Manufacturing Science and Technology Group
Room 305 - Session MS+HI-MoM

Machine Learning for Microelectronics Manufacturing Process Control and Materials Discovery R&D

Moderators: Tina Kaarsberg, U.S. Department of Energy, Advanced Manufacturing Office, **Gary Rubloff**, University of Maryland, College Park

8:20am **MS+HI-MoM-1 Advancing Semiconductor Industry Process Control via Data-Centric AI*, Jeff David,*** PDF Solutions **INVITED**

Data-Centric AI.Over the last several decades, much of the research and work in machine learning has been based on a model-centric approach or software centric approach.[1] In recent years, some of the codebase for core algorithms such as neural networks have reached a level of maturity where those base algorithms now essentially represent a solved problem for a lot of applications.This has created the opportunity to focus more on the data to improve results, where there is still a significant amount of room to improve the approaches to boost overall outcomes.Areas of Data-Centric AI that have received a growing amount of attention recently include[1]: Measuring data quality, Data iteration, Data management tools, and Data augmentation and data synthesis.

While there has been increasing application of Data-Centric AI in other industries, there are also opportunities for the application of Data-Centric AI in the semiconductor industry as well.Below are a two examples of approaches that have been explored and implemented:

Metabinning and generalized model across chip products

It is difficult to train a model using available data that can be applied to other chip products, due to the unique bin assignment across potentially many thousands of products, even though the underlying failure mechanisms at the device level may be similar.A solution to this challenge is to create metabins that group together bin ID's that are the same or very similar, across chip products.By generating metabins as new labels and overriding the original hardbin/softbin ID's (which again may be disparate), a generalized model can be trained with more previously available data and quickly applied to new chip products for failure prediction.

Classification of wafer failure patterns

Key difficulties in applying machine learning to the classification of spatial failure patterns on wafers are the limited number of wafer classifications (labels) and wafer data available to train the model for a new chip product.To address these issues, a Data-Centric AI approach can be applied.The first step is to generate Augmented Data: Wafers with known patterns are generated randomly.Then the new patterns can be quickly added to the model, by expanding the pattern definition library and retraining.Unlike actual data, new patterns from one product type might be considered general learning and useable to upgrade models.

References:

(1)Andrew Ng, NeurIPS Workshop, Data Centric AI, December 2021

9:00am **MS+HI-MoM-3 Paths Toward Autonomous Plasma Process Tool Operation by Pairing of Plasma and Machine Learning Technologies*, Jun Shinagawa,*** *P. Ventzek,* Tokyo Electron America Inc., **INVITED**

"Smart manufacturing" initiative is a means to meet automation and process control requirements set by semiconductor device technology that is now far below the 10 nm critical dimension in manufacturing[1]. We present our holistic approach on pairing first principle in-situ plasma diagnostics with machine learning techniques to build key components of autonomous plasma process tool operation system or advanced equipment control (AEC) system. AEC is a multi-module system consisting of plasma monitor and control and fault detection and classification(FDC) modules. Machine learning techniques are used to enhance accuracy and reliability of embedded models in the aforementioned modules.

REFERENCE

[1] SEMI, "What is smart manufacturing?" [Online], Available: https://www.semi.org/en/industry-groups/smart-manufacturing/what-is-smart-manufacturing [Accessed Nov. 30, 2021]

9:40am **MS+HI-MoM-5 Compliant Hybrid Bonding for Large CTE Mismatched Electronic Materials*, Mieko Hirabayashi Hirabayashi,*** *M. Wiwi, S. Herrera, E. Madison, M. Jordan,* Sandia National Laboratories

We will discuss methods for hybrid bonding utilizing low modulus materials to enable heterogeneous integration with high density (< 20 µm pitch) interconnections of CTE (coefficient of thermal expansion) mismatched materials. Hybrid bonding techniques, where mechanical stability is provided from a direct bond and electrical connection through a metal-to-metal bond are used to make high-density electrical connections for materials like hybridized CMOS imagers. The temperature of formation of the direct bond and the stiffness of the bonding material result in highly stressed interfaced, limiting the materials that can be used for a hybrid bond.

We demonstrate the joining dissimilar materials utilizing a compliant bonding layer. By changing materials, we allow stress moderation in the bonding layer which reduces the stress on the top and bottom chips. Reliability decreases significantly when the differences in coefficients of thermal expansion (CTE) are large. With large CTE mismatch between a top chip and a bottom chip, one chip expands more than the bottom chip during thermal cycling. If the interface is brittle, the stress due to the difference in CTE induces cracking at the interface. The current method for addressing this issue is to limit the total area bonded –but this limits the advancement of the technology.

This has ramifications for infrared imagers and other applications that combine compound semiconductors with silicon microchips. The size of hybridized infrared imagers, for example, is limited by the mismatch between the different materials that go into them. This presentation will include the methods we have developed to integrate polymers between dissimilar materials to reduce stress due to CTE mismatch and thus increase reliability.

SNL is managed and operated by NTESS under DOE NNSA contract DE-NA0003525

10:40am **MS+HI-MoM-8 Machine Learning Accelerated Scale-up for Microporous Materials - An Industrial Perspective*, Di Du,*** *P. Kamakoti,* ExxonMobil Technology and Engineering Company **INVITED**

Microporous materials such as zeolites and MOFs play a crucial role in producing energy and energy products at scale. Traditional approaches for materials development and scale-up are time consuming and involve experience-based trial and error. Two key areas for materials understanding are the critical variables that impact the synthesis and optimization of material properties which are usually described by quantitative synthesis-property relationships (QSPR). This presentation provides an overview of statistical and machine learning approaches to build QSPR. These methods provide a highly efficient path to optimize synthesis parameters towards targets such as purity, crystal size and surface area, and enable us to significantly speed up our materials workflow.

Our workflow combines design of experiments, machine learning, and high-throughput experimentation (HTE). In order to build QSPR, we featurized the characterization data using machine learning and deep learning approaches. For example, we quantified crystal purity using peak deconvolution of powder XRD pattern. We used a deep learning model to calculate crystal size and aspect ratio from scanning electron microscopy (SEM). We performed functional principal component analysis to select the linear region of Brunauer-Emmett-Teller (BET) adsorption curve which is found to be more accurate than Rouquerol's rules. Since the synthesis space for microporous materials is large and complex, we combined Bayesian Optimization and HTE to further accelerate the workflow. The prior knowledge for Bayesian optimization often comes from a sparse matrix. We used an iterative machine learning model to predict and fill the missing values with uncertainty quantification. After optimization, we used feed-forward neural networks to summarize QSPR for extended investigation at different scales.

We validated the accelerated workflow with a known zeolite. Without referring to historical data, we used the workflow to systematically probe a large and complex synthesis parameter space and obtain small pure crystals of the material. The new workflow demonstrated a significant reduction in the number of experiments needed to meet the same goals as past experiments.

11:20am **MS+HI-MoM-10 Optimizing Copper Deposition in High Aspect Ratio Through Silicon Vias*, Jessica N. McDow*,** *R. Schmitt, M. Hirabayashi, J. McClain, M. Jordan,* Sandia National Laboratories

We show an optimization method for filling high aspect ratio through silicon vias (TSVs) that provides insights into the diffusion and suppression kinetics of a superfilling electroplating chemistry. In general, TSV copper filling processes are designed to be used with thinned wafers (<200 µm), but some TSV last and microelectromechanical systems (MEMS) require full wafer thicknesses. To electroplate full-wafer thickness TSVs, a suppressor only solution utilizes an s-shaped negative differential resistance (S-NDR) mechanism.[1,2]

This suppression/fill mechanism is sensitive to the via geometry as well as the overpotential during the electroplating. Using a suppressor only chemistry consisting of copper sulfate, sulfuric acid, potassium chloride, and Tetronic 701, we demonstrate a time-dependent process window where early on too high of an overpotential results in suppressor breakdown and too low of an overpotential results in complete suppression of the deposition process. By controlling the voltage between -520 mV (MSE) and -560 mV (MSE), we were able to demonstrate complete fill of the TSVs in 30% of the time previously required for filling. We also hypothesize that there is a maximum void-free fill rate for suppressor only chemistries.

Understanding the filling kinetics provides a throughput target for microelectronic devices. Copper filled TSVs are a key technology for 3D heterogeneous integration. TSV designs improve device functionality, increase bandwidth per volume, simplify assembly, and enable system miniaturization. In this work, understanding of copper deposition kinetics in a suppressor only electrolyte and the development of optimized plating parameters utilizing the S-NDR mechanism is outlined.

SNL is managed and operated by NTESS under DOE NNSA contract DE-NA0003525. SAND2022-7316A

[1] Rebecca P. Schmitt et al 2020 J. Electrochem. Soc. 167 162517.

[2] D. Josell and T. P. Moffat 2018 J. Electrochem. Soc. 165 D23.

11:40am **MS+HI-MoM-11 Advanced Manufacturing using Virtual Metrology and Equipment Intelligence®** *, **David Fried,*** Lam Research Corporation **INVITED**

The semiconductor industry is now confronting a number of metrology and manufacturing challenges due to critical technology requirements at next-node architectures.Advanced patterning techniques, such as EUV, frequency multiplication and selective deposition, are needed to meet cost and variability challenges at smaller line dimensions.Memory technologies, such as NAND, DRAM, and others, are requiring new materials and the transition to 3D topologies that are more challenging to manufacture.Advanced logic (such as GAA architectures) and heterogenous integration are being pursued in order to reduce power, footprint, and speed in next generation devices, but also require new, higher density and more complex manufacturing techniques.These technology requirements are creating additional metrology challenges, such as a need to measure smaller dimensions in complex 3D structures, increased measurement frequencies and additional demands for metrology data integration and analysis.

In this talk, we will discuss innovative concepts to address some of these next node metrology challenges.We will review the concepts of virtual fabrication and virtual metrology, and how they can be used in conjunction with conventional metrology to better support defect analysis and yield optimization at the latest technology nodes.We will also discuss how physical metrology can be used to calibrate a virtual process model, along with how a virtual process model can be used to validate physical metrology measurements made on a 3D NAND device.

In addition, our presentation will review the concept of Equipment Intelligence®, and how sensor-based metrology is being used to improve chamber and fleet variability.We will discuss how data from in-situ and standalone metrology, using machine learning/artificial intelligence, calibrated models, and advanced analytics, can drive real-time feed-forward and feedback optimization. We will show a specific example of next-generation metrology-based optimization, by presenting an advanced, in-situ etch-depth metrology system that uses spectral analysis and machine learning to deliver significant improvements in wafer-to-wafer etch depth control.

In our conclusion, we will summarize the challenges of next node architectures, and discuss how the concepts discussed in this presentation can be used by all participants in the semiconductor technology space to measure, characterize and address these upcoming challenges.

Atomic Scale Processing Focus Topic
Room 317 - Session AP+AS+EM+HI+PS+SS+TF-TuM

Area Selective Processing and Patterning II

Moderators: Michelle Paquette, University of Missouri-Kansas City, **Christophe Vallee**, SUNY College of Nanoscale Science and Engineering

8:00am **AP+AS+EM+HI+PS+SS+TF-TuM-1 New Precursors and Approaches to ALD and AS-ALD of Metals*, Mikko Ritala,*** University of Helsinki, Finland **INVITED**

Metal ALD is a topic where high technological relevance combines with inspiring and challenging scientific questions. As always, the success of ALD builds on chemistry. There is constant need for new precursors enabling ALD of metals of interest with improved characteristics. A major challenge arise from the strong tendency of metals to agglomerate, hence preventing achieving continuous films at the smallest thicknesses. Lowering of the deposition temperature is of utmost importance to limit the agglomeration. This requires highly volatile and reactive metal precursors and reducing agents. 1,4-bis(trimethylgermyl)-1,4-dihydropyrazine ((Me_3Ge)DHP) is a new reducing that is found more efficient than its earlier reported silicon analogue. $NiCl_2(PEt_3)_2$ in turn represents a series of metal halide adduct compounds of nickel and cobalt where the poorly volatile parent halides are made volatile by proper adduct ligands. The $NiCl_2(PEt_3)_2$ - (Me_3Ge)DHP combination affords deposition of Ni at 110 °C which is the lowest temperature for thermal ALD of Ni so far. (Me_3Ge)DHP enables also deposition of gold. This is the first reductive thermal ALD process of gold.

Area-selective ALD of metals is an important topic for self-aligned thin-film patterning. An entirely new approach to this is area-selective etching of polymers. In these etching processes the selectivity arises from the materials underneath the polymer layers. Both O_2 and H_2 can be used as an etchant gas. Etching gas molecules diffuse through the polymer film, and if they meet a catalytic surface underneath, the molecules become dissociated into their respective atoms which then readily react with the polymer etching it away. On noncatalytic surfaces the polymer film remains. When combined with area-selective ALD, self-aligned etching of polymers opens entirely new possibilities for the fabrication of the most advanced and challenging semiconductor devices. An example is given where the area-selective etching of polyimide from Pt was followed by area-selective ALD of iridium using the patterned polymer as a growth-inhibiting layer on SiO_2, eventually resulting in dual side-by-side self-aligned formation of metal-on-metal and insulator (polymer)-on-insulator.

8:40am **AP+AS+EM+HI+PS+SS+TF-TuM-3 Comparing Interface and Bulk Physicochemical Properties of TiO_2 Deposited by PEALD Assisted by Substrate Biasing on Thermal SiO_2 and TiN Substrates, for Area Selective Deposition Application*, Jennifer Not,*** LTM - MINATEC - CEA/LETI, France*; L. Mazet,* STMicroelectronics, France*; T. Maindron,* Minalogic, France*; R. Gassilloud,* CEA-LETI, France*; M. Bonvalot,* LTM - MINATEC - CEA/LETI, France

To bypass the limitations implied by the miniaturization of electronic components, area selective deposition (ASD) is becoming a key point of focus, as photolithography steps are avoided. This bottom-up promising technique, as opposed to the top down approach inherent to etching, relies on nucleation mechanisms resulting from substrate - precursor interactions. Differing nucleation kinetics may indeed be observed under very same experimental conditions,[1] allowing a growth delay on a surface type while simultaneously promoting growth on a different surface.

Atomic Layer Deposition (ALD) remains a technique of choice to obtain area selective deposition. Based upon the self-limiting nature of surface reactions, this technique enables a conformal deposition with atomic-scale thickness precision, and is gradually becoming a major deposition process in the microelectronic industry.

The ALD reactor used in this study includes an ICP deported plasma source and is equipped with an additional RF polarization kit at the back side of the chuck, enabling plasma ion extraction from the source towards the substrate surface. Depending on the ion incident kinetic energy, which can be tuned as a function of the applied polarization bias, this ion flux can modulate the properties of the thin film under growth, opening new perspectives of physicochemical properties. These properties may also vary according to the substrate surface, making this RF polarization kit an interesting experimental knob for the development for ASD processes[2]. PEALD TiO_2 layers of various thicknesses have been deposited with no air break on a 15 nm-thick TiN layer, as well as on a 100 nm-thick thermal SiO_2 substrate, under various polarization bias power from 0 W to 80 W. The purpose of this work is to physically and chemically characterize the obtained thin films with respect to substrate surface, and to understand how these properties evolve with the film thickness and for various bias values. X-Ray Reflectivity (XRR), Grazing Incident X-Ray Diffraction (GIXRD), *in-situ* and *ex-situ* ellipsometry and Angle Resolved X-ray Photoelectron Spectrometry (AR-XPS) measurements have been performed, providing detailed information on chemical bond formation during nucleation and within the bulk TiO_2 layer, and thin film physical properties, such as thickness, density, roughness and crystallinity. The outcome of this study gives some insight into the benefit of bias for area selective deposition of TiO_2 thin films on TiN against SiO_2.

References

1. C. Vallée, M. Bonvalot *et al.*, J. Vac. Sci. Technol. A 38(3) May/June **2020**
2. T. Faraz *et al.*, ACS Appl. Mater. Interfaces, 10, 13158−1318 (**2018**)

9:00am **AP+AS+EM+HI+PS+SS+TF-TuM-4 Area Selective Atomic Layer Deposition of SnO_2 as An Etch Barrier*, Xin Yang,*** University of Texas at Austin*; B. Coffey,* Lam Research Corp*; J. Ekerdt,* University of Texas at Austin

Reactive ion etching (RIE) is widely used in semiconductor nanofabrication processes since it can provide high etch rate, high selectivity, and high anisotropy. Traditional etch masks such as organic photoresists suffer from shortcomings such as low etch selectivity. Other material systems have been investigated to improve the selectivity. $Sn^{(0)}$-containing block copolymers were demonstrated as materials for nanolithographic applications.

Here we propose SnO_2 as a RIE etch mask in fluorine-based etching processes. Tin forms nonvolatile compounds with fluorine enabling tin to function as an etch mask. We establish processes that create SnO_2 grid patterns, which can be transferred into the Si native oxide substrate using SF_6 RIE. The concept is illustrated using a 1000-mesh copper TEM grid as an ultraviolet light shadow mask to generate patterns in polystyrene. SnO_2 patterns are achieved by area selective atomic layer deposition (ALD) using tetrakis(dimethylamino) tin(IV) and H_2O as ALD precursors on a Si native oxide at 170 °C. The selective growth can be directed by the hydrophilicity of the substrate surface. ALD growth of SnO_2 shows no nucleation delay on Si native oxide, which is hydroxylated. By coating the substrate with a polymer such as polystyrene (PS) the reactive sites can be passivated to accomplish selective growth. SnO_2 growth can be blocked up to 50 cycles on H-terminated Si(001), and 200 cycles on cured polystyrene and possibly beyond. Atomic force microscopy (AFM) results show that SnO_2 grown on native oxide has a low roughness of 75 pm, while SnO_2 grown on H-terminated Si has a relative higher roughness of 250 pm indicating a 3-D growth process. To create SnO_2 patterns 20, 50, and 100 ALD cycles of SnO_2 are selectively deposited onto Si native oxide with estimated SnO_2 thicknesses of 1.2 nm, 3 nm and 6 nm, respectively. Samples are then etched with SF_6 RIE for 30 s to 1 min at room temperature and 200 mTorr. AFM results show that SnO_2 grid patterns are transferred into the substrate with a depth of around 300 nm to 1 µm for all three samples. X-ray photoelectron spectroscopy results show that some SnO_2 is transformed into SnF_4 for 100 ALD cycle samples, while all of the SnO_2 is transformed into SnF_4 for 20 and 50 ALD cycle samples.

9:20am **AP+AS+EM+HI+PS+SS+TF-TuM-5 Selective Deposition Two Ways: Chemical Bath Deposition of Metal Sulfides on Organic Substrates***, T. Estrada, **Amy Walker,*** University of Texas at Dallas

Selective deposition has many technological applications. While area selective deposition (ASD) has been widely investigated using atomic layer deposition (ALD), there have been few studies of composition-selective deposition or ASD in which growth occurs at material boundaries. In this talk we shall illustrate these alternate selective deposition methods using two examples.

First, we demonstrate that the composition of tin sulfides is controlled by the bath pH and the interaction of sulfur-containing species with -CH_3, -OH and -COOH terminated self-assembled monolayers (SAMs). On -OH terminated SAMs, as the bath pH increases from 10 to 12, the tin sulfide deposited changes from SnS_2 to Sn_2S_3. On -COOH terminated SAMs the deposit is S_2S_3 at pH 10 and SnS at pH 12. In contrast, on $-CH_3$ terminated SAMs the deposit changes from SnS at pH10 to Sn_2S_3 at pH 12. We attribute this behavior to a competition between the repulsion of the chalcogenide ions by the negatively charged carboxylic acid and hydroxyl terminal groups and an increase in chalcogenide ion concentration with increasing bath pH.

Second, we exploit the interaction of the chalcogenide ions with different SAM terminal groups to deposit CuS nanowires at the junction of micropatterned $-OH/-CH_3$ terminated SAMs. We term this method SEmiconductor Nanowire Deposition On Micropatterned substrates (SENDOM). In SENDOM the deposition reaction is kinetically favored on the $-CH_3$ terminated SAMs but transport of reactants is preferred on the hydrophilic -OH terminated SAM. Thus at short deposition times a nanowire forms at the junction of the -OH and $-CH_3$ terminated surfaces.

9:40am **AP+AS+EM+HI+PS+SS+TF-TuM-6 Anatase Crystalline Phase Discovery on Ultra-Thin Layer TiO2Films During Low-Temperature Ald on Fluorine-Rich Carbon Substrates***, **Brian Butkus**, S. Dabas, C. Feit, J. Ganesan, Z. Parsons, X. Feng, P. Banerjee,* University of Central Florida

A novel approach to crystalline anatase phase in atomic layer deposition (ALD) of $TiO_2$by deposited on a fluorine-rich carbon substrate using titanium (IV) isopropoxide (TTIP) and O_2 plasma. In films deposited at temperatures as low as 100 °C and with a thickness of only 4 nm, highly crystalline anatase phases have been observed. Furthermore, when deposited on glass or silicon substrates other than carbon, TiO_2 films consistently produce amorphous films under these conditions. This talk will highlight the unique substrate-driven crystallization of ALD TiO2 and lays the ground rules for selective crystallization using surfaces with suitable initiation chemistries.

An ALD process using a Veeco® Fiji Gen2 ALD system was used to deposit TiO_2 on hydrophobic, polytetrafluoroethylene-coated carbon substrates (AvCarb GDS2230 from Fuel Cell Store). Temperatures of the ALD ranged from 100 °C to 200 °C, and O_2 plasma (300 watts) and water were used as oxidants. Target film thickness ranged from 4 nm to 22 nm. To characterize the films, Raman, Fourier transform infrared spectroscopy (FTIR), and x-ray photoelectron spectroscopy (XPS) were used. AvCarb GDS2230 substrates with an O_2 plasma as an oxidant resulted in anatase TiO_2 films irrespective of thickness, even when deposited at temperatures below 100 °C. However, the anatase phase is significantly weaker when H_2O is used as the oxidant. An interfacial layer of ALD Al_2O_3 suppresses the growth of the anatase phase. Data from XPS indicates that Ti-F bonds form at the pre-deposition stages of films with anatase TiO_2. On non-fluorinated substrates, where the Ti-F bond does not exist, this structurally distinguishes amorphous TiO_2. This fluorine on the surface of the carbon paper serves as a directing agent[1-5] for the application of TTIP to PTFE in a flourolysis reaction, which drives the TiO_2 to crystallize into anatase films.

Fluorine doped crystallization in Ti-O systems has been reported in sol-gel and hydrothermal approaches to synthesize TiO_2 powders.[6, 7]Here, we report the first gas-phase analog of the above reaction mechanism to synthesize crystalline anatase TiO_2 films. With this strategy, surface initiation chemistries can be used to achieve area-selective and in situcrystallization of films.

11:00am **AP+AS+EM+HI+PS+SS+TF-TuM-10 Site-selective Atomic Layer Deposition: Targeting Electronic Defects***, **Alex Martinson**,* Argonne National Laboratory **INVITED**

While ALD is most commonly employed in uniform conformal growth, more selective precursors and processes may allow for more precise synthetic strategies including targeted reaction at subtly unique surface sites including those that lead to electronic defects. We apply a selective hydration strategy to target reaction at the step edges and/or oxygen vacancies of rutile TiO2 and In_2O_3. We computationally and experimentally investigate the feasibility of facet- and site-selective ALD through accurate asymmetric slab models from which the free energy of adsorption at unique surface sites is leveraged to predict step selectivity. Computational evaluation of ALD precursor adsorption free energies on multiple dehydrated facets further refine the feasibility of a temperature-dependent selective hydration strategy. Initial experiments of Al2O3 and Ga_2O_3ALD nucleation on TiO_2single crystals and MgO ALD on In_2O_3broadly support the computational predictions and strategy. The strategies outlined here provide one possible route to selectively target growth at structural defects of oxide surface that may also act as surface or interface electronic defects.

11:40am **AP+AS+EM+HI+PS+SS+TF-TuM-12 Low Temperature Area-selective ALD and ALE of Pd***, H. Nallan, X. Yang, B. Coffey, **John Ekerdt**,* University of Texas at Austin **INVITED**

Thin film deposition and etching approaches may be constrained by the temperature limits for flexible substrates in roll-to-roll processes.We describe a low temperature ALD route to Pd metal film growth at 100 °C that uses $Pd(hfac)_2$ and H_2.The Pd ALD reaction proceeds in the presence of atomic hydrogen; Pd growth nucleates without delay on a Ni metal seed layer that catalyzes H_2 dissociation.Once nucleated, the evolving Pd surface catalyzes H_2 dissociation.To generate the Ni seed layer, a NiO film is first deposited and reduced with an atomic hydrogen source. The 100 °C ALD of NiO using bis(N,N'-di-*tert*-butylacetamidinato)nickel(II) and H_2O as coreactants is highly selective on exposed oxide surfaces and blocked on organic surfaces.NiO films as thin as 0.5 nm, once reduced to Ni^0, effectively seed Pd growth through catalytic area activation.NiO reduction employs an e-beam heated tungsten capillary at 2.5×10^{-6} Torr H_2 that generates atomic hydrogen to reduce NiO films at 100 °C.

In the event non-selective nucleation occurs on passivated regions, we describe a low temperature ALE route to etch clusters/islands on the passivated regions faster than the desired film.This low temperature route exploits the general findings that metal oxides can be etched by a variety of vapor phase etchants, such as formic acid – the issue is controlled oxidation while minimizing damage to adjacent surfaces.We present a low temperature route that involves VUV ($115 \text{ nm} < \lambda < 180 \text{ nm}$) activation of O_2 to produce atomic oxygen and ozone, with atomic oxygen being the dominant species that oxidizes the near surface region of Pd at 100 °C.Oxidation extents differ between continuous films and discontinuous films enabling the removal of metal islands without significantly etching the film.Density functional theory is used to study the adsorption of oxidants (O and O_3) and describe O diffusion into the films to understand the kinetic limitations of the oxidation step.

Advanced Ion Microscopy and Ion Beam Nano-engineering Focus Topic
Room 301 - Session HI+AP-WeA

Emerging Ion Sources, Optics, & Applications

Moderators: Rosa Cordoba, University of Valencia, Spain, **Florian Vollnhals**, INAM, Germany

2:20pm **HI+AP-WeA-1 Rationalizing and Controlling the Composition and Properties of Materials Deposited Using Charged Particles***, **Howard Fairbrother**,* Johns Hopkins University **INVITED**

Focused ion beam-induced deposition (FIBID) and focused electron beam-induced deposition (FEBID) are charged particle nanofabrication techniques able to directly fabricate 2D and 3D metal-containing nanostructures from organometallic precursors. These techniques provide for precise control over beam position for nanoscale pattern writing without the use of a mask or organic solvents and as such are more environmentally benign and less wasteful than traditional lithography techniques. However, one of the biggest issues with charged particle deposition techniques is the difficulty in controlling the composition of the deposits. Indeed, the creation of pure metal nanostructures in FEBID or FIBID is often a goal for achieving optimal materials properties, but is rarely realized in practice without the need for post deposition processing. In this presentation, and using Pt as an example, I will describe how UHV surface science studies can be used to aid in the design of precursors and selection of deposition conditions that favor the creation of pure metal deposits. Examples will include, (i) the electron beam induced deposition of Pt nanostructures from $Pt(CO)_2Cl_2$, where surface science studies have identified the two sequential steps which can lead to Pt formation, notably the initial deposition event ($Pt(CO)_2Cl_{2(ads)}$ + e^-à $PtCl_{2(ads)}$ + $2CO_{(g)}$) as well as the subsequent removal of Cl via an ESD type process ($PtCl_{2(ads)}$ + e^-à $Pt_{(ads)}$ +$Cl^-_{(g)}$) and (ii) ion-beam induced deposition of Pt from $MeCpPtMe_3$ and $Pt(CO)_2X_2$ precursors, where surface science studies have revealed the greater utility of effecting deposition with lighter ions (e.g. H_2^+, He^+), due to their ability to access deposition conditions over which nearly pure Pt can be deposited as compared to heavier ions (e.g. Ar^+) where Pt sputtering rates preclude the formation of Pt. The microstructure and properties of these Pt deposits as well as the possibilities to use analogous approaches for the charged particle deposition of other metals (e.g. Au) will also be discussed.

3:00pm **HI+AP-WeA-3 Ion Beam Induced Reactions and Deposition of $Pt(CO)_2Br_2$ and $Pt(CO)_2Cl_2$***, **Mohammed Abdel-Rahman**, P. Eckhert,* Johns Hopkins University*; J. Yu, A. Chaudhary, L. McElwee-White,* University of Florida*; H. Fairbrother,* Johns Hopkins University

Direct-write lithography techniques, such as focused ion beam induced deposition (FIBID), are an attractive alternative to traditional lithographic techniques. However, traditional precursors ($Me_3PtCpMe$, $Pt(acac)_2$, for chemical vapor deposition perform poorly as FIBID precursors and result in carbon-contaminated metal deposits. To overcome this limitation, $Pt(CO)_2X_2$ (X = Br, Cl) were chosen as alternative precursors because of its low carbon content and desirable volatility for FIBID. FIBID deposits were created from commercially available $Pt(CO)_2Cl_2$ and lab synthesized $Pt(CO)_2Br_2$, complemented by *in situ* UHV studiesusing XPS and MS to determine the sequence of ion-induced processes that underly the deposition mechanism. Deposition and UHV studies were conducted with Ar^+, He^+, and H_2^+ to determine the effect of ion properties (mass and reactivity) on the deposition process. Results obtained on thin films of precursors molecules deposited on inert substrates at temperatures < 200K under UHV conditions indicate that, regardless of the ion or precursor, the deposition mechanism proceeded via rapid and complete CO loss forming PtX_2. Subsequent reactions are dominated by sputtering of the PtX_2 species with halogen loss proceeding quicker than Pt loss. Consequently, pure Pt deposits are formed beyond a critical ion dose. Although varying the ion does not alter the deposition mechanism, the relative reaction rates for the deposition and sputtering processes increase with ion size following the pattern $Ar^+ > He^+ > H_2^+$. This information can be used to select the steady-state conditions best suited to form pure Pt deposits in the absence of post-deposition purification steps.

3:20pm **HI+AP-WeA-4 Next Generation Ion Beam Resists: Sub-10 nm Helium Ion Beam Lithography***, **Scott Lewis**, G. Derose,* California Institute of Technology

A new class of metal organic resist materials that is based on a heterometallic ring (Figure 1) has been demonstrated with helium ion beam lithography while demonstrating extraordinarily high etch selectivity for silicon of >5:1 (at 8 nm half pitch (HP)) when subjected to a pseudo-Bosch inductively coupled plasma–reactive-ion etch (ICP-RIE). The resist was designed using our latest Monte Carlo simulator (Figure 2a), which we developed because there are no simulators for ion beam lithography. Ion simulation packages such as SRIM/TRIM provide accurate data for ion penetration and propagation into a material for applications such as ion implantation, it fails to provide data on the creating a secondary electron (SE) and creating SE's cascade with further orders of SE's which are essential as they will have large effect on the sensitivity of the resist and will have a large contribution on the proximity effect. Exposing the resist to 35 keV helium ions, produced a nano pattern with a resolution of 8.5 nm HP (Figure 2b and c for a plan and tilted view), and obtained a low exposure dose of 22 pC/cm. This dose is 3 orders of magnitude lower than what is required with EBL when comparing to 100 and 30 keV electrons. To account for the low doses, this talk describes how the helium ion interacts with the resist and shows that the exposure mechanism occurs in two parts. Firstly, the helium ions are confined to the incident beam because of the resist properties of high molecular weight and low density limits the number of scattering sites that the ion encounters. Secondly, the helium ion interaction yields significantly more SE's per incident ions than electrons which had a significant contribution to the exposure efficiency. This is why that a dose lower by three orders of magnitude when compared to electron beam lithography. Through Monte Carlo simulation and X-ray photoelectron spectroscopy we will explain how the resist achieves the extraordinary high dry etch selectivity seen here (Figure 2d) where the 8.5 nm HP pattern was successfully transferred via the dry etching process into the silicon substrate. We will show through the use of Monte Carlo simulations how we were able to dramatically improve the resist by increasing its sensitivity and improving its LER while maintaining the same resolution and its high dry etch electivity. The resist has demonstrated the flexibility to be exposed by ions, electrons and EUV, which makes it a versatile lithographic material with the potential for further customization to address a wide range of nanofabrication challenges.

4:20pm **HI+AP-WeA-7 Novel Source Development for Focused Ion Beam Implantation and Irradiation***, **Edward S. Bielejec**, M. Titze, A. Katzenmeyer, A. Belianinov,* Sandia National Laboratories*; Y. Wang,* Los Alamos National Laboratory*; B. Doyle,* Sandia National Laboratories **INVITED**

We will present on Sandia National Laboratory's Ion Beam Laboratory (IBL) development of novel sources for focused ion beam implantation and irradiation capabilities.The IBL operates seven focused ion beam (FIB) systems that range in ion energy from less than 1 keV to greater than 70 MeV, including a wide range of ion species from protons (H) to lead (Pb) over a range of spot sizes from nm to mm.In particular, we will concentrate on the development of liquid metal alloy ion sources (LMAIS) for our two mass filtered FIB systems, the A&D nanoImplanter and the Raith Velion, both of which include high spatial resolution with CAD based patterning to enable the formation of arbitrary patterned implantation.

We will present an overview of the available LMAIS for the mass filters systems as well asfabrication details and results using both novel Pb and N sources.The Pb source is based on a SnPb alloy using a custom Kovar wire tip in place of the standard tungsten tip usually used for FIB applications.This source has demonstrated a long lifetime comparable to our other alloy-based sources of greater than 2000 mA*hr and less than 50 nm resolution without optimization of the tip design.The atomic N source is based on an AuSn alloy implanted with nitrogen up to the saturation limit and verified using elastic recoil detection (ERD).This N source has demonstrated a relatively short lifetime of less than 100-200 mA*hr and is limited to a total N ion production rate of approximately 2,000 to 10,000 ions/s corresponding to up toapproximately 1 fA of current of singly charged nitrogen ions.The demonstration of these sources works to continue the development of high resolution localized implantation and irradiation capabilities enabling the fabrication of custom implanted samples for cutting edge physics and quantum optics experiments.

Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

5:00pm **HI+AP-WeA-9 Focused Ion Beams from GaBiLi LMAIS for Nanofabrication and Ion Imaging**, ***Torsten Richter**, P. Mazarov, A. Nadzeyka, L. Bruchhaus, U. Mantz,* Raith GmbH, Germany

Focused Ion Beams (FIB) are broadly used in nanoscale science related applications, and they are inherently applied for direct nano-patterning, nanofabrication as well as for nano-analytics. FIB has become established as a direct, versatile, and precise fabrication method of smallest features at high fidelity. High demands are made on the ion beam that is used for direct FIB in terms of beam stability, patterning resolution and adjusting of the sputter yield. Liquid Metal Alloy Ion Source (LMAIS) is an emerging FIB source technology that provides a versatile solution to deliver various ion species from a single source for FIB nanofabrication to enhance resulting nanostructures [1]. However, beside nanofabrication FIB is utilized as a primary beam for SIMS analysis [2] and light ions such as Lithium in particular are well suited for sample imaging due to their low sputter yield and surface sensitive properties.

GaBiLi ion sources provide light and heavy ions from a single Liquid Metal Alloy Ion Source (LMAIS) fulfil requirements for both nanofabrication and nano-analytic [3]. Lithium, Gallium and Bismuth ions are emitted simultaneously, and ion species are separated subsequently in an ExB filter. Therefore rapid, easy, and reliable switching between light Lithium ions, and heavy Bismuth or Gallium ions enables not only novel nanofabrication processes but also satisfies analytical demands. GaBiLi allows 3D mill&image by imaging at highest lateral resolution by light Li ions and permits sample delayering at highest depth resolution with Bi ions or even Bi clusters [4]. SIMS takes advantage of optimizing either sputter yield or ionization yield of ejected ions.

In this contribution we present an overview of direct nanopatterning applications and related workflows such as 2-step bowtie nanofabrication with Lithium and Bismuth from GaBiLi (Figure 1) and ion beam imaging results that were obtained with Lithium ions (Figure 2).

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Applied Surface Science Division
Room 320 - Session AS+AC+BI+CA+HI-ThM

Unraveling the Composition of Complex Systems with SIMS

Moderators: Steve Consiglio, Tokyo Electron, **Gregory L. Fisher**, Physical Electronics

8:20am **AS+AC+BI+CA+HI-ThM-2 Ex-Situ, Surface and Bulk Investigations of Defluxing Chemistry Effects on Solder Mask***, **J. Elliott Fowler**,* Sandia National Laboratories*; R. Gerhardt,* Georgia Institute of Technology*; J. Ohlhausen, R. Callaway,* Sandia National Laboratories*; M. Watt,* Georgia Institute of Technology*; S. Grosso, S. Rosenberg,* Sandia National Laboratories

Solder masking is an integral process in the manufacture of printed circuit board assemblies (PCBAs) – it serves to protect the unfinished copper traces from environmental effects, define the soldering pads and provide an adherable surface for conformal coating. Liquid photo-imageable (LPI) solder mask is one of the most popular choices for PCBAs due to ideal electrical and physical properties as well as chemical. LPI solder mask is a heterogeneous epoxy acrylate-based matrix, thus its surface chemistry can be very distinct from its overall bulk chemistry. The surface chemistry is of particular interest as it must be compatible with and resistant to a wide range of chemistries and environments which the PCBA will experience during its production and fielding lifetime. For instance, sensitivity to moisture is a well-known issue. Several studies of solder mask moisture absorption reveal that it reaches a saturation of ~1 weight percent in high-humidity environments, and corresponding changes in insulative properties occur. Risks to mask performance have arisen as a result of the use of new flux formulations which require increasingly aggressive aqueous defluxing chemistries for removal. We hypothesize that alkaline defluxing chemistries will significantly modify the surface chemistry of solder mask, making it more vulnerable to moisture and thus deteriorating their insulative properties.

A commercially available LPI solder mask material was prepared on squares of FR4 board and exposed to increasingly alklaine cleaning chemistries: including DI water, a pH neutral and a pH >10 defluxing chemistry. Samples were tested with three complementary surface-sensitive analytical techniques, x-ray photoelectron spectroscopy (XPS), time of flight secondary ion mass spectrometry (TOF-SIMS) and contact angle goniometry. TOF-SIMS results show that both pH neutral and alkaline chemistries alter the surface, removing PDMS and leaving organic residues. Contact angle goniometry shows that the alkaline chemistry makes the board's surface significantly more hydrophilic. XPS results show a decrease in Si at the surface correlating to a decrease in PDMS versus the control, with the largest decrease occurring due to the alkaline chemistry. In all cases, the surface is somewhat recoverable; however, recovery is less pronounced with increasing pH. AC impedance spectroscopy and DC Surface Insulation Resistance (SIR) testing of exposed samples in elevated humidity environments was performed to resolve changes in insulative performance due to observed changes in mask surface chemistry.

SNL is managed and operated by NTESS under DOE NNSA contract DE-NA0003525

8:40am **AS+AC+BI+CA+HI-ThM-3 Unraveling the Composition of Complex Systems with SIMS***, **Birgit Hagenhoff**,* Tascon GmbH, Germany **INVITED**

For more than four decades SIMS has proven to be a valuable tool in academic research as well as for industrial applications. Whereas the first years focussed on understanding the underlying physical processes using mono-elemental samples, the advent of ToF-SIMS (Time-of-Fligt Secondary Ion Mass Spectrometry) instrumentation in the 80s of the last century opened the path to using SIMS as a screening tool for characterising "the unknown", i.e. samples about which hardly anything is known before the analysis, where sample preparation should be as scarce as possible and where the analysis is performed without any chromatographic pre-step to separate out sample compounds.

Over the years, intensive discussions between those developing instruments and those applying them on a daily basis lead to a fruitful and steep learning curve in the SIMS communities. Meanwhile SIMS instruments offer a multitude of operational modes and are capable of analysing as 3D volume pixel by pixel. The lateral resolution has almost reached the physical limit of the collision cascade with values well below 100 nm and a depth resolution in the nm-range can be achieved. The development of cluster ion sources leads to the possibility to also probe organic materials in depth. At the same time data evaluation routines have become more powerful to address the large amount of data (several GByte for a 3D data set).

Generally, users would like to address three main questions: what (identification) is sitting where (localization) and how much is it (quantification)? Whereas the localization problem, as mentioned above, meanwhile is comparatively straightforward, identification and quantification still are challenging. In identification, the parallel presence of many elements and organic compounds in the SIMS spectra still asks for an expert to solve the analytical puzzle. Here, the application of multivariate statistical techniques and, more recently, machine learning approaching offer promising paths into the future. For a reliable quantification, the influence of the SIMS matrix effect on the results has to be taken into account. Here, the availability of suited reference samples plays a key role for closing the gap to quantitative techniques, like e.g. XPS (X-ray Photoelectron Spectroscopy).

The talk will therefore focus on identification and quantification issues including the use of multivariate statistics, MS/MS approaches and the use of reference sample comparing SIMS with the quantitative techniques XPS and LEIS (Low Energy Ion Scattering).

9:20am **AS+AC+BI+CA+HI-ThM-5 Time of Flight Secondary Ion Mass Spectrometry (ToF-SIMS) as a Novel Approach to the Characterization of Coatings and Interfaces of Porous Transport Layers***, **Genevieve Stelmacovich**, M. Walker, J. Foster,* Colorado School of Mines*; D. Cullen ,* Oak Ridge National Laboratory*; A. Paxson ,* Plug Power*; G. Bender, T. Schuler, S. Ware,* National Renewable Energy Laboratory*; S. Pylypenko,* Colorado School of Mines

The United States energy infrastructure aims to move towards the integration of hydrogen energy. As such, the production of reliable hydrogen generation through the optimization of water electrolyzers is imperative. In proton exchange membrane water electrolyzes (PEMWE's), the porous transport layer (PTL) plays an important role. Due to the harsh conditions of the cell, titanium is the current state-of-the-art anode PTL material. However, titanium quickly forms a layer of titanium oxide which significantly decreases conductivity of the PTL and respectively decreases the overall efficiency of the PEMWE system. To mitigate oxide effects, coatings are commonly applied to the PTL.

Focused Ion Beam Scanning Electron Microscopy (FIB-SEM) in conjunction with Scanning Transmission Electron MicroscopyEnergy-dispersive X-ray Spectroscopy (STEM-EDS) analysis is a commonly used technique to characterize PTL materials and PTL coatings, specifically to look at coating homogeneity and unfavorable oxide layer formation. Unfortunately, this approach is both time-consuming and labor intensive. Additionally, STEM-EDS analysis only provides elemental information, so if several oxide layers preside, it can be difficult to differentiate them, and thus lead to a lack of understanding fundamental degradation mechanisms. These technique deficiencies have motivated the development of an alternative approach that allow more efficient characterization of these materials. Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) is a valuable technique that is commonly used to characterize thin films and buried interfaces. ToF-SIMS can be performed relatively quickly, provides chemical information, and is sensitive to trace elements. This technique also enables visualization of elemental distribution, which provides more detailed surface and interface information. This presentation will demonstrate capabilities of ToF-SIMS for characterization of PTLs comparing results to TEM analysis of cross-sections obtained with FIB-SEM. This study will highlight similarities and differences between the techniques, expand on technique optimization for these morphologically challenging samples, and suggest paths for future investigation moving forward.

9:40am **AS+AC+BI+CA+HI-ThM-6 Construction of Accurate 3D NanoSIMS Depth Profiling Images of Cells in the Presence of Lateral Variations in Sputter Rate***, M. Brunet, B. Gorman, **Mary Kraft**,* University of Illinois Urbana-Champaign

We have developed a strategy for constructing accurate 3D NanoSIMSdepth profiling images of cells in the presence of lateral variations in sputter rate. In this strategy, we use the secondary electrons acquired in parallel with the negative ions during depth profiling to reconstruct the 3D morphology of the cell every time a depth profiling image is acquired. The morphologies created for each raster plane in the depth profile are adjusted to ensure that the height at every pixel decreases with increasing image plane. The resulting reconstructions of the cell's morphology are used to define the z-positions of the voxels in the component-specific 3D NanoSIMS images. We validated this strategy by comparing morphology reconstructions for secondary electron depth profiling images acquired with focused ion beam - secondary electron microscopyand AFMdata acquired from the cell before depth profiling. The shape, curvature, and relative height of the reconstructed morphology agreed well with the AFM data. Depth correction of 3D NanoSIMSdepth profiling data of a metabolically labeled mammalian cell using this strategy improved visualization of the^{18}O-cholesterol and ^{15}N-sphingolipids distributions intransport vesicles and organelle membranes. Accurate 3D NanoSIMS images that show the intracellular distributions of molecules of interest may now be constructed in the presence of variations in sputter rate and the absence of correlated topography data.

11:00am **AS+AC+BI+CA+HI-ThM-10 Innovations in Nuclear Materials Analysis with SIMS*, Christopher Szakal,*** National Institute of Standards and Technology (NIST) **INVITED**

Global nuclear safeguards efforts, coordinated by the International Atomic Energy Agency (IAEA), require precision measurements to answer questions related to nuclear treaty compliance. Despite those questions seeming relatively simple, and the elemental composition of the target analytes often consisting of just uranium and oxygen, the complex processes employed to answer those questions reveal the importance of subtle nuances to meet analytical objectives. This presentation will explore the complexity of this type of analyte system and how innovations in secondary ion mass spectrometry (SIMS) resulted in a routinely utilized tool to address nuclear safeguards requirements for environmental sampling analysis. The complexity is largely driven by the small amounts of nuclear particle material available for analysis, including challenges for 1) representative sampling of analyte material to represent actual nuclear processes, 2) accuracy and precision of uranium isotopic analyses across a wide dynamic range, and 3) determining when a nuclear process created the analyte material. Precision SIMS measurements provide the opportunity to address these analytical complexities to answer key nuclear safeguards questions, but the presentation will also highlight when limitations are inevitable for which questions can be answered.

11:40am **AS+AC+BI+CA+HI-ThM-12 Understanding Surface Bonding and Molecular Structure with MS/MS Imaging: From Click-Chemistry to Biogenesis*, Gregory L. Fisher,*** Physical Electronics

A TOF-TOF imaging mass spectrometer allows TOF-SIMS (MS^1) imaging and tandem MS (MS^2) imaging to be achieved in a lossless fashion [1,2]. Secondary ions for MS^1 and MS^2 analysis are produced from the same surface area by a primary ion nanoprobe. Monolayer film samples may be characterized without undesired erosion or degradation; even sub-monolayer 2D films are readily characterized. Kilo-electron volt collision-induced dissociation (keV-CID) enables compositional identification and structural elucidation of precursor ion moieties. This analytical capability has been brought to bear for straightforward molecular identification as well as multifaceted studies involving surface modification, polymers, composites, catalysis, forensic and failure analysis, biology and pharmaceuticals. TOF-SIMS tandem MS imaging was employed to unravel the click-chemistry of sub-monolayer films [3] and shed new light to unlock the mystery of molecular biogenesis [4,5].

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12:00pm **AS+AC+BI+CA+HI-ThM-13 Probing Grain Boundary Segregation in 304L Stainless Steel using Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS)*, James Ohlhausen,*** *E. Barrick, D. Susan, C. Robino, K. Hattar, J. Herrmann, P. Duran, J. Rodelas,* Sandia National Laboratories

Boron-rich phases in austenitic stainless steels can promote liquation cracking in the heat affected zone during welding. Stainless steels with boron concentrations even as low as 20 wt. ppm are susceptible to grain boundary cracking after heat treatments. The kinetics of phase transformations during heat treatment that generate the crack susceptible microstructure are currently unknown. Time-of-flight Secondary Ion Mass Spectrometry (ToF-SIMS) was used to map Boron at grain boundaries and in the bulk at trace levels for a series of 304L stainless steel coupons that were heat treated across a range of temperatures and cooling rates to investigate these phase transformations.ToF-SIMS and Scanning electron microscopy (SEM) were used to observe the distribution of chromium borides before and after heat treatment. These results will be used to enable quantitative prediction of thermal processing conditions to avoid weld cracking.Sample preparation methods, ToF-SIMS acquisition conditions and data analysis will be discussed.

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Advanced Ion Microscopy and Ion Beam Nano-engineering Focus Topic

Room 301 - Session HI+AS-ThM

Advanced Ion Microscopy & Surface Analysis

Moderators: Armin Golzhauser, Bielefeld University, Germany, **Shida Tan**, Intel Corporation

8:00am **HI+AS-ThM-1 Defect Engineering on the Atomic Scale with the Helium Ion Microscope*, Frances I. Allen,*** UC Berkeley **INVITED**

The use of ion beams to tune the properties of materials through the introduction of defects is a well-established technique. In this area, focused ion beam microscopes have the advantage that they allow the researcher to irradiate materials locally in a highly controlled manner. Over the last 15 years, the Helium Ion Microscope (HIM) has been employed for a range of defect engineering applications, in particular concerning thin films and 2D materials. Properties tuned include electrical, magnetic, optical and thermal behavior, achieved by varying the concentration of defects and local disorder, controlled by varying the ion dose [1]. In the case of freestanding atomic monolayer materials, it has been shown that irradiation with helium (and neon) ions using the HIM in raster mode (as opposed to e.g. spot mode), can form single vacancy defects and vacancy defect clusters due to single-ion hits [2,3]. Such sub-nanometer pores have applications in gas separation [4] and for selective ion transport in liquid [5]. In this talk, I will discuss the fabrication of sub-nanometer pores in 2D materials using the HIM. I will present characterization results from Raman spectroscopy and high-resolution transmission electron microscopy, and will also discuss the merits of multi-step fabrication workflows in which vacancy “seeds” are first introduced into the 2D material by ion irradiation, that are then expanded into the final nanopores of desired size and shape by plasma treatment and/or electron beam irradiation.

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8:40am **HI+AS-ThM-3 Effects of Defects and Si Doping on Ion Motion in TaOx Bilayer Memristors*, Matthew Flynn-Hepford,*** University of Tennessee Knoxville*; J. Keum, I. Kravchenko, S. Randolph, A. Ievlev, B. Sumpter,* Oak Ridge National Laboratory*; M. Marinella,* Arizona State University*; O. Ovchinnikova,* Oak Ridge National Laboratory

TaOx materials have promising properties for memristive applications such as long state retention time and consistent resistive switching.If the mechanism of the resistive switching can be controlled, these materials could be the foundation for the next generation of neuromorphic computing. A material design approach was implemented with the goal of lowering the operational voltage of these devices.Radial distribution functions (RDF) of the modeled TaOx materials with added defects and Si doping were used to predict the bonding strength of the materials.Experimentally, in order to increase ion mobility, defects were introduced into the active layer by He ion irradiation.Local strong bonding was induced in the form of local Si doping by Si irradiation, in an attempt to induce ion mobility channels where ion motion can be better controlled.In order to probe the mechanism of this resistive switching, conductive atomic force microscopy (c-AFM) and kelvin probe force microscopy (KPFM) were used to induce ion motion in the thin films and probe the change in surface potential, respectively.Specially resolved time-of-flight secondary ion mass spectroscopy (ToF-SIMS) was used to probe the chemical change in the film with applied tip bias.These c-AFM and KPFM experiments along with ToF-SIMS were used to probe the locally defected and Si-irradiated areas in order to better understand the effects of defects and Si doping on ion mobility in TaOx bilayer memristors.

9:00am **HI+AS-ThM-4 Advantages of Using Helium Ion Microscopy for Morphological Analysis of $BiScO_3$-$PbTiO_3$ Piezoelectric Ceramics***, S. Chen, A. Bunevich, Y. Yuan,* ***Karen Kavanagh****, Z. Ye,* Simon Fraser University, Canada

Piezoelectric materials can convert mechanical energy into electrical and vice versa. Imaging by Scanning Electron Microscopy (SEM) is commonly used for initial morphological analysis of the grain size, uniformity and porosity, properties that correlate with the piezoelectric quality of interest. However, piezoelectric ceramic is highly insulating, requiring a conductive coating to inhibit charging while imaging through secondary electron collection in most SEMs. Thus, SEM images may not be representative of the sample surface. We have found that there are numerous advantages to using Helium Ion Microscopy (HIM) instead. In a HIM, a positively-charged focussed He ion beam is used to excite secondary electrons, with sample charging neutralized by a simultaneous, large-area electron flood gun. Samples are analysed directly without any surface modification, enabling rapid comparisons of a sintered batch for selection of the best quality ceramic for electrical testing. Higher resolution is achieved for better images of grain boundaries and textural irregularities that are not visible by SEM. In this talk, we will compare SEM and HIM secondary electron images of various compositions of $BiScO_3$-$PbTiO_3$ ceramics, a high temperature piezoelectric material. We will show examples of nanometer-wide ceramic grain boundaries and triple points that were not visible using SEM. Grain boundaries are regions of changes in the lattice structure that have significant implications for piezo and other electronic properties. We have found HIM images to show surface topography and regions of dramatically different contrast that are invisible in the SEM. Furthermore, the lack of conductive coating allows us to see variations in the grain boundary itself, which may explain why piezoelectric properties fluctuate with region in a single sample. Ceramics with large grains (> 20 mm) and flat surfaces correlated with high ferroelectricity at 200°C, with a P_{max} = 282 mC/cm^2. These samples were also more physically robust and able to be poled at higher temperatures and voltages than previous samples of the same composition, improving their piezoelectric properties. Incorporating HIM into the design and synthesis process allows us to quantify the effects of factors such as sintering temperature and die conditions on the physical quality of the ceramic, which ultimately determines its electronic properties and the feasibility of material commercialization.

Acknowledgments

This work is supported by the Natural Science and Engineering Research Council of Canada (NSERC grant RGPIN-2017-06915).

9:20am **HI+AS-ThM-5 Low-Energy Ion Implantation - Range Comparisons between Theory and Experiment***,* ***Michael Titze,*** Sandia National Laboratories*; J. Poplawsky,* Oak Ridge National Laboratory*; A. Belianinov, E. Bielejec,* Sandia National Laboratories

The continued decrease in size of microelectronic devices has created a need for shallower implanted dopant layers. With the recent discovery of two-dimensional (2D) materials, the ultimate limit for shallow layer implant is incorporating material into a single monolayer. Multi-specie focused ion beams (FIB) can operate with a variety of ion species and enable direct-write implantation of specific ions tailored for an exact application. Prior to any ion irradiation experiment, the range of ions in the material needs to be calculated, often predicted by using freely available Stopping and Range of Ions in Matter (SRIM) simulation.

SRIM simulations are in excellent agreement with experiment for high energy light ions, however, for low energy heavy ions, discrepancies between SRIM and observed experimental values have been reported. We use Rutherford backscattering spectrometry (RBS), Secondary ion mass spectrometry (SIMS) and Atom-probe tomography (APT) to measure the depth of heavy ions in silicon following FIB implantation with energies from 1 – 150 keV. The resolution limit of RBS and SIMS is on the order of nanometers, comparable to the implantation depth for few keV ion implants, requiring the use of APT for measuring lowest energy implants because APT is capable of almost angstrom resolution in the 100 direction of single crystalline Si. The difference between SRIM and experimental result is < 10 nm for all investigated ion energies, however due to the low overall range of the ions, the relative error is larger for lower ion energies with 1 keV as the minimum energy investigated showing > 500 % relative discrepancy.

This work was performed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International, Inc., for the U.S. DOE’s National Nuclear Security Administration under contract DE-NA-0003525. The views expressed in the article do not necessarily represent the views of the U.S. DOE or the United States Government.APT research was supported by the Center for Nanophase Materials Sciences (CNMS), which is a US Department of Energy, Office of Science User Facility at Oak Ridge National Laboratory.

11:00am **HI+AS-ThM-10 Correlative Microscopy Using HIM and HIM/SIMS***,* ***Florian Vollnhals,*** Institute for Nanotechnology and Correlative Microscopy - INAM, Germany*; G. Sarau,* Fraunhofer Institute for Ceramics Technology and Systems - IKTS, Germany*; A. Kraus,* Institute for Nanotechnology and Correlative Microscopy - INAM, Germany*; S. Christiansen,* Fraunhofer Institute for Ceramics Technology and Systems - IKTS, Germany **INVITED**

The Helium Ion Microscope (HIM) has changed the world of charged particle microscopy [1]. The attainable spot sizes have enabled advances in imaging as well as nanotechnological applications such as ion beam lithography, nanopatterning or material modification.

An area that has been challenging for HIM, especially in comparison to scanning electron microscopy (SEM), is sample analysis beyond secondary electron (SE) imaging. While most SEMs are equipped with some form of X-ray detection systems (e.g., EDX) for chemical analysis, and many more analytical modes available for further characterization of physical or chemical sample properties, such capabilities have been limited for HIM [3].

The HIM community has made considerable efforts to improve this situation by introducing ion beam specific detection tools like Rutherford backscattering (RBS), Scanning Transmission Helium Ion Microscopy (STHIM) or Secondary Ion Mass Spectrometry (SIMS) using the neon ion beam provided by the latest generation ORION NanoFab HIM [2].

In HIM-SIMS, the focused ion beam is scanned across and thus sputters the surface, resulting in the emission of atoms and ions. The ions are collected and guided to a mass analyzer, allowing for the detection of ions and small clusters ranging from light elements like hydrogen, lithium and boron to heavy elements like Lead. Especially the detection of lithium is a valuable new tool for battery research.

Recently, the SIMS detection system developed by Wirtz et al. at Luxembourg Institute of Technology (LIST) has been upgraded from four individual detectors into a prototype focal plane detector within the npSCOPE project (npscope.eu, funded by the European Commission), allowing for the detection of many masses in parallel, which is beneficial for many applications for which a compositional analysis is required [3].

In addition to the detector development efforts at LIST, a focus has been set on the development of workflows for correlative microscopy using the HIM in a context of additional analytical modalities. The correlation of high resolution HIM and HIM-SIMS imaging with complementary analytical modalities like atomic force microscopy, optical or Raman microscopy to allow for new insights and overcome some of the limitations of the individual tools [4].

This contribution aims at showcasing applications of HIM-SIMS and offers some insights into correlative microscopy workflows involving the HIM.

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11:40am **HI+AS-ThM-12 Electronic vs. Nuclear Sputtering of Coronene***,* ***Lars Breuer****, T. Heckhoff, M. Herder,* University of Duisburg-Essen, Germany*; H. Tian, N. Winograd,* The Pennsylvania State University*; A. Wucher,* University of Duisburg-Essen, Germany

Electronic sputtering induced by swift heavy ion (SHI) irradiation of solids has been suggested as a relatively soft desorption mechanism for intact molecules in Secondary Ion Mass Spectrometry (SIMS). In order to evaluate the prospects of this “MeV-SIMS” technique as compared to the standard SIMS methodology utilizing nuclear sputtering with projectile energies in the keV range, we have performed a case study using time-of-flight (ToF) mass spectrometry to detect both ionized and neutral particles sputtered from a coronene film. In particular, secondary ion and neutral mass spectra obtained under 4.8 MeV/u Ca, Bi and Au ion impact were compared with those measured under irradiation with keV Ar, C_{60} and Ar_{1500} ions. While secondary ions were directly detected using a reflectron ToF spectrometer, sputtered neutral particles were post-ionized using two different laser photoionization schemes, namely vacuum ultraviolet single photon ionization at 157 nm and infrared strong field ionization at wavelengths between 800 and 1300 nm, respectively. The measured spectra are interpreted in terms of partial sputter yields, fragmentation patterns, emission velocity distributions and ionization probabilities with emphasis on the emission and/or formation of intact molecular ions.

The obtained data clearly demonstrate that the MeV-induced electronic sputtering process results in much cleaner molecule spectra than the keV-induced nuclear sputtering process even if cluster projectiles are used in the keV experiment. In particular for the Ca SHI and SPI post-ionization, the measured spectra are completely dominated by unfragmented neutral coronene molecules detected at m/z 300, followed by some fragmentation via the loss of one or more hydrogen atoms. Interestingly, the spectra measured under SHI impact are even cleaner than those measured under thermal evaporation conditions, thereby illustrating a fundamental difference between macroscopic thermal evaporation and the electronic sputtering process. Comparing the secondary neutral and ion spectra, one finds an ionization probability of the intact molecule of the order of 1 % under SHI impact, which may be slightly higher than that measured under keV C_{60}^{+} ion impact (several 10^{-3}). Apart from the hugely different fragmentation characteristics, no significant difference is found between SHI and keV cluster ion impact regarding the emission velocity distributions of the emitted molecules, thereby indicating that the measured signals largely represent the respective partial sputter yields.

12:00pm **HI+AS-ThM-13 Scanning Transmission Helium Ion Microscopy-How Does It Compare to TEM?***,* ***Annalena Wolff****,* Caltech*; R. Fieth,* QUT, Australia

This work explores the HIM’s analysis capabilities of unstained biological samples using a self-built dark field scanning transmission ion microscopy holder. For thin enough samples, such as thin sections of biological specimen on TEM grids, the high energy helium ions can penetrate through the sample. While the ion transverses through the thin foil, it undergoes collisions with the sample atoms and is deflected. The ion exits the sample at a deflection angle which is specimen thickness, ion energy as well as sample material dependent. The deflection angle can be determined using Monte Carlo simulations. The freeware program Stopping and Range of Ions in Matter was used in this work. This effect can be used to design a dark field scanning transmission ion microscopy holder (DF-STIM). The holder design is based on a previously reported experiment [1]. In principle, ions, which are deflected by a specific angle hit a metal conversion plate, which is mounted at a specified distance h below the sample. Here, the transmitted ions create a secondary electron signal which can be collected by the HIM’s Everhart-Thonley Detector. Ions which are deflected less than the acceptance angle enter a hole in the holder which his located directly below the specimen. This hole acts as a Faraday cup. For this case, no secondary electron signal is created. For biological samples, areas with higher carbon density create signal while areas with lower carbon density create less signal and can this be distinguished in the DF STIM image.

The DF STIM holder is tested by imaging stained and unstained biological samples and the results are compared to TEM measurements.

[1] Emmrich D, Wolff A, Meyerbröker N, Lindner JKN, Beyer A, Gölzhäuser A. Scanning transmission helium ion microscopy on carbon nanomembranes. Beilstein J Nanotechnol. 2021 Feb 26;12:222-231. doi: 10.3762/bjnano.12.18. PMID: 33728240; PMCID: PMC7934706.

[2] Dr. Crystal Cooper is thanked for the many useful discussions and the sample preparation suggestions.

Advanced Ion Microscopy and Ion Beam Nano-engineering Focus Topic

Room 301 - Session HI-ThA

Novel Beam Induced Material Engineering and Nano Patterning

Moderators: Frances Allen, UC Berkeley, **Annalena Wolff**, Caltech

2:20pm **HI-ThA-1 Additive Nano-Manufacturing of Advanced Superconductors, and Devices Using Focused Ion Beam Technology***, **Rosa Córdoba**,* Institute of Molecular Science (ICMol), University of Valencia, Spain **INVITED**

Superconducting materials are dissipationless carriers of electric current and provide macroscopic and robust quantum coherence. These properties render them highly valuable as parts for electrical generators, magnetic sensors, and powerful magnets. To achieve the required performance employed in those applications, bulk superconductors often need nanoengineering. Moreover, when these materials are reduced to the nanoscale becoming **nanosuperconductors**, exciting new physical phenomena emerge. This has encouraged the study of their performance as 1D quantum oscillators and Josephson junction arrays as essential elements to be implemented in circuits.

Ground-breaking proposals have taken advantage of the third dimension (3D) for the development of advanced electronic components, opening fascinating novel routes in the fields of material science, physics and nanotechnology. Thus, **3D nanosuperconductors** could be implemented in future highly-efficient electronic elements. However, their fabrication and characterization remain a challenge.

In this contribution, we introduce a direct-write additive nano-manufacturing method based on focused ion beam technologies to fabricate advanced nano-superconductors at-will. This technique called focused ion beam induced deposition (FIBID) is based on CVD process assisted by an ion beam focused to a few nanometers.

We have prepared 3D superconducting hollow nanocylinders with controllable inner and outer diameters (down to 32 nm), and nanohelices with at-will geometries, by decomposing a precursor with a He$^+$ FIB [1,2]. These nanostrucutres become superconducting at 7 K and show large critical magnetic field and critical current density.Remarkably, these nanohelices display superconductivity up to 15 T depending on the direction of the field with respect to the nanohelix axis. This suggest that their helical 3D geometry and their orientation in a magnetic field play a significant role in the superconducting phase transition. Moreover, fingerprints of vortex and phase-slip patterns are also experimentally identified and supported by numerical simulations based on the time-dependent Ginzburg-Landau equation[3].

Additionally, we present an experimental work on the modulation of electric field-induced superconductivity in 45 nm-wide nanowires fabricated using Ga$^+$ FIB[4]. A theoretical model based on the GL theory explains this modulation by the squeezing of the superconducting state by the electric field.

[1]R. Córdoba et al., *Nano Lett.***2018**, *18*, 1379.

[2]R. Córdoba et al., *Beilstein J. Nanotechnol.***2020**, *11*, 1198.

[3]R. Córdoba et al., *Nano Lett.***2019**, *19*, 8597.

[4]P. Orús et al., *Sci. Rep.***2021**, *11*, 17698.

3:00pm **HI-ThA-3 On Demand Spatially Controlled Fabrication of Single Photon Emitters in Si***, **Gregor Hlawacek**, N. Klingner, M. Hollenbach, U. Kentsch, G. Astakhov,* Helmholtz-Zentrum Dresden - Rossendorf, Germany

Single photon emitters (SPE) are fundamental building blocks for future quantum technology applications. However, many approach lack the required spatial placement accuracy and Si technology compatibility required for many of the envisioned applications. Here, we present a method to fabricate at will placed single or few SPEs emitting in the telecom O-band in Silicon [1] . The successful integration of these telecom quantum emitters into photonic structures such as micro-resonators, nanopillars and photonic crystals with sub-micrometer precision paves the way toward a monolithic, all-silicon-based semiconductor-superconductor quantum circuit for which this work lays the foundations. To achieve our goal we employ home built AuSi liquid metal alloy ion sources (LMAIS) and an Orsay Physics CANION M31Z+ focused ion beam (FIB). Silicon-on-insulator substrates from different fabrication methods have been irradiated with a spot pattern. 6 to 500 Si^{2+} ions have been implanted per spot using an energy of 40 keV. For the analysis and confirmation of the fabrication of true SPEs a home build photo luminescence setup has been used. G-centers formed by the combination of two carbon atoms and a silicon atom are confirmed by measurements of zero phonon lines (ZPL) at the expected wave length of 1278 nm for the case of carbon rich SOI wafers. In the case of ultra clean SOI wafers and high ion fluxes emission from tri-interstitial Si complexes is observed. The SPE nature of these so called W-centers has also been confirmed by ZPL measurements at 1218 nm. The achieved lateral SPE placement accuracy is below 100 nm in both cases and the success rate of SPE formation is more than 50%. After a discussion of the formation statistic we also present an approach how our FIB based approach can be upscaled to wafer-scale nanofabrication of telecom SPEs compatible with complementary metal oxide semiconductor (CMOS) technology for very large scale integration (VLSI).

[1] M. Hollenbach, N. Klingner, N. S. Jagtap, L. Bischoff, C. Fowley, U. Kentsch, G. Hlawacek, A. Erbe, N. V. Abrosimov, M. Helm, Y. Berencén, and G. V. Astakhov, “Wafer-scale nanofabrication of telecom single-photon emitters in silicon,” (2022), arXiv:2204.13173 [quant-ph].

3:20pm **HI-ThA-4 Towards FIB Patterning of Reconfigurable Plasmonic Arrays***, **Ivan Kravchenko**, N. Lavrik,* Center for Nanophase Materials Sciences, Oak Ridge National Laboratory

Arrays of deterministic plasmonic nanoparticles have numerous applications in areas spanning from analytical chemistry, to catalysis, to biomedicine. E-beam lithographic patterning of metal films on inorganic substrates is a well-established technological strategy that enables implementation of 2D arrays of plasmonic particles of various shapes and arrangements with arbitrary complexity [1]. However, existing approaches to creating such arrays are limited to solid substrates that are insoluble in organic solvents since development of e-beam resists relies on the use of ketones or similar solvents that tend to attack or dissolve polymers. Here we explore a novel system in which advanced FIB milling is used to pattern arrays of plasmonic nanoparticles on a polymer film that can be liquified by increasing its temperature above its glass transition point and, therefore, provide a nanofluidic interface and, in turn, a pathway to changing the nanoparticle arrangement by external stimuli. Our initial proof of principle experiments focused on arrays of Au nanoparticles with broken symmetry (Figure 1, Supplemental Document) that can attain mobility due to photothermal or thermophoretic excitation [2].

A Raith Velion focused ion beam/scanning electron microscope (FIB/SEM) was used to mill 25 nm thick Au film sputtered on ZEP520A electron beam resist that coated a silicon substrate. ZEP520A e-beam resist was selected as a copolymer compatible with spin coating and commonly available in a cleanroom setting. Its glass transition point of about 180C makes it a good candidate for experiments in which it can be liquified under photothermal or thermal excitation.

To avoid contamination of the Au film, the FIB milling was done by a doubly ionized gold (Au^{++}) ions at an energy of 35 kV. The beam current of 22 pA provided a sub 20 nm resolution. The dose of ion beam exposure was varied from 500 to 5000 mC/cm^2. The arrays of patterned nanoparticles were characterized by SEM, optical microscopy and Raman spectroscopy. Evolution of the nanoparticle shapes and arrangements were monitored during the FIB milling and subsequent thermal activation experiments.

Acknowledgement

This work was supported by Center for Nanophase Materials Sciences (CNMS), which is a US Department of Energy, Office of Science User Facility at Oak Ridge National Laboratory

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3:40pm **HI-ThA-5 Low Energy Ion Beam Backside Circuit Edit Applications in FinFET Devices***, M. Raza, R. Livengood , T. Malik, O. Sidorov, Z. Malamud, I. Ronen, **Shida Tan**,* Intel Corporation*; M. Wong,* Thermofisher Scientific

Circuit Edit (CE) using Focused Ion Beams (FIB) has been widely adopted in the industry to validate known circuit and design marginality issues, test circuit design changes, and generate engineering samples [1]. An approach, commonly known as backside CE, is to access the transistor cell and lower level interconnects of interest through the bulk silicon. The highly localized FIB milling continues until Shallow Trench Isolation (STI) region or the transistor fins are exposed. This FIB exposed trench is commonly referred to as a Node Access Hole (NAH) and it is within this area that subsequent CE tasks such as, accessing, cutting, or rerouting of lower level signals take place [2]. At the most commonly used ion beam energy of 30 KeV, Ga^+ Ion beam penetration depth, can negatively affect circuit performance by altering the intrinsic device parameters. In order to preserve the functionality of the neighboring active devices, machining geometries need to be limited by the transistor cell size and ion beam material interaction volume

An obvious approach in reducing ion material interaction volume is to operate at lower ion beam energy [3].At reduced beam energy, the lateral machining geometry can increase in size due to reduction in the ion material interaction range.In a FinFET device, the transistor channel is located at the tip of the fin, 50 to 70 nm from the bulk substrate - STI interface. Sufficient reduction in the ion beam material interaction volume also allows non-invasive machining directly over-active transistors.

In this work, we present a new approach in using low energy Ga^+ Ion beams at 5 KeV energy for NAH preparation. At low ion beam energy, we demonstrated that the NAH dimension is no longer limited by the cell size.We will present simulation data on interaction volume of the ion beams relative to the depth of the channel in FinFET, showcasing the effectiveness of low energy Ga^+ beams. Finally, we will present empirical results measuring timing impact of ion beam machining on free running ring oscillator test structures and will show example edit results on latest generation process node devices.

Author Index

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