PCSI

Room Ballroom South - Session PCSI-WeA1

Characterization of Interfaces and Devices

Moderator: Paul M. Koenraad, Eindhoven University of Technology, Netherlands

2:00pm **PCSI-WeA1-1 Imaging the Properties of Atoms and Fields at the Picometer Scale inside Materialsand Devices*, David Muller,*** Cornell University **INVITED**

Electron microscopes use electrons with wavelengths of a few picometers, and are potentially capable of imaging individual atoms in solids at a resolution ultimately set by the intrinsic size of an atom. Even with the rapid advances in aberration-corrector technology, both residual aberrations in the electron lenses and multiple scattering of the incident beam inside the sample, the best resolution possible was an order of magnitude worse than this limit. However, with recent advances in detector technology [1] and ptychographic algorithms to unscramble multiple scattering, the resolution of the electron microscope is now limited only by the dose to the sample, and thermal vibrations of the atoms themselves [2].At high doses, these approaches have allowed us to image the detailed vibrational envelopes of individual atom columns as well as locating individual interstitial atoms that would be hidden by scattering of the probe with conventional imaging modes.The three-dimensional nature of the reconstruction means surface relaxations can be distinguished from the bulk structure, and interface roughness and step edges inside devices can be resolved – including gate-all-around transistors and Josephson junctions. Even the location of all atoms in thin amorphous films now seems within reach.These approaches have also allowed us to image the internal structures of both magnetic and ferroelectric vortices, skyrmions and merons, including their singular points that are critical for accurately describing the topological properties of these field textures.

[1] M. W. Tate, P. Purohit, D. Chamberlain, K. X. Nguyen, R. Hovden, C. S. Chang, P. Deb, E. Turgut, J. T. Heron, D. G. Schlom, D. C. Ralph, G. D. Fuchs, K. S. Shanks, H. T. Philipp, D. A. Muller, and S. M. Gruner. "High Dynamic Range Pixel Array Detector for Scanning Transmission Electron Microscopy" *Microscopy and Microanalysis***22**, (2016): 237–249.

[2] Z. Chen, Y. Jiang, Y.-T. Shao, M. E. Holtz, M. Odstrčil, M. Guizar-Sicairos, I. Hanke, S. Ganschow, D. G. Schlom, and D. A. Muller. "Electron Ptychography Achieves Atomic-Resolution Limits Set by Lattice Vibrations" *Science***372**, (2021): 826–831

2:45pm **PCSI-WeA1-10 Cryogenic Growth and *in-Situ* Fabrication of Superconducting Tantalum Devices*, Teun van Schijndel,*** UC Santa Barbara*; A. McFadden,* NIST-Boulder*; A. Engel, J. Dong, S. Chatterjee,* UC Santa Barbara*; R. Simmonds,* NIST-Boulder*; C. Palmstrøm,* UC Santa Barbara

Superconducting devices are crucial in various fields of quantum information technology, including superconducting qubits and topological quantum computing. The vast majority of either of these qubit technologies use Aluminum as the superconducting component. Al is generally grown at low temperatures to achieve smooth thin films. This allows for easy integration of Al-based devices with material systems such as sapphire, Si, Ge, or III-V materials due to minimal interfacial reactions. Also, Al is often used for Josephson Junctions (JJ) that require *in-situ* oxidation to form AlO_x barriers. While Al is the most common superconductor, other superconductors show promising results as well. In particular, Tantalum-based superconducting qubits on sapphire show low loss and long coherence times.[1,2] One of the contributing factors to the enhancement of qubit performance is their higher chemical resistance during device fabrication. However, only a few substrates can be used to stabilize the required α-Ta phase with a BCC lattice structure. Due to its resilience to high temperatures, low-loss sapphire can withstand the growth of Ta at elevated temperatures necessary for the realization of desirable superconducting properties. Growth of Ta on Silicon or III-V substrates remains a challenge.

In this work, we explore the MBE growth and *in-situ* fabrication of superconducting tantalum films. The growth at ultralow temperatures below 10K shows the stabilization of the required superconducting phase of Tantalum (α-Ta). Moreover, Figure 1 shows that α-Ta can be stabilized at low temperatures regardless of the substrate choice. In each case, a superconducting transition temperature of above 4K is observed. Furthermore, this deposition technique can be combined with *in-situ* shadow masks, which allows for patterns with at least 1 µm precision. This can be used to realize $Ta/Ta_2O_5/Ta$ JJ's by using the native oxide, which is something that has never been shown before, or by depositing other dielectrics *in-situ* such as Silicon or Germanium. Our work demonstrates the growth of high-quality superconducting devices, which enables the exploration of different superconductors and dielectric combinations for use in quantum information technology.

[1] Nat Commun 12, 1779 (2021).

[2] npj Quantum Inf 8, 3 (2022).

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2:50pm **PCSI-WeA1-11 Multi-Technique Characterization of GaN-Based Devices: A Powerful Tool to Probe the in-Depth Chemistry*, Kirène Gaffar,*** CNRS, Université Paris-Sud, France*; S. Béchu, G. Patriarche, M. Bouttemy,* CNRS, France

Gallium Nitride (GaN) technology has proven to be a contender for power electronic applications and has shown its suitability for GaN-based devices such as High Electron Mobility Transistors (HEMT) for high-frequency applications. However, the miniaturization of the device dimensions, such as gate length, requires a thorough mastering of device fabrication process with the help of suitable analytical techniques. In particular, systematic electrical characterization has shown that the interface states have a significant impact on the electrical performance and long-term reliability of GaN HEMT devices. This work ambitions to develop a robust methodology to perform advanced chemical characterization of GaN transistors and better understand how the chemistry of the constitutive layers and interfaces properties impact the electrical response. In addition to STEM/EDX analysis on cross sections, conventionally used to access quantitative information and epitaxy quality, an innovative methodology combining X-ray photoemission spectroscopy (XPS) and Auger Electron Spectroscopy (AES) is developed. Indeed, these techniques not only provide access to elemental compositions but also to key information on chemical environments, especially modifications induced by each technological step during device fabrication. In particular, (nano)-Auger spectroscopy, with its high spatial resolution (12 nm), is a very promising tool to access buried interfaces directly on cross-section, bringing complementary information to STEM/EDX such as oxidation states or contaminant presence. If the added value of this multi-technique approach is obvious, the direct implementation and interpretation of XPS and Auger analyses is not straightforward. In fact, access to the ultimate composition of any GaN-based structure is conditioned by the fitting procedure to ensure a reliable nitrogen content determination. We propose here a reliable methodology to decompose gallium $L_2M_{4.5}M_{4.5}$ transition and nitrogen N 1s photopeakoverlap for precise quantification. We will show how these preliminary results obtained by XPS and STEM/EDX analysis are crucial for an accurate interpretation of Auger spectrum acquired on the same materials with the nano-probe for chemical state identification and to refine the quantification. The correlation between structural observations, chemical information and electrical performances measured on a HEMT transistor will be illustrated on a concrete case.

2:55pm **PCSI-WeA1-12 Mo-SiN_x Granular Metal High-pass Filters*, Laura Biedermann,*** *M. McGarry, S. Gilbert, W. Bachman, M. Meyerson, L. Yates, P. Sharma, J. Flicker, P. Kotula, M. Siegal,* Sandia National Laboratories

Granular metals (GMs) comprise a 3D network of metal nanoparticles embedded in a dielectric matrix. Over the past ~50 years, GM investigations have spanned fundamental physics to unique applications, including Au-SiO_2 GMs used as insulating contacts in vidicons, video cameras used in NASA's Apollo and Voyager missions [1]. As a controlled platform for electron transport studies, GMs exhibit tunneling transport (*e.g.* variable-range hopping, Poole-Frenkel conduction in Ni-SiO_2 GMs) and frequency-dependent conductivity $\sigma(\omega)$ in Pt-SiO_2 and Pd-ZrO_2 GMs [2-4]. These prior GM investigations focused almost exclusively on metal-oxide GMs. Our goal—to develop nanosecond-responsive high-pass filters for electrical grid applications—has advanced development of Mo-SiN_x GMs that exploit these conductivity mechanisms.

High-dielectric strength SiN_x is an attractive matrix for GMs, enabling Mo-SiN_x and Co-SiN_x GMs [5]. However, initial Mo-SiN_x GMs showed weak $\sigma(\omega)$; thermally-excited resistive transport through defective SiN_x overwhelmed the desired transport mechanisms. Fortunately, sputtering Mo-SiN_x in a partial N_2 environment ameliorates these SiN_x matrix defects. X-ray photoemission spectroscopy (XPS) analysis shows deleterious $MoSi_2$ is further reduced by annealing in H_2/N_2 forming gas (Fig. 1a). Improvements in SiN_x insulator quality resulted in the desired many decades reduction in σ_{DC} (Fig. 1b). This evaluation of nanostructure and chemical structure has enabled optimization of high-frequency and high electric (E) field transport

(Fig. 1c, d), key properties of high-pass filters for electric grid applications [6].

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3:00pm **PCSI-WeA1-13 Restructuring Cracks in Rutile TiO_2 with Radiolysis-Driven Rolling of Octahedral Units***, **Silu Guo**, H. Yun, S. Nair, B. Jalan, K. Mkhoyan,* University of Minnesota, USA

When energetic electrons interact with crystals in transmission electron microscope (TEM), a combination of "knock-on" and radiolysis effects takes place [1]. Radiolysis, in particular, is known to either amorphize or crystalize materials and limit the accuracy of the measurements [2]. However, the precise atomistic mechanisms of these transformations are still under debate. Here, we use scanning TEM (STEM) imaging and electron energy loss spectroscopy (EELS) to study the bond-breakage, atomic movements and crystallization mechanisms in rutile-TiO2 driven by radiolysis [3].

Thin-film rutile IrO_2 was grown on top of rutile TiO_2, introducing nano-meter width cracks due to an anisotropic epitaxial strain (Figure 1a). To assess the impact of electron beam exposure on the structural healing process of the atomically sharp cracks in rutile-TiO_2,high angle annular dark-field STEM (HAADF-STEM) time-lapse images of the cracks were obtained (Figure 1b). With the accumulation of electron doses, the crack undergoes a self-healing restructuring process. Based on these observations and quantitative EELS analysis, we propose a "2-step rolling" model for the TiO_6 octahedral building blocks located at the crack's edge of rutile-TiO_2 as a possible mechanism for radiolysis-driven atomic migration (Figure 1c and d). With radiolytic bond breakage (Figure 1c), the TiO_6 octahedral units from the edge of the crack can roll and occupy new sites and, in the process, move materials from both sides of the crack into the gap (Figure 1d) [3]

[1] L. Reimer and H. Kohl, *Transmission Electron Microscopy: Physics of Image Formation* (Springer, Berlin, 2008), 5th edn.

[2] L. W. Hobbs, *Introduction to Analytical Electron Microscopy*, edited by J. J. Hren, J. I. Goldstein, and D. C. Joy (Scanning Microscopy International, Chicago, 1979), p. 437

[3]S. Guo, H. Yun, S. Nair, B. Jalan and K. A. Mkhoyan (arXiv: 2304.03482)

3:05pm **PCSI-WeA1-14 UPGRADED: Growth and Angle-Resolved Photoemission of Strain- and Thickness- Tuned Epitaxial α-Sn Thin Films***, **Aaron Engel**, H. Inbar,* University of California, Santa Barabara*; P. Corbae, C. Dempsey, S. Nishihaya, Y. Chang,* University of California, Santa Barbara*; A. Fedorov,* Advanced Light Source, Lawrence Berkeley National Laboratory*; M. Hashimoto, D. Lu,* SLAC National Accelerator Laboratory*; C. Palmstrøm,* University of California, Santa Barbara

α-Sn, the diamond structure allotrope of Sn, is a zero-gap semiconductor with band inversion. Calculations suggest that epitaxial tensile strain induces a 3D topological insulator (TI) phase, while epitaxial compressive strain induces a 3D Dirac semimetal (DSM) phase [1,2]. When this DSM phase is confined in a thin film, it has been suggested to form a quasi-3D TI phase [3]. Transitions to other phases instead, such as 2D TI, have been suggested as well [4].

We first explore the topological phase of ultrathin unintentionally doped α-Sn thin films. Using spin- and angle-resolved photoemission spectroscopy (ARPES), we study compressively strained α-Sn films on InSb(001). We find clear evidence of the confinement-induced quasi-3D TI phase in compressively strained α-Sn. We also find that the spin-polarization of the topological surface states differs markedly from reports in the literature where the films are intentionally doped with Te [5], indicating this intentional doping (a frequently used procedure) could have a significant effect on the electronic structure of α-Sn.

With the previous behavior benchmarked, we then alloy the α-Sn films with Ge to decrease the bulk lattice constant and switch from compressive to tensile strain when grown on InSb(001). Morphology changes as a function of Ge alloying were studied with *in-situ* scanning tunneling microscopy, and strain was confirmed through X-ray diffraction. The presence of a topological phase transition induced by tensile strain *away* from the expected 3D TI phase is found in ARPES (Fig. 1). Our results pave the way for a better understanding of the effect of strain and confinement on α-Sn's band structure.

[1]Phys Rev B **97**, 195139 (2018).

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[3]Phys Rev Lett **111**, 216401 (2013).

[4]Advanced Materials **33**, 2104645 (2021).

[5]Phys Rev B **97**, 75101 (2018).

3:25pm **PCSI-WeA1-18 Characterization of Buffer Layers for Remote Plasma-Enhanced Chemical Vapor Deposition of Germanium-Tin Epitaxial Layers***, **Stefan Zollner**, C. Armenta,* New Mexico State University*; B. Rogers,* Vanderbilt University*; G. Grzybowski, B. Claflin,* Air Force Research Laboratory

Germanium-tin alloys are of interest for infrared light detectors to increase capabilities in image and data capture and transmission, because they can have a direct band gap with more than about 7% tin. Remote plasma-enhanced chemical vapor deposition (RPECVD) is attractive for growth of Ge-Sn alloys because it enables low-temperature epitaxy on Si using common precursors GeH4 and SnCl4. The growth of such epilayers can be optimized with an initial high-temperature buffer layer. This work focuses on the characterization of this buffer layer using atomic force microscopy, ellipsometry, thin-film powder x-ray diffraction, and x-ray photoelectron spectroscopy (XPS) for different growth conditions.

Thin Ge and Ge-Sn buffer layers with 10-20 nm thickness were deposited on Si (100) substrates for one minute at temperatures from 360°C to 500°C with varying SnCl4 precursor flows mixed with GeH4 and helium. Ellipsometry spectra for all films show critical point structures in the E1, E1+Δ1, and E2 region of Ge, indicating that all layers are crystalline. A layer grown at 360°C without SnCl4 can be described reasonably well as an 11 nm thick layer of crystalline germanium with 2 nm of roughness. Adding SnCl4 to the gas flow significantly reduces the height of the ε2 maximum at E2, indicating that the layer is rough. In addition, a new broad peak appears near 1.3 eV, which is attributed to plasmonic effects arising from metallic β-tin inclusions. The plasmon peak disappears in the layers grown at 490°C with the same SnCl4 flow. We conclude that depositing the buffer layer with SnCl4 at low temperatures leads to β-Sn precipitates, where plasmon oscillations can be excited, but are not present for high-temperature growth.

The tin contents in the layers were also estimated by x-ray photoelectron spectroscopy. While XPS measures the total amount of tin in the layers, the presence of substitutional tin in Ge1-xSnx alloy buffers is best determined with x-ray diffraction. The (002) diffraction peak is forbidden in pure Ge, because the contributions from the two Ge atoms in the primitive unit cell cancel. It is absent in our buffers grown without SnCl4 or at high temperature. The (004) XRD peak position in these layers is also very similar to pure Ge. The Ge1-xSnx (002) peak does appear in buffers grown at temperatures lower than 460°C. From the position of the (004) XRD peak, we can estimate the tin content to be below 7%, ignoring the effects of residual stress. This tin content determined from XRD shifts is much lower than the total tin content of about 20% estimated by XPS.

3:30pm **PCSI-WeA1-19 Near Zero-Field Magnetoresistance and Defects in GaN pn Junctions***, M. Elko, A. Higgins, D. Hassenmayer, **Patrick Lenahan**,* Pennsylvania State University*; M. Flatte, D. Fehr,* University of Iowa*; T. Larsen, M. Craven,* NexGen Power Systems

We report on observation of near zero-field magnetoresistance (NZFMR) in GaN devices, in this case, pn junction diodes. NZFMR is a new technique with great potential in electronic materials physics. [1,2] The NZFMR response is due to recombination centers within the diode depletion regions. A representative NZFMR amplitude versus magnetic field plot is shown in figure 1. Figure 2(a) illustrates the anticipated depletion region recombination current versus bias predicted by standard first order expressions.[3] Figure 2(b) shows the measured NZFMR amplitude versus junction bias. The agreement between the calculated response of figure

2(a) and the experimental observations of figure 2(b) should be considered reasonably convincing, considering multiple approximations involved. The NZFMR pattern peaked near the built-in voltage is expected from recombination within the depletion region. [3,4] The NZFMR phenomena are somewhat similar to low field magnetoresistance phenomena observed in some organic semiconductors. The NZFMR response can be understood within the framework of the stochastic quantum Liouville expression. [1,2] Preliminary analysis of traces represented by figure 3, based upon this framework, indicates that the NZFMR response is consistent with nitrogen vacancies.

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