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## 2012 FALL MEETING of the AVS HUDSON MOHAWK CHAPTER

### Talks, posters, networking, pizza

**3:00 to 7:00 pm  
Fri Oct 12, 2012**

**NFS auditorium and rotunda  
University at Albany**

**final page = map**

## **ORAL PRESENTATIONS**

**T1: APPLICATION OF XPS IMAGING ANALYSIS IN UNDERSTANDING OF INTERFACIAL DELAMINATION AND RELATED PROBLEMS**, Hong Piao<sup>a</sup>, Neal Fairley<sup>b</sup>, and John Walton<sup>c</sup>, <sup>a</sup>General Electric Co., Niskayuna, NY 12309, U.S., <sup>b</sup>Casa Software Ltd, UK, <sup>c</sup>The University of Manchester, UK

**T2: QUANTITATIVE XPS IMAGING – PRACTICAL CONSIDERATIONS FOR DATA ACQUISITION AND PROCESSING**, David Surman, Kratos Analytical Inc., 100 Red Schoolhouse Road, Chestnut Ridge, NY 10977, Jonathan Counsell, Kratos Analytical Ltd., Wharfside, Trafford Wharf Road, Manchester, M17 1GP, UK

**T3: USE OF MUELLER MATRIX – SPECTROSCOPIC ELLIPSOMETRY FOR SCATTEROMETRY BASED MEASUREMENT OF CRITICAL DIMENSIONS DURING SEMICONDUCTOR MANUFACTURING**, Gangadhara Raja Muthinti<sup>1</sup>, Brennan Peterson<sup>2</sup>, and Alain C. Diebold<sup>1</sup>, 1. College of Nanoscale Science and Engineering, 257 Fuller Rd., Albany, NY, 12203, USA 2. Nanometrics Inc., Milpitas, CA, USA

**T4: IN-SITU ELLIPSOMETRIC MODELING OF INTERFACE FORMATION DURING ATOMIC LAYER DEPOSITION**, Han Wang<sup>a</sup>, Xiaoqiang Jiang, and Brian G. Willis, Department of Chemical, Materials & Biomolecular Engineering, University of Connecticut, Storrs, Connecticut 06269

**T5: SURFACE CHARACTERIZATION OF SUPPORTED METAL CATALYSTS BY X-RAY PHOTOELECTRON SPECTROSCOPY (XPS)**, Brian R. Strohmeier<sup>1</sup>, Tim S. Nunney<sup>2</sup>, and Andrew E. Wright<sup>2</sup>, 1. Thermo Fisher Scientific, 5225 Verona Rd, Madison, WI 53711, USA, 2. Thermo Fisher Scientific, The Birches Industrial Estate, Imberhorne Lane, East Grinstead, West Sussex, RH19 1UB, UK

**T6: SPECTROSCOPIC ELLIPSOMETRY AND ABBERATION-CORRECTED STEM FOR THE OPTICAL AND STUCTURAL CHARACTERIZATION OF GRAPHENE**, F. Nelson<sup>1</sup>, D.P. Sinha<sup>1</sup>, A. Sandrin<sup>2</sup>, D. Dougherty<sup>2</sup>, D. Aspenes<sup>2</sup>, J. Rowe<sup>2</sup>, J.U. Lee<sup>1</sup> and A.C. Diebold<sup>1</sup>, Tim S. Nunney<sup>2</sup>, and Andrew E. Wright<sup>2</sup>, 1. College of Nanoscale Science and Engineering, 257 Fuller Rd., Albany, NY, 12203, USA 2. Dept. of Physics, North Carolina St. Univ., Raleigh, NC, 27695, USA

## **POSTER PRESENTATIONS**

**P1: GROWTH OF GRAPHENE ON SINGLE CRYSTAL CU SURFACES BY CATALYTIC DECOMPOSITION ETHYLENE**, Tyler R. Mowll, Parul Tyagi, Zachary R. Robinson, Carl A. Ventrice Jr., College of Nanoscale Science and Engineering, University at Albany-SUNY

**P2: SPIN PRECESSION MEASUREMENT IN GRAPHENE**, A.Jayanthinarasimham<sup>1</sup>, W. Nolting<sup>1</sup>, J. Abel<sup>1</sup>, A. Matsubayashi<sup>1</sup>, J. Garramone<sup>1</sup>, C.Dimitrakopoulos<sup>2</sup>, D.Farmer<sup>2</sup>, A.Grill<sup>2</sup>, C.YSung<sup>2</sup> and V. LaBella<sup>1</sup>, <sup>1</sup>College of Nanoscale Science and Engineering, University at Albany,  
<sup>2</sup>IBM T.J. Watson Research Center, Yorktown Heights

**P3: OXIDE GROWTH ON CVD GRAPHENE FOR EFFICIENT SPIN INJECTION**, Akitomo Matsubayashi, J. Abel, D. P. Shinha, S. Shahrezaei, J. U. Lee and Vincent P. LaBella, College of Nanoscale Science and Engineering, University at Albany, SUNY

**P4: ToF-SIMS DEPTH PROFILING OF ORGANIC LAYERS USING AN Ar CUSTER ION SOURCE**, Vincent S. Smentkowski, Gilad Zorn, Amanda Misner, Gautam Parthasarathy, and Aaron Couture, GE Global Research, Niskayuna NY 12309, Elke Tallarek, Birgit Hagenhoff, Tascon, Muenster Germany

**P5: IMPROVING CATALYST PERFORMANCE IN PEM FUEL CELLS USING TiO<sub>x</sub> CATALYST SUPPORTS**, Richard Phillips, Alexander O'Toole, Xiaoli He, Robin Hansen, Robert Geer, and Eric Eisenbraun, College of Nanoscale Science and Engineering, The University at Albany-State University of New York, Albany, New York 12203

**P6: ADVANCED ANALYTICAL CHARACTERIZATION OF MULTILAYERED THIN FILMS FOR CORROSION INHIBITION**, Gilad Zorn<sup>1</sup>, Mallikarjun Karadge<sup>1</sup>, Claudia C. Pierce<sup>2</sup>, Jeffrey I. Melzer<sup>2</sup> and Martin M. Morra<sup>1</sup>, <sup>1</sup> GE Global Research, Niskayuna, NY, <sup>2</sup> GE Power & Water, Trevose, PA

**P7: EFFECT of GERMANIUM CONCENTRATION ON THE DIELECTRIC FUNCTION OF STRAINED Si<sub>1-x</sub>Ge<sub>x</sub> FILMS**, Manasa Medikonda<sup>a</sup>, Gangadhara. R. Muthinti<sup>a</sup>, Thomas. N. Adam<sup>a</sup>, Alexander Reznicek<sup>b</sup>, Vamsi Paruchuri<sup>b</sup>, Alain. C. Diebold<sup>a</sup>, <sup>a</sup>College of Nanoscale Science and Engineering, University at Albany, NY, USA, <sup>b</sup>IBM Research at Albany Nanotech, Albany, NY, USA.

**P8: AN *IN SITU* TECHNIQUE FOR USING BALLISTIC ELECTRON EMISSION MICROSCOPY TO MEASURE HOT ELECTRON TRANSPORT AT METAL SEMICONDUCTOR INTERFACES**, Robert Balsano and Vincent P. LaBella, University at Albany, NY, USA

**P9: KAUST-CORNELL CENTER FOR ENERGY AND SUSTAINABILITY: A NIMs-BASED RESEARCH CENTER**, David R. Jung, Lynden A. Archer and Emmanuel P. Giannelis, KAUST-CU Center for Energy and Sustainability, Cornell University, Ithaca, NY 14853

**P10: ELECTRICAL CHARACTERIZATION OF ULTRATHIN Ag FILMS DEPOSITED ON Cu**, E. Tatem, E. Eisenbraun, A. E. Kaloyeros, University at Albany, College of Nanoscale Science and Engineering, 257 Fuller Rd, Albany NY, 12222

**P11: ATOMIC LAYER DEPOSITION OF THIN FILM CATALYSTS FOR HYDROGEN FUEL CELLS**, Robin Hansen and Eric Eisenbraun, University at Albany, College of Nanoscale Science and Engineering, Albany, NY

**P12: SPIN LAB MEASUREMENT SYSTEM**, W. Nolting, A. Matsubayashi, A. Jayanthinsimham. R. Balsano and, V.P. LaBella, College of Nanoscale Science and Engineering, University at Albany, SUNY

T1

## APPLICATION OF XPS IMAGING ANALYSIS IN UNDERSTANDING OF INTERFACIAL DELAMINATION AND RELATED PROBLEMS

Hong Piao<sup>a</sup>, Neal Fairley<sup>b</sup> and John Walton<sup>c</sup>

<sup>a</sup>*General Electric Co., Niskayuna, NY 12309, U.S.*

<sup>b</sup>*Casa Software Ltd, UK*

<sup>c</sup>*The University of Manchester, UK*

*piao@research.ge.com*

The recent development of X-ray Photoelectron Spectroscopy (XPS) instrumentation with near-micron spatial resolution has advanced the capability of elemental and chemical state imaging. This work extends the application of imaging XPS to the analysis of real world samples. The presentation also focuses on description of radiation damage of polymers encountered in XPS imaging analysis. The imaging analysis can cause extensive damage to polymers since the acquisition time for creating datasets can be excessive. Understanding of radiation damage in polymers is necessary for successful and validated application of XPS spectromicroscopy.

**Keywords:** XPS, chemical states, imaging, delamination.

T2

**QUANTITATIVE XPS IMAGING – PRACTICAL CONSIDERATIONS FOR DATA  
ACQUISITION AND PROCESSING**

David Surman

Kratos Analytical Inc., 100 Red Schoolhouse Road, Chestnut Ridge, NY 10977

[dsurman@kratos.com](mailto:dsurman@kratos.com)

Jonathan Counsell

Kratos Analytical Ltd., Wharfside, Trafford Wharf Road, Manchester, M17 1GP, UK

**Abstract:**

Although XPS imaging is a technique that has been available on many instruments for quite some time, the conversion of these images into quantitative distributions is a relatively recent development. There are a variety of methodologies available to the analyst to obtain and process XPS images, and there are many practical aspects that must be considered in order to achieve good results within a reasonable time-frame. For example spatial resolution needed to identify the features of interest, acquisition times per pixel and pixel density need to be considered for the acquisition while sensitivity factors, instrument transmission functions and backgrounds need to be considered during the data processing stage. This talk will discuss all the various aspects for acquisition and processing and illustrate these effects with examples.

T3

## **Use of Mueller Matrix – Spectroscopic Ellipsometry for Scatterometry based measurement of critical dimensions during semiconductor manufacturing**

Authors: Gangadhara Raja Muthinti<sup>1</sup>, Brennan Peterson<sup>2</sup> and Alain. C. Diebold<sup>1</sup>

<sup>1</sup>College of Nanoscale Science & Engineering, University at Albany, SUNY, NY.

<sup>2</sup>Nanometrics Inc., Milpitas, CA.

### **Abstract**

Scatterometry is one of the most useful metrology methods for the characterization and control of critical dimensions (CD) during nano-electronic manufacturing. Most Scatterometry is based on Spectroscopic Ellipsometry (SE) and Normal Incidence Reflectometry (NI) measurement and the simulation of the measured spectra through the Rigorous Coupled Wave Approximation. Evolution of better optical hardware and faster computing capabilities led to the development of Mueller Matrix (MM) based Scatterometry (MMS). Typically, spectroscopic ellipsometry based Scatterometry uses  $\Psi$  and  $\Delta$  measured at each wavelength. In this presentation we discuss dimensional metrology using full Mueller Matrix (16 element) Scatterometry in the wavelength range of 245nm-1000nm measured using a dual rotating compensator Spectroscopic Ellipsometer. Unlike SE and NI, MM data provides complete information about the optical reflection and transmission of polarized light through a sample. The advantage of MMSE over traditional SE Scatterometry is its ability to measure samples that have anisotropic optical properties and depolarize light. We demonstrate this using a series of structures fabricated by e-beam lithography.

T4

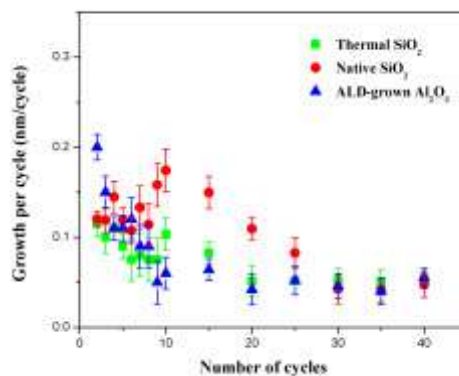
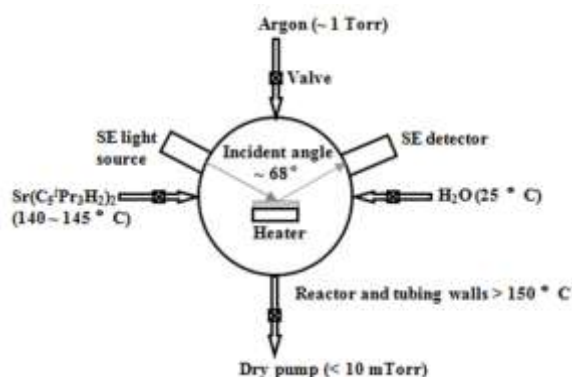
## IN-SITU ELLIPSOMETRIC MODELING OF INTERFACE FORMATION DURING ATOMIC LAYER DEPOSITION

Han Wang<sup>a)</sup>, Xiaoqiang Jiang, and Brian G. Willis

Department of Chemical, Materials & Biomolecular Engineering, University of Connecticut, Storrs, Connecticut 06269.

<sup>a)</sup>Electronic mail: [haw09003@engr.uconn.edu](mailto:haw09003@engr.uconn.edu)

Spectroscopic ellipsometry (SE) is an optical technique which is highly surface-sensitive, fast as well as non-destructive. Atomic layer deposition (ALD) is known for its ability to control film growth and properties on the atomic level. Essentially, ALD is a surface-controlled process and independent of the precursor flow in the reactor. Many open questions remain about non-ideal behaviors in ALD processes, such as the initial non-linear growth rate, crystallization-growth rate relation, and stoichiometry-pulse ratio relation in ALD of ternary oxides. *In-situ* characterization of ALD by ellipsometry enables a fast way for optimizing process parameters (dosing duration and deposition temperature) and determining film thickness and optical properties ( $n$  and  $k$ ). In this work, we investigated strontium oxide (SrO) as a model system to better understand the fundamental origins of substrate and temperature effects. SrO ALD is critical for the ALD of the ternary strontium titanate (STO), which is of major interest for use with high-density metal-insulator-metal (MIM) capacitors. SrO is also of interest for the growth of epitaxial perovskite oxides on semiconductors where it acts as a buffer layer between the reactive semiconductor and the metal oxide layers.



Left Figure: Schematic representation of a warm wall stainless steel ALD reactor. Sr precursor and oxidizer lines are shown as well as the Ar purge and dry pump lines. Also the implementation of a spectroscopic ellipsometer is shown with a fixed angle of incidence (68°).

Right Figure: Growth per cycle (GPC) as a function of ALD cycles for SrO growth on native/thermal SiO<sub>2</sub> and ALD-grown Al<sub>2</sub>O<sub>3</sub> surfaces at deposition temperature of 250 °C

## **SURFACE CHARACTERIZATION OF SUPPORTED METAL CATALYSTS BY X-RAY PHOTOELECTRON SPECTROSCOPY (XPS)**

Brian R. Strohmeier<sup>1</sup>, Tim S. Nunney<sup>2</sup>, and Andrew E. Wright<sup>2</sup>

1. Thermo Fisher Scientific, 5225 Verona Rd, Madison, WI 53711, USA.
2. Thermo Fisher Scientific, The Birches Industrial Estate, Imberhorne Lane, East Grinstead, West Sussex, RH19 1UB, UK.

brian.strohmeier@thermofisher.com

Heterogeneous catalysts typically consist of an active material dispersed upon a high surface area (i.e., ~200-300 m<sup>2</sup>/g) support. The active component is often a dispersion of transition metal particles, which may be in a completely or partially reduced form. The support material is commonly a metal oxide, such as alumina, silica, or titania, and may also have a role in the catalytic process, either directly or by influencing the active material. Activated carbon and other nanoparticle carbon and carbon nanotube materials are also being used as a catalyst supports. Catalysts may be supplied in a precursor form, which needs activating prior to usage. As an optimized industrial process relies on fixed operating conditions, any deviation from the correct precursor catalyst will result in less than optimum operating conditions or possible failure of the process. X-ray photoelectron spectroscopy (XPS) is a widely used technique for catalyst analysis, delivering both elemental and chemical state information. Although the lateral resolution of XPS is typically greater than 10  $\mu\text{m}$ , XPS has the benefit of being a very surface selective technique; the data collected is representative of the outer 10 nm of the sample. This means that although the surface chemistry of individual particles cannot be measured, the average surface chemistry of an aggregate of the catalyst particles can be determined and contrasted with that obtained from more bulk-sensitive methods. This presentation will discuss how XPS was used to determine the surface elemental composition and the chemical states of the detected components for a variety of catalyst systems. These examples will also include results obtained from field emission scanning electron microscopy (FESEM), scanning transmission electron microscopy (STEM), energy dispersive X-ray spectroscopy (EDS), and low energy ion scattering spectroscopy (LEIS).



**T6**

**Spectroscopic Ellipsometry and Aberration-Corrected STEM for the Optical and Structural Characterization of Graphene**

F. Nelson<sup>1</sup>, D. P. Sinha<sup>1</sup>, Andreas Sandin<sup>2</sup>, Dan Dougherty<sup>2</sup>, Dave Aspnes<sup>2</sup>, Jack Rowe<sup>2</sup>, J. U. Lee,<sup>1</sup>  
A.C. Diebold<sup>1</sup>

<sup>1</sup>College of Nanoscale Science and Engineering, University at Albany, Albany, NY 12203 <sup>2</sup>Department of Physics, North Carolina State University, Raleigh, North Carolina 27695

Contact email: FNelson@ albany.edu

Hydrocarbon-based Chemical Vapor Deposition (CVD) on copper, and thermal decomposition of SiC, have become two main growth methods for the fabrication of large-area graphene sheets. The control of film area by the area of the substrate makes these growth methods attractive candidates for industrial applications due to their scalability. However, integration of graphene into industrial-scale device fabrication will require accurate thickness determinations, as the number of layers in graphene stack determines its electronic/optical properties. Spectroscopic Ellipsometry is a common inline optical measurement that is used here to determine the complex refractive index/dielectric function for both CVD graphene and graphene grown on 6H-SiC. The latter rests on a “buffer”, or interface layer, the dielectric function of which is shown here to possess a significantly different optical response than purely sp<sup>2</sup>-bonded graphene. CVD graphene is polycrystalline and consists of domains/grains of graphene, the sizes of which may be controlled by growth process conditions. The presence of grain boundaries affects the electronic properties of graphene, therefore making them of interest for further study. Aberration-corrected Scanning TEM (STEM) images obtained at Oak Ridge National Lab will be shown for CVD graphene in order to demonstrate the challenge of locating grain boundaries between domains of different crystallographic orientations.

\*Submitted for: Talk

**P1**

## **Growth of Graphene on Single Crystal Cu Surfaces by Catalytic Decomposition of Ethylene**

Tyler R. Mowll, Parul Tyagi, Zachary R. Robinson, Carl A. Ventrice Jr.

*College of Nanoscale Science and Engineering  
University at Albany-SUNY*

Graphene growth by chemical vapor deposition on Cu substrates is one of the most promising techniques for the production of large area graphene for technological applications. However, it is not uncommon for graphene grown on Cu foil substrates to have carrier mobilities that are a couple of orders of magnitude lower than what has been achieved for exfoliated graphene. The primary reason for this is that the graphene films are typically polycrystalline. This results from the growth on foil substrates, which do not have well defined surface terminations. In order to study the influence of Cu orientation on graphene growth, a systematic study of graphene growth on Cu(100) and Cu(111) single crystals oriented within  $0.1^\circ$  from the surface normal and a vicinal Cu(111) crystal oriented  $5^\circ$  off-axis has been studied. Initial attempts to grow graphene by annealing each crystal to temperatures as high as  $900^\circ\text{C}$  in UHV, followed by backfilling the chamber with  $\text{C}_2\text{H}_4$  at pressures up to  $5 \times 10^{-3}$  Torr did not result in graphene formation on either the on-axis Cu(100) and Cu (111) surfaces. For the vicinal Cu(111) surface, some graphene was formed under the same conditions. This indicates that step edges have a higher catalytic activity than the terrace sites. By using an argon overpressure, it was determined that the growth of single-domain epitaxial overlayers could be achieved on both the on-axis and off-axis Cu(111) substrates. This result indicates that the sublimation of Cu atoms at the elevated temperatures needed for ethylene dissociation will inhibit graphene growth.

## SPIN PRECESSION MEASUREMENT IN GRAPHENE

A.Jayanthinarasimham<sup>1</sup>, W. Nolting<sup>1</sup>, J. Abel<sup>1</sup>, A. Matsubayashi<sup>1</sup>, J. Garramone<sup>1</sup>,  
C.Dimitrakopoulos<sup>2</sup>, D.Farmer<sup>2</sup>, A.Grill<sup>2</sup>, C.YSung<sup>2</sup> and V. LaBella<sup>1</sup>

<sup>1</sup>College of Nanoscale Science and Engineering, University at Albany

<sup>2</sup>IBM T.J. Watson Research Center, Yorktown Heights

ajayanthinarasimham@albany.edu

**Abstract:**

The understanding of spin transport and relaxation in graphene is crucial to advancing the use of electron spin to achieve low-power, high-speed nanoscale logic devices or for more exotic applications such as quantum computing<sup>1,2</sup>. Specifically we are focusing on the All Spin Logic Device proposed by Behtash<sup>2</sup> at Purdue. Graphene is an ideal candidate for the transport channel due to the long spin lifetimes and diffusion lengths that have been observed at room temperature<sup>3</sup>. This arises due to the small intrinsic spin orbit interaction, and the low hyper-fine interaction of the electron spins with the carbon nuclei. In addition, transparent ferromagnetic Co contacts can be readily fabricated for spin injection and readout by depositing and patterning Co directly upon the graphene. Here we demonstrate spin injection, diffusion, and readout utilizing a four contact “non-local” spin device fabricated on epitaxial graphene grown on silicon carbide[4].

## Reference:

[1] H. Dery, P. Dalal, L. Cywinski, L. J. Sham, Nature **447** 573 (2007) .

[2] Behtash Behin-Aein, Deepanjan Datta, Sayeef Salahuddin, Supriyo Datta, Nature Nanotech. **5** 266 (2010)

[3] B.Behin-Aein, D.Datta, S. Salahuddin and S. Datta, “Proposal for an all-spin logic device with built-in memory”, Nature Nanotech, 4,p.266, (2010).

[4] J. Abel, A. Matsubayashi, T. Murray, C. Dimitrakopoulos, D. B. Farmer, Ali Afzali, A. Grill, C. Y Sung, V. P. LaBella, Journal of Vacuum Science and Technology B 30 04E109 (2012).

P3

## OXIDE GROWTH ON CVD GRAPHENE FOR EFFICIENT SPIN INJECTION

Akitomo Matsubayashi, J. Abel, D. P. Shinha, S. Shahrezaei, J. U. Lee and Vincent P. LaBella

College of Nanoscale Science and Engineering, University at Albany, SUNY  
amatsubayashi@albany.edu

### **Abstract:**

Utilizing the spin of electron as well as its charge has a potential to create devices which are more energy efficient with faster operating speeds. The all spin logic device was proposed as one such example in 2010<sup>[1]</sup>. In order to realize such a device, channel materials with two important metrics are needed; long spin relaxation times and efficient spin injection. Graphene is an ideal channel material because it has intrinsically low spin-orbit coupling and minimal hyperfine interaction with carbon nuclei give it a long spin lifetime at room temperature<sup>[2]</sup>. Our previous work has demonstrated that introducing a tunnel barrier between the ferromagnetic metal and an epitaxial graphene channel improves the measured lifetime and spin injection efficiency<sup>[3]</sup>. In this poster, we are presenting our recent work on growing aluminum oxide under ultra-high vacuum conditions for tunnel barriers on graphene grown by chemical vapor deposition (CVD). The chemical and structural composition of the oxide both with and without the use of a Ti seed layer is investigated using atomic force microscopy and X-ray photoelectron spectroscopy. The Ti seed layer is shown to significantly reduce the surface roughness of the dielectric film and if kept thin enough remain completely oxidized.

### References:

- [1] B. Behin-Aein *et al.*, *Nat. Nanotechnol.*, **5**, 266 (2010)
- [2] N. Tombros *et al.*, *Nature*, **448**, 571 (2007)
- [3] J. Abel *et al.*, *J. Vac. Sci. Technol. B*, **30** 04E109 (2012)

**P4**

**ToF-SIMS Depth Profiling of Organic Layers using an Ar Cluster Ion Source**

Vincent S. Smentkowski, Gilad Zorn, Amanda Misner, Gautam Parthasarathy, and Aaron Couture  
GE Global Research, Niskayuna NY 12309

Elke Tallarek, Birgit Hagenhoff, Tascon, Muenster Germany

**Abstract**

Time of Flight Secondary Ion Mass Spectroscopy (ToF-SIMS) is a very powerful technique for the analysis of organic and biological samples since it is able to detect and image high mass molecular species, such as polymer additives as the ion fluence is low enough to prevent decomposition of the organic/molecular species. Depth profiling, in contrast, uses a high ion fluence in order to remove material between analysis cycles. Unfortunately, the high ion fluence results in decomposition of the organic species in addition to erosion when monoatomic projectile's are used. Recently, high mass Ar cluster ion sources have become available and are enabling depth profile analysis of organic layers. In this poster, we demonstrate that we can obtain and maintain molecular information throughout the entire organic layer when erosion is performed using an  $Ar_{1500}^+$  cluster ion beam.

P5

## IMPROVING CATALYST PERFORMANCE IN PEM FUEL CELLS USING $\text{TiO}_x$ CATALYST SUPPORTS

Richard Phillips, Alexander O'Toole, Xiaoli He, Robin Hansen, Robert Geer, and Eric Eisenbraun

College of Nanoscale Science and Engineering  
The University at Albany-State University of New York  
Albany, New York 12203  
rphillips@albany.edu

A few well-known challenges face current proton exchange membrane fuel cell (PEMFC) technologies which utilize Pt on a colloidal carbon black cathode support material. While effective in its application, the current Pt on carbon electrode technology suffers from some well-known issues, such as, carbon corrosion, and electrode flooding. Carbon corrosion occurs when the colloidal carbon catalyst support oxidizes during typical fuel cell operation. This causes the Pt catalyst particles to detach from the carbon support. Agglomeration of Pt particles results which limits the active surface area where catalytic reactions take place. Electrode flooding results from over accumulation of water at catalyst sites which prevents complete access for unreacted gases. Water accumulation occurs when inadequate gas transport pathways exist in the cathode layer of PEMFCs. Such is the case for the current tightly packed colloidal carbon black based cathodes. These two issues lead to degradation of the overall fuel cell efficiency.

This work investigates strategies for overcoming carbon corrosion and electrode flooding. It is suggested that changing the catalyst support material (to prevent corrosion) and material structure (to avoid flooding by increasing gas transport pathways) may lead to improved fuel cell performance.  $\text{TiO}_x$  is suggested as a replacement for carbon based PEMFC catalyst support materials due to its high conductivity and corrosion resistance. Atomic layer deposition (ALD) of  $\text{TiO}_x$  over anodic aluminum oxide (AAO) and silicon nanowire (SiNW) templates was used to form nanostructures with high aspect ratios. Aspect ratios of 100:1 have been observed.  $\text{TiO}_x$  films underwent post deposition anneals in hydrogen (to reduce oxygen). Four point probe measurements of annealed  $\text{TiO}_x$  films indicate a large increase in conductivity with only a slight reduction in oxygen (as measured by x-ray photoelectron spectroscopy (XPS)). Liquid phase deposited Pt and plasma enhanced atomic layer deposition (PEALD) of Pt were used for the metallization of nanostructured  $\text{TiO}_x$ . Electrochemical measurements were performed using cyclic voltammetry (CV) and rotating disk electrode (RDE) analysis. Electrochemical data measures high activity for Pt on  $\text{TiO}_x$  catalyst materials when compared to the typical Pt on carbon materials.

## ADVANCED ANALYTICAL CHARACTERIZATION OF MULTILAYERED THIN FILMS FOR CORROSION INHIBITION

Gilad Zorn<sup>1</sup>, Mallikarjun Karadge<sup>1</sup>, Claudia C. Pierce<sup>2</sup>, Jeffrey I. Melzer<sup>2</sup> and Martin M. Morra<sup>1</sup>

<sup>1</sup> *GE Global Research, Niskayuna, NY*

<sup>2</sup> *GE Power & Water, Trevose, PA*

Advanced corrosion inhibitors developed by General Electric Power and Water can have complex multilayered structures that incorporate metal, ceramic and polymeric structures. For optimal performance it is important to understand the structure, morphology and composition of different layers. However, characterizing these nano scale films is very challenging, as they can be sensitive to preparation technique and damage. Moreover, surface roughness and homogeneity of the layers should be considered.

The challenges in characterizing these multilayered structures will be discussed as an example for thin film characterization in the industrial R&D world. A multi technique approach that provides a detailed view of complex structures and compositions will be presented. Transmission Electron Microscopy (TEM) equipped with EDS was used to define local morphologies, crystalline structures and chemical composition; and Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) depth profiles were used to determine the molecular distribution within the different layers. TEM provides local information in the range of a few nanometers over Focused Ion Beam (FIB) cross sections while ToF SIMS allows the analysis of larger areas, in the range of hundreds of nanometers, and provides top down views of the layers. The talk will emphasize how these two methods complement each other to achieve a detailed picture of complex structures within thin films.

P7

## EFFECT of GERMANIUM CONCENTRATION on the DIELECTRIC FUNCTION of STRAINED $\text{Si}_{1-x}\text{Ge}_x$ FILMS

Manasa Medikonda<sup>a</sup>, Gangadhara. R. Muthinti<sup>a</sup>, Thomas. N. Adam<sup>a</sup>, Alexander Reznicek<sup>b</sup>, Vamsi Paruchuri<sup>b</sup>, Alain. C. Diebold<sup>a</sup>

<sup>a</sup>College of Nanoscale Science and Engineering, University at Albany, NY, USA, [mmedikonda@albany.edu](mailto:mmedikonda@albany.edu), <sup>b</sup>IBM Research at Albany Nanotech, Albany, NY, USA.

### Abstract

Transistors fabricated using strained silicon-germanium alloy channels provide improved carrier mobility. While the electrical properties of  $\text{Si}_{1-x}\text{Ge}_x$  for a variety of values of  $x$  and the optical properties of strain-free  $\text{Si}_{1-x}\text{Ge}_x$  for  $x=0$  to 1 have been reported, reports of the optical properties of pseudomorphic  $\text{Si}_{1-x}\text{Ge}_x$  were limited to  $x < 0.3$ . For Ge concentrations of less than 30%, the optical properties follow the low shear elastic response [1, 2]. Here, we report an in-depth study of the optical properties of bi-axially strained pseudomorphic  $\text{Si}_{1-x}\text{Ge}_x$  alloys for Ge concentrations ranging from 0.05 to 0.75. Optical properties (complex refractive index / dielectric function) of semiconductors are dominated by strong absorption at critical points (CP). Strain often shifts the  $E_1$  and  $E_1+\Delta$  CPs. The spin orbit splitting  $\Delta$  increases with germanium concentration. Strained semiconductors are described by an elastic theory approach that predicts the shifts of the critical points. There are two approximations for the optical response depending on the magnitude of spin-orbit coupling versus the shift in CP energy for shear stress. The low shear approximation used previously for the calculation of elastic response of critical points (direct gap transitions) is no longer valid for pseudomorphic  $\text{Si}_{1-x}\text{Ge}_x$  alloys having germanium concentrations greater than 40%. The optical properties of these alloys follow a high shear approximation and high shear effects can be seen very prominently for concentrations above 50%, where two peaks are readily apparent for  $E_1$  with an energy separation greater than the spin orbit splitting. Undoped  $\text{Si}_{1-x}\text{Ge}_x$  films, with Ge concentrations from 0.05 to 0.75 were grown on Si (001) substrates using ultra-high vacuum and reduced pressure chemical vapor deposition. Layer thickness and composition was measured using high resolution X-ray diffraction. Figure 1 shows the  $\Omega$ -2 $\Theta$  004 rocking curves for some of the wafers starting from 5% Ge concentration. Relaxation scans and reciprocal space maps confirmed the alloys as fully strained, low defect and high quality structural formations. The dielectric function of pseudomorphic alloys was evaluated using spectroscopic ellipsometry from 0.73 eV to 5.17 eV (245nm to 1700nm). Figure 2 shows two peaks near the  $E_1$  and  $E_1+\Delta$  critical points for higher germanium concentrations. Errors in ellipsometric based thickness measurements occur if the complex refractive index of relaxed SiGe alloys is used to measure pseudomorphic SiGe films.



AN *IN SITU* TECHNIQUE FOR USING BALLISTIC ELECTRON EMISSION MICROSCOPY  
TO  
MEASURE HOT ELECTRON TRANSPORT AT METAL SEMICONDUCTOR INTERFACES

Robert Balsano and Vincent P. LaBella

University at Albany  
rbalsano@albany.edu

**Abstract:**

Ballistic electron emission microscopy (BEEM) is a scanning tunneling microscopy (STM) technique that can measure transport of hot electrons through materials and interfaces with high spatial and energetic resolution. BEEM requires an additional contact to ground the metal base layer of a metal semiconductor junction. Performing BEEM *in situ* with the sample fabrication requires a custom built STM or modifying a commercial one to facilitate the extra contact, which leaves the technique to highly trained experts. This presentation will describe our work to develop a special silicon substrate that has the extra contact and an oxide hard mask built in to enable *in situ* BEEM without modifications to the STM. Electrically isolated contact traces are lithographically patterned *ex situ* onto the silicon substrate. Then a hard mask is grown and lithographically patterned. The substrate is then mounted on the BEEM sample plate which is then inserted into the ultra-high vacuum chamber. The metal is deposited through the hard mask over the patterned contact. The sample plate is then mounted *in situ* onto the STM for BEEM measurements. BEEM measurements comparing both *in situ* and *ex situ* deposited films will be presented. This new modification enables BEEM measurements of materials and interfaces that were previously not feasible, which will be discussed.

KAUST-Cornell Center for Energy and Sustainability: a NIMs-based Research Center

David R. Jung, Lynden A. Archer and Emmanuel P. Giannelis

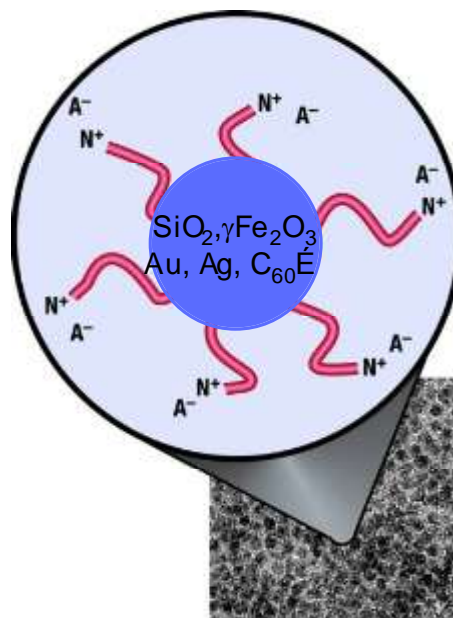
KAUST-CU Center for Energy and Sustainability, Cornell University, Ithaca, NY 14853

dj59@cornell.edu

The KAUST-Cornell University (KAUST-CU) Center for Energy and Sustainability is developing Nanoscale Ionic Materials (NIMs), a hybrid materials platform technology.

A wide variety of NIMs can be created by attaching a charged oligomer to an inorganic nanoparticle. The charge on the core is balanced by a counter ion species, A, which can be an oligomer. Through variation of the kind and relative proportion of the inorganic and organic species, a wide range of properties can be attained, including materials that flow like liquids at room temperature as solventless, self-suspended fluids.

The KAUST-CU Center integrates the complementary expertise in materials synthesis, simulation, theory, property and structural measurement of faculty, post-doctoral associates, and graduate students from 6 departments at Cornell and from 5 partner institutions in research activities organized around the following four Thrust topics:



#### Photovoltaics and Energy Storage

- Composites of inorganic semiconductor nanoparticles and conjugated polymers are being developed to improve the efficiency vs. cost trade-off in solar cells and rechargeable batteries.

#### Water Treatment and Desalination

- Research focuses on novel nanocomposite materials and their applications as membranes for advanced water purification by forward and reverse osmosis.

#### CO<sub>2</sub> Capture and Conversion

- Research focuses on designing a nanoscale hybrid materials technology that facilitates enhanced high-temperature carbon dioxide capture and catalytic sequestration in a single materials platform.

#### Enhanced Oil & Gas Exploration & Production

- A suite of stimuli-responsive, nanoscale materials are being developed for mapping and managing subsurface fluid flows in oil and gas reservoirs.

**ELECTRICAL CHARACTERIZATION OF ULTRATHIN Ag FILMS DEPOSITED ON Cu**

E. Tatem, E. Eisenbraun, A. E. Kaloyeros,

University at Albany, College of Nanoscale Science and Engineering,  
257 Fuller Rd, Albany NY, 12222  
etatem@albany.edu

As the semiconductor industry continues to scale feature sizes, scattering from phonons, surfaces, and grain boundaries result in an increase of metal interconnect resistance. Although surface and grain boundary scattering are reported to be the dominating contributors to resistivity in the sub 32nm regime<sup>1, 2</sup>, phonon scattering is still a significant portion of the total resistivity at room temperature and in recent years efforts have been made to reduce temperature dependent contributions to resistivity<sup>3</sup>. In this work, the thermal coefficient of resistivity of ultra thin Ag films deposited on Cu is used as a measure of inelastic scattering due to electron-phonon coupling within the metal. Previous studies of the Ag Cu multilayer system showed that there is a relationship between the thickness of the Ag overlayer and the electron-phonon scattering parameter<sup>4</sup>. It has been reported that there are critical thicknesses of Ag on Cu such that electron-phonon scattering is reduced up to thirty percent for Ag/V films<sup>5</sup>.

In this work the thermal coefficient of resistivity is observed for various thicknesses of Ag deposited on Cu and compared to pure Cu films. An interdependence between Ag film thickness and the thermal coefficient of resistivity is observed for ultra-thin Ag films deposited on Cu. Also, a reduction in the rate of increase of the resistivity with respect to temperature is observed for all samples when compared to bare Cu films. A lower thermal coefficient of resistivity is indicative of a decreased contribution of the temperature dependent portion of resistivity to the total resistivity and may prove critical for the viability of future metallic conductor-based interconnect architectures.

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## ATOMIC LAYER DEPOSITION OF THIN FILM CATALYSTS FOR HYDROGEN FUEL CELLS

Robin Hansen and Eric Eisenbraun  
phansen@albany.edu

College of Nanoscale Science and Engineering

Current fuel cell technology requires the use of large quantities of platinum in the cathode for the oxygen reduction reaction (ORR). A major roadblock to the commercialization of fuel cells is the high cost of Pt. Current efforts to reduce the fuel cell costs have focused on the minimization of the amount of Pt.

Work on substoichiometric titanium dioxide thin film development through atomic layer deposition (ALD) has already been reported [1]. These films have demonstrated conductive properties in oxidizing environments, making them an ideal supports for catalytic materials. This current work outlines the deposition of catalysts on top of support materials. When under 300 cycles of ALD Pt are deposited on a TiO<sub>x</sub> substrate, well dispersed Pt particles form. With increased ALD cycles the particles grow larger and form continuous films. After continuous films are formed no increase is expected in the mass activity of platinum due to a decrease in the Pt surface area to volume ratio. As a result pt depositions of 300 cycles or less are examined here.

Films were made using methylcyclopentadienyltrimethyl platinum (IV) (MeCpMe<sub>3</sub>Pt) with NH<sub>3</sub> and H<sub>2</sub> as reactant gases. The ALD Pt process was optimized to yield the maximum growth rate and conductivity of the Pt/TiO<sub>x</sub> film stack. Characterization was performed with scanning electron microscopy (SEM), Auger electron spectroscopy (AES), x-ray photoelectron spectroscopy (XPS), Rutherford backscattering spectrometry (RBS), four point probe analysis, and cyclic voltammetry (CV).

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## SPIN LAB MEASUREMENT SYSTEM

**W. Nolting, A. Matsubayashi, A. Jayanthinsimham. R. Balsano and, V.P. LaBella<sup>1</sup>**

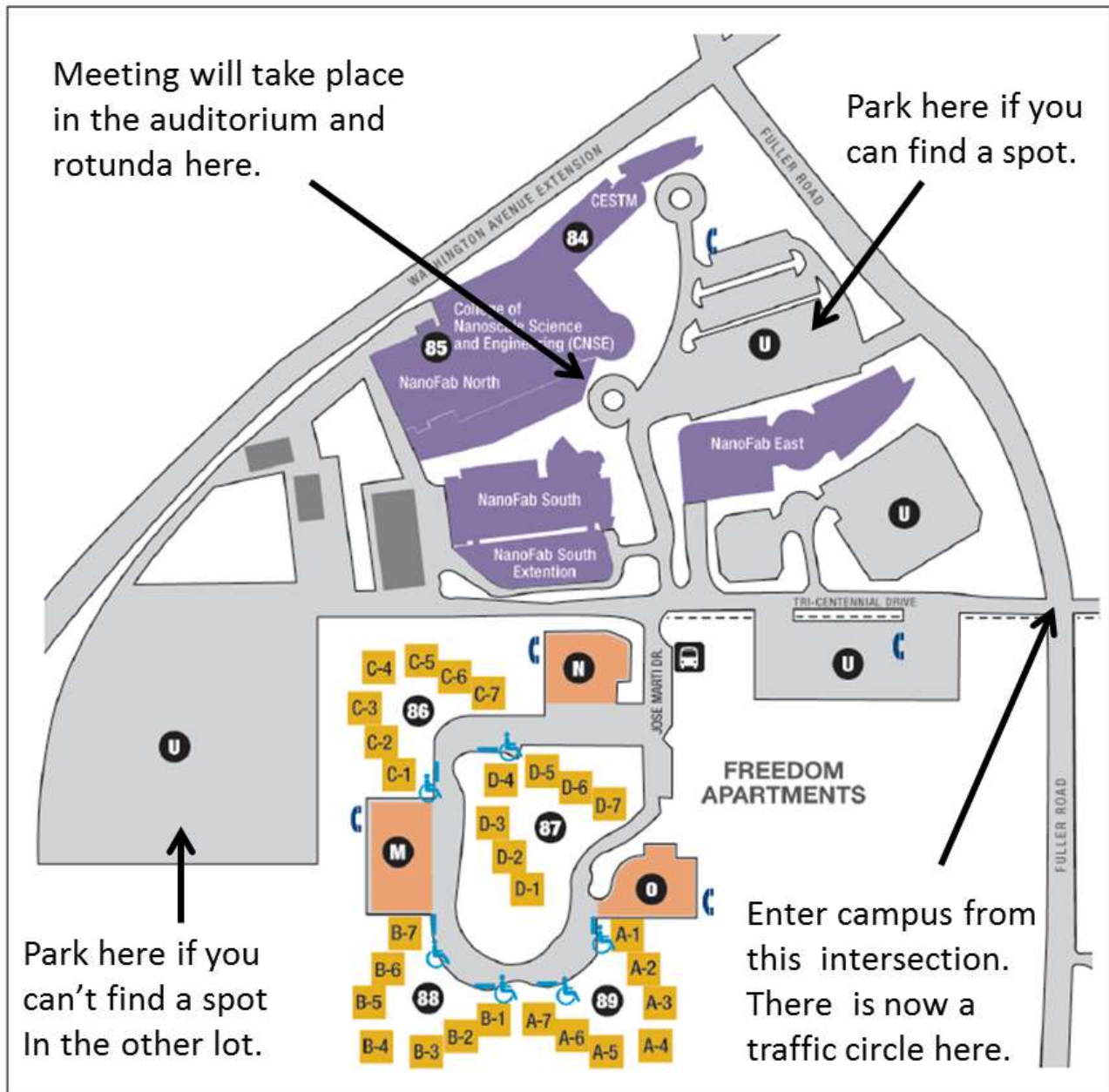
College of Nanoscale Science and Engineering, University at Albany, SUNY  
wnolting@albany.edu

**Abstract:**

Measuring electron spin transport metrics of materials and devices is becoming increasingly important as there is a great desire to fabricate devices that utilize the spin of the electron as well as its charge. This creates a need to measure ultra-low current without creating an increase in the signal-to-noise ratio with variable temperature and magnetic fields. In this poster we will present our custom built spin transport measurement system called the “Spin Lab Measurement System” (SLMS). This system can perform spin relaxation time measurements using non-local spin detection with spin precession (i.e. Hanle technique), along with other standard measurements such as Hall Effect, spin Hall Effect, Kondo Effect and other transport measurements.

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- 87 Lazarus Hall, F3
- 86 Northup Hall, F2
- 88 Stanton Hall, G2
- 89 Truth Hall, G3