Science and Technology
of Materials, Interfaces, and Processing

2018 Spring Meeting
Hudson Mohawk AVS Chapter
Monday, April 30, 2018
4:00 – 8:00 PM

General Electric Global Research Center
1 Research Circle
Niskayuna, NY 12309
Conference Center Rooms 4 – 5

Meeting Agenda*:

4:00 PM – 4:05 PM	**Introductory Comments (Conference Center Rooms 4 – 5)**
4:05 PM – 6:05 PM	**Focused session: Additive Manufacturing**
6:05 PM – 7:30 PM	**Poster presentations and networking with pizza and beverages**
7:30 PM – 7:40 PM	**Awards Ceremony**
7:40 PM – 8:00 PM	**AVS Hudson Mohawk Chapter Executive Committee Meeting**

Complete presentation schedule is available in the next page.

***Presentation Schedule:**
(Talks are of 15 minutes with 5 more minutes for Q&A.)

4:00 – 4:05 PM	Welcome note
	Focused Session on *Additive Manufacturing*
4:05 – 4:25 PM	William T. Carter, GE-GRC -- Direct Metal Laser Melting.
4:25 – 4:45 PM	Laura Dial, GE-GRC -- Materials Development for Beam-Based Powder Bed Additive Manufacturing.
4:45 – 5:05 PM	Justin Gambone, GE-GRC -- In-Process Analytics and Controls for Powder Bed Fusion Additive manufacturing.
5:05 – 5:25 PM	Andrey Meshkov, GE-GRC -- High Fidelity Melt Pool Modeling of Powder Bed Fusion Additive Manufacturing.
5:25 – 5:45 PM	Arun Natarajan, GE-GRC -- Binder Jet Technology for Additive Manufacturing.
5:45 – 6:05 PM	Steven Attanasio, Naval Nuclear Laboratory (NNL) -- Microstructure of AM-Fabricated Stainless Steel and Effects on Materials Properties.
6:05 – 7:30 PM	**Poster Presentations**

DIRECT METAL LASER MELTING

William T. Carter

GE Global Research, 1 Research Circle, Niskayuna, New York 12309

The Powder Bed Fusion process got its start in 1986 with an invention at the University of Texas at Austin by an undergraduate student. In the 30+ years since, it has seen steady growth resulting in a new manufacturing technology that has evolved from "rapid prototyping" to "functional prototyping," and now into full-scale production of metal parts.

Interest at General Electric started 10 years ago with investigations into reducing the cost of a fuel injector for an aircraft engine. GE quickly realized that additive manufacturing could free designers from constraints of conventional manufacturing processes, opening paths for novel fuel-saving designs. More importantly, multiple parts could be consolidated into single parts to reduce cost. This talk will feature some of the lessons learned in that development, and highlight some of the active research in developing the process for higher speed, higher accuracy, and larger parts.

ADDITIVE MANUFACTURING STUDIES WITH FOCUS ON TYPE 316L STAINLESS STEEL MICROSTRUCTURES

Steve Attanasio, Steve Sabol, Tressa White, Nathan Lewis,
Robert Morris, Trevor Hicks, Tyler Tenkku, and Justin Kurp

Naval Nuclear Laboratory (NNL)
Schenectady, NY; West Mifflin, PA

Metal Additive Manufacturing (AM) is a growing technology in industry, where digital 3D files are used to drive fabrication of components. Most commonly, laser or electron beam heat sources are directed in a programmed pattern to locally melt powder or wire feedstock one layer at a time, which solidifies to produce the desired shape. Typically, near-net shape parts are produced, with heat treatment and final machining needed. The capabilities of AM processes have spurred changes to fabrication methods in the aerospace and medical industries with modest changes to date in other areas such as the nuclear industry. This presentation will provide an overview of potential benefits of testing and qualifying AM technology to support pressurized water reactor design, testing, and fabrication. Prospective benefits include performance and manufacturing gains (e.g., reduced delivery time, fabrication of hard-to-source parts, part consolidation) for tooling, test articles, rapid prototyping, repairs, or hard-to-fabricate components.

Microstructure studies have been performed to develop insight to AM process/structure/property relationships, with focus on the laser powder bed fusion (L-PBF) process. A range of techniques were used including metallography, scanning electron microscopy, electron backscatter diffraction, transmission electron microscopy, and chemical mapping of local regions of the metal at the atomic level using atom probe tomography. Advanced characterization performed on Type 316L Stainless Steel (316L SS) fabricated via L-PBF shows differences in microstructure and properties when compared to wrought or conventionally-welded 316L SS. The rapid solidification rates (and oxygen pickup) in the L-PBF process affect microstructure and properties. Process modeling is also being used in an effort to predict distortion and residual stress in AM builds. Understanding process/structure/property relationships is key to deployment of AM-fabricated metals, particularly for demanding service conditions and/or long lifetimes.

Poster presentation

CHARACTERIZATION TO SUPPORT ADDITIVE MANUFACTURING

Vincent S. Smentkowski, James Grande, Yan Gao

GE Global Research Center, Niskayuna NY 12309

A variety of methods and materials are being used by the additive manufacturing community to fabricate parts which cannot be made using other techniques. The six talks in the symposium will highlight the techniques being used at locally at General Electric -Global Research Center (GE-GRC) and Knolls Atomic Power Laboratory (KAPL). This electronic poster (scrolling power point slides) provides a high-level overview of characterization to support additive manufacturing; these slides were assembled for the 2016 America Makes Symposium which took place at GE-GRC in the summer of 2016.

Keywords:
Characterization, additive manufacturing, microscopy, bulk analysis, surface analysis, synchrotron, diffraction

Poster presentation

INVESTIGATION OF COPPER DIFFUSION BARRIER BY X-RAY PHOTOELECTRON SPECTROSCOPY

Yibin Zhang[1], Zhiguo Sun[1]

[1]*GLOBALFOUNDRIES, 400 Stone Break Rd Extension, Malta NY USA*

Refractory metals such as reactively sputtered TaN and/or sputtered Ta films have been commonly used as copper diffusion barriers to prevent Cu diffuses into Ultra Low K materials at back end of line in semiconductor industry. In this study, Ta or TaN films were deposited on Ultra Low K materials by self-ionized plasma system with various plasma conditions. The variations of TaN compositions are revealed by X-ray photoelectron spectroscopy (XPS). Different Ta chemical states were found at the interface of Ta or TaN /ULK. They were formed through chemical reaction between Ta or TaN with underneath ULK materials. And the Carbon (C) was depleted in ULK layer which might degrade the performance of devices, because K value will change significantly due to C change in ULK. A failure mechanism will be discussed in this study based on the interfacial characterization results.

Poster presentation

STRUCTURAL AND ELECTRONIC CHARACTERIZATION OF ATOMIC LAYER DEPOSITED HAFNIA- ZIRCONIA NANOSCALE FILMS

Vineetha Mukundan* and Alain Diebold

Colleges of Nanoscale Science and Engineering, SUNY Polytechnic Institute, Albany, NY

*Email: vmukundan@sunypoly.edu

There is a continual push for electrical performance improvements in both gate and channel materials with the scaling down of the metal oxide semiconductor devices for future technology nodes. Hafnia-based gate dielectric materials have recently been successfully integrated into high volume manufacturing. To continue device scaling and enhance gate dielectric performance beyond the 10nm technology nodes, crystal engineering via atomic layer deposition (ALD) of hafnia-based dielectrics is attracting significant attention due to ability to obtain higher K-values, while engineering lower leakage current and reduced equivalent oxide thickness. The recent discovery of ferroelectric behavior in this material has led to attractive applications such as negative differential capacitance field effect transistors and ferroelectric memory devices. The thermodynamically stable phase of pure HfO_2 is the monoclinic phase and other metastable phases are the tetragonal, orthorhombic and cubic phases. The cubic and tetragonal phases of HfO_2 are predicted to have the highest dielectric constant [1]. Although the monoclinic phase is thermodynamically preferred, the various metastable phases can be stabilized at higher pressure and/or temperature. Alloying with similar oxides like ZrO_2, doping with specific elements, novel processing methods, and annealing schemes are also some of the techniques that are being explored actively to target structural modifications with a simultaneous improvement in electrical properties. Synchrotron based x-ray diffraction in the grazing in plane geometry reveals a rich mixture of crystalline phases in hafnia-zirconia compositional alloys explored in this study. Advanced processing techniques, like the cyclical deposition and anneal method for the ALD growth of hafnia-zirconia materials, have been shown to form higher k-tetragonal phase on silicon and germanium substrates [2-4]. Observation of ferroelectric behavior when capped and annealed with a metal such as TiN makes these materials highly attractive for non-volatile memories and ferroelectric field effect transistors [5,6].

References:

[1] X. Zhao et al., Phys. Rev. B 65, 233106 (2002)
[2] R. D. Clark et al., ECS Trans. 35(4), 815 (2011)
[3] K. Tapily et al., ECS Trans. 45(3), 411 (2012)
[4] S. Dey et al., J. Appl. Phys. 120, 125304 (2016)
[5] M. H. Park et al., Adv. Mater. 27, 1811, (2015)
[6] S. Dey et al., Frontiers of Characterization and Metrology for Nanoelectronics: 2017 (NIST, 2017)

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Poster presentation

EPITAXIAL ZnTe THIN FILMS GROWN ON GLASS USING SINGLE-CRYSTALLINE GRAPHENE AS BUFFER

Xin Sun[1,*], Zhizhong Chen[2], Yiping Wang[2], Zonghuan Lu[1], Jian Shi[2], Morris Washington[1], Toh-Ming Lu[1]

[1]Center for Materials, Devices and Integrated Systems and Department of Physics, Applied Physics and Astronomy, Rensselaer Polytechnic Institute, Troy, New York, 12180, USA
[2]Department of Materials Science and Engineering, Rensselaer Polytechnic Institute, Troy, New York 12180, USA

*Email: sunx12@rpi.edu

Graphene template has long been promoted as a promising host to support van der Waals flexible electronics. However, van der Waals epitaxial growth of conventional semiconductors in planar thin film form on transferred graphene sheets is challenging because the nucleation rate of film species on graphene is notoriously low due to the passive surface of graphene. In this work, we demonstrate the epitaxy of zinc-blende ZnTe thin film on single-crystalline graphene supported by an amorphous glass substrate. Given the amorphous nature and no obvious remote epitaxy effect of the glass substrate, this study clearly proves the van der Waals epitaxy of a 3D semiconductor thin film on graphene. X-ray pole figure analysis reveals the existence of two ZnTe epitaxial orientational domains on graphene, a strong X-ray intensity observed from the ZnTe $[\bar{1}\bar{1}2]$ || graphene [10] orientation domain and a weaker intensity from the ZnTe $[\bar{1}\bar{1}2]$ || graphene [11] orientation domain. Furthermore, this study systematically investigates the optoelectronic properties of this epitaxial ZnTe film on graphene using temperature-dependent Raman spectroscopy, steady-state and time-resolved photoluminescence spectroscopy, and fabrication and characterization of a ZnTe-graphene photodetector. The research suggests an effective approach towards graphene-templated flexible electronics.

Poster presentation

EFFECT OF OXIDATION ON THE RAMAN AND PHOTOLUMINESCENCE PROPERTIES OF UNPASSIVATED AND NOVEL-PASSIVATED LAYERED GaTe

Mounika Kotha[1,*], Spyros Gallis[1]

[1]*Colleges of Nanoscale Sciences and Engineering, SUNY Polytechnic Institute, Albany, New York 12203, USA*

*Email: mkotha@sunypoly.edu

Emerging two-dimensional gallium chalcogenides, such as gallium telluride (GaTe), are considered promising layered semiconducting materials that can serve as vital building blocks towards the implementation of nanodevices in the fields of nanoelectronics, optoelectronics and quantum photonics. However, surface-induced electronic, structural and optical changes observed in ambient-exposed gallium chalcogenides need to be further investigated and addressed. Herein, we report on the thickness-dependent effect of air and oxygen exposure on the Raman and photoluminescence (PL) properties of GaTe flakes, with thicknesses spanning in the range of a few layers to 65 nm. We have developed a novel chemical passivation technique that results in complete encapsulation of the as-exfoliated GaTe flakes in ultrathin hydrogen-silsesquioxane (HSQ) film. A combination of correlation and comparison of Raman and PL studies reveal that the HSQ-capped GaTe flakes are effectively protected from oxidation in oxygen and air ambient over the studied-period of 25 days, and thus, preserving their structural and optical characteristics. This contrasts with the behavior of uncapped GaTe, where we observe a significant reduction of the GaTe-related PL (~100×) and Raman (~4×) peak intensities. Furthermore, the time-evolution of the Raman spectra is accompanied with the appearance of two new prominent peaks, at ~130 cm^{-1} and ~145 cm^{-1}, which are attributed to the oxidation of the uncapped GaTe. The oxidation of uncapped GaTe is consistent with our Auger spectroscopy findings. Our surface-passivation offers a new approach to further explore and reveal the physical properties of layered GaTe, with the potential of fabricating reliable nanophotonic devices with electronic, structural and optical stability.

Graduate student.

Poster presentation

OPTIMIZATION OF THE GROWTH OF GRAPHENE ON Cu FOIL SUBSTRATES BY CHEMICAL VAPOR DEPOSITION

Siddarth Laveti[1], Jodi Hotalen[2], and Carl A. Ventrice, Jr.[1,2]

[1]*Colleges of Nanoscale Science and Engineering, SUNY Polytechnic Institute*
[2]*College of Nanoscale Science & Engineering, University at Albany-SUNY*

Email: SLaveti@sunypoly.edu

The most common method for producing large area graphene films is by performing chemical vapor deposition (CVD) on Cu foil substrates. The reason for using Cu as a substrate is that it has a very low solubility for C at the temperature that the CVD is performed, which ensures a self-limited growth of a single monolayer of graphene. The goal of this research project is to determine the optimal procedure for producing graphene films with a low defect density on Cu foil substrates. The graphene films were grown on 99.8% pure Cu foils 0.001" thick, 0.25" wide, and 6" long. The Cu films were heated by passing a current through the film, and the temperature was monitored using a disappearing filament optical pyrometer. The precursor used was ethylene (C_2H_4).

Three methods were used to grow the graphene films. The initial step in each growth process was to anneal the Cu foil in 1 x 10^{-5} Torr of H_2 at 800 °C for 30 min to remove the native oxide. The first growth procedure involved annealing the film in 1 x 10^{-6} Torr of O_2 at 500 °C for 10 min after the initial H_2 anneal to remove residual carbon from the surface of the Cu foil, followed by annealing in 1 x 10^{-5} Torr of H_2 at 800 °C for 10 min to remove chemisorbed oxygen from the surface from the previous anneal in O_2. The growth of the graphene film was then performed by introducing 5 mTorr of H_2, 5 mTorr of C_2H_4, and 5 Torr of Ar and annealing at 850 °C for 30 min. The second growth procedure also used the oxygen treatment to remove residual carbon but did not use H_2 during the graphene growth process. The third growth procedure did not use the oxygen anneal to remove residual carbon and used H_2, C_2H_4, and Ar during the graphene growth. The reason for not doing the O_2 anneal was to determine how residual carbon might affect the subsequent graphene growth.

The samples were characterized using angle-resolved X-ray photoelectron spectroscopy (AR-XPS) and scanning electron microscopy (SEM). The XPS data showed that all samples had a very low oxygen content, which indicates that the Cu foil was completely covered by graphene. The area under the C-1s peak for all of the samples was approximately the same, which indicates that all of the samples were a monolayer thick. The SEM data show a similar growth morphology for the samples annealed in O_2 before graphene growth. The sample grown without the oxygen anneal had a rougher topography, indicating that the residual carbon adversely affected the graphene growth process.

Undergraduate student.

Poster presentation

SIMULATED ELECTROMAGNETIC FIELD MANIPULATION FOR OPTIMIZED ELECTROHYDRODYNAMIC PRINTING

Matthew, Strohmayer, Atul Dhall, Natalya Tokranova, and James Castracane

SUNY Polytechnic Institute, Colleges of Nanoscale Science and Engineering, Albany, NY

Email: mstrohmayer@sunypoly.edu

In 3-D printing, obtaining high resolution (<1 um) printing is difficult and costly. Electrohydrodynamic printing (EHD) is a method used to 2-D print dots and fibers on the nanoscale at a much lower cost than most current methods. Currently EHD has some hurdles to make it a viable high resolution 3-D printing technique; including wavy prints, proximity to substrate, and careful electric field strength control required for printing as structures are built up. In order to reduce the waviness and allow for more variation in substrate proximity, an electromagnetic lens system is proposed. Electromagnetic field manipulation through electrostatic or magnetic lenses will enable high resolution, high precision and repeatable prints from nanoscale EHD 3-D printing. In EHD, the droplets carry a charge because of the polarization build up on the Taylor cone surface prior to release from the needle. This native charge can then be redirected using forces from externally applied fields. A simulation of several different methods of focusing was performed to confirm its functionality. The approximate charge and weight of a drop was calculated and then used as input for these simulations. This modeling also functions as a means to determine other relevant parameters to the system, such as applied current to the lens, and the resulting field configurations. The current status of the simulations and initial experimental setup will be presented.

Graduate Student.

Poster presentation

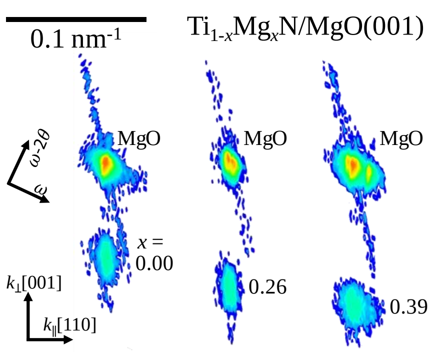
GROWTH AND PROPERITIES OF EPITAXIAL $Ti_{1-x}Mg_xN(001)$ LAYERS

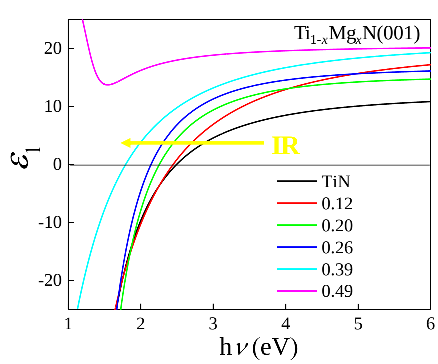
Baiwei Wang and Daniel Gall

Department of Materials Science and Engineering, Rensselaer Polytechnic Institute, Troy, NY 12180, USA

wangb12@rpi.edu

The goal of this research is to grow a completely new semiconductor, $Ti_{0.5}Mg_{0.5}N$. This compound has never been synthesized yet but has been theoretically predicted to exhibit a 1.3 eV band gap and to have promising properties for thermoelectric and plasmonic devices without compromising its refractory and CMOS-compatible performances. More specifically, in this work, epitaxial $Ti_{1-x}Mg_xN(001)$ layers were deposited on MgO(001) by reactive magnetron co-sputtering from titanium and magnesium targets in 5 mTorr pure N_2 at 600 °C. The combination of X-ray diffraction ω - 2θ, φ-scans, pole figures, and high resolution reciprocal space maps (RSMs) show that rock-salt $Ti_{1-x}Mg_xN$ layers are epitaxial single crystals which grow cube-on-cube with respect to their substrates: $(001)_{TiMgN} \| (001)_{MgO}$ and $[100]_{TiMgN} \| [100]_{MgO}$. $Ti_{1-x}Mg_xN/MgO(001)$ layers, ~ 50 nm thick, are fully strained after deposition, with in-plane lattice parameters at constant 4.212 ± 0.001 Å and out-of-plane lattice parameters increasing from 4.254 Å (x = 0.00) to 4.308 Å (x = 0.49). The in-plane x-ray coherence length decreases from 212 to 25 nm as x increases from 0.00 to 0.49, while the out-of-plane x-ray coherence length is confined by the layer thickness d for $x \leq 39$, but is smaller than d for $x \geq 0.45$, indicating a decreasing crystalline quality with increasing Mg content and local strain variations along the film growth direction. The electrical resistivity of $Ti_{1-x}Mg_xN$ layers increases from 13 to 358 $\mu\Omega$·cm for x = 0 and 0.49, respectively. Optical transmission and reflectivity spectra are described by a Drude–Lorentz model. Optical data fitting indicates that the free carrier density drops by a factor of four, from 1.3×10^{22} cm^{-3} for TiN to 3.3×10^{21} cm^{-3} for $Ti_{0.51}Mg_{0.49}N$, while the optically determined resistivity agrees well with the values from 4-point probe measurements for alloys with compositions $x \geq 0.26$. The real part of the dielectric function indicates that the plasma frequency ω_p moves into the infrared region for compositions with $x \geq 0.39$, indicating that $Ti_{1-x}Mg_xN$ is promising for plasmonic applications.





Graduate Student.

Poster presentation

SINGLE CRYSTAL VAN DER WAALS PEROVSKITE $(C_4H_9NH_3)_2PBI_4$ BY VAPOR PHASE EPITAXY WITH EXCITON BINDING ENERGY ~300 meV

Zhizhong Chen[1], Xin Sun[2], Xi Wang[3], Hanwei Gao[3], Toh-Ming Lu[2], Jian Shi[1]

[1]Department of Materials Science and Engineering and
[2] Department of Physics, Applied Physics and Astronomy, Rensselaer Polytechnic Institute, Troy, NY, 12180
[3]Department of physics, Florida State University, Tallahassee, FL, 32306

Similar to III-V semiconductors-based quantum wells, 2D van der Waals perovksites $(RNH_3)_2PbX_4$ are self-assembled alternating layers of inorganic lead halide and organic ligands. Due to reduced screening from organic layers, the exciton confined in inorganic lead halide layers show binding energy of several hundred meV, much higher than in ZnO or GaN. The highly stable excitons, along with direct bandgap nature, render $(RNH_3)_2PbX_4$ an ideal candidate for exciton or polariton studies. While most of these materials/devices were synthesized/fabricated from solution methods, vapor-based synthesis are still needed to minimize impurity and defects. In this work, we present the vapor-phase growth of single crystalline $(C_4H_9NH_3)_2PbI_4$ flakes with high optical quality. Individual single crystalline domains with lateral size about 5-10 µm and thickness 15-100 nm were deposited on Si, SiO_2/Si or muscovite mica, showing well-defined rectangular shape. Epitaxial relation were observed between perovskite flakes and mica/Si substrates. Due to the substrate effect therein, the structural phase transition at around 240 K were hindered and room-temperature phase were stabilized till liquid nitrogen temperature. Room temperature photoluminescence (PL) showed full width at half maximum (FWHM) of 70 meV and decay lifetime of several nanoseconds, indicating comparably high quality with mechanically exfoliated counterparts. Based on temperature dependent PL intensity, exciton binding energy 279 ± 46 meV and electron–phonon coupling (Fröhlich) strength around 20 meV are revealed. This vacuum-based method showed better controllability over thickness and structure, and may provide a solution for integrating layered perovskites into optoelectronic devices and systems.

Graduate Student.