantum Emitters Tzu-Yung Huang¹, Richard Grote¹, Sander Mann^{2, 3}, David A. Hopper^{1, 4}, Annemarie Exarhos^{1, 5}, Gerald Lopez⁶, Erik Garnett² and Lee Bassett¹; ¹Electrical and Systems Engineering, University of Pennsylvania, Philadelphia, Pennsylvania, United States; ²Center for Nanophotonics, AMOLF, Amsterdam, Netherlands; ³Electrical and Computer Engineering, University of Texas at Austin, Austin, Texas, United States; ⁴Physics, University of Pennsylvania, Philadelphia, Pennsylvania, United States; ⁶Singh Center of Nanotechnology, University of Pennsylvania, Philadelphia, Pennsylvania, United States.

Quantum emitters such as the diamond nitrogen-vacancy (NV) center are the basis for a wide range of quantum technologies. However, refraction and reflections at material interfaces impede photon collection, and the emitters' atomic scale necessitates the use of bulky, intricate optical measurement setups that prevent packaging of quantum devices. To overcome these limitations, we present the design and fabrication of a metasurface composed of nanoscale diamond pillars that acts as an immersion objective to collect and collimate the emission of an individual NV center [1]. Excellent agreement between metalens design and performance can be seen by comparing the 3D finite-difference time-domain simulations and the measured transverse and axial profiles.

Photoluminescence (PL) images are collected simultaneously from the metalens and an oil-immersion objective focused through the backside of the high-pressure, high-temperature diamond substrate. Both images exhibit an NV center at the same lateral position, and the backgroundsubtracted spectra of both paths clearly show the NV zero phonon line at 637 nm and characteristic phonon side band. The background-corrected photon cross-correlation function, $g^{(2)}$, measured between both paths exhibits the antibunching dip characteristic of a single NV center. The metalens path demonstrates a saturation count rate of 121.7±2.2 photons/ ms, as compared to the objective with a saturation count rate of 33.5±0.6 photons/ms. Furthermore, we measured a transmission of >88% in the metalens path and a numerical aperture greater than 1.0, outperforming a traditional free-space objective. The flexible metasurface design will enable miniaturization of quantum devices in a wide range of host materials, is compatible with on-chip electronics for spin control, and it can be readily adapted to create metasurfaces with arbitrary phase profiles that shape single-photon emission for coupling to optical cavities or route photons based on their quantum state. [1] R. R. Grote, T.-Y. Huang, et al. "Imaging a Nitrogen-Vacancy Center with a Diamond Immersion Metalens.", arXiv:1711.00901.

4:30 PM C3.06

CVD Growth and Characterization of NV Center Probes for Low Temperature Nanoscale Magnetic Imaging Simon A. Meynell, Alec Jenkins, Claire A. McLellan, Susanne Baumann-Venzin, Matthew Pelliccione, Jeff Cady, Preeti Ovartchaiyapong, Christopher Reetz and Ania Jayich; Physics, University of California, Santa Barbara, Goleta, California, United States.

The nitrogen vacancy (NV) center in diamond is a highly sensitive atomic defect that can be used to sense sub-microTesla magnetic fields with nanometer-scale resolution. The NV center is a promising candidate for quantum sensing over a broad range of temperatures. In fact, an imaging technique that utilizes a single NV center in a scanning probe geometry has been used to image magnetic order in systems such as vortices in superconductors and magnetic solitons known as skyrmions at both room and low temperature. An important limitation to increasing the sensitivity and spatial resolution of such an NV-based sensor is the quantum decoherence induced by the diamond surface. As the exact origins of this decoherence are presently not well understood, we explore chemical vapor deposition diamond growth techniques with nitrogen delta-doping and NV creation methods which allows us to create samples that are limited only by the surface. These techniques give us the ability to understand the specific decoherence mechanisms with the hopes of eventually surmounting them and approaching the magnetic and spatial resolution necessary to ultimately image a single nuclear spin.

EP3: Innovation and Entrepreneurship Session III Session Chair: David Eon Wednesday Afternoon, May 23, 2018 Room: Doyle/Rees

5:00 PM *EP3.01

Synthetic Diamond—The Industrial Potential and Challenges <u>Adam Hoffman</u>; ICDAT-Israeli Center of Advanced Diamond Technology, Nesher, Israel.

ICDAT was conceived in my mind some 20 years ago while I was wondering with my father from one academic conference to another. Hearing all the excellent qualities of synthetic diamond I couldn't help asking myself: If CVD diamond is so useful why isn't it widely used? ICDAT was founded in 2014. Early days setting up, the biggest challenges were to establish a highly sophisticated infrastructure and recruit multi domains global matter experts. Luckily fund raising was lesser a concern due to family and friend network believing in my dream.Once setup, the initial barrier was to create a focal point and to prioritize applications to generate income and create a viable working business. Analyzing the market, we noticed the "Gorilla" in the marketplace selling diamond

material slabs letting the customer decide how to integrate it. This left not much room for competition due to lack of production capabilities and financial backing. We had to find other global partners to have a significant footprint. However, once we started to talk with potential customers we found out that without proper integration, they were receiving only a small portion of the CVD diamond benefits – this was our opportunity! The realization that material creation was an important but still know-how. Our claim to fame is material matching and integration. A slim slice of diamond integrated correctly creates a line of premium products to our customer. This revelation also led to our business model of partnerships and revenue sharing. Beginning with scanning the field of shining opportunities due to the wide spread industrial options the first stop on the way to glory was reaping the low-lying fruit of the uprising multibillion-dollar lab grown GEM stone industry. Combining forces with the largest diamond trade body in the Israeli diamond exchange - slowly converting to the next generation of high profit and turnover. This market is supply limited and has a window of opportunity slowly closing on small scale production players. The first necessity was to create a strong branding based on our enterprises relative advantages opening the existing north America market to new horizons. At this point, once we got basic cash flow we can afford going to the real passion and meet the challenge of the industrial world led by microelectronics uprising of high power and frequency applications needing diamond for thermal management. This is done with government support and defense partners to prove the concept and extreme added value. Other preliminary initiatives with global partners are being developed with Medical Equipment manufacturer and Machining tools leader for composite materials. My personal lessons so far: Dare to dream, plan your dream and partner the leaders.

^{*} Invited Paper

G4: Graphene and 2-D Materials IV Session Chair: Bingbing Liu Thursday Morning, May 24, 2018 Room: Agassiz/Fremont

8:30 AM G4.01

Modified Tour Method for Synthesis of Graphene Oxide and Reduced Graphene Oxide for Photovoltaic Solar Cell Applications <u>Dulce K. Becerra</u>¹, Hailin Z. Hu¹ and Merida Sotelo²; ¹Instituto de Energías Renovables, Universidad Nacional Autónoma de México, Temixco, Mexico; ²Universidad de Sonora, Hermosillo, Mexico.

In this paper it is presented the synthesis, characterization and reduction of graphene oxide (GO) for its future application in hybrid or organic solar cells. The GO was prepared by variations of the Tour method [2], we added a different protective agent (H₂BO₂) that the commonly has been used in the method of Tour (H₂PO₄) and we reduce the oxidation time. The GO's was synthesized by the oxidation of natural graphite powder (~45 μm with purity of ~85 % wt) obtained from Mina de Sahuaripa (Sonora, México) and procedure is described as follows: for oxidation stage, a mixture of concentred sulfuric acid and boric acid (H₂SO₄/H₂BO₂) was added to a mixture of graphite powder and potassium permanganate (KMnO₄) producing a slight exotherm. The reaction was stirred for 30-90 min to 20-30 °C and then was heated to 40-55°C and stirred for 1-2 h. For exfoliation stage, deionized (DI) water was added to the suspension and then was heated to 80-90°C and stirred for 15-45 min. The reaction was then finished by adding hydrogen peroxide (H₂O₂ 50%). The resulting product has a brown/yellowish color and was separated by centrifugation from the solution. The resulting GO powders were washed 3-5 times with diluted HCl (20%) and DI water to remove the remnant Mn ions and acid respectively; for each wash, the mixture was centrifuged (6000 rpm for 20-40 min) and the supernatant decanted away. The solid obtained was dried overnight to 50-60°C, obtaining 30-40 mg of product. For reduction stage [3], 100 mg of GO was suspended in DI water and sonicated for 1-2 h, yielding an aqueous GO dispersion. This dispersion was treated with ascorbic acid (30-60 mM) and stirred for 15-24 h to 50-70°C under reflux. The resulting product has a dark color and was separated by centrifugation from the solution. The resulting rGO powders were washes 3-4 times with DI water to remove the remnant acids; for each wash the mixture was centrifuged (6000 rpm for 40-60 min) and the supernatant decanted away. The solid obtained was dried overnight to 50-60°C, obtaining 50-60 mg of product. To study the contribution of the reaction conditions to the chemical composition, presence/quantity of functional groups, exfoliation level, number of layers, oxidation and reduction degree, the samples were characterized by different techniques such as UV-Visible spectroscopy, X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and Energy Dispersive X-ray spectroscopy (EDS) [4]. The results of XRD, EDS and UV-Vis reveal that the oxygenated groups that were present have been widely removed, this can be seen reflected in the atomic ratio C/O of rGO (3.7), it increase close to 70% with respect to the GO (1.3), so that almost 50% of the oxygenated groups were removed, but the challenge is remove completely the oxygenated groups remaning and can it apply in photovoltaic solar cells. The designed synthesis strategy was to looking for a simple, easily controlled and an alternative approach for large scale production of graphene oxide (GO) for solar cell applications. This method decreases time of reaction around of 70% than the original Tour method, with the same quality and yields of graphene oxide sheets.

8:45 AM G4.02

Using a Layered Graphene Oxide/Graphene-Based Flexible Bioelectrical Sensor for RNA Detection <u>Ting T. Huang</u> and Hsien C. Huang; Ming Chi University of Technology, New Taipei City, Taiwan.

In the past decade, micro ribonucleic acids (miRNAs) have been researched extensively in several different human cancers and identified as acting as both oncogenes and tumor suppressors. miRNAs are short, single-stranded ribonucleic acid (RNA) molecules, about 19–23 nucleotides in length, that can play important regulatory roles in animal and plant cells. Dysregulated miRNA expression has been associated with

a variety of diseases, including cancer. miRNA-21 has been detected at relatively high concentrations in different cancer patients as compared to those without cancer. Using indium tin oxide (ITO) as an electrode, we transferred a graphene oxide/graphene layered structure onto a soft substrate. Due to the flexibility of the soft substrate, and the improvement of the mechanical properties of the ITO by graphene[1], the flexible component can be applied extensively to various fields. After single-layer graphene was stacked onto a graphene/copper foil, an annealing process was performed to enhance the coupling effect between the two graphene layers. The stacked double-layer chemical vapor deposited(CVD) graphene was then transferred onto an interdigital electrode, after which we used oxygen/hydrogen low damage plasma treatment (O/H-LDPT)[2] to modify it. The top layer of graphene was functionalized, through the controllable modification of the O/H-LDPT process, to form a top layer of graphene oxide. In this graphene oxide/graphene layered structure, only the top layer of graphene was oxidized as a bio-receptor; the bottom layer of graphene was kept natural as an electrical transmission layer. A synthetic probe (5'-UCA, ACA, UCA, GUC, UGA, UAA, GCU, A-3') was immobilized on the surface of the graphene oxide layer with covalent bonds. The synthetic probe was then hybridized with miRNA-21 in phosphate-buffered saline solution via the complementary property of RNA, after which we used an electrical meter to measure the variation in resistance as a function of miRNA-21 concentration. Once different concentrations of miRNA-21 are tested to determine the specificity of the components, we will detect targets that are not complementary to each other and characterize them using electrical detection. Finally, we will bend the samples to a certain extent during the testing process to analyze the influence of bending on the component and better understand its capability to detect miRNA-21.

9:00 AM G4.03

Nano-Bio Interactions of Graphene Oxide with Blood-Related Entities and Their Effects on Hematological Disorders <u>- Kenry</u>; Chemical and Biomolecular Engineering, National University of Singapore, Singapore, Singapore.

In recent years, nanomaterial-based strategies have emerged as attractive approaches in combating hematology-related diseases, such as thrombosis and malaria. With their unique physicochemical properties and biocompatibility, graphene and its related derivatives, specifically graphene oxide (GO), have been increasingly explored for this purpose. Here, we assess the nano-bio interactions between GO and blood-related entities, such as plasma proteins, blood cells, and malaria parasites, to elucidate the biophysicochemical activity of GO for hematological applications. We first investigate numerous facets of the GO-protein interactions, particularly, protein adsorption, equilibrium binding, and conformational stability. We observe that GO possesses a high loading capacity and affinity for the major blood plasma proteins, including albumin, globulin, and fibrinogen. Also, we note that these nano-bio interactions are protein-specific and may be significantly influenced by the lateral size distribution and concentration of GO. On the basis of the robust biomolecule adsorption on GO and the size- and concentrationdependent nano-bio interactions, we rationally functionalize GO and probe the antithrombotic and antimalarial properties of different GO nanomaterials in order to evaluate their potential in combating hematological disorders. We observe that functionalized GO exhibits antithrombotic characteristic while pristine GO, regardless of size distribution and concentration, inherently possesses antimalarial activity by inhibiting the invasion of malaria parasites on red blood cells and delaying the maturation of these parasites. We anticipate that this study will offer a broad perspective on the GO-blood interactions and potentially facilitate further exploration into the development of nanotechnologybased strategies for hematological and other biological applications.

9:15 AM G4.04

Graphene Oxide with Controlled Content of Oxygen Groups as a Filler for Polymer Composites Used for Infrared Radiation Shielding Marta Mazurkiewicz-Pawlicka, Maksymilian Nowak, Artur Malolepszy and Leszek Stobinski; Faculty of Chemical and Process Engineering, Warsaw University of Technology, Warsaw, Poland.

Graphene oxide (GO) is obtained by oxidizing graphite powder using modified Hummers' method. It has completely different properties comparing to the graphite powder – it is very hydrophilic and has low

electric conductivity. Due to many functional groups present in GO it shows high absorption of the infrared (IR) radiation, especially in the mid-IR region. GO can be reduced by thermal or chemical treatment. Depending on the reduction method reduced graphene oxide (rGO) with controlled content of oxygen can be obtained. After reduction GO becomes more similar to graphite - it is hydrophobic and has good electrical conductivity. Depending on the content of functional groups rGO exhibits different IR shielding properties. Based on the properties of these materials, new polymer composites with polyvinyl alcohol as the matrix were proposed for IR shielding applications. In this work, graphene oxide was reduced by different methods. Thermal (heating in air at 220°C and in nitrogen at 1000°C) and chemical (reduction with hydrazine hydrochloride and L-ascorbic acid) treatments were proposed. The obtained materials were analyzed with FT-IR spectroscopy, elemental analysis and thermogravimetric analysis (TGA) in order to evaluate the reduction degree. The polymer composites with the graphene fillers were prepared by a solution mixing method. Various amounts of GO and rGO (from 0.05 wt% to 5 wt%) were added to the polymers and the properties of the obtained materials were analyzed. The composites were characterized with FT-IR spectroscopy (in attenuated total reflectance -ATR and in transmission mode), optical microscopy, and TGA. Additionaly, thermal insulation measurements were performed for the obtained materials. The results show that the properties of the graphene oxide filler strongly influence the infrared shielding properties of the obtained polymer composites. The best IR shielding properties were obtained for the composites where rGO with the lowest oxygen content was used.

9:30 AM G4.05

Tuning of Magnetic Behaviour of Nitrogen-Functionalized Graphene-Oxide Sekhar C. Ray; Physics, University of South Africa, Johannesburg, South Africa.

Graphene-oxides are functionalized with nitrogen using different N-based precursors and hence studied their electronic structure and magnetic properties. The electronic structure of nitrogen functionalized graphene oxide were investigated using C, O and N 1s X-ray photoelectron spectroscopy (XPS), ultraviolet photoemission spectroscopy (UPS), and their magnetic behaviours are correlated with SQUID-measured magnetization M-H loops. The XPS results indicates that the different nitrogen-based precursors functionalized graphene oxide forms different N-bonding and changes their electronic structure and bonding properties. These change of electronic structure change the magnetic properties drastically. Work function and valence band maximum were estimated from the UPS measurements and are correlated with electronic behaviours of Graphene-oxide. Magnetic studies show that the magnetization is changes due to conversion due to formation of different nitrogen bonded carbon and oxygen. The change in magnetization in graphene-oxide on nitrogen functionalization using different precursors are another routes to tailor the magnetic properties of graphene-oxide for magnetic device applications.

9:45 AM G4.06

Newly Laser-Assisted Multilayer Graphene Patterning and Its Capabilities in Biosensorics and Optoelectronics Levgeniia Kovalska, Pauline Lesongeur¹, Petro Deminskyi², Coskun Kocabas³ and Anna Baldycheva¹; ¹College of Engineering, Mathematics, and Physical Sciences, University of Exeter, Exeter, United Kingdom; ²Bilkent University, Ankara, Turkey; ³The University of Manchester, Manchester, United Kingdom.

An appropriate candidate in sensing systems (as an electrode) is graphene or other 2D materials. The large surface area and superior electrical conductivity of graphene make it an excellent «electron wire» between the redox centers of an interested molecule and the electrode's surface. Owing to the extraordinary electronic transport property and high electrocatalytic activity of graphene, the electrochemical reactions of analyte are greatly promoted on the graphene film, resulting in an enhanced voltammetric response. Graphene is now a more mature technology, it can be produced in a cost-effective way, in large scale and in a short time, it shows good biocompatibility, and the source material is abundant and inexpensive. Graphene is extremely stable to the environmental conditions and does not degrade with time, which makes it an ideal material for stable sensor performance. It also has a range of surface chemistry which can be used to

modify the graphene surface and make it amenable to detecting different (bio)markers. Thus, graphene is a promising candidate for advanced electrode materials which has found its way into a wide variety of sensing schemes. Herein we report new laser-assisted patterning of graphene films which can selectively enhance certain sensory and (opto)electronic capabilities of graphene-based devices and can offer its progress of the next generation for energy storage, photonics, and bioelectronics. Basically, device's advanced multifunctionality (sensitivity, selectivity, reactivity) is driven by exerting the periodicity and forming active centers on the surface of graphene electrodes. Therefore, we represent the new approach of multilayer graphene (MLG) patterning employing nonlinear laser lithography (NLL) which is contamination-free, rapid and low-cost pattern replication technique. Via patterning, we demonstrate tuning of chemical and electro-optical properties of MLG due to laser power adjustment (310 – 380 mW). We control the hydrophobic/hydrophilic properties which define multifunctionality of graphene-enabled devices due to the graphene surface (dis)organizing, and consequently, a formation of reactive oxygen-based functional groups. Distinct graphene structure alignment (which prevents defects appearance) assisting by strongest laser power (380 mW) contributes to patterned MLG electro-optical changes (sheet resistance decreasing and optical transmittance enhancement) as well. In conclusion, we discuss the great promise of fabricated devices with sensor, supercapacitor and battery designs by using as an electrically reconfigurable medium NLL-assisted graphene patterning. The patterned MLG-based sensor demonstrates high sensitivity to exhaling molecules of humans that would be helpful for an early stage disease detection. The patterned MLG-based supercapacitor testing results reveal two times transmittance value rising (in comparison with MLG-based supercapacitor) caused by light interaction with patterned structures where its thickness is reduced. Meanwhile, the patterned MLGbased battery indicates long-life viability (500 charging/discharging cycles) with 0.01% capacity-loss per cycle. We anticipate a proposed NLL-assisted approach for MLG patterning demonstrates a new avenue to advance graphene for multifunctional device engineering particularly in energy storage, wearable electronics, and biosensorics.

10:00 AM BREAK

B4: Diamond Electronics IV Session Chair: TBA Thursday Morning, May 24, 2018 Room: Doyle/Rees

8:30 AM *B4.01

Semiconductor Integrated Diamond Materials and Devices—The Pathway to Optimal Heatsinking Martin Kuball; University of Bristol, Bristol, United Kingdom.

Many semiconductor technologies, including GaN, are limited by challenges related to an excessive temperature rise in the active device region when operated. Diamond with its high thermal conductivity can provide pathways, when integrated with semiconductors, to extract the heat from the devices, to cool at the active device region. Advances in the integration of diamond with GaN and other semiconductors will be discussed. A detailed understanding of the thermal properties of the diamond on the nano-scale is critically important for the optimal use of diamond to cool semiconductor electronics.

9:00 AM B4.02

Chemical Vapor Deposition of Diamond on GaN Using MOCVD Grown *In Situ* SiNx as Dielectric Adhesion Layer <u>Anwar Siddique</u>¹, Raju Ahmed¹, Jonathan Anderson¹, Mohammad Nazari¹, Mark Holtz^{1, 2} and Edwin Piner^{1, 2}; ¹Materials Science, Engineering and Commercialization, Texas State University, San Marcos, Texas, United States; ²Department of Physics, Texas State University , San Marcos, Texas, United States.

Effective heat spreading within a GaN-on-diamond wafer does not solely depend on thermal conductivity of Chemical Vapor Deposition (CVD) diamond, but also the effective thermal boundary resistance (TBR_{eff}) at the

^{*} Invited Paper

GaN/diamond interface. TBR_{off} is a result of phonon mismatch between materials which includes the dielectric adhesion layer used for diamond seeding, and the defective transition region at the dielectric layer/diamond interface, also referred as the nucleation layer, or nano crystalline diamond (NCD) layer. It has been reported that the contribution to the thermal resistance by dielectric adhesion layers is approximately 80 % of the TBR_{aff} . Moreover, it has also been reported that TBR_{aff} is proportional to the thickness of the dielectric adhesion layer. Therefore, the thinnest possible dielectric adhesion layer is desirable for efficient heat dissipation of GaNon-diamond. One of the biggest challenges of growing diamond on GaN is the etching of the underlying III-Nitride layer due to the harsh CVD diamond growth environment. Therefore, it is necessary to reduce the etching of the protective adhesion layer to ensure a thinnest possible interface layer. Another impact of high temperature diamond growth on nitride semiconductor is excessive thermal stress which may affect the electrical properties of the underlying nitride semiconductor device. In this study, we have demonstrated in-situ SiNx grown by MOCVD at elevated temperature followed by polycrystalline diamond growth without adversely affecting the underlying nitride semiconductor layers. SiNx/ GaN(cap)/AlGaN(barrier)/AlN(profile)/GaN(buffer) High Electron Mobility Transistor (HEMT) structures were grown using a custom built cold wall, shower head Metalorganic CVD (MOCVD) system on low resistivity (40 Ω-m) Si (111) substrates. Using X-ray reflectometry (XRR) the thickness of the SiNx is determined to be ~45 nm, which was also confirmed by Transmission Electron Microscope (TEM)measurement. Very smooth and continuous surface morphology with a root mean square (RMS) roughness of 0.14 nm for 1 µm x1 µm scan area is observed using Atomic Force Microscopy (AFM). Subsequently, seeding of the wafer is performed by spin coating using nano diamond seed suspended DMSO mixed photoresist. Poly crystalline diamond was grown in a Hot Filament CVD (HFCVD) system using 2 % CH₄ in H₂ at 700-750 °C and a chamber pressure of 20.8 torr. Uniform coverage of the diamond is observed by Scanning Electron Microscopy (SEM). Grain size, measured by linear intercept method from SEM, was 950 ± 200 nm.HFCVD grown polycrystalline diamond on AlGaN/GaN HEMT structures is realized using MOCVD grown in-situ SiNx as a dielectric adhesion layer. The interface characteristics of the GaN-SiN_-Diamond region, and the thickness of SiN after diamond growth, is evaluated by TEM. From TEM observations, in-situ MOCVD SiN_x, grown at high temperature (~ 1000 °C) is found to be two times more resistant to etching of the underlying III-Nitride film during HFCVD diamond deposition than Plasma Enhanced CVD (PECVD) grown SiN₂. High resolution X-ray Diffraction (HRXRD) reciprocal space mapping (RSM) was performed around both GaN asymmetric (1 -1 4) and symmetric plane (0 0 2) to find the stress and composition of the constituent layers before and after the diamond growth. Ultraviolet Raman spectroscopy based stress measurement of the GaN layer was performed before and after the diamond growth. XRD and Raman stress measurements indicated a compressive stress increase of ~ 0.3 GPa in GaN due to diamond growth. Having a thin, high etch resistant in-situ SiNx as dielectric adhesion layer, with reduced nucleation layer thickness as observed in TEM, this GaN-SiNx-Diamond interface is expected to be have reduced TBR_{aff}.

9:15 AM B4.03

Fabrication of Diamond Field-Effect Transistors on High-Quality Heteroepitaxial Diamond Wafer Using Microneedle Technology Makoto Kasu¹, Naru Fukami¹, Yuma Ishimatsu¹, Satoshi Masuya¹, Toshiyuki Oishi¹, Daiki Fujii² and Seongwoo Kim²; ¹Department of Electrical and Electronic Engineering, Saga University, Saga, Japan; ²Adamant Namiki Precision Jewel Co., Ltd., Tokyo, Japan.

Diamond semiconductor is expected to be high-power and high-efficient devices owing to its unexceptional physical properties such as high breakdown field strength, high carrier mobility, and highest thermal conductivity. By using NO_2 hole doping and low temperature-deposited $\mathrm{Al}_2\mathrm{O}_3$ passivation technologies we achieved, diamond FETs operate stably and reproducibly¹. Nevertheless, diamond wide wafers remain an issue. In this work, we demonstrate the operation of diamond FETs on high-quality heteroepitaxial diamond wafers. First, (001) heteroepitaxial diamonds (8 x 8mm²) were grown on Ir-deposited MgO substrates. Then, the micropattern was formed by Ni deposition and annealing². Next, epitaxial overgrowth was performed on it and dislocation density was drastically reduced. Microneedles were made on the surface and a diamond bulk layer was grown on it. By using microneedle technology², the diamond bulk was

separated very smoothly during the cooling. Then, both faces were mechanically polished, and the surface underwent chemical-mechanical polishing. The FWHM of (004) rocking curve was as low as 0.07° and the curvature was 0.29 m. These values suggest high-quality heteroepitaxial diamond. Moreover, homoepitaxial diamond was grown on it by CVD. Diamond field-effect transistors were fabricated using NO₂ hole doping and low temperature-deposited Al₂O₂ gate-oxide and passivation layers¹ on the heteroepitaxial diamond. The DC output characteristics of diamond FETs with a gate length of 3 um on heteroepitaxial diamond. The drain current at a gate voltage of -4V reached 91 mA/mm. Its drain current value were at the same level as for that of a conventional homoepitaxial single-crystal layer. In conclusion, we have demonstrated diamond FETs on high-quality heteroepitaxial diamond. The performance of these FETs is as efficient as that on the conventional homoepitaxial diamond layer. The characteristics are expected to improve by optimizing the dimension and condition. References 1. M. Kasu. Jpn. J. Appl. Phys. 56 (2017) 01AA01. 2. H. Aida, S. -W. Kim, K. Ikejiri, Y. Kawamata, A. Sawabe, et. al. Appl. Phys. Express 9 (2016) 035504.

9:30 AM B4.04

Electrical Contact Considerations for Diamond Electronics Franz A. Koeck, Manpuneet Kaur and Robert J. Nemanich; Arizona State University, Tempe, Arizona, United States.

The demonstration of diamond devices has substantiated the superior capability of diamond in high power electronics that relied on the preparation of p-type and n-type diamond through boron and phosphorus doping, respectively, and the growth of high purity intrinsic diamond. Practical devices require suitable electrical contacts and their advancement is of ongoing interest. Electrical contacts to p-type diamond are readily achieved with low contact resistance of $\sim 10^{-5}\Omega$ cm² reported. [1] Equivalent developments for n-type diamond were challenged by difficulties in the growth of highly P-doped diamond particularly on (100) oriented surfaces where elaborate micro-structuring processes to form (111) facets were suggested. [2, 3] We present an approach for electrical contacts to homoepitaxial, phosphorus doped, n-type, diamond that utilizes an interfacial layer of highly nitrogen doped, nanostructured carbon grown by plasma enhanced CVD (PECVD). This contact strategy was realized in a pin diamond diode for electron source applications. The pin-nano-carbon structure was prepared on HPHT type IIb (111) oriented substrates with intrinsic, n-type, and nano-carbon layer grown in dedicated PECVD systems. The nanostructured nitrogen doped carbon layer was synthesized with a process gas mixture of nitrogen/methane/argon/hydrogen and flow rates of 100sccm/20sccm/10 ccm/5sccm, respectively. A growth rate of ${\sim}1\mu\text{m/hour}$ was achieved using a microwave power of 900W and a chamber pressure of 20Torr with the growth temperature of ~900°C controlled by a substrate heater. Diodes from this pin-nano-carbon structure were prepared by lithography utilizing an aluminum hard mask to etch the 200µm diameter mesa contacted by Ti/Pt/Au metallurgy on the backside of the p-type substrate and top of the nano-carbon layer. The final device was processed in a hydrogen plasma for 5min at a temperature of ~850°C to induce negative electron affinity properties for electron emission. Electric characterization of the diodes commenced in vacuum after annealing at ~600°C for 15min. Observation of exciton light emission indicated bipolar transport where at a forward bias of 14V a current of 0.1A was measured and at 17V its increase to 0.5A corresponded to >1500A/cm². Compared to conventional pin diodes, the introduction of the nano-carbon layer enhanced the diode and electron emission current by more than an order of magnitude. Light emission and diode operation at temperatures >750°C indicated superior stability of the electrical contact. The n-type layer was characterized by SIMS communicating a phosphorus incorporation of ~2x10¹⁹cm⁻³ and the nanocarbon layer a nitrogen incorporation of ~5x10²⁰cm⁻³. Addressing contact limitations to n-type diamond through the growth of moderately P-doped epilayers and highly nitrogen doped nano-carbon layers could provide a preferential approach for electronic devices and we will discuss its viability extended to (100) surfaces. This research was funded by the Office of Naval Research under grant #N00014-17-1-3002. [1] Ohmic contacts on p-type homoepitaxial diamond and their thermal stability, Yigang Chen, Masahiko Ogura, Satoshi Yamasaki and Hideyo Okushi, Semicond. Sci. Technol. 20 (2005) 860-863 [2] Selective Growth of Buried n+ Diamond on (001) Phosphorus-Doped n-Type Diamond Film, Hiromitsu Kato, Toshiharu Makino, Masahiko Ogura, Norio Tokuda, Hideyo Okushi, and Satoshi Yamasaki, Applied Physics Express 2 (2009)

055502 [3] New Process for Electrical Contacts on (100) N-type Diamond, Nephi Temahuki, Remi Gillet, Vincent Sallet, Francois Jomard, Ekaterina Chikoidze, Yves Dumont, Marie-Amandine Pinault-Thaury, and Julien Barjon, Phys. Status Solidi A 2017, 214, 1700466

9:45 AM B4.05

Diamond Radiation Detectors for Proton Therapy-Lifetime and Spatial Uniformity Mengnan Zou¹, Tianyi Zhou¹, James Distel², Jen Bohon³, John Smedley⁴ and Erik Muller¹; ¹Stony Brook University, Stony Brook, New York, United States; ²Los Alamos National Laboratory, Los Alamos, New Mexico, United States; ³Case Western Reserve University, Cleveland, Ohio, United States; ⁴Brookhaven National Laboratory, Upton, New York, United States.

The most common dosimeters for particle therapy are gas filled ion chambers, which have issues with varied deviation correction according to radiation conditions and inconvenient installation due to their largevolume. One alternative is to construct compact devices from diamond, which is known for its radiation hardness, with high spatial and temporal resolution. To ensure the reliability of dose measurements, tests of the lifetime and spatial uniformity of diamond detectors were performed. We have irradiated several diamond detectors with the high flux 800MeV proton beam at LANSCE (Los Alamos National laboratory) and the ion beam induced current (IBIC) was real-time monitored for lifetime measurement. Spatial uniformity was confirmed by response maps of irradiated diamond detectors using small non-destructive X-ray beams. During the proton irradiation, an exponential decay in detector response was observed. This was explored further by X-ray beam induced current (XBIC) measurement and SRIM simulations, which reveal that the carbon vacancies introduced from displacement effect act as hole trap centers in charge collection, and a model was built to evaluate the extent of radiation damage. Currently, we are adapting our diamond x-ray imaging technology to measuring proton flux, profiles and center-of-mass, with high spatial resolution (down to 15 µm) and high temporal resolution (per pulse, ~ µs scale).

10:00 AM BREAK

A3: Diamond Growth III / NDNC Closing Session Chair: Timothy Grotjohn Thursday Morning, May 24, 2018 Room: Doyle/Rees

10:30 AM A3.01

High Resolution Selective Area Deposition of Hot Filament Chemical Vapor Deposited Diamond Films on Si, GaN, and AlN Raju Ahmed¹, Anwar Siddique¹, Jonathan Anderson¹, Mohammad Nazari¹, Mark Holtz¹.² and Edwin Piner¹.²; ¹Materials Science, Engineering and Commercialization, Texas State University, San Marcos, Texas, United States; ²Department of Physics, Texas State University , San Marcos, Texas, United States.

Diamond-on-GaN is a promising material system for thermal management in high frequency and high temperature electronics due to diamond's excellent thermal conductivity. Low cost chemical vapor deposition (CVD) of polycrystalline diamond has made this technology even more attractive from a manufacturing point of view. From a practical point of view, however, significant challenges still exist in realizing this material system because damage to the nitride semiconductor during CVD of diamond and the difficulty of patterning diamond on nitride materials. To leverage the tremendous thermal conductivity of CVD diamond, it is necessary to minimize damage to the GaN and control the selectivity of diamond when growing on nitride semiconductors such as GaN and AlN. In this study, we have demonstrated successful selective area deposition of CVD diamond on GaN and AlN with a lateral resolution as small as 5 µm and without any significant surface damage to the underlying nitride semiconductor. A selective deposition process of CVD diamond was developed and optimized on Si substrates, first. Diamond nanoparticles suspended in dimethyl sulfoxide were mixed with photoresist, and then standard photolithography techniques were employed to pattern a 100 mm

Si wafer. Reactive ion etching was performed afterward to increase selectivity. Diamond deposition was carried out in a hot filament CVD reactor designed for 100 mm wafers. Hot tungsten filaments were placed 6 mm above the substrate resulting in a surface temperature of 700-720 °C during diamond deposition. Scanning electron microscopy (SEM) images indicate excellent selectivity and cross wafer uniformity. Ultraviolet micro-Raman spectroscopy indicates the presence of diamond with a small non-diamond carbon (NDC) percentage in the deposited films. The selective seeding technique developed for Si was then used to deposit CVD diamond on metal organic CVD (MOCVD) grown GaN and AlN. Initially, significant etching of the nitride semiconductor was observed by SEM and the selectivity of the diamond was poor. Experiments were conducted that evaluated the impact of the methane concentration during CVD diamond deposition. The methane concentration was varied between 1.5% and 3% to observe changes in the diamond growth rate and its effect on etching of underlying layers. Higher methane concentrations resulted in higher growth rates, quicker surface coverage, and reduced surface etching, however, it also increased the amount of NDC. Based on growth rate, surface etching and NDC data, diamond deposition was performed with 3 % methane for its relatively higher growth rate and moderate NDC percentage. However, under these conditions, significant etching of the nitride semiconductor was observed from SEM images and selectivity of diamond was still poor. To mitigate this effect, a thin layer of plasma enhanced CVD (PECVD) SiNx was deposited on the nitride semiconductors before selective seeding and diamond deposition. This resulted in excellent selectivity of the CVD diamond with feature sizes as small as 5 µm across a 100mm wafer. The reduction in damage was confirmed by comparison of X-ray diffraction and Raman spectroscopy data collected before and after diamond deposition, and by SEM images of the final structures. In this presentation, a detailed discussion on the processing of the wafers with characterization results at different stages of the research will be discussed.

10:45 AM A3.02

Fundamentals of Bias Enhance Nucleation/Bias Enhanced Growth of Ultrananocrystalline Diamond Films on Tungsten/Silicon Substrates by Hot Filament CVD for Large Areas Application Jesus J. Alcantar-Peña²¹¹, Elida de Obaldia³¹¹, Jorge Montes²¹¹, Karam Kang¹, María J. Arellano-Jimenez⁴, Jose E. Ortega Aguilar⁴, Dainet Berman-Mendoza⁵, Miguel J. Yacaman⁴ and Orlando Auciello¹; ¹Materials Science and Engineering, The University of Texas at Dallas, Richardson, Texas, United States; ²Departamento de Investigación en Física, Universidad de Sonora, Hermosillo, Mexico; ³Facultad de Ciencias y Tecnología, Universidad Tecnológica de Panamá, Panama City, Panama; ⁴Department of Physics and Astronomy, University of Texas at San Antonio, San Antonio, Texas, United States.

Polycrystalline diamond films have been and are investigated due to their unique combination of properties such as high wear resistance, highest hardness relative to any other film, lowest friction coefficient compared with metal and ceramic coatings, chemical inertness, excellent thermal conductivity (~1500 W/K m, close to that of single crystal diamond ~2100 W/K m) perpendicular to the film surface and in the film plane for microcrystalline diamond (≥1 µm grain size), tunable electronic properties, and biocompatible properties. This presentation will focus on describing the results from systematic polycrystalline film synthesis research and system development for Bias Enhance Nucleation and Bias enhance Growth (BEN-BEG) to growth ultrananocrystalline diamond (UNCD) films on medium size areas (100 mm diameter) Silicon wafers coated with 40 nm thick Tungsten film to induce denser films. The BEN-BEG process to growth diamond films eliminates the conventional chemical seeding process used today. The studies discussed are focused on understanding the BEN mechanism by varying the BEN time between 0.5 and 2.5 hrs. Following the BEN process, films were grown for 2.0 hrs without bias. A H2 (49%)/ CH4 (2%)/Ar (49%) gas mixture was flown in the HFCVD chamber previously evacuated to $2 \sim 3 \times 10^{-6}$ Torr. The substrate surface was heated to 650-700 C via a combination of heat provided by an underlying substrate heater and radiation from an array of ten parallel filaments heated by electronic current to ~ 2300 C. An electric field was created between the filaments (bias positively) and the substrate (biased negatively) to attract positively charged $\text{CH}x^{\scriptscriptstyle +}, \text{Ar}^{\scriptscriptstyle +},$ and $\text{H}^{\scriptscriptstyle +}$ ions created in plasma stablished on the substrate surface, to induce the BEN and growth of the UNCD films. Computer simulations and experiments showed that electric fields concentrated on the substrate edges initiating the film growth, which

propagated towards the center of the substrate, as the insulating UNCD film growth inducing electric field displacement towards the uncoated area to finally induce a uniform film growth across the substrate. Evidence of very homogeneous nucleation by BEN appears at 2.0 and 2.5 hrs. However, at 2.5 hrs of BEN, signs of etching of the UNCD film is observed, probably due to long exposure ion bombardment-induced sputtering or ion bombardment-enhanced chemical reaction between H atoms and C atoms on the UNCD film surface, inducing formation of volatile CH4 molecules, or a combination of both processes. According to the studies made until now, BEN for 2.0 hrs appears to be the optimum process to growth very dense film across 100 diameter wafer. HRTEM studies show that 2.0 hrs. BEN induce the growth of tungsten carbide layer with crystals orientation (001 and 101), which induce the formation of diamond grains (111), followed by change of diamond grain orientation to (220) and (311). These results show that the BEN process could be used to tailor orientation of UNCD film grains. In addition, it was demonstrated that the HFCVD BEN-BEG process can be used to grow diamond films selectively on electrically conductive patterns produced on insulating substrate surfaces, providing a pathway for fabrication of diamond based devices on SiO₂/Si substrates.

11:00 AM A3.03

Laser-Nucleated Cleaving of (111)-Faced Single-Crystal Diamond Plates Samuel M. Parks, Richard Grote, David A. Hopper and Lee Bassett; University of Pennsylvania, Philadelphia, Pennsylvania, United States.

Single-crystal diamond plates with surfaces oriented in a (111) crystal plane are required for high-performance solid-state device platforms ranging from power electronics to quantum information processing architectures. However, producing plates with this orientation has proven challenging. We demonstrate a method for reliably and precisely fabricating (111)-faced plates from commercially available, chemical-vapor-deposition-grown, type-IIa single-crystal diamond substrates with (100) faces [1]. Our method uses a nanosecond-pulsed visible laser to nucleate and propagate a mechanical cleave in a chosen (111) crystal plane, resulting in faces as large as 3.0 mm × 0.3 mm with atomically flat surfaces, negligible miscut angles, and near zero kerf loss. We will discuss the underlying physical mechanisms of the process along with potential improvements that will enable the production of millimeter-scale (111)-faced single-crystal diamond plates for a variety of emerging devices and applications. The (111) crystal plane is the hardest face, making it more resilient to damage and desirable for industrial scale diamond cutting tools. Similarly, the Raman gain coefficient is strongest when the optical polarization axis is aligned along [111]. For this reason, integrating on-chip Raman lasers requires precise crystallographic orientation of the diamond layer to maximize the laser efficiency. High-power electronic devices also benefit from (111) faces, which provide improved donor incorporation efficiency and correspondingly higher mobility. Finally, point defects in SCD that are used as single-photon sources and spin qubits, such as the nitrogen-vacancy and silicon-vacancy centers, have a symmetry axis aligned along (111) directions. Thus, a (111) face maximizes the interaction with normally-incident light fields and optimizes the photon collection efficiency from these emitters, which is critically important for light-matter quantum interfaces and high-sensitivity magnetometry. In contrast to traditional mechanical cleaving of diamond, laser-nucleated cleaving (LNC) provides the opportunity for precise positioning and control of the cleave propagation. Since kerf loss is minimized, LNC may be desirable for faceting diamond gems or cutting tools. Additionally, the LNC process has potential for eliminating plate wedge, which adversely affects diamond photonic devices. LNC will ultimately prove useful for fabricating seed crystals required for improving CVD growth of customized [111]-oriented samples, and for generating (111)-faced SCD substrates for diamond electronics, photonics, and quantum technologies. [1] S. M. Parks, R. R. Grote, D. A. Hopper, and L. C. Bassett, "Fabrication of (111)-Faced Single-Crystal Diamond Plates by Laser Nucleated Cleaving." arXiv:1712.03882 [cond-mat.mtrl-sci].

11:15 AM A3.04

Fundamentals of Processes Involved in the Synthesis of Electrically Conductive Ultrananocrystalline Diamond Films with Grain Boundary Inserted Nitrogen Atoms Grown by Hot Filament Chemical Vapor Deposition Gerardo Garibaldi^{2,3}, Jesus J. Alcantar-Peña³, Pablo A. Tirado^{3,4}, Amanda Gorby³, <u>Elida de Obaldia^{2,3} and Orlando Auciello¹</u>;

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A discussion will be presented on systematic studies focused on investigating the fundamentals of processes involved in the growth of electrically conductive grain-boundary incorporated ultrananocrystalline diamond films named N-UNCD, grown using the Hot Filament Chemical Vapor Deposition (HFCVD) technique. Parameters investigated to develop an optimum process to achieve the highest electrical conductivity in N-UNCD films include; a) the flow rate (critical) of each gas in the mixture of gases used to grow the films, b) the filament-to-substrate distance, c) the temperature of the filaments (set for all grows at 2300 C), d) the temperature of the substrate surface (600-650 C), and e) the substrate material (high conductivity Si and insulating SiO, (300 nm thick)/Si substrates). Insulating UNCD films were grown first, as control for comparison, using a mixture of Ar (10 sccm)/CH₄ (2) sccm)/H₂ (20 scm), which produced films with high sheet resistance (M Ω /sq.), as measured by the four-point probe method. Subsequently, the first N-UNCD film was grown with Ar (10 sccm)/CH₄ (2) sccm)/H₂ (20 scm)/ and N₂ (1 sccm) with a filament-to-substrate distance of 10 mm, which also produced an insulating film. An hypothesis that could explain the growth of insulating UNCD film, although N, gas was flowing, was that the 10 mm filament-to-substrate distance resulted in N atoms recombination to N₂ molecules during the flight path from the filament to the substrate, thus inhibiting atomic N insertion into the grain boundaries, as required to produce high electrical conductivity N-UNCD films, demonstrated in extensive prior research growing N-UNCD films using microwave plasma chemical vapor deposition (MPCVD). Thus, the next series of N-UNCD films grown using HFCVD involved N, flow rates of 1, 1.5 and 2.0 sccm and a filament-to substrate distance of 5 mm, which produced films with 14-23 $\Omega/\text{sg.}$, $0.09 - 0.32 \Omega/\text{sg.}$, and $0.05-0.14 \Omega/\text{sg.}$ sheet resistance, respectively. Indicating that N, flow is very important once the other parameters have been optimized. The chemical nature of the UNCD and N-UNCD films were studied using Raman spectroscopy, which revealed the characteristic spectrum for insulating UNCD and conductive N-UNCD, and X-ray photoelectron spectroscopy (XPS), which revealed the incorporation of N atoms in the N-UNCD films, which exhibited high electrical conductivity. The surface morphology and thickness of the films were characterized using top and cross-section views scanning electron microscopy (SEM), respectively, the latter revealing relatively uniform film thickness (~ 250 nm) across 100 mm diameter wafers. Finally, Hall effect measurements were performed to determine the type of electrical behavior (p or n type) in the N-UNCD films. The high conductivity N-UNCD films will be tested integrated on different media, seeking to identify their degree of efficiency for key technological applications as corrosion resistant coatings for encapsulation of electrodes of Li-ion batteries to protect the base material from Li-induced degradation and for high efficiency photovoltaic cells.

11:30 AM A3.05

Boron Doping of Ultrananocrystalline Diamond Films on Silicon Substrates by Thermal Diffusion Pablo A. Tirado^{1, 2}, Jesus J. Alcantar-Peña², Elida de Obaldia^{2, 3}, Rafael García¹ and Orlando Auciello^{2, 4}; ¹Departamento de Investigación en Física, Universidad de Sonora, Hermosillo, Mexico; ²Material Science and Engineering, University of Texas Dallas, Richardson, Texas, United States; ³Facultad de Ciencia y Tecnología, Universidad Tecnológica de Panamá, República de Panamá, Panama; ⁴Department of Bioengineering, University of Texas Dallas, Richardson, Texas, United States.

The physics and applications of Ultrananocrystalline Diamond (UNCD) films are been investigated due to their unique combination of properties such as high wear resistance, highest hardness relative to any other film, lowest friction coefficient compared with metal and ceramic coatings,

chemical inertness, negative electron affinity, low work function, and the high electrical conductivity for boron doped and nitrogen grain boundary incorporated diamond films. The combination of these properties make doped diamond films suitable for many applications like electrodes for water purification, thermionic and field emission devices, and high power electronic devices. Boron doping of UNCD films during growth has the drawback of Boron contamination of the chamber where the film is grown, which can only be used for the growth of B-doped diamond films. This presentation will focus on describing the results from research and development of a novel process for Boron doping large area UNCD films by thermal diffusion after growth, thus eliminating the problem of Boron contamination of the diamond film growth chamber. The research is focused on understanding the chemical, structural and electrical properties of the UNCD films before and after doping with Boron by thermal diffusion. The UNCD films were grown by Microwave Plasma Chemical Vapor Deposition (MPCVD) technique on (1 0 0) Silicon substrates. Afterwards, a 200-nm thick Boron containing film was deposited, as a B source, on the UNCD films's surface via spin coating, using a commercial Spin on Dopant (SOD) solution (Boroflim 100 ®). A spinning speed of 3000 rpm and a process time of 20 seconds was used for depositing the SOD film on the UNCD film surface, followed by annealing in an atmospheric oven at 200°C for 20 minutes in order to evaporate any excess solvent from the SOD film. The diffusion process was carried in a Jetfirst 200 Rapid Thermal Processor (RTP) in a low oxygen atmosphere at 800 °C, 900 °C, and 1000 °C for 180s. Successive chamber evacuations and purges with 1500 sccm of N, were performed to minimize O, content in order to avoid the C-based UNCD film being etched by oxygen at the high temperatures needed for Boron diffusion. Once the diffusion process was over, the UNCD films were ultra-sonically cleaned with acetone, methanol and isopropanol in order to remove any remaining Spin on Dopant film from the surface. The boron doped, and as deposited films were characterized by Raman, XRD, XPS and Hall effect measurements. Raman and XRD characterizations were done to confirm that there was no induced graphitization or damage in the films during the diffusion process, while XPS, and Hall effect characterizations were carried to confirm the boron doping and the change in electrical properties (sheet resistance, charge carrier concentration) during the diffusion process. This new process to produce B-doping of UNCD films can in principle be extended to NCD ad MCD films to explore the development of a new generation of diamond based electronics.

11:45 AM CLOSING COMMENTS

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