Spring 2016 Meeting of the Hudson Mohawk Chapter of AVS

General Electric Global Research Center
1 Research Circle
Niskayuna, NY 12309
Conference room 4 and 5
Monday May 9, 2016 4pm to 8pm

Agenda:

Talks:

X-ray Sources and Systems

Need for Advanced Medical Imaging
Vincent S. Smentkowski, PhD Chem/Surface Scientist, Engineered Materials Chemistry & Physics, Nanostructures & Surfaces Laboratory, GE GRC

History of GE, GRC, and X-ray Sources
J. Scott Price, PhD Physicist/X-ray Technologist, Radiation Systems Laboratory, Diagnostics, Imaging, and Biomedical Technologies, GE GRC
GE's role in early X-ray production
X-ray physics and sources

X-ray Detectors
Brian D. Yanoff, PhD Manager Detector Laboratory, Diagnostics, Imaging, and Biomedical Technologies, GE GRC

X-ray Systems – CT and beyond
Peter M. Edic, PhD Principal Engineer, Diagnostics, Imaging, and Biomedical Technologies, GE GRC
Eric J. Tkaczyk, PhD Principal Scientist, Diagnostics, Imaging, and Biomedical Technologies, GE GRC

Non-Medical Uses of X-rays—Non-Destructive Evaluation
Clifford Bueno, PhD Metrologist/NDE Investigator, Manufacturing & Materials, Inspection & Metrology Laboratory, GE GRC

Impact of AVS on X-ray Source Development
J. Scott Price, PhD Physicist/X-ray Technologist Radiation Systems Laboratory, Diagnostics, Imaging, and Biomedical Technologies, GE GRC

Poster Presentations:

X-RAY POLE FIGURE ANALYSIS OF TEMPERATURE-DEPENDENT EPITAXY IN $Ge_{1-x}Sn_x$ FILMS GROWN ON CaF_2

A. J. Littlejohn,[ab†] T.-M. Lu,[ab] L. H. Zhang,[c] K. Kisslinger,[c] and G.-C. Wang[ab]

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$Ge_{1-x}Sn_x$ alloy has been studied over the past decades because several of its properties, namely its high mobility and IR bandgap which can be tuned by changing the Sn alloying concentration, x, make it an attractive candidate for implementation in Si-based optoelectronics. In this study, sequential thermal evaporations of Sn and Ge layers were used to grow $Ge_{1-x}Sn_x$ alloy films at below the crystallization temperature of Ge on single crystal (111) and (100) CaF_2 substrates to assess the role of Sn alloying in Ge crystallization. Single crystal $Ge_{1-x}Sn_x$(111) films were achieved by deposition on CaF_2(111) substrates, as indicated by X-ray diffraction theta *vs.* two-theta ($\theta/2\theta$) scans. However, $\theta/2\theta$ scans reveal that films grown on CaF_2(100) substrates exhibit a temperature-dependent superposition of (111) and (100) orientations at growth temperatures above 250 °C. This is the first report of temperature-dependent epitaxy of $Ge_{1-x}Sn_x$ films and can be explained by the enhancement of adatom diffusion at higher substrate temperatures during growth, which serves to lower the surface energy of the film. Results are confirmed by X-ray diffraction pole figure analysis (figure below), which further indicates the existence of crystal twinning and multiple rotational domains. In addition to the heteroepitaxial relations between the films and substrates, we used scanning electron microscopy and atomic force microscopy to identify trends in the morphological properties of films grown at several growth temperatures ranging from 250 °C to 400 °C.

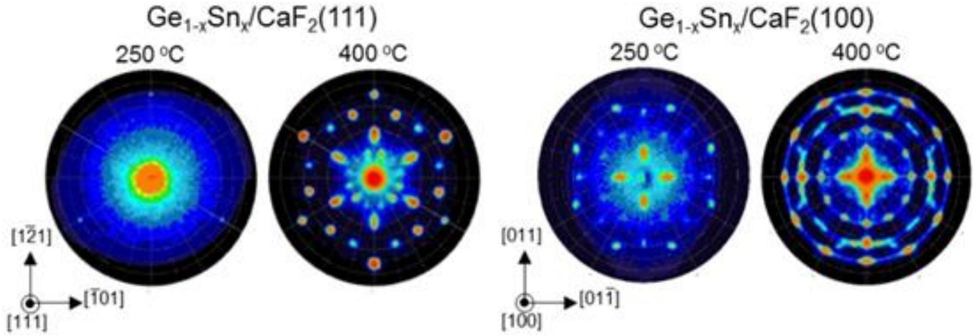

Figure: X-ray diffraction pole figures of films grown on single crystal CaF_2(111) substrates (left) and single crystalCaF_2(100) substrates (right) at 250 °C and 400 °C.

Acknowledgement: This work is supported by NSF DMR, NYSTAR through Focus Center-New York, and Rensselaer. This research used TEM at the Center for Functional Nanomaterials, which is a U.S. DOE Office of Science Facility, at BNL.

ROLE OF THE STRONG METAL SUPPORT INTERACTION ON THE CATALYTIC ACTIVITY OF PLATINUM DEPOSITED ON TiO_2 SUPPORTS

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Several roadblocks prevent the large-scale commercialization of hydrogen fuel cells, including the stability of catalysts and their substrates and the high cost of the Pt involved in the oxygen reduction reaction (ORR). The former of these problems can be solved by replacing the traditional carbon support with a conductive metal oxide such as reduced TiO_2, which will not easily corrode and should result in longer lasting fuel cells. The Pt is necessary in the cathode of the fuel cell to overcome the slow kinetics of the ORR. In this study, Pt was deposited either by atomic layer deposition (ALD) or physical vapor deposition (PVD). The typical size of the Pt islands that were grown using these deposition techniques was 5-8 nm. One factor that can inhibit the catalytic activity of a metal catalyst on a metal oxide is the strong metal support interaction (SMSI). This is where a metal on a reducible metal oxide can be encapsulated by a layer of the metal oxide support material at elevated temperatures. The processing of materials through atomic layer deposition can exceed this temperature. The TiO_2 substrates used in this study were either grown by ALD, which results in a polycrystalline anatase film, or were single-crystal rutile $TiO_2(110)$ samples prepared in ultra-high vacuum (UHV). The Pt/TiO_2 samples were tested electrochemically using cyclic voltammetry (CV) to determine the level of catalytic activity. To determine the effect of the SMSI interaction on the catalytic activity of the PVD grown samples, CV was performed on samples that were annealed in high vacuum after Pt deposition. Additional characterization was performed with scanning electron microscopy (SEM), Auger electron spectroscopy (AES), x-ray photoelectron spectroscopy (XPS), Rutherford backscattering spectrometry (RBS), and four point probe analysis.

GROWTH OF GRAPHENE ON CU SINGLE CRYSTAL SUBSTRATES

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Copper foils are frequently used to grow single-layer graphene by chemical vapor deposition (CVD). The primary reasons for this are the low costs of the Cu foils, the low solubility of carbon in Cu, and the relatively low cost of the equipment needed to grow the films. However, the polycrystalline nature of Cu foils can lead to numerous crystalline defects in the graphene, resulting in a reduction in the transport properties. To provide a systematic study of the CVD graphene growth process, a study of the growth of graphene on single crystal Cu substrates, with terminations along the (100), (110), and (111) planes, was performed. Synthesis was performed in an ultra-high vacuum (UHV) chamber using a modified setup to allow growth at pressures as high as 1 Torr. Ethylene was used as the precursor gas. To control Cu sublimation at the elevated growth temperatures, an Ar overpressure was used. This arrangement allowed for the preparation of clean Cu surfaces by sputtering and annealing the Cu crystals in UHV, followed by graphene growth at low pressure, and analysis with low energy electron diffraction in UHV without breaking vacuum. This avoided exposure of the crystal to atmospheric contaminants. It was found that surface termination plays a strong role in the rotational alignment of the nucleating graphene grains and the decomposition rate of the ethylene. It was observed that single-domain epitaxy is possible on Cu(111) when the ethylene pressure is 5 mTorr or less. However, growth on both Cu(100) and Cu(110) result in a minimum of two domains. In addition, ex-situ EELS is currently being performed on graphene films grown on Cu substrates to determine the effect of the graphene-Cu interaction on the electronic properties of the graphene.

This research was supported by NSF (DMR-1006411)

ELECTRON TRANSPORT AND INELASTIC ELECTRON TUNNELING SPECTROSCOPY OF 1,8-OCTANEDITHIOL IN A MOLECULAR JUNCTION

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Inelastic Electron Tunneling Spectroscopy (IETS) can be used to identify vibrational modes excited by the interaction of tunneling electrons with a molecule. 1,8-octanedithiol was used as a test molecule because of its low cost and thiol (-SH) functional groups which create a covalent bond to gold, forming a junction. IETS for 1,8-octanedithiol has already been reported via a break junction technique[1] and a Metal-SAM-Metal technique[2]. Future testing will switch focus to various porphyrin molecules, as they have shown conductance state/electrical switching behavior[3].

Molecular junctions were prepared via a Zig-Zag electromigration technique[4]. An intact 100x50x50nm nanowire with a Self Assembled Monolayer (SAM) of drop-dried 10µM 1,8-octanedithiol was lowered into a cryostat and put in low vacuum (10^{-3} Torr, 300K). Electromigration was performed, leaving a small 5 nm gap. SEM images of two successful nanogaps can be seen in Figure 1. The samples were cooled to 4.2 K and 10^{-6} Torr before IETS was performed. The IV, dI/dV, and d^2I/dV^2 (IETS) curves were taken simultaneously, Figure 2 shows the IETS curves obtained. While the IETS curves for these two samples are similar, they show modes that do not coincide with each other or are asymmetric. This can be attributed to the gap's asymmetry and geometry, as 1,8-octanedithiol is a symmetric molecule.

[1] J. Hihath, C. Bruot, NJ Tao. *ACS Nano*. **4**, 7, 3823-3830 (2010).
[2] T. Lee, W. Wang, M. A. Reed. *Jap. J Ap Phys*. **44**, 523-529 (2004).
[3] S. Saha, A. Nicolai, J. R. Owens, A. Krawicz, P. H. Dinolfo, V. Meunier, K. M. Lewis. *ACS Appl. Mater. Interfaces*. **7**, 10085-10090 (2015).
[4] Strachan, D.R., et al. *Ap. Phys. Lett*. **86**, 043109 (2005).

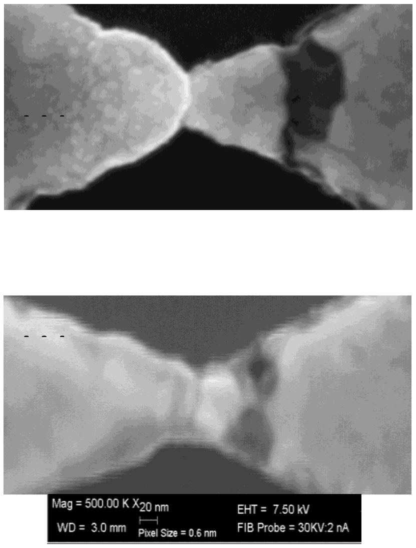

Figure 1. SEM images of two electromigrated nanogaps used for IETS.

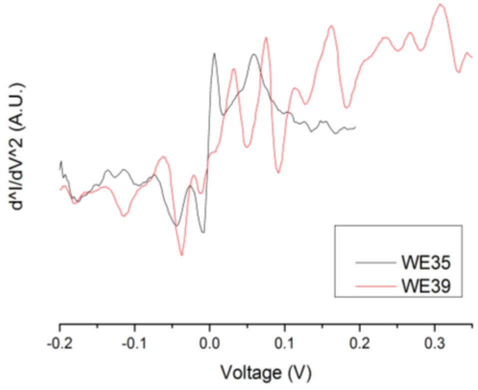

Figure 2. IETS curves for 1,8-octaneditihiol.

THE BENEFITS OF USING ALL OF THE MEASURED MASS CHANNELS DURING MVSA OF ToF-SIMS DATA SETS

Vincent Smentkowski[1], Mike R. Keenan[2], Henrik Arlinghaus[3]
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Time of flight secondary ion mass spectrometry (ToF-SIMS) data sets are very large and contain a wealth of information about the material being analyzed. A typical image data set can be comprised of 256 x 256 pixels with a 0 to 900 amu (or greater) mass spectrum collected at high (M/ΔM ~10,000) mass resolution at every pixel. Data sets are often comprised of >1 x 1015 spectral channels. The challenge for a ToF-SIMS analyst is to scrutinize all of the measured information without bias in order to provide for the most robust understanding of the material being analyzed; this is especially important in an industrial setting where unknown samples are analyzed. Multivariate statistical analysis (MVSA) algorithms have assisted in ToF-SIMS data work up [1,2], however commercially available software is not able to handle data sets this large and the analysts often select mass intervals to utilize and/or degrades the mass resolution prior to MVSA analysis. In this poster, we will report first results obtained using MVSA software that is able to handle massive ToF-SIMS data sets. We demonstrate two important benefits of unbiased analysis of the massive ToF-SIMS data sets: (1) finding unexpected elements in real world samples (this is a reason why the authors never use peak lists for MVSA analysis) and (2) the ability to obtain high mass resolution results from data sets collected at nominal mass resolution (e.g., the beam alignment pulsing mode on ION-TOF instruments). The importance of these two benefits will be highlighted.

References:
[1] Surface and Interface Analysis, Special issue on Multivariate Analysis. Volume 41, issue 2 Feb
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[2] Surface and Interface Analysis, Special issue on Multivariate Analysis II. Volume 41 issue 8, Aug 2009

MATERIALS CHARACTERIZATION: COMBINING BULK AND SURFACE ANALYTICAL METHODS

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Industrial components are extremely complex and contain several dissimilar and possibly highly incompatible materials that are placed in close proximity to each other, in order to achieve the desired properties in an end application. Examples are metal-ceramic interfaces, and multilayer coatings – to name a few. This could lead to instability and degradation-related issues under the conditions for the end application - such as inter-diffusion, interfacial reactions, delamination and so on. In some situations, in contrast, such changes might result in enhanced stability.

This presentation showcases several examples highlighting the significance of using multiple analytical methods in characterization of complex material systems consisting of a combination of oxides, ceramics, coatings, polymers, metals and alloys. In addition, the choice of the correct materials characterization technique(s) is key to getting the desired information.

Unambiguous Molecular Identification with TOF-SIMS Imaging MS/MS

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A new tandem TOF-TOF imaging mass spectrometer exploiting the unique characteristics of the TRIFT analyzer used in the PHI *nanoTOF* II has been developed [1]. This design allows for conventional TOF-SIMS spectra and product ion spectra of a specific precursor to be acquired in parallel, providing the maximum information from a given analytical volume. It has been recognized for many years that MS/MS is required to unambiguously identify peaks above 200 *m/z* due to the limitations of mass resolution and mass accuracy in commercial TOF-SIMS instrumentation. In the design reported here, a single nominal mass can be picked from the stream of secondary ions after it emerges from the 3rd electrostatic analyzer (ESA) and deflected into a collision cell for collision induced dissociation (CID). The entire TOF-SIMS spectrum, minus the precursor, is acquired in the standard way in the primary mass spectrometer (MS1). The selected precursor ion and fragment ions that emerge from the collision cell are further accelerated into a linear TOF mass spectrometer (MS2). Full mass spectra at both MS1 and MS2 are simultaneously acquired for each pixel in the image. Advantages of this new approach of TOF-SIMS with MS/MS for unambiguous molecular identification with imaging experiments will be demonstrated.

1. P.E. Larson, J.S. Hammond, R.M.A. Heeren and G.L. Fisher, Method and Apparatus to Provide Parallel Acquisition of MS/MS Data, *U.S. Patent 20150090874*, April 2015.