Characterization of Thin Films and Materials

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A (Very Incomplete) Report of AVS 2015

We recently attended the 62nd American Vacuum Society (AVS) International Symposium in San Jose, California. The AVS conference is a terrific meeting for those that work in the fields of microfabrication, thin film deposition, nanotechnology, and surface and thin film characterization. The conference ran from October 18 – 23, 2015 and each day featured 14 - 18 parallel sessions, with topics such as applied surface science, 2D materials, additive manufacturing/3D printing, thin films, and vacuum technology, to name only a few. In the evenings, there were great poster sessions as well as organizing meetings for the different divisions. It's quite easy to get involved at AVS. There are lots of opportunities to help organize sessions and participate in moving the society forward, which are terrific ways to serve and meet people. In our experience, the AVS meet-

ing draws an outstanding group of scientists and engineers. The next AVS International Symposium will be from Nov. 6 – 11, 2016 in Nashville, TN. We hope to see you there.

In this article, we will review a few of the presentations/posters we enjoyed at the AVS conference. As a disclaimer, we wish to emphasize that with only two of us, we could only attend a small fraction of the presentations. In addition, the presentations/posters we review here reflect our own research interests, which tend towards surface/material analysis and synthesis/functionalization. So, freely admitting that we are barely scratching the surface (sorry for the pun) of the high quality work that was presented at the meeting, we proceed by highlighting four talks and one poster that we saw presented at AVS 2015. We will also briefly mention the work we presented.

Highlights from AVS 2015

"Sensitivity Factors in XPS: Where Do They Come From and How Accurate Are They?"

By John T. Grant

John Grant has extensive experience in surface science, having worked in this area for over 40 years. He is an expert in Auger electron spectroscopy (AES), X-ray photoelectron spectroscopy (XPS or ESCA), ion bombardment of solids, ion scattering spectroscopy (ISS), secondary ion mass spectroscopy (SIMS) scanning electron microscopy (SEM), low energy electron diffraction (LEED), ionization loss spectroscopy, soft X-ray appearance potential spectroscopy (APS), surface conductivity, surface photovoltaic effects, gas adsorption and desorption, electron

beam interactions with solid surfaces, surface cleaning procedures, ultrahigh vacuum techniques, specimen preparation techniques, and controlled cleavage or fracture of specimens. He has studied polymers, metals, semiconductors, and insulators. Dr. Grant has worked in these fields in Australia, Europe, and the United States and is an internationally-recognized authority in surface science. Currently, his main area of research is on plasma polymerized thin films for optical applications.

Dr. Grant gave an invited talk on quantitation in XPS. XPS is the most widely used surface analysis tool, where its popularity is largely due to its quantitative nature. Quantitation in XPS depends on having accurate sensitivity factors. Most

often, sensitivity factors are supplied by the instrument manufacturer, and too often the user knows little about how they were selected or derived. Incorrect sensitivity factors can result in large errors in quantitation, which may lead to incorrect conclusions about a material's surface composition. As evidence of this, results from two round robin studies were presented.[1, 2] One study indicated that relative areas (the ratio) between two widely separated photoelectron lines varied by as much as a factor of four between instruments, even those from the same manufacturer. Further analysis of this data indicated that this variation could not be attributed to differences in signal attenuation from adventitious carbon overlayers.[1]

A subsequent study concluded that it was not possible to empirically calibrate the responses between different instruments.[2]

Several different types of sensitivity factors have been defined by the International Organization for Standardization (ISO). Indeed, factors that can impact the accuracy of quantitation have been identified, including the excitation source used, instrument geometry, the selected energy resolution, and the degree of surface contamination from adventitious carbon. Each instrument has its own transmission function, which must be measured individually. Sensitivity factors also depend on the sample matrix. In many cases, the provenance of the sensitivity factors provided with the instrument is not well known. In one case, a manufacturer incorrectly used the same sensitivity factors for both Al and Mg X-ray sources. Dr. Grant additionally demonstrated that the analysis software can strongly affect quantitation results, e.g., when the same spectrum is analyzed using the same sensitivity factors, but with different software packages. A simple test for checking the instrument sensitivity factors is to measure several peaks from the same pure element and to check whether they give the same result. For example, if the Au $4d_{5/2}$, $4p_{3/2}$, $4p_{1/2}$ and 4f peaks are quantified from the same pure sample, each peak should give the same fraction of gold (25 at. % here). If they do not, the sensitivity factors are in need of adjustment.

Specific methods for improving XPS quantitation were discussed. Reference materials available from the National Physics Laboratory (NPL) can be used to calibrate any instrument to an accuracy of ± 2%.[3] Software from NPL is also available to help calibrate XPS instruments.[3] A 1992 round robin study across 58 different instruments demonstrated that with calibration, variations in quantitation across instruments could be reduced to about 3%.[4] Without calibration, most XPS instruments are best described as precise, but not accurate.

Dr. Grant will be teaching short courses on analysis and data processing for both Auger electron spectroscopy (AES) and XPS this coming year. More information can be found at www.surfaceanalysis.org.

"The Satellites of the 2p Core Level Transition Metals"

By Alberto Herrera-Gomez

Alberto Herrera-Gomez is deeply interested in data fitting in X-ray photoelectron spectroscopy (XPS). His topics of current study include correct background selection, the theoretical basis for XPS line shapes, and the calculation of uncertainty. He has a series of publications on these topics.[5-9] He also studies the thermal stability of nanofilms using angle-resolved XPS (AR-XPS). He is a professor at the Centro de Investigación y de Estudios Avanzados (CINVESTAV) — Campus Querétaro, Mexico.

Peak fitting is a challenging aspect of XPS data analysis.[10-19] In his presentation, Dr. Herrera-Gomez discussed how applying correct line shapes and backgrounds in the fitting of the 2p peaks for various transition metals led to the discovery of previously unreported satellite peaks. This careful fitting approach also allowed the composition of the corresponding metal oxides to be quantified by applying basic physical parameters, such as the photoelectron cross-section, effective attenuation length, and spectrometer transmission function.

Transition metal 2p spectra consist of several overlapping peaks, as seen in **Figure 1** for nickel. Consequently, they are expected to have a complex background shape. A key tool in the fitting process
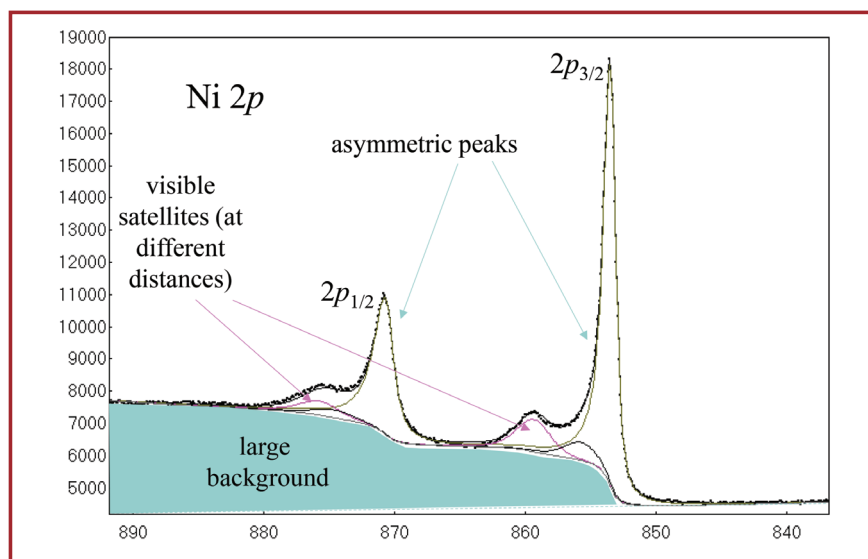

Figure 1. The Ni 2p spectrum, fit using the active background approach and an SVSC background. *Figure obtained from A. Herrera-Gomez.*

used by Dr. Herrera-Gomez was the use of the Shirley-Vegh-Salvi-Castle (SVSC) background in conjunction with an active background fitting approach.[5] In the active background approach, the endpoints of the background and other parameters controlling the background shape are fit together with the peaks in the spectrum. The optimum values are determined by minimizing χ^2. This approach always produces a more accurate fit than the more conventional static approach, where the background endpoints and shape are selected by the user prior to any peak fitting. The active approach also avoids potential user bias and allows the background to consist of different contributions from multiple background types.[5] For example, the SVSC background in the active approach allows the strength of the Shirley background to be varied across each peak in the fit, and therefore is especially suited for fitting regions of overlapping peaks where each peak can have a different background strength. Using an active background, the uncertainties in peak areas can be calculated using a covariance matrix. **Figure 2** shows the SVSC background and the more commonly used (iterative) Shirley background as applied to
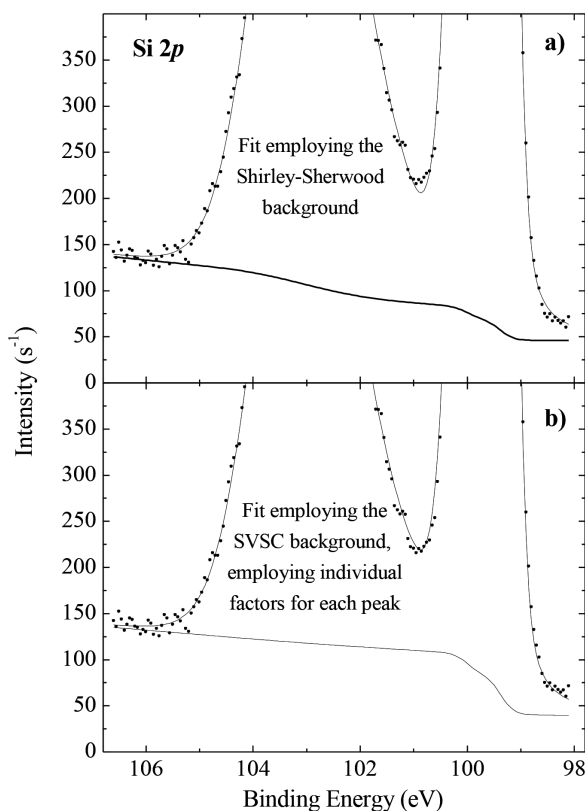
Figure 2. Si 2p spectrum from a silicon wafer with a thin layer of silicon oxynitride fit using the iterative Shirley background (top) and the SVSC background (bottom), both under the active approach. *Figure obtained from A. Herrera-Gomez.*

the Si 2p spectrum from a silicon wafer with a thin oxynitride layer. The SVSC background gives a significantly different background than the Shirley background here. The utility of the active SVSC back-

ground is even more apparent in **Figure 1**, where the satellite peaks would have been lost had the Shirley background been used.

"3D Organic Structure Characterization by FIB-TOF Tomography"

By David M. Carr, Gregory L. Fisher, Shin-ichi Iida, and Takuya Miyayama

Advances in surface analysis are often driven by improvements in instrument technology. Consequently, we are excited to hear from instrument manufacturers about their latest equipment and analytical approaches. Dr. David M. Carr, a Senior Staff Scientist at Physical Electronics (PHI), gave an excellent presentation on the powerful combination of focused ion beam (FIB) tomography and time-of-flight secondary ion mass spectrometry (ToF-SIMS) imaging using the nanoTOF II instrument from PHI. At PHI, David analyzes samples for potential customers using the nanoTOF II. He also works ac-

tively with the R&D and software teams at PHI to improve the instrument.

Chemical imaging is a powerful tool for understanding the distribution of chemical species in a material. Of the surface analysis techniques, ToF-SIMS is especially useful for chemical imaging because it has high spatial resolution, fast data acquisition, high surface sensitivity, and trace level detection limits. With ToF-SIMS, an entire mass spectrum is obtained at every pixel, allowing for retrospective data analysis.

ToF-SIMS is often used with sputter beams to obtain depth profiles. This is a well-established approach, but it also has obvious drawbacks. One challenge in sputter depth profiling is the potential for differential sputtering, which com-

promises the integrity of the 3D depth distribution data. Voids, pores, and embedded particles within the sample may also scramble 3D depth distribution data. In addition, the sputter beam may damage the sample, resulting in a loss of molecular information.

The combination of focused ion beam (FIB) tomography and ToF-SIMS imaging (FIB-TOF) is an exciting alternative to sputter depth profiling as well as to other prominent tomography/imaging techniques such as FIB-scanning electron microscopy (FIB-SEM). Artifacts from differential sputtering are avoided, and porous samples or samples with embedded particles can be analyzed without scrambling the 3D depth distribution data. That is, it brings the advantages of ToF-SIMS to tomography studies, providing highly sensitive, spatially resolved molecular information that cannot be obtained with FIB-SEM. Its trace level sensitivity and the molecular information it provides also give it significant advantages over other tomography/chemical imaging techniques such as energy dispersive X-ray analysis (EDX) and electron energy loss spectroscopy (EELS).

Organic samples are difficult to analyze using FIB-TOF because they are highly susceptible to sputter damage. One option for preparing organic sample cross-sections is to partially mask the sample with a ca. 50 μm metal mask and to then sputter using an argon gas cluster ion beam (Ar-GCIB). The Ar-GCIB sputters the metal mask at an extremely low rate, but the organic sample at a higher rate. Additionally, the GCIB limits the chemical alteration of the organic sample. While this is a simple and effective approach, it only allows one cross-section to be prepared, making it unsuitable for tomography studies. In addition, it only works on purely organic samples, making it unsuitable for mixed organic/inorganic materials. In his presentation, Dr. Carr presented an improved approach, where multiple beams were used for preparing the cross-section. This technique was tested on a sample of polycarbonate (PC) covered with a thin layer of platinum. An FIB was used for preparing the cross-sections. The FIB left a layer of damaged organic material on the cross-section surface, which was
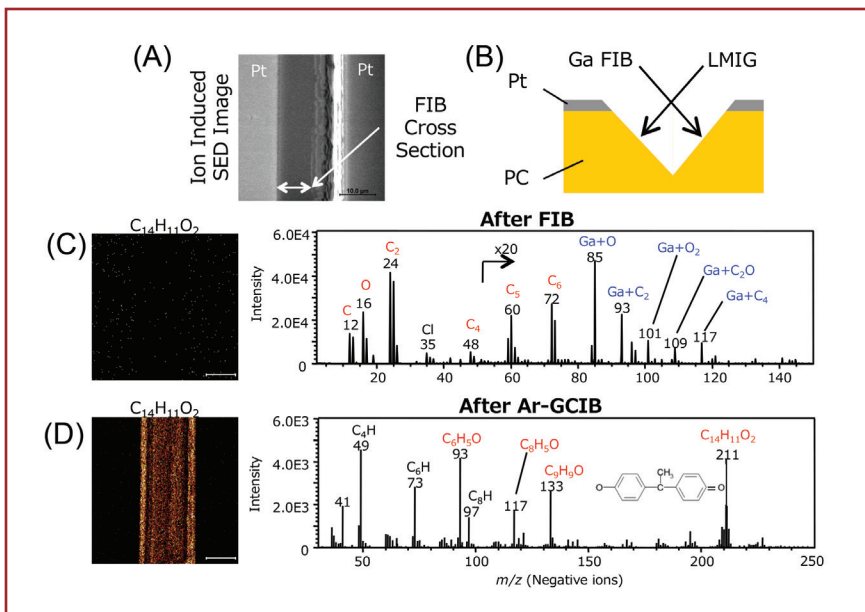
Figure 3. (**A**) Ion induced secondary electron detector (SED) image of a polycarbonate sample coated with a layer of platinum, showing the direction of the FIB cross section. (**B**) Cartoon representation of the polycarbonate sample, showing the direction of the FIB and LMIG beam incidence. (**C**) (Left) A sample image, showing absence of $C_{14}H_{11}O_2^+$ ions after ion milling by FIB (Right). Negative ion spectrum of FIB cross section showing implantation of Ga and organic damage; (**D**) (Left) A sample image, showing recovery of $C_{14}H_{11}O_2^+$ ions after polishing the FIB milled surface with the Ar-GCIB. (Right) Negative ion spectrum, showing the recovery of several organic fragments characteristic of polycarbonate after polishing the FIB milled surface with an Ar-GCIB. *Figure obtained from David M. Carr.*

removed by polishing the cross-section surface with the Ar-GCIB. Finally, the sample was analyzed using a liquid metal ion gun (LMIG). The FIB was effective in cutting through both the platinum and the polycarbonate. Prior to polishing the cross-section surface with the Ar-GCIB, little molecular information from the PC could be obtained. After polishing, several important organic fragments typical of PC were detected. Thus, this method may provide a versatile approach for performing FIB-TOF studies on samples of mixed organic/inorganic composition. A representation of this approach is provided in **Figure 3**.

"Stress-Directed Compositional Patterning of SiGe Substrates for Lateral Quantum Barrier Manipulation"

By Swapnadip Ghosh, Daniel Kaiser, Jose Bonilla, Talid Sinno, and Sang M. Han

Dr. Han is a Regents Professor in the Department of Chemical and Biological Engineering at the University of New Mexico. His current research topics include (1) thin film processing and nanoscale surface corrugation for enhanced light trapping for photovoltaic devices, (2) technology development for energy harvesting in urban areas, (3) metal matrix composite development for high-efficiency multijunction solar cells, (4) heteroepitaxial films on Si and quantum barrier manipulation for photovoltaic, electronic, and sensor applications, and (5) hybrid micro/nanofluidic systems for advanced bioseparations and analysis.

Dr. Han gave a presentation on compositionally patterning SiGe substrates by elastically deforming the wafer at high temperature using a silicon nanoindenter array. In this approach, the array was pressed against a $Si_{0.8}Ge_{0.2}$ wafer in a clamping fixture with contact pressures ranging from 20-45 GPa, and the wafer was annealed under this pressure at 900-1000 °C for 3 h. A schematic representation of this process is shown **Figure 4**. Under these conditions, the larger Ge atoms diffuse away from the deformed region to form Si-enriched islands surrounded by bulk SiGe. This process was described in detail in a recent publication.[20] This
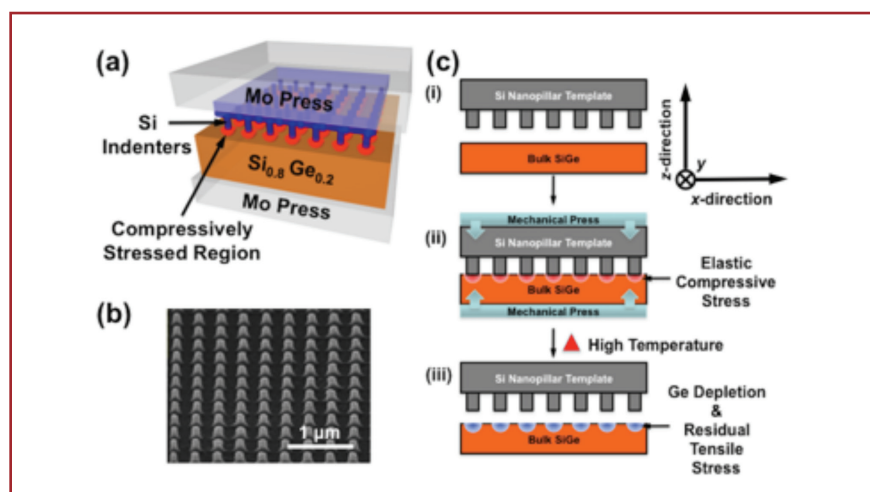

Figure 4. (**a**) Schematic representation of the clamping fixture used by Dr. Han. (**b**) Scanning electron micrograph of the silicon nanoindenter array. (**c**) Schematic representation showing compositional patterning with the nanoindenter array. *Reprinted with permission from* Ghosh, S.; Kaiser, D.; Bonilla, J.; Sinno, T.; Han, S. M., "Stress-directed compositional patterning of SiGe substrates for lateral quantum barrier manipulation." *Applied Physics Letters* **2015**, *107* (7), 072106.
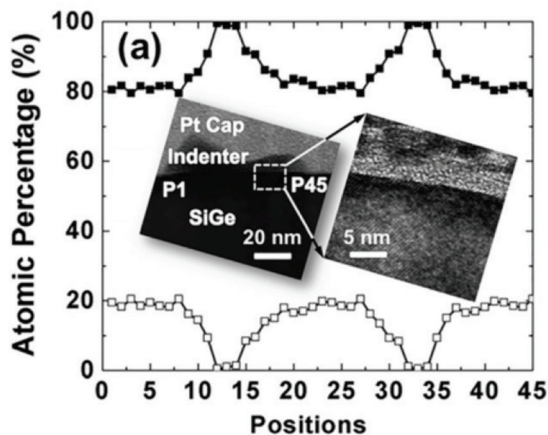
Figure 5. EDS results showing Ge depletion and Si enrichment in the regions contacted by the nanoindenter in Dr. Han's study. Inset TEM image showing complete Ge depletion under elastic deformation. Sample conditions were T = 1000 °C and P = 35 GPa. *Reprinted with permission from* Ghosh, S.; Kaiser, D.; Bonilla, J.; Sinno, T.; Han, S. M., "Stress-directed compositional patterning of SiGe substrates for lateral quantum barrier manipulation." *Applied Physics Letters* **2015**, *107* (7), 072106.

technology could potentially be used to eliminate strain-driven Stranski-Krastanov-type growth and to form quantum dots or dashes with high uniformity and high lateral resolution.

Dr. Han's materials were characterized by scanning electron microscopy (SEM), cross-sectional tunneling electron microscopy (XTEM), scanning tunneling microscopy (STEM), and nanoprobe en-

ergy dispersive spectroscopy (EDS). In order to understand the process in more detail, computational simulations were also performed. SEM showed that at the pressures and temperatures near the maximum used in his study, the substrate could be plastically deformed. When this deformation occurred, there was virtually no compositional change. Some pillars from the nanoindenter array also adhered to the surface under these conditions. However, when pressures/temperatures in the elastic deformation regime were used, the surface composition was significantly altered. TEM/EDS results shown in **Figure 5** revealed that Ge concentrations approaching 0 at. % could be achieved in the modified regions. Computational simulations suggested that pressures as low as 9 GPa will modify the surface composition, with complete depletion of Ge in the compressed regions at pressures of ca. 15 GPa. The simulations also indicated that Ge-depleted regions are under tensile stress.

"Ethylenediamine Grafting on Oxide-Free H-, 1/3 ML F-, and Cl- Terminated Si(111) Surfaces"

By Tatiana Peixoto Chopra,
R. C. Longo, K. Cho, M. D. Halls,
P. Thissen, and Y. J. Chabal

Tatiana is studying Materials Science at the University of Texas at Dallas under the guidance of Prof. Yves Chabal and will earn her doctorate in December 2015. We became aware of her work at AVS through a poster she presented. She also gave a talk on this work. Tatiana's research focuses on the wet-chemical attachment of ammonia and ethylene diamine to modified, oxide-free Si(111) surfaces. In addition, she has studied silicon nitride functionalization in collaboration with Intel.

Ethylene diamine (EDA, en, or $NH_2CH_2CH_2NH_2$) is a versatile organic molecule. For example, it can be used as a linker between self-assembled monolayers and quantum dots or nanoparticles. However, the direct attachment of diamines, and in particular EDA, to silicon surfaces has not been widely studied. EDA may physisorb to surfaces or chemisorb

through one or both of its nitrogens. **Figure 6** shows density functional theory (DFT) calculations of EDA binding to silicon as a monodendate (single attachment point) or bidentate ligand (two attachment

points). Chopra and coworkers studied this amine reaction systematically using three well-defined, atomically flat Si(111) surfaces with different surface terminations: (**1**) an H-terminated silicon sur-
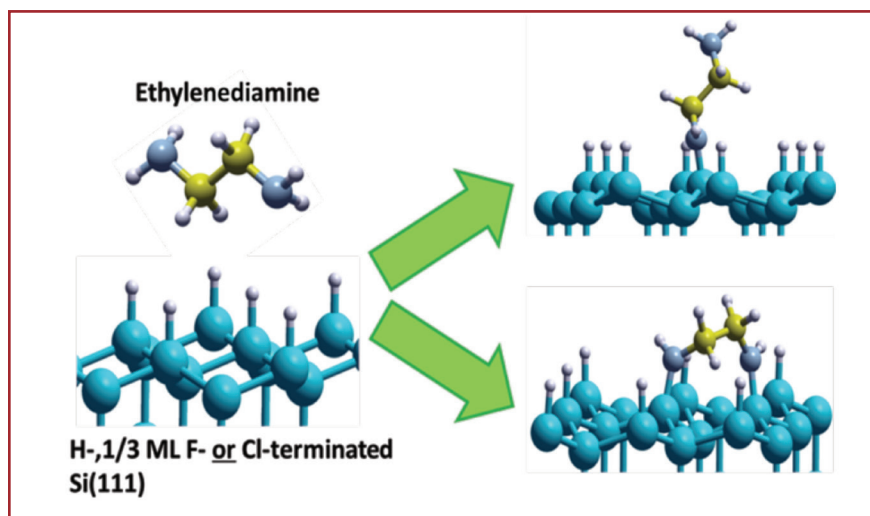

Figure 6. DFT calculations of the two possible reaction outcomes for ethylenediamine chemisorption on a silicon surface. One possibility involves the reaction of only one amine end group (top) leading to monodentate attachment. The second possibility involves the reaction of both amine end groups (bottom), creating a bridging structure. *Reprinted (adapted) with permission from* "Ethylenediamine Grafting on Oxide-Free H-, 1/3 ML F-, and Cl-Terminated Si(111) Surfaces" by T. P. Chopra, R. C. Longo, K. Cho, M. D. Halls, P. Thissen, and Y. J. Chabal. *Chem. Mat.* **2015** *27* (18), 6268-6281. Copyright 2015, American Chemical Society.
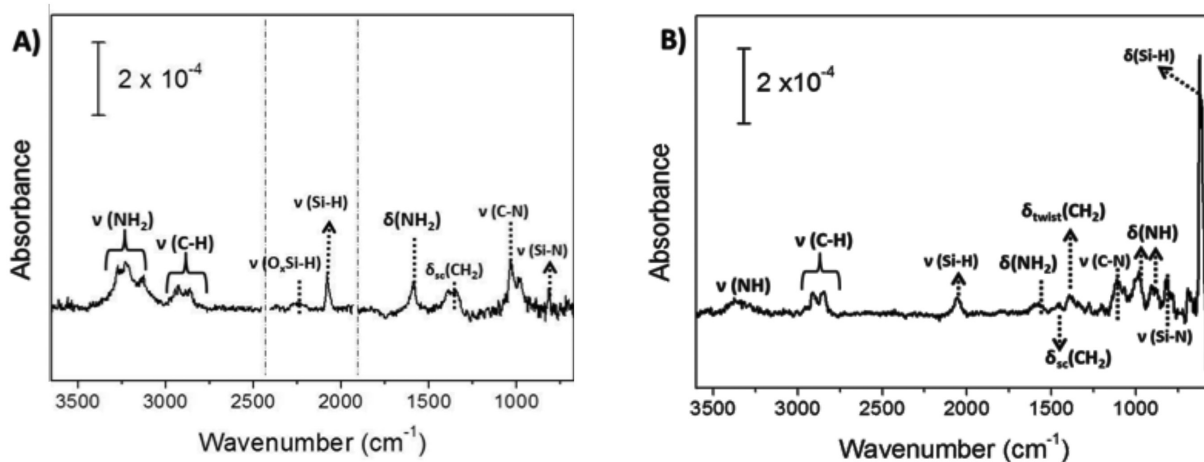
Figure 7. Differential infrared spectra of ethylenediamine vapor phase reaction on the (**A**) 1/3 fluorine- 2/3 hydrogen-terminated Si(111) surface and (**B**) chlorine-terminated Si(111) surface. Each spectrum is referenced to its untreated surface, with the 1900-2400 cm⁻¹ region of **Figure 7** A referenced to the silicon oxide surface in order to show Si-H bonds after EDA reaction. *Reprinted (adapted) with permission from* "Ethylenediamine Grafting on Oxide-Free H-, 1/3 ML F-, and Cl-Terminated Si(111) Surfaces" by T. P. Chopra, R. C. Longo, K. Cho, M. D. Halls, P. Thissen, and Y. J. Chabal. *Chem. Mat.* **2015** *27* (18), 6268-6281. Copyright 2015, American Chemical Society.

face, (**2**) an H-terminated silicon surface with 1/3 of the hydrogens replaced with fluorine, and (**3**) and a chlorine-terminated silicon surface. Both gas phase and liquid phase surface amination reactions were performed, and the surfaces were analyzed using Fourier transform infrared spectroscopy (FTIR) and XPS. DFT calculations supported the experimental results and conclusions.

FTIR and XPS revealed clearly that the surface termination plays a key role in determining the final configuration of adsorbed EDA at the surface. For the H-terminated Si(111) surface exposed to EDA, FTIR showed no Si-N vibration, indicating that the molecule simply physisorbed. However, the fluorine and chlorine terminated silicon did show an Si-N bond vibration after they were exposed to EDA, indicating chemisorption (see

Figure 7). Differences between the two infrared spectra in **Figure 7** indicated that EDA had different binding configurations for the two different surface terminations. For the surface that was partially terminated with fluorine, the Si-N stretch indicated surface chemisorption, while the presence of an NH₂ stretch and scissor modes indicated that the molecule still contained a free amine group. The position of the C-N stretch vibration, XPS results, and DFT calculations all suggested a monodentate attachment of EDA on this surface. In contrast, the experimental results indicated that both amine groups bonded to the more reactive chlorine-terminated silicon surface. Characteristic IR absorption bands for the N-H, CH₂, and C-N bonds, XPS N 1s binding energies, and accessible DFT reaction barrier and reaction energy confirmed the formation

of this bridge configuration. This bridge formation is unprecedented for EDA reactions on modified silicon surfaces. It has only previously been observed under UHV conditions for bare silicon surfaces. IR and XPS also suggested the presence of residual NH₂ groups at the chlorine-terminated surface. Thus, it was concluded that the surface contained a mixture of monodentate and bridging surface groups. In addition, the appearance of Si-H after the surface was exposed to EDA suggested that subsequent reactions can occur after the initial bonding of EDA. Hydrogenation of the initially fully Cl-terminated surface occurred both for vapor and neat EDA reaction exposures, with the exposure method having little influence on the quantity of Si-H bond formation. This work was recently published in *Chemistry of Materials*.[21]

"Quantitative Analysis of Advanced Commercial Glasses for Display Technologies"
By Cody V. Cushman, Nicholas J. Smith, Thomas Grehl, Phillip Brüner, and Matthew R. Linford

"Development of Nanoporous Solid Phase Microextraction (SPME) Fibers by Sputtering"
By Matthew R. Linford, Cody V. Cushman, Bhupinder Singh, Anubhav Diwan

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We also spoke at the conference. Because the results we presented have not yet been published, we are hesitant to provide too much information about them. Cody discussed the surface characterization of display glasses. These are the substrates of microfabricated flat panel displays, which are ubiquitous – they are found

in cellular phones, television screens, tablets, laptops, etc.[22] These advanced electronic materials require special substrates (the display glasses) with carefully controlled compositions, surface quality, and dimensional stability. During the manufacturing process, these substrates undergo cleaning steps that may include

exposure to acids, bases, etching chemistries, detergents, and plasmas. These treatments can modify the surfaces of the glasses, and therefore affect subsequent manufacturing. Angle-resolved (AR-) XPS is a powerful tool for identifying gradients in surface concentrations. We showed AR-XPS data from an HCl-treat-

ed display glass that indicated leaching of network and non-network forming components of the glass. ToF-SIMS supported the AR-XPS conclusions, where the low detection limits of ToF-SIMS were especially useful in studying some of the trace components of the glass. Cody's talk also included a discussion of how to prepare the glass samples in order to minimize hydrocarbon contamination during the analysis. Parenthetically, a week after AVS, we traveled to Münster, Germany to the headquarters of IONTOF. IONTOF produces time-of-flight secondary ion mass spectrometers and low energy ion scattering (LEIS) instruments. We have previously discussed LEIS in a series of *VT&C* articles – we consider LEIS to be a powerful technique that is complementary to XPS and ToF-SIMS.[22-26] At IONTOF we had the opportunity to analyze a series of glass samples using their TOF.SIMS 5 ToF-SIMS instrument and their Qtac 100 LEIS instrument. In next month's article, we hope to publish a pictorial review of these two instruments.

We also presented on the development of new nanomaterials for solid phase micro-extraction (SPME). In SPME, a coated fiber that is ca. 1 cm long is exposed to one or more analytes (target molecules) of interest. This exposure may take place in the gas phase, i.e., if the analyte has a reasonable vapor pressure, the SPME fiber can be put above the liquid (solution) where it is present, sampling/extracting and concentrating it from the vapor (headspace) that is above the solution. The fiber is then inserted into another analytical instrument like a gas chromatograph, which desorbs the analytes from the fiber and separates them. All of this allows the identification of the analytes, and often their quantitation. Part of the importance of SPME stems from the fact that sample preparation is often the most time consuming and uncertain part of a chromatographic analysis. We hope to have a paper accepted on this topic within the next month – it is submitted. This work follows previous work in our group on making new materials for high performance liquid chromatography and thin layer chromatography.[27-31]

Conclusion

The latest AVS International Symposium was again a success. It was an excellent opportunity to learn about recent developments in surface analysis, thin film deposition, and other related topics. We encourage you to attend next year's meeting in Nashville.

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