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Science and Technology of Materials, Interfaces, and Processing

2021 Fall Meeting Hudson Mohawk AVS Chapter Wednesday, October 6, 2021 3:30 – 7:30 PM

CESTM Auditorium and Rotunda SUNY Polytechnic Institute 253 Fuller Road, Albany, New York 12203

Meeting Agenda:

	3:30 – 3:45 PM	Reception (coffee and cookies served)
222	3:45 – 3:50 PM	Introductory comments
229 224	3:50 – 4:10 PM	Yamini Kumaran, CNSE, SUNY Polytechnic Institute Noble-Metal Free Electrode for Hydrogen Generation in Alkaline Medium
S	4:10 – 4:30 PM	<u>Minghua Zhang</u> , Dept. of Materials Science & Engr., RPI Resistivity Size Effect in Epitaxial Ti₄SiC ₃ (0001), CuTi(001) And CuAl₂(001) Layers
223	4:30 – 4:50 PM	<u>Tristen Head</u> , CNSE, SUNY Polytechnic Institute Exploring Optical Lithography for Micro-Nozzle Fabrication: Computational Simulations and Experimental Validation
	4:50 – 5:10 PM	<u>Atharv Jog</u> , Dept. of Materials Science & Engr., RPI Effect of Surface Adatoms on Electron Scattering at Cu(001), Co(0001), and Rh(001) Surfaces
rt	5:10 – 5:30 PM	<u>Rubab Ume</u> , CNSE, SUNY Polytechnic Institute Phase Change Memory With Multilevel Resistance States Based On Group III-Sb Binary Alloys
s	5:30 – 5:50 PM	<u>Joshua Roys,</u> Dept. of Chemistry and Biomolecular Science, Clarkson University <i>Polymerization Under Spatial Confinement</i>
kova ore	5:50 – 6:10 PM	<u>Amir Hegazy</u> , CNSE, SUNY Polytechnic Institute Porous Anode Structures for Lithium-Ion Batteries
ird	6:10 – 7:20 PM	Poster presentations (pizza and beverages served)
0	7:20 – 7:30 PM	Awards ceremony



CNSE Nanotechnology Complex

Parking Information

Parking is free in any of the designated Visitor Lots. However, the number of available non-reserved slots is limited in the A, A1, and H Lots. Therefore, it is preferred that you park in the F Lot.

Directions to F Lot

Enter Tricentennial Drive from the Fuller Road traffic circle. Drive past the NFE building (on your right). Proceed straight at the first stop sign. Drive past the NFS building (on your right). At the second stop sign, take a left into the F Lot.

Visitor Information

Non-CNSE attendees should check in at security desk in the NFE rotunda before proceeding to the CESTM rotunda and auditorium. Visitors need to present a government issued ID (driver's license, non driver's license, or passport) to security in the NFE Rotunda to obtain a Visitor's badge. Non US citizens that possess a Permanent Resident Card (green card) must present the card when they register at the security desk. Non US citizens who do not posses a Permanent Resident Card, should contact Prof. Carl Ventrice (cventrice@sunypoly.edu) for additional security clearance procedures.

Virtual Access to Meeting

Virtual Access

The Fall 2021 meeting of the Hudson Mohawk Chapter of the AVS will be held in-person in the CESTM auditorium and rotunda at the College of Nanoscale Science and Engineering in Albany, NY. For those who cannot attend in-person, the oral presentations will be streamed live via Zoom.

Zoom Link

Topic: AVS Hudson Mohawk Chapter Fall 2021 Meeting Time: Oct 6, 2021 03:45 PM Eastern Time (US and Canada)

Join Zoom Meeting https://us02web.zoom.us/j/89364116635?pwd=VVd3TmpJbXdZaFFwMXdYSW9YM3VLUT09

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COVID-19 Safety Protocol

Masking Policy

It is required by all SUNY Poly faculty, staff, students, and visitors to wear a face covering at all times indoors while on campus, regardless of vaccination status. Therefore, all attendees will be required to wear a mask during the meeting, except when eating or drinking. In addition, the seating capacity of the CESTM auditorium has been reduced to 27 to allow for social distancing.

NOBLE-METAL FREE ELECTRODE FOR HYDROGEN GENERATION IN ALKALINE MEDIUM

Yamini Kumaran¹, Iulian Gherasoiu², Haralabos Efstathiadis¹

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The emerging need of clean and renewable energy drives the exploration of effective strategies to produce molecular hydrogen. With the assistance of highly active non-noble metal electrocatalysts, electrolysis of water is becoming a promising candidate to generate pure hydrogen with low cost and high efficiency. High purity hydrogen is used in many applications such as portable power for cars and in houses and to grow epitaxial layers in silicon devices. This reaction takes place almost exclusively on Pt/C catalysts at the cathode which is expensive and need to be replaced by a metal-based catalyst which can show a comparable HER activity. Transition metal oxides, nitrides and sulfides have been widely explored as catalysts for HER due to their good electronic conductivity and superior corrosion resistance. In this research, MoNi₄ embedded MoO₃ nanorods are synthesized using facile hydrothermal method. Further, Molybdenum Vanadium Nitride is coated on top the synthesized electrode using RF/DC magnetron co-sputtering. This combination of hydrothermal and magnetron sputtering fabrication methods of the electrodes results in high surface area of the electrodes thereby improving the reaction kinetics of hydrogen production. The performance of the electrodes is tested in N_2 saturated 1M KOH solution. The electrodes are characterized by X-ray diffraction and SEM for structural and morphological analysis. This work provides a reliable approach to the production of low cost and high-effectiveness electrodes for the application in commercial electrolyzers.

RESISTIVITY SIZE EFFECT IN EPITAXIAL Ti₄SiC₃(0001), CuTi(001) and CuAl₂(001) LAYERS

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Compound conductors are considered as a replacement material for Cu in highly scaled interconnect lines. They are particularly promising if their higher bulk resistivity is more than compensated by a small resistivity size effect, such that they are more conductive than Cu for narrow lines. Epitaxial Ti₄SiC₃(0001), CuTi(001) and CuAl₂(001) layers are deposited by magnetron co-sputtering onto $Al_2O_3(0001)$ and MgO(001) substrates. The epitaxy is confirmed by x-ray diffraction, the surface morphology quantified by x-ray reflectivity and atomic force microscopy, and the composition measured by photoelectron spectroscopy and Rutherford backscattering. The resistivity size effect is quantified by measuring the resistivity ρ both in situ and ex situ as a function of layer thickness d. The resistivity of MAX-phase Ti₄SiC₃(0001) increases only slightly from ρ = 35.2 ± 0.4 to $37.5 \pm 1.1 \mu\Omega$ cm with decreasing thickness d = 92.1 to 5.8 nm, indicating only a minor effect of electron surface scattering. Data fitting with the Fuchs-Sondheimer model yields a room-temperature bulk resistivity $\rho_0 = 35.1 \pm 0.4 \ \mu\Omega$ cm in the basal plane and an effective mean free path λ = 1.1 ± 0.6 and 3.0 ± 2.0 nm at 293 and 77 K, respectively, assuming completely diffuse electron surface scattering. The intermetallic compounds CuTi(001) and CuAl₂(001) exhibit bulk resistivities of 19.1 ± 0.8 and 7.7 ± 0.4 $\mu\Omega$ cm, and $\rho_{0}\lambda$ values of (18.8 ± 3.2) × 10⁻¹⁶ and (11.9 ± 1.4) × 10⁻¹⁶ Ω m², respectively. Air exposure causes a resistivity increase for both intermetallic compounds which is attributed to a transition from partially specular (p = 0.6) to completely diffuse surface scattering caused by surface oxidation. The overall results indicate that the explored materials or related compounds have great potential to replace conventional Cu as interconnect metal for advanced technology nodes and therefore need to be further investigated.

EXPLORING OPTICAL LITHOGRAPHY FOR MICRO-NOZZLE FABRICATION: COMPUTATIONAL SIMULATIONS AND EXPERIMENTAL VALIDATION

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Conventional microfabrication of hollow micro-needles and micro-nozzles requires multiple processing steps and advanced exposure tools to achieve high aspect ratio structures with good control of side-wall profiles. Using dry-film photoresist sheets and photolithography, we have demonstrated the fabrication of hollow, tapered sidewall SU-8 micro-nozzle structures in a single pass with a simple exposure setup. This is accomplished using various mask patterns in a proximity-based photolithography mode to utilize a diffractive effect followed by post-exposure lamination of the activated photoresist onto the final device. The resulting tapered micro-nozzle geometry has been shown to improve fluid delivery into tissue mimics by reducing interfacial flow at the hydrogel surface. To predict the resulting form of fabricated micro-nozzles, the following numerical simulations are performed: light distribution dependence on mask design and gap distance, light propagation and photoacid generation in the photoresist during exposure, and acid diffusion and SU-8 crosslinking during the post-exposure bake step.

EFFECT OF SURFACE ADATOMS ON ELECTRON SCATTERING AT Cu(001), Co(0001), AND Rh(001) SURFACES

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Electron transport in metals is adversely affected by surface scattering as their dimensions approach the electron-phonon scattering mean free path. In situ transport measurements on 10-nm-thick epitaxial Cu(001), Co(001), and Rh(001) layers demonstrate that electron surface scattering and the consequent increase in resistivity is determined by the perturbation in the flat surface potential caused by surface adatoms. Adding Ti capping layers (0.1-13.0 monolayers thick) on epitaxial Cu(001), Co(0001), and Rh(001) layers causes the measured sheet resistance R_s to increase by 40%, 10% and 5%, respectively, compared to uncoated layers. Similarly, exposing these layers to 0.8 Torr O₂ results in a 26%, 22%, and <5% increase in R_s , respectively. These results suggest that adatoms on Cu and Co surfaces considerably disturb the surface potential, leading to diffuse electron scattering and a resulting resistance increase. In contrast, these effects are quite small for Rh, indicating its potential as a high-conductance metal at the nanoscale. More generally, the measured resistivity increase during air exposure of 10-nm-thick epitaxial layers is quite small (< 5%) for electronegative metals including Ru, Rh, Ir, W, and Mo, but increases to 11-35% for more electropositive metals including Cu, Ag, Co, Ni, and Nb. This correlation suggests that the higher charge transfer at electropositive metals and the related surface potential perturbation causes diffuse electron scattering. Conversely, electronegative metals facilitate specular electron surface scattering and therefore mitigate the resistivity size effect which limits downscaling of interconnect lines in integrated circuits.

PHASE CHANGE MEMORY WITH MULTILEVEL RESISTANCE STATES BASED ON GROUP III-Sb BINARY ALLOYS

<u>Rubab Ume¹</u>, Haibo Gong¹, Vadim Tokranov¹, Michael Yakimov¹, Devendra Sadana¹, Kevin Brew², Guy Cohen³, Christian Lavoie³, Sandra Schujman¹, Karsten Beckmann¹, Nathaniel Cady¹, and Serge Oktyabrsky¹

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The potential of tellurium free AI-Sb and Ga-Sb binary alloys thin films for application in multi-level phase change memory is presented here. The thin films were deposited under UHV conditions from molecular beams. The bulk films demonstrate controllable crystallization temperature between 160°C -280°C, higher than in widely used Ge₂Sb₂Te₅ (GST) alloy. The films with 30% group III fraction demonstrate data retention of up to 150-160 °C projected to 10 years. Annealing of bilayer stack showed two crystallization temperatures indicating that the alloys with different composition can be assembled into multilayer structures for multilevel memory operation. X-ray diffraction (XRD) analysis shows crystallization into rhombohedral doped Sb (A7) and cubic zincblende AISb or GaSb phases with almost linear dependence of phase volume ratio upon increasing composition. Furthermore the recrystallization of the two phases after melting from in-situ synchrotron XRD analysis indicates that the AISb cubic phase crystalizes at a much higher temperature as compared to the A7 phase. This implies that the resistance switching of AI-Sb alloy is mostly due to amorphization/crystallization of the A7 phase and the cubic phase stays crystalline throughout cycling. Measurements of mushroom cells with ~100nm TiN heater demonstrated reversible switching with comparable resistance drift to GST. The reset/set resistance ratio of ~300 for Ga-Sb and ~4000 for Al-Sb alloy has been demonstrated with low drift power coefficient ranging from 0.003 (set) to 0.015 (Reset). The controllable high crystallization temperature, study of phase structure and preliminary switching of mushroom cells reveal a path toward developing multilevel PCM devices.

POLYMERIZATION UNDER SPATIAL CONFINEMENT

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Developing a mechanistic understanding of surface reactions is crucial to increasing control over surface modification processes. Graphene layers have been shown to effectively confine water, alcohols, and other small molecules on various substrates. resulting in two-dimensional confinement at the surface. This process can be extended into understanding surface polymerization by investigating morphological changes in chemical reactions of covalent organic framework precursors under graphene confinement. Here, the condensation reaction of 1,4-benzenediboronic acid (BDBA) to form the boroxine-linked COF-1 is used to develop a preliminary understanding of selfassembly, molecular diffusion, and condensation reaction kinetics under a graphene layer. A solution of BDBA in THF was drop-cast on mica substrates, followed by the deposition of graphene layers on the sample via mechanical exfoliation. AFM images of the resulting molecular assembly before and after annealing steps are presented, depicting BDBA assembly both with and without graphene confinement. AFM phase images show clear morphological changes for graphene-confined domains that are not observed in the molecules outside of those graphene flakes. Further study of the system will aid in the development of a crystalline growth model for confined system reactions.

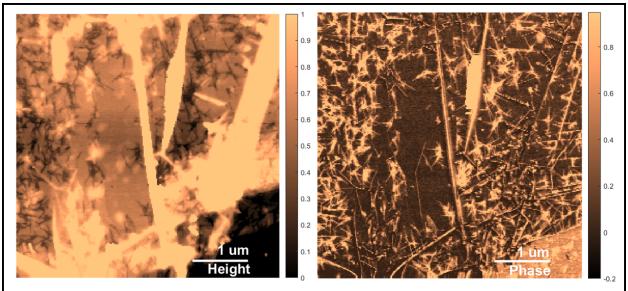


Figure 1: Height and phase AFM images of crystalline BDBA domains under a graphene flake following 2 hours of annealing at 120 °C. The sharp domain boundaries separating the crystallites beneath the graphene layer are highlighted by a positive phase contrast.

POROUS ANODE STRUCTURES FOR LITHIUM-ION BATTERIES

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Lithium-ion batteries (LIBs) have been dominating the development of electronics shaping our daily life owing to their good specific and volumetric energy densities. However, the technological advancement calls for higher energy, safer, lower cost, and large scale manufacturing batteries. Different materials have been investigated as anode to increase the energy density of LIBs. Lithium metal is found to be an excellent candidate for anode material. This is due to: (1) it has high theoretical capacity of 3860 mAh/g, (2) it has low potential of -3.04V vs SHE, and (3) it has low density of 0.53 gm/cc. However, using lithium anodes raises many challenges; (1) lithium dendrite formations, (2) significant volumetric changes, and (3) safety hazards due to reactivity with organic liquid electrolytes (OLE).

Previous studies showed that lithium dendrite formation could be mitigated by using porous substrate for lithium deposition. Porous anode structures provide space where lithium can be deposited and confined away from the electrolyte thus reducing the dendrite formation and short-circuiting hazards. This concept has also been proven in lithium sulfur battery systems, where the microstructure porous layers serve as electrode supports and increase the mechanical strength by ~9×, making the cells strong enough to handle with ease. The porous structure mitigated the polysulfide shuttle effect increasing the cyclability. Moreover, solid state electrolytes have been investigated as a safer alternative to OLE. They provide high stiffness suppressing the dendrites growth. Moreover, they offer wider electrochemical stability window. Hence, they allow usage of pure lithium anodes that would increase the energy density of the battery.

This study focuses on lithium metal batteries where pure lithium is an anode. The electrochemical performance of lithium metal battery with different configurations is studied and compared. NMC Lithium ion battery showed capacity fading after 4 cycles indicating loss of material and aggravated side reactions. However Lithium cobalt oxide cells showed more cycling stability at different charging rates. Lithium anode surface is analyzed by different techniques; nuclear reaction analysis (NRA), SEM, and XRD. The concept of using porous nanofibers as substrate for lithium deposition coupled with solid state electrolytes is demonstrated.

PLASMA-INDUCED ROUGHNESS AND CHEMICAL MODIFICATIONS OF TIN BOTTOM ELECTRODE AND THEIR IMPACT ON HfO₂-MIM PROPERTIES

Sophia Rogalskyj^{1,2}, Hunter Frost^{1,2}, Amber Palka¹ Nicholas Joy¹, Dina Triyoso¹, Robert Clark¹, Cory Wajda¹, Gert Leusink¹, Angelique Raley¹, Kathleen Dunn²

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Metal-insulator-metal (MIM) stacks, though simple in design, are the backbone device for resistive random access memories (ReRAM) and, as such, play a vital role in emerging memory technologies. In this work, we characterize the impact of Ar, Ar/N₂, and BCl₃/Cl₂ plasma processes on the physical properties of the TiN bottom electrode (BE) surface. The BCl₃/Cl₂ process increases roughness by 40%. Ar and Ar/N₂ processes both decrease PVD TiN roughness by 50%. Surface morphology shown by AFM and XSEM in Fig. 2. This reduction propagates through the entire MIM stack. X-ray photoelectron spectroscopy (XPS) indicates that both plasma processes alter the Ti-ON concentration on the TiN surface Example shown in Fig. 1. The impact of BE surface roughness and composition on MIM electrical properties is currently under investigation. (Graduate Student)

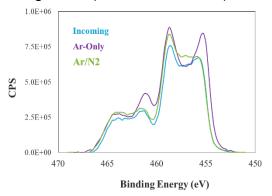


Figure 1: High resolution Ti2p spectra of wafers with an (blue) as-deposited, (purple) Ar-plasma processed, and (green) Ar/N_2 -

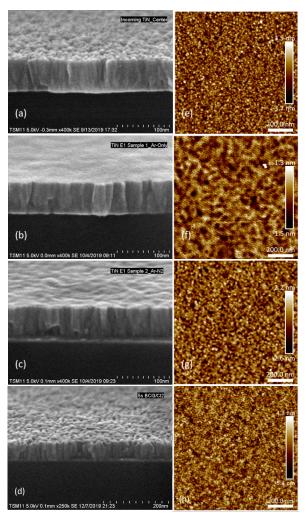


Figure 2: Tilted cross-sectional SEM images of 60nm ALD Ti/TiN film on coupon (a) as deposited, (b) after 30s Ar-only, (c) 120s Ar/N₂, and (d) 5s $BCl_3/Cl_2.1 \ \mu m \ x \ 1 \ \mu m \ AFM$ scan of 60nm ALD Ti/TiN film (e) as deposited, (f) after 30s Ar-only, (g) 120s Ar/N₂, and (h) 5 s BCl_3/Cl_2 plasma processes.

CONSTANT ELECTRIC FIELD STUDY OF TIME DEPENDENT DIELECTRIC BREAKDOWN IN A LOW-K INTERLEVEL DIELECTRIC

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Time dependent dielectric breakdown (TDDB) is when a dielectric suddenly breaks down after being exposed to an electric field below its breakdown field for extended periods of time. This breakdown is caused by a buildup of defects that cause the dielectric to change from an insulating material to a conducting one. TDDB is an important reliability risk and it is important to understand the mechanisms behind TDDB in a low-k dielectric to as the semiconductor industry moves towards smaller structures ensure product lifetimes.

Over the past several years, many TDDB models with different breakdown mechanisms have been proposed. Currently the two best fitting models are the lucky electron model (LEM) and a power law model. In the LEM the dielectric is damaged by a collision between an electron being accelerated by the field and an atom in the dielectric. If the electron hits a hydrogen atom in the low k dielectric it could knock it out, creating a defect. The failure time is related the current density and the probability of the electron causing damage. The LEM is the only model to have an energy and field dependance

To test if the LEM is the most accurate model an experiment was performed with serpentine-comb capacitor structures with varying dielectric thicknesses. The different thicknesses cause the field to remain constant while varying the voltage. If the failure is solely dependent on the applied electric field then the failure times at all voltages should be equivalent. If the failure is dependent on a momentum transfer as the lucky electron model suggests the lower voltages on the smaller dielectric spacings would have an increased failure time.

The test structures were metal-insulator-metal serpentine comb capacitors with different dielectric width between the combs. It was assumed that the failure would occur at the narrowest inter-comb spacing in the structure. To find the minimum spacing the dielectric between the combs was measured by TEM images. The spacings were observed to have a bimodal distribution of two normal distributions with no overlap. The minimum spacing was calculated by taking the ratio of the area of observed spacing to the entire structure for the smaller of the bimodal distributions.

When the devices were tested at a constant field calculated by the minimum spacing most tests showed an increase in lifetime with a decrease in voltage as predicted by the LEM. Some tests at high fields and at fields calculated from the median spacing instead of the minimum had ambiguous results. The major finding was that there is energy dependence in TDDB.

SILICON CARBON ANODES OF LI-ION BATTERIES

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Li-ion batteries (LIBs) have been successfully adopted as a power source for wide range of applications including portable electronics, electric/hybrid electric vehicles (EVs/HEVs), and in large systems for grid storage applications. LIBs offer high energy density, high voltage, and high cycling efficiency. Graphite is currently the most popular choice for commercial LiB-anodes due to its low manufacturing cost and high stability over many charge-discharge cycles. However, graphite anode is quite limited in its applications due to its low specific energy (372 mAh/g) making it unviable for applications that require a high-energy capacity within a small volume. Silicon has been studied as an alternative anode material due to its significantly high specific energy (4200 mAh/g) but is held back by its high volumetric expansion (~ +300% fully lithiated) due to the internal entropy-increasing intercalation and de-intercalation of lithium ions. This high volumetric expansion causes batteries to break down and fail.

In this work, we report the effect on cell life cycling of batteries due to the modification of graphite anodes with silicon. Thin films of amorphous and microcrystalline columnar growth silicon were deposited via plasma enhanced chemical vapor deposition (PECVD). The batteries with the microcrystalline columnar silicon structure showed minor improvements of 1-2% to cycling stability over time vs their Graphite-Only counterparts. The amorphous-silicon composite anodes, however, showed a decrease in cycling efficiency for all deposition heights. Postmortem nuclear reaction analysis was performed on cell anodes to study the Li ion depth profile trapped in the solid electrolyte interface and silicon film to better explain battery degradation.

NEAR INFRARED SINGLE PHOTON EMISSION MICROSCOPY

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Single-photon emitters (SPEs) associated with point-defects and ions in semiconductors are currently considered a significant resource for the solid-state implementation of photonic quantum information processing, quantum imaging, and guantum key distribution technologies. Specifically, non-classical single-photon light sources emitting in the near-infrared region of the electromagnetic spectrum around 1.5 µm, falling in the lowest loss wavelength range of fiber optics networks, are critical chipscale building components for the development of fiber-based quantum networks. The realization of scalable on-chip quantum devices, such as single-photon sources and guantum memories, requires novel nanostructured materials that must be compatible and can be integrated with existing electronic circuits, waveguide architectures, and current chip-scale and silicon process technology. The precise placement of rare-earth ions into silicon-based wide-bandgap nanostructured materials with high integration functionality, such as silicon carbide (SiC) nanowires (NWs), can serve as critical building-blocks towards the implementation of such quantum devices. To this end, we introduce a modular interface to our micro-PL spectroscopy setup to enable automated and highly repeatable measurements, where the same positions along the NW sample can be probed across multiple trials. Benchmarking and optimization of the novel micro-PL module in conjunction with our state-of-the-art near-infrared single photon detector system were performed to enable the study of single-photon behavior from these Er:SiC NW nanophotonic structures at telecom wavelengths. This upgrade benefits current and future PL spectroscopic measurements for our samples.

ANALYSIS OF TEMPERATURE EFFECTS ON Li_{1+x}Al_xTi_{2-x}(PO₄)₃ THIN FILMS FOR ALL SOLID-STATE LI-ION BATTERY APPLICATIONS

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A promising alternative to traditional Li-ion batteries for next-generation energy storage devices would be all-solid-state batteries (ASSBs) with solid electrolytes (SEs). This is due to their decrease in cost and increase in safety, reliability, and energy density.

A NASICON-type (sodium superionic conductor) oxide $Li_{1+x}Al_xTi_{2-x}(PO_4)_3$, (LATP) is a favorable SE with its high ionic conductivity (~10⁻³ S/cm) and good electrochemical stability. However, most fabrication processes are done by chemical methods, such as sol-gel, which is a hindrance for large scale manufacturing. In this study, we focused on developing a growth process for polycrystalline LATP solid electrolyte thin films for Li-ion battery systems via plasma vapor deposition (PVD) technique. A 99.9% pure single LATP target was used for the RF-magnetron sputtering. Annealing parameters during deposition and post-deposition were changed to determine which method would grow polycrystalline films with high ionic conductivity.

Scanning electron microscopy (SEM) depicted a uniform thin film and provided thickness measurements. Secondary ion mass spectroscopy (SIMS) was coupled with X-ray photoelectron spectroscopy (XPS) to receive compositional measurements and determine depth profiles for each element including Li. It was found that annealing the films at higher temperatures produced polycrystalline films with more uniform lithium distribution in bulk of the films.

FEASIBILITY OF ROOM TEMPERATURE ION IMPLANTATION IN SIC PROCESSING

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Ion Implantation involves the ion bombardment of the substrate in order to introduce impurities into the substrate. These impurities are then activated in order to contribute carriers (holes or electrons) for the operation of the devices. We can use the P/N junction to form the junction to support voltage. Ion implantations in SiC are generally conducted at a high temperature (HT). There are concerns associated with this process because 1) it is more complicated than room temperature (RT) implant process, and 2) SiC surface can be etched and damaged during the etch of blocking oxide. This poster reviews the different techniques and characterizations of ion implantation of SiC JBS diodes at different temperatures.