

Applied Surface Science

Room Ballroom BC - Session AS-ThP

Applied Surface Science Poster Session

AS-ThP-1 EUV Induced Degradation Studies on Reticles by XPS, *Shriparna Mukherjee*¹, *Alessandro Troglia, Véronique de Rooij-Lohmann*, TNO Science and Industry, the Netherlands

EUV reticles play an important role in the semiconductor manufacturing since their quality directly impacts the resolution and accuracy of the transferred image onto the wafer. This, in turn, affects the performance and yield of the resulting chips. Meanwhile, these reticles are very costly and subject to degradation. Better understanding of degradation mechanisms is therefore necessary to improve the designs and further increase the lifetime. Under the framework of the European project 14ACMOS, TNO develops metrology for reticle degradation assessment. Test samples are produced for metrology studies by inducing degradation on various types of reticles. One of the criteria is that the degradation should be non-reversible upon exposure to the ambient, as the samples will be transported to metrology equipment in ambient conditions. Also, it is preferred to have lateral variations and real EUV-induced degradation. Different types of reticles (multilayer blank, absorber blank, patterned reticles) were exposed to EUV at TNO's EUV beam line 2 (EBL2). To facilitate oxidation, water and oxygen were introduced into the exposure chamber. XPS analysis was performed before and after the EUV exposures. Severe oxidation of Ru and Si were primarily observed on the multilayer blank. Moreover, compared to the pre-XPS, the post XPS analysis showed significant decrease in Ru while no such decrease was observed in case of Mo/Si. This might indicate that EUV induced oxidation resulted in intermixing or surface segregation and delamination in the multilayer blanks.

AS-ThP-2 Advanced Characterization of Sputter Induced Effects on the Work Function Using a Combined ToF-SIMS/SPM Instrument, *Bertram Schulze Lammers, Julia Zakel, Andreas Pelster, Derk Rading, Thomas Grehl*, IONTOF GmbH, Germany

The work function is the minimum energy required to remove an electron from a solid surface. This quantity can be directly related to the Fermi level which is of major interest for solid-state physics, material science, and semiconductor applications. Kelvin Probe Force Microscopy (KPFM) combines Scanning Probe Microscopy (SPM) with the electrostatic Kelvin probe method. It can laterally resolve the work function difference between the probing tip and the sample surface together with the corresponding surface topography.

Although the work function describes a macroscopic property of a solid, it may vary locally due to doping, surface contamination or surface oxides. Therefore, clearly defined measurement conditions are required to avoid artifacts and to gain reliable results.

For this work, a combined instrument for time-of-flight secondary ion mass spectrometry (ToF-SIMS) and SPM is used. It enables working under clean UHV conditions, preparation and measurement take place completely in-situ. As mentioned above, the work function of the sample is not measured absolutely but relatively to the probing tip. Once the chemical termination of the tip changes due to e.g. wear or oxidation, the reference is changed, and a general comparison is not possible anymore.

Furthermore, the resulting voltage differences from varying work functions causes additional contributions to the tip-sample force interaction leading also to artifacts in topography measurements. This variation may be sample specific but can also be artificially induced by the ion beam. Separating the work function effect from the topography signal enhances the reliability of the SPM results.

For comparative studies or quality control purposes it is mandatory to control the reference, determined by the termination of the tip. This can be achieved by in-situ tip cleaning, as is performed by the ion sources of the ToF-SIMS. The ion milling removes unknown contaminations and potentially sharpens the tip apex, resulting in a well-defined tip as a reproducible reference.

As a model system, a silicon wafer is bombarded with different doses of different ions, to compare the effect on the work function. Known tip and sample conditions allow a clear correlation between work function

variations and the ion bombardment to characterize the sputter induced effects on the work function.

This work demonstrates the possibilities of KPFM for the investigation of implants, doping or compound semiconductors. In addition, it aims at separating the effects of the work function in topographic measurements and therefore remove artifacts.

AS-ThP-3 Effect of Pulse Duration and Multi-shot Ablation in Femtosecond Laser Ablation (fs-LA) XPS Depth Profiling of Indium Phosphide, *Charlie Chandler*², *University of Surrey, UK; Dhilan Devadasan, Simon Bacon, Tim Nunney*, Thermo Fisher Scientific, UK; *Mark Baker*, University of Surrey, UK

Femtosecond laser ablation (fs-LA) is a newly developing XPS depth profiling technique which avoids the chemical damage observed using traditional monatomic and gas cluster ion beam sputtering [1]. The laser pulse duration plays a key role in determining the involvement (or not) of thermal processes in the ablation mechanism. InP is a thermally sensitive compound semiconductor material, as shown by enhanced preferential sputtering effects being observed when profiled using a gas cluster ion beam compared to a monatomic ion beam [1]. As such, it is a useful test material for studying the effects of laser pulse length on chemical composition during profiling. fs-LA XPS depth profiles of bulk InP were recorded using a 1030 nm laser for pulse durations varying between 160 fs and 6 ps. To ensure the true chemical composition could be retained at ultrashort pulse lengths, a multi-shot regime at a laser energy below the ablation threshold was required. The effect of laser pulse duration and variation of the number of shots per ablation level on the chemical composition, ablation threshold energy and crater surface morphology during profiling will be presented and discussed.

[1] M.A.Baker et al, *Applied Surface Science* 654 (2024) 159405

AS-ThP-4 Applications of Femtosecond Laser Ablation (fs-LA) XPS Depth Profiling, *Mark Baker, Charlie Chandler*, University of Surrey, U.K.; *Simon Bacon, Dhilan Devadasan, Adam Bushell, Tim Nunney, Richard White*, Thermo Fisher Scientific, UK

XPS depth profiling is widely employed to determine the chemical composition and offer chemical state information for thin films, thin film devices, coatings, surface treatments and surface degradation processes. Traditionally, XPS depth profiling has been performed through sputtering, using a monatomic or gas cluster ion beam (GCIB). However, many materials suffer from ion beam induced chemical damage during profiling, resulting in distorted chemical compositions and incorrect chemical state information being recorded during the depth profile. Recently, in a new approach to XPS depth profiling, ion beam sputtering has been replaced by femtosecond laser ablation (fs-LA). This new methodology has been shown to offer significant advantages over sputtering: (i) avoidance of chemical damage; (ii) profiling to much greater depths (several 10s microns); (iii) faster profiling speeds; (iv) ease of varying the ablation rate for different materials [1]. Using a 1030 nm wavelength, 160 fs pulsed laser, fs-LA XPS depth profiles will be shown for selected thin films, coatings, devices, surface treatments and oxidised surfaces, demonstrating the capabilities of this new technique.

[1] M.A.Baker et al, *Applied Surface Science* 654 (2024) 159405

AS-ThP-5 Standardless, Semi-quantitative ToF-SIMS depth profiling using the Full Spectrum Method (FSM), *Nicolas Molina Vergara*, University of Texas at Austin; *John Curry, Tomas Babuska*, Sandia National Laboratories; *Filippo Mangolini*, University of Texas at Austin

The quantitative evaluation of the depth-dependent chemical composition of thin films plays a pivotal role in the development of novel technologies across several sectors, from electronics to medicine. While Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) offers exceptional chemical sensitivity and spatial resolution as well as the possibility of acquiring data as a function of depth from the surface (through sputtering), standardless quantification has remained a significant challenge due to matrix effects and the complex physics of secondary ion generation. Here, we demonstrate the first successful implementation of the Full Spectrum Method (FSM) for quantitative concentration depth profiling of inorganic thin films using ToF-SIMS. The FSM approach—though documented in only six publications over two decades—effectively minimizes matrix dependencies by leveraging large ion clusters that incorporate numerous neutral atoms, thereby decreasing the ratio of charged particles per cluster. In this study, we systematically quantified molybdenum, sulfur, and oxygen concentrations in physical vapor deposited MoS₂ thin films with varying

¹ JVST Highlighted Poster

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stoichiometries. Our ToF-SIMS measurements achieved excellent agreement with complementary Rutherford Backscattering Spectrometry performed on reference samples from identical deposition batches. This validation not only establishes FSM as a viable pathway for standardless, semi-quantitative ToF-SIMS analysis of complex inorganic systems, but also enhances the analytical capabilities of ToF-SIMS for characterizing complex organic specimens, layered structures, and heterogeneous thin films central to emerging technologies in electronics, energy storage, and catalysis.

AS-ThP-6 Insights Into Battery Chemistry Using TOF-SIMS, XPS, and AES, Jacob Schmidt, Sarah Zaccarine, Amy Ferryman, Physical Electronics USA

Battery devices are complex, multi-layered systems with many surfaces and interfaces that contribute directly to performance. Increased global energy demands and environmental concerns have driven the need for next-generation battery materials with excellent performance and stability, low cost, and improved safety. But the multi-component interfaces and dynamic nature of these systems leads to challenges with their characterization. Developing new materials and technologies to meet energy storage needs requires physicochemical characterization approaches with high-spatial resolution, chemical and morphological information, and correlation of properties.

Time-of-flight secondary ion mass spectrometry (TOF-SIMS), X-ray photoelectron spectroscopy (XPS), and Auger electron spectroscopy (AES) are complementary techniques that, when utilized together, can provide a holistic understanding of complex systems such as batteries. TOF-SIMS offers ppm-level insight into molecular bonding and structural composition, and allows for a wide variety of samples, both in composition (organic or inorganic) and in format (powders, thin films, electrodes). AES provides high-spatial-resolution spectra, images, and maps for nm-range analysis of defects and small sample features. XPS and hard X-ray XPS (HAXPES) can be used to obtain short-range chemical state information. Additionally, these multi-technique instruments have features beneficial to battery device analysis including air-free handling via an inert environment transfer vessel; co-located images, and in-situ/operando analysis of chemical changes as they occur. By using these complementary techniques, the mass spectra, elemental and chemical-state maps, sputter depth profiles, and electronic structures can all be determined. This poster will highlight these powerful combined capabilities on a range of battery materials that can be used to drive next-generation stability and performance.

AS-ThP-7 Update on New Guides and Tools to Encourage and Facilitate Generation and Reporting of Reliable and Reproducible Information Using Surface Analysis Methods, Don Baer, Lyndi Strange, Pacific Northwest National Laboratory

A review of recent literature has revealed several common flaws and limitations in surface analysis using various methods, particularly XPS, as well as a notable deficiency in reporting critical sample, instrument, and analysis parameters essential for assessment of the reported information by readers and any efforts to replicate the results. The core assumption is that new and casual method users want to do quality research, but with the increasing number of techniques involved in many research activities, it is a challenge to have expertise for each method. Multiple efforts have been made in response to the identified problems, each intended to provide easily accessible and useful information and tools that can assist an analyst in avoiding the limitations and faulty analyses found in the literature. This poster will provide updated information on some of these efforts. Topics to be addressed include: 1) as a reviewer or reader, you do not need to be an XPS expert to recognize many peak fitting and analysis issues in XPS, 2) an overview of topics addressed in the topical paper collections Reproducibility Challenges and Solutions I and II appearing in the Journal of Vacuum Science and Technology A, 3) an overview of the topics discussed in the shorter Notes and Insights papers now appearing in Surface and Interface Analysis, 4) information about a series of detailed instrument papers starting to appear in Surface Science Spectra to help with instrument parameter reporting and providing descriptions of instrument operation modes, 5) overview of two new ISO standards for reporting on the selection, handling, storage and preparation of samples for surface analysis to be recorded and included as part of sample provenance information, and 6) other journal and web-based papers and platforms intended to assist peak fitting, parameter reporting and error identification.

AS-ThP-10 XPS Characterization of Thiol-Carbazole Self-Assembled Monolayers for Work Function Modulation in Organic Electronics, Mohamed Nejib Hedhili, Yu-Ying Yang, Shadi Fatayer, Martin Heeney, King Abdullah University of Science and Technology (KAUST), Saudi Arabia

Tuning electrode work functions through self-assembled monolayers (SAMs) is a key strategy for optimizing charge injection in organic electronic devices. In this work, we investigate the interfacial chemistry of thiol-carbazole SAMs bearing bromo (Br) and tert-butyl (tBu) substituents using X-ray photoelectron spectroscopy (XPS) and correlate the findings with changes in the gold work function. XPS confirms successful chemisorption of all SAMs onto Au through characteristic S 2p peaks near ~ 162.0 eV, indicating formation of Au-S bonds. Despite similar anchoring, the electronic nature of the substituents significantly influences the resulting interfacial dipole. The electron-withdrawing Br group in Br-2SCz induces a strong upward shift in the gold work function by +0.71 eV, reaching ~ 5.48 eV, which favors hole injection. In contrast, the bulky electron-donating tBu group in tBu-2SCz lowers the work function to ~ 4.52 eV, enabling improved electron injection. These results highlight how molecular substituents can modulate the electrostatic landscape at the metal interface, offering a precise method for controlling energy level alignment in organic semiconductor devices.

AS-ThP-11 Study of F₂ Thermal Etching combined Remote Plasma treatment for Channel Release in Process of Gate-all-around FET, Junjie Li, Institute of Microelectronics of Chinese Academy of Sciences, China; Mingmei Wang, Lam Research Corporation

Introduction: Gate-all-around (GAA) transistor will replace fin field-effect transistor (FinFET) in technology nodes below 3nm compared with FinFET process[1], GAA process mainly adds three process modules, among which channel release is extremely important. This step requires complete removal of the SiGe sacrificial layer while preserving the Si channel. Although some excellent SiGe selective etching methods have been reported such as gas-phase etching[2] or remote plasma source (RPS) etching [3], However, most of these literatures focus on optimizing etching profile, yet rarely mention the interface state of the Si channel after etching. In this article, we focus on the morphology after different etching depths, obtain interface state density through different schemes, and ultimately develop gate-all-around devices (GAA) to study the effects of different schemes on the electrical characteristics of the devices. This study provides insights and references for the industry in selecting channel release methods.

Results and Discussion: Figure 1 shows the morphology obtained by combining F₂ gas reaction with remote plasma source at different etching depths, and the results show good morphology control from 5nm to 50nm. Figure 2 shows that the scheme exhibits high etching selectivity under high-resolution transmission electron microscopy, and the channel atoms remain intact. Figure 3 shows that adding RPS processing can reduce the interface state. Figure 4 shows the channel of the GAA device released by the combination of F₂ gas and RPS, and the electrical characteristics are better than those released by F₂ alone (with lower subthreshold (ss) characteristics). Conclusion: We propose a new etching method combining F₂ gas and remote plasma, which respectively helps enhance channel mobility and improve subthreshold swing (SS). This offers references and insights for path finding in channel release method and facilitates the volume manufacturing of GAA.

Reference:

- [1] Basker V. S., VLSI, (2024).
- [2] Yi Chen., J. Vac. Sci. Technol. A 43, 042604 (2025).
- [3] Erwine Pargon., et al., J. Vac. Sci. Technol. A 37, 040601 (2019).

AS-ThP-12 Which Instrument Should You Use for High Lateral Resolution Chemical Analysis - SEM/EDS or ToF-SIMS?, Vincent Smentkowski, Deliang Guo, GE Vernova Advanced Research Center; Felix Kollmer, ION-TOF GmbH, Germany

Scanning electron microscopy (SEM) coupled with energy dispersive spectroscopy (EDS) is often the instrument of choice when researchers desire rapid high lateral resolution chemical analysis. Time of flight secondary ion mass spectrometry (ToF-SIMS) instrumentation has matured over the past few decades – state of the art instruments can now perform rapid high lateral resolution chemical analysis of all elements (including H and Li), however its use as a microscopy technique is often overlooked.

BAM-L200, is a certified reference material prepared from a cross-sectioned epitaxially grown layer stack of Al_(0.70)Ga_(0.3)As and In_(0.2)Ga_(0.8)As on a GaAs substrate. The surface of the cross sectional BAM-L200 sample provides a

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flat pattern with layer widths down to 1 nm. Calibration distances, grating periods and layer widths have been certified by TEM with traceability to the length unit. The combination of gratings, isolated narrow lines and sharp edges of wide lines offers plenty of options for the determination of lateral resolution, sharpness and calibration of length scale for analytical instruments.

In this poster, we will compare line scan traces generated using ToF-SIMS and EDS on a SEM. We will show that the lateral resolution using the 20% 80% rule, is 21 nm for ToF-SIMS and 54 nm for EDS. In addition to being faster and providing higher lateral resolution imaging, ToF-SIMS also allows for the collection of a full mass spectrum (all elements from H to U, and high mass molecular fragments) at every volume element; ToF-SIMS also has much higher chemical sensitivity (detection limit for most elements is in the ppm to ppb range); and ToF-SIMS does not require the deposition of a thin conductive coating.

AS-ThP-13 Time Evolution of Chemical Reactions During Aluminium Oxide Atomic Layer Deposition on InAs, Eleni Charitoudi, Nishant Patel, Sina Ritter, Lund University, Sweden; Rosemary Jones, Max IV Laboratory, Sweden; Joachim Schnadt, Lund University, Sweden; Esko Kokkonen, MAX IV Laboratory, Sweden; Rainer Timm, Lund University, Sweden

In this project, we are studying the atomic layer deposition (ALD) of Al_2O_3 on InAs. The interest in this system stems from the intrinsic properties of III-V semiconductors for electronic applications. Specifically, InAs has a narrow, direct band gap and high electron mobility, making it a good candidate to outperform Si-based Metal Oxide Semiconductor Field Effect Transistors (MOSFET) regarding speed and power consumption¹. The detrimental native oxide should thereby be replaced by a so-called high-k oxide such as Al_2O_3 , allowing a smaller gate size. However, the performance of this system is limited due to high defect density in the InAs/ Al_2O_3 interface², which is related to incomplete removal of the native oxide layer. The precise deposition of Al_2O_3 on InAs is achieved through the ALD process, enabling the production of thin films with atomic-scale precision. It is characterized by its layer-by-layer mechanism and self-limiting interaction of two gaseous precursors with the substrate³. In our case, two half-cycle depositions were performed using trimethylaluminum (TMA) and water as precursors. A common method to study the chemical composition and interface quality of ALD films is to perform X-ray photoelectron spectroscopy (XPS) measurements after the entire process or after each half-cycle. Here, we went a step further and used ambient pressure XPS (AP-XPS) to observe time-resolved surface chemical reactions occurring during the ALD half-cycles. For this purpose, the ALD process was implemented in the ALD cell of the SPECIES beamline at the MAX IV Laboratory⁴.

During the first half-cycle, i.e. the deposition of TMA, the As-oxide was removed entirely, simultaneously with the formation of Al components and organic TMA ligands on the sample surface. In contrast, the In-oxide decreased, but still a small amount remained at the interface. Also, it was observed that the formation of Al_2O_3 started already during the initial TMA deposition. During the water deposition, a slight increase in In-Oxide was taking place, indicating the reoxidation of the InAs/ Al_2O_3 interface, which is in contrast to the findings from previous AP-XPS studies of HfO_2 ALD on InAs⁵. These results help to understand the reaction dynamics of ALD processes and the role of the metalorganic precursor, as well as to improve the interface in future MOS-based electronics.

1. D'Acunto et al., Surf. and Interf. 39, 1029272
2. Brennan & Hughes, J. Appl. Phys 108, 0535163
3. Karnopp et al., Coatings 14, 5784
4. Kokkonen et al., J Synchrotron Rad 28, 5885
5. D'Acunto et al., ACS Appl. Electron. Mater. 2, 3915

AS-ThP-14 Enhanced Sensitivity in Low-Energy Inverse Photoemission Spectroscopy with an Off-Axis Parabolic Mirror for Efficient Light Collection, Jong-Am Hong, Kyu-Myung Lee, Min-Jae Maeng, Yongsup Park, Kyung Hee University, Republic of Korea

Inverse photoemission spectroscopy (IPES) is a powerful tool for investigating unoccupied electronic states where the electron beam is incident upon the sample and the emitted photons are detected. However, its broader application is hindered by inherently low sensitivity, caused by the low probability of photon emission during the transition of free electrons to the unoccupied states. To enhance sensitivity through improved photon collection, an off-axis parabolic (OAP) mirror is designed and fabricated for the low-energy version of the IPES (LEIPS). Optical simulations showed that the OAP mirror increased photon collection efficiency from 3.06% to 63.3%, which is experimentally validated in the lowest unoccupied molecular orbital (LUMO) spectra of C60 thin films. The

OAP mirror-LEIPS system is applied to study the energy level alignment (ELA) of pentacene films on substrates with different work functions (WF). By measuring both the highest occupied molecular orbital (HOMO) and LUMO levels, the evolution of transport gaps and the ELA of HOMO and LUMO with the Fermi level are analyzed. Pentacene exhibited n-type behavior on a low WF substrate (Cs_2CO_3) and switched to p-type on a high WF substrate (ITO). The OAP mirror-enhanced LEIPS system significantly reduced the time required for such experiments, enabling efficient and reliable measurements.

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