Steven J. Randolph, Ph.D.

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Education

2005; Ph.D. *Materials Science and Engineering*; University of Tennessee, Knoxville Dissertation: "Experimental, Theoretical, and Device Application Development of Nanoscale Focused Electron-Beam-Induced Deposition"

2004; M.S. *Materials Science and Engineering;* University of Tennessee, Knoxville Thesis: "Nanoscale Materials Processing: Electron-Beam-Induced Etching of Silicon and Silicon Dioxide"

2002; B.S. *Chemistry*; University of Tennessee, Knoxville; Focus: Physical and Biochemistry Undergraduate Research: Electron spin resonance spectroscopy (ESR) for structural analysis of free radicals

Professional Experience

2021-Current: Group Leader, Nanofabrication Research Laboratory, ORNL

Summary: Management of personnel, budget controls, and organization of team of scientists and engineers for our user program within the Center for Nanophase Materials Sciences. Collaborate with high impact visiting scientists to execute their projects involving all aspects of focused ion beam nanofabrication using helium, neon, gallium, silicon, and gold ion sources. Recruitment of new academic and industrial scientists to fulfill our DOE mission of advancing science by providing community access to our user facilities and expertise. Development of research program/emphasis on focused beam processing of nanomaterials through ORNL's Strategic Hire program and associated project management activities ensuring execution of this research plan. Mentorship of interns, graduate students, and post-doctoral researchers; serving as advisor on thesis committees for graduate students.

Group Management

- Interview and hiring of a new research staff member to further the group's focus on beam-induced direct write processes.
- Responsible for advocating for promotions, awards nominations, and Laboratory recognition for direct reports.
- Responsible for performance management, goal setting, and strategizing for staff members' career trajectory desires.
- Leadership of staff in resource planning to facilitate user science and ensure our visiting scientists can execute complex tasks in a limited time and come away with quality data and a positive impression of the user program.
- Provide a supportive and positive environment that encourages openness and collaboration among our staff scientists to drive internal theme science.
- Maintain control and understanding of a dynamic budget situation that mandates we not only maintain our facility, but slowly recapitalize equipment and expand capability annually.

Scientific Responsibilities

- Develop and promote the group's scientific emphasis on direct-write nanomaterials processing through inter- and intragroup collaborative science.
- Execute a two-year plan for fundamental research in the use of electron, ion, and photon mediated manipulation of 2D and quantum materials.
- Build collaborative relationships with closely aligned groups at ORNL to diversify our expertise and broaden our impact on the nanomaterials field.
- Provide a three-year strategic plan for fundamental science using direct-write expertise to support our internal directed nanoscale transformations scientific theme.
- Mentoring graduate students and interns in science and co-authoring publications with them to facilitate their career advancement.
- Involvement in the scientific community through society memberships, meeting attendance, presenting our science, organizing symposia.
- Utilize the community network of scientists and engineers to help with career placements for post-docs and students wherever possible.
- Encourage and motivate our staff to contribute to internal theme science by engaging them in my own scientific interests and finding commonalities.

2007-2021: Staff Scientist; ThermoFisher Scientific, Advanced Technology

Summary: Progressed from Scientist II, Senior Scientist, to Staff Scientist developing IP and publishing on business-relevant topics including femtosecond laser, electron, and ion induced beam chemistry for fabrication. Developed 3D light and electron correlative workflows for biological microscopy. Developed chemistries responsible for fabrication of high conductivity nanostructures as well as differential etch chemistries. Developed in-situ microfluidics platform for wet chemistry in environmental SEM. Developed expertise in a wide array of electron and ion microscopy and fabrication techniques targeted to semiconductor, life science, and materials science application space. Managed early adopter capital equipment development projects and supported all aspects from safety and regulatory compliance to end-user application and training. Ownership and responsibility for multiple prototype microscopes and a development laboratory. Member of environmental health and safety committee. Leading and mentoring numerous recent graduates of University of Oregon's Semiconductor Processing and Optics Masters internship program.

Femtosecond Laser FIB/SEM Development

- Developed novel workflow using serial cryogenic fs-laser tomography to study intact lithium metal coin cells.
- Managed project developing and integrating a femtosecond laser onto a plasma FIB / SEM system to allow high rate, low damage materials characterization.
- Extensive experience optimizing laser conditions and optics for improving cut quality for a given application
- Assisted marketing and sales to demonstrate the technology through customer facing demos and scientific publication / presentations.
- Transferred knowledge to production and service to all commercialization and support of the newly developed Helios 5 Laser PFIB.
- Continuing application support for our early adopter customers to ensure they get the most out of their investment.
- Gained valuable experience in safety and regulatory compliance to integrate and develop the first class-IV laser product in our division.

• Modification and characterization of cryogenically cooled samples to enable energy storage materials, such as lithium metal batteries, to be analyzed from the millimeter to the nanoscale domain.

Biological Specimen Preparation for Correlative Microscopy

- Super-resolution imaging of sub-diffraction limit biological structures.
- Correlative light & electron microscopy workflow development.
- Biological sample preparation for light, electron, and ion imaging.
- Developed imaging conditions for HER2 labeled SKBR3 cells for 3D analysis of filopodial protein organization
- Developed intellectual property on genetically expressible, EM contrast agents.
- Developed intellectual property on multiphoton- and electron-induced metal labeling of *C. elegans*
- Investigated cryo-sectioning of *C. elegans* with non-gallium focused ion beam including study of novel contrast mechanisms in cryosectioned and resin embedded samples.
- Designed, organized, and converted a physics lab space to accommodate wet chemistry, simple cell culture, fixation, labeling, embedding, ultramicrotomy, and fluorescence imaging.

Light Optics for Imaging and Sample Modification

- Developed expertise in super resolution imaging, specifically dSTORM to produce 3D reconstruction of immunolabeled structures.
- Investigated optimization of redox buffer chemistries for improved super resolution imaging.
- Developed synthetic biological system for rapid assessment of super resolution imaging conditions.
- Investigated use of super resolution microscopy with resin-embedded biological specimens.
- Femtosecond laser based serial sectioning tool and workflow development
- Multiphoton-induced surface chemistry using ultrashort pulsed laser induced deposition.
- Investigated multiphoton-driven biological ultrastructure heavy metal labeling for image contrast enhancement.

Technical Program Management

- Facilitated and fostered the collaborative efforts of Project 2 of the OHSU/FEI Living Lab joint work on 3D super resolution CLEM
- Technical lead for full workflow development enabling correlation between 3D super resolution image and TEM tomography of immunolabeled U2OS microtubule structures.
- Project manager and researcher on development of early adopter femtosecond laser equipped DualBeam for large scale tomography applications

Beam-Induced Surface Chemistry

- Beam chemistries for biofunctionalization of nanostructures with proteins, etc. via amine termination of surfaces and amide bond formation.
- Self-assembly of nanostructures during ion bombardment of surfaces
- Catalytic deposition through beam-induced surface functionalization.
- Identification of novel precursors and/or processes for electron- and ion-beam-induced CVD and gas-assisted etching.
- Discovery, development, and implementation on novel low resistivity ion-beam-induced metal CVD (multiple patents to be filed).
- Discovery, development, and implementation of gas-assisted electron-beam-induced etch chemistries using gas mixtures to optimize material selectivity.

- XeF₂ as an oxidizing agent combined with a variety of fluorocarbon species results in differential etching of silicon/silicon dioxide/silicon nitride.
- Investigation of post-processing and *in-situ* purification techniques for improving electrical and compositional properties of ion and electron beam CVD materials
- Novel beam chemistry applications with non-standard ion sources (non-gallium ion, optical).
- Sub-diffraction-limit pattern transfer using material-dependent laser ablation threshold.
- In-situ fluid manipulation in environmental SEM (ESEM).
- Deterministic and non-deterministic room-temperature, spontaneous metal CVD.
- Member of Environmental, Health, and Safety Committee.

2005-2007: Post-doctoral researcher; Oak Ridge National Laboratory

- Responsible for maintenance and upkeep complex semiconductor equipment including stepper, contact aligner, RIE/ICP dry etch tool, PECVD, e-beam evaporator, profilometer, reflectometer, resist spinners, d.c. sputter coater, LPCVD, CMP.
- Training users and facilitation of user projects involving the application of the above tools.
- General cleanroom maintenance, organization, establishment of safety procedures.
- Investigated conventional microfabrication processes as well as novel fabrication processes for creating field emission cathodes.
- Electron-beam-induced tungsten nanopillar deposition in prefabricated diode and triode wells for testing field emission properties.
- Continuation of earlier established, self-aligned carbon nanofiber field emission device fabrication.
- Microfabrication process optimization for RIE/ICP dry etching, PECVD, PVD (sputter), optical stepper and contact lithography, wet etch, e-beam lithography.
- Investigated stress-induced and/or thermally induced preferred orientation of nickel films and subsequent effects on dewetting properties following catalytic nanoparticle formation.
- Evaluated parameter space of pressure, power, d.c. substrate bias, temperature of r.f. nickel sputter deposition process.
- Characterization of films as deposited and post-dewetted by x-ray diffraction and electron microscopy.
- Dewetted nanoparticles used as catalysts for carbon nanofiber growth by PECVD.

2002-2005: Research Assistant, The University of Tennessee, Knoxville

- Investigated various aspects of electron-beam-induced processing.
- Studied effects of scan parameters and beam conditions on the effects of e-beaminduced etching of silicon and silicon dioxide. Proposed possible mechanism.
- Studied and reported on material properties of tungsten as-deposited from e-beaminduced deposition from WF₆ via AES, TEM, SEM, STEM, EDS.
- Finite element thermal modeling of beam-induced heating effects during deposition of ebeam-induced dielectric nanopillars. Correlated to experiment.
- Implemented Monte-Carlo electron-scattering model to determine energy distribution profiles during electron-beam-induced deposition.

- Application of electron-beam-induced deposition for high-resolution single- and bi-layer e-beam lithographic patterning with hard masks.
- Application of electron-beam-induced deposition for fabrication of nanoscale field emitters.
- Microfabrication process development for microscale vacuum encapsulation devices.
- Process development for device fabrication from CAD layout to final device testing.
- Gained experience with wide array of standard microfabrication techniques such as optical lithography, PECVD, evaporation and sputtering (PVD), dry etching, wet etching, resist processing, thin film characterization and analysis.
- Helped design and build in-house d.c. sputter tool to facilitated vacuum encapsulation project.
- Completed vacuum encapsulation and successfully tested a vacuum-sealed microchamber (encapsulation).

Research Interests

- Electron microscopy of in situ laser material interactions
- Fabrication, materials modification, and chemistry utilizing reactive ion beams.
- Atypical uses of electron microscopy, such as beam-induced chemistry for materials synthesis.
- Beam-induced reactions: electron, ion, photon / gas, liquid, solid, adsorbed phases
- Beam chemistry for biological contrast enhancement in electron microscopy
- Cryogenic microscopy techniques in lithium metal battery research.
- Direct write beam chemistry and fabrication techniques in sample preparation for cryo TEM tomography and single particle analysis
- Nanofabrication and materials analysis utilizing ultrashort pulse laser technology.
- Super resolution fluorescence microscopy and correlative EM imaging of cellular ultrastructure.
- Ultrashort (femtosecond) pulsed laser materials processing and serial sectioning.
- Biofunctionalization of surfaces and nanostructures via novel surface chemistry for molecular attachment and immobilization
- Environmental SEM as a tool for imaging and manipulating samples that are non-equilibrium in high vacuum. Imaging of liquid stabilization and phase transformations phenomena *insitu*.
- Structural characterization of materials via electron and x-ray diffraction.
- Mathematical modeling of complex physical systems
- Materials synthesis and structural, morphological, compositional characterization
- General nanofabrication of multilevel structures, such as MEMS and microfluidic devices

Relevant Skill Set

Microscopy

- Electron, Ion, and Scanning Probe Microscopies: SEM, FIB, TEM, STEM, ESEM, AFM, Helium Ion Microscopy
- Light Microscopies: Widefield fluorescence, Confocal fluorescence, Super-resolution PALM, dSTORM, 3D Super-resolution interferometric PALM (iPALM)

Sample Preparation

• Immunolabeling for fluorescence microscopies

- Plastic resin embedding of biological samples (U2OS, SKBR3 cancer cell lines; yeast) for correlative light and electron microscopies
- Preservation of fluorescence in resin embedded, immunolabeled biological systems.
- Ultramicrotomy for biological and materials TEM analysis
- DualBeam-based TEM lamellae preparation of biological, semiconductor, and materials samples.
- Micro-Computed-Tomography (µCT) sample preparation via ultrashort (femtosecond) pulsed laser ablation.

Characterization and Analysis

• EDS (Energy Dispersive X-ray Spectroscopy), X-ray Diffraction (XRD), Electron Backscatter Diffraction (EBSD), Mass Spectrometry (MS), Residual Gas Analysis (RGA), Electron diffraction, Profilometry, ESR and NMR resonance spectroscopy, electrical property determination via 2- and 4-point probing.

Wet Laboratory Skills

- Quantitative chemical analysis such as titrations, dilutions, solution preparation, neutralizations, electrochemistry, basic cell culture, labeling, fixation
- Lab and facility design and management; Environmental Health and Safety compliance
- Hazardous material/chemical handling and waste management

Microfabrication and Sample Modification

- Charged particle induced surface chemistry
- Ultrashort pulsed laser material processing (high-rate material removal; deposition)
- PECVD, thermal CVD (LPCVD), PVD (evaporation, r.f. and d.c. sputter, reactive sputter)
- RIE and ICP dry etching, wet etching, CMP,
- Electrochemistry (electrodeposition, anodization, etc.)
- Lithography (optical, e-beam, novel techniques)
- Microfluidics device fabrication

Computer simulation, programming, and CAD

- LabView
- SIMION charged particle optics simulation
- Finite element methods for partial differential equation numerical solution
- Finite difference differential equation numerical solution
- MATLAB programming environment, Mathematica
- Monte-Carlo modeling methods
- L-Edit, Layout Editor, AutoCAD
- COMSOL Finite Element Physics Simulations

Funding and Grants

- Principle Investigator: Next-Generation Microelectronics for Advanced Sensing and Computing.Laboratory. Directed Research and Development Program 2023-2024.
- Co-Investigator: In situ laser-material interactions for additive manufacturing laser powderbed fusion. Directed Research and Development Program 2023-2024.
- Principle Investigator: Electron, Ion, and Photon Mediated Modification of 2D and Quantum Emitter Materials for Precision Manufacturing, Strategic Hire Grant for Oak Ridge National Laboratory's Laboratory Directed Research and Development Program 2021-2023.
- Co-author/investigator: *Real-Time Electron Beam Studies of Dynamic Processes In Nano Scale Materials Physics.* 4 year Joint proposal between FEI and University of Technology at Sydney funded by the Australian Research Council, LP11020090.

Scientific Community Involvement

- Co-organizer: NNCI Nano + Additive Manufacturing Summit, Louisville, KY (2024).
- Steering Board: Focused Electron Beam Induced Processing (FEBIP) Workshop.
- Co-organizer: NNCI Nano + Additive Manufacturing Summit, Louisville, KY (2023).
- Co-organizer: NNCI Nano + Additive Manufacturing Summit, Louisville, KY (2022).
- Symposium Organizer and Chair: *Ultrashort Pulse Lasers: Microscopy, Simulations, and Material Interactions.*, Microscopy and Microanalysis, Portland, OR (2022).
- Member: Microscopy Society of America.
- Member: Microanalysis Society

Patents and Inventions

- Authored or co-authored approximately 50 novel invention disclosures in 15+ years
- Patents and Applications:
 - De Marco, Alex, Sergey Gorelick, Chad Rue, Joseph Christian, Kenny Mani, Steven Randolph, and Matthias Langhorst. "Method, device and system for the treatment of biological cryogenic samples by plasma focused ion beams." U.S. Patent 11,735,404, issued August 22, 2023.
 - Liu, Kun, and Steven J. Randolph. "Charged particle beam source." U.S. Patent Application 17/836,611, filed January 12, 2023.
 - Gancarcik, Tomas, Ivan Dekan, David Krobot, Tomas Trnkocy, Steven J. Randolph, and Remco Geurts. "In-situ laser redeposition reduction by a controlled gas flow and a system for reducing contamination." U.S. Patent Application 17/211,657, filed September 29, 2022.
 - Randolph, S., Miyasaki, J., Straw, M. Fiducial-based correlative microscopy, US Patent 9368321, (2016).
 - Botman, A.P.J.M, Toth, M., Randolph, S., Narum, D., Localized, in-vacuum modification of small structures, US Patent 9255339 B2, (2016).
 - Chandler, C.D., Randolph, S., Hartigan, G., Gas delivery for beam processing systems, US Patent 9,150,961, (2015).
 - Botman, A.P.J.M., Randolph, S., Utlaut, M.W., *Microfluidics delivery systems*, US Patent 9,044,781, (2015).
 - Randolph, S., Chandler, C.D., High Selectivity, Low Damage Electron-Beam Delineation Etch, U.S. Patent 8,778,804, (2014).
 - Botman, A.P.J.M., Randolph, S., Toth, M., *Method of Depositing Material*, US Patent 8,853,078, (2014).
 - Straw, M., Toth, M., Randolph, S., Lysaght, M., Utlaut, M., Charged Particle Beam Masking for Laser Ablation Micromachining, U.S. Patent 8,629,416, (2014).
 - Toth, M., Lobo, C., Randolph, S., Chandler, C., Method of Using Nitrogen Based Compounds to Reduce Contamination in Beam-Induced Thin Film Deposition, US Patent 8,617,668, (2013).
 - Randolph, S., Chandler, C.D., Beam-Induced Deposition of Low-Resistivity Material, US Patent 20,120,308,740, (2012).

Invited Review Articles and Presentations

- Randolph, SJ *et al.*, Energetic, focused, beam-induced surface chemistry: A nanofabrication perspective. 153rd TMS Annual Meeting and Exhibition, Orlando, Florida 2024.
- 2. Randolph, SJ, Multiple Ion Plasma: Emerging opportunities in FIB Materials Processing, Plasma FIB Workshop, University of Tennessee, Knoxville, TN 10/6/2022.
- 3. *NRL capabilities:Helium, Neon, Gallium ion beams, Electron and pulsed laser beam chemistry,* Center for Nanophase Materials Sciences User Workshop (virtual 2022).
- 4. Randolph, SJ, Selective Photothermal Functionalization of 3D Nanostructures, NNCI Nano + Additive Manufacturing Summit, Louisville, KY 2022.
- 5. Randolph, SJ, *Helios Laser PFIB: fs-Laser Enabling New Applications,* Focused Ion Beam User Group, EDFAS 2021 (Remote Conference).
- 6. Randolph SJ, Fowlkes JD, Rack PD. *Focused, nanoscale electron-beam-induced deposition and etching.* Critical Reviews in Solid State and Materials Sciences. 2006;31(3):55-89.
- 7. Botman, A., Randolph, S. J., Bahm, A., Straw, M. & Toth, M. Spontaneous nanostructure assembly with new focused charged particle beam-induced processes. in *Abstracts of Papers of The American Chemical Society* **247**, (2014).
- 8. Randolph SJ, Toth M, Chandler CD, *Present and future challenges in electron-beaminduced processing*, User Meeting of the Center for Nanophase Materials Sciences, Oak Ridge National Laboratory, Oak Ridge, TN, September 2008.

Peer-Reviewed Journal Articles

- 1. Lasseter, J. et al. Selected Area Manipulation of MoS2 via Focused Electron Beam-Induced Etching for Nanoscale Device Editing. ACS Appl. Mater. Interfaces (2024) doi:10.1021/acsami.3c17182.
- 2. Flynn-Hepford, M. et al. Direct Visualization of Charge Migration in Bilayer Tantalum Oxide Films by Multimodal Imaging. Advanced Electronic Materials 10, 2300589 (2024).
- 3. LC Merrill, RN Gannon, KL Jungjohann, SJ Randolph, et al. Evaluation of Lithium Metal Anode Volumetric Expansion through Laser Plasma Focused Ion Beam Cross-Sectional Imaging, Journal of The Electrochemical Society 170 (8) 2023, 080527.
- Olha Popova, Steven J. Randolph, Sabine M. Neumayer, Liangbo Liang, Benjamin Lawrie, Olga S. Ovchinnikova, Robert J. Bondi, Matthew J. Marinella, Bobby G. Sumpter, Petro Maksymovych; Nanoscale imaging of He-ion irradiation effects on amorphous toward electroforming-free neuromorphic functions. *Appl. Phys. Lett.* 9 October 2023; 123 (15): 153503. <u>https://doi.org/10.1063/5.0158380</u>
- 5. Wen, X., Pierce, J., Lavrik, N., Randolph, S. J., Guo, W., & Fitzsimmons, M. R. (2023). Flow of the normal component of He-II about bluff objects observed with He 2* excimers. *Physical Review B*, *107*(17), 174501.
- Lasseter, J.; Rack, P.D.; Randolph, S.J. Selected Area Deposition of High Purity Gold for Functional 3D Architectures. *Nanomaterials* 2023, 13, 757. https://doi.org/10.3390/nano13040757
- Lasseter, J., Rack, P.D., Randolph, S.J., Selected Area Deposition of PtC_x Nanostructures: Implications for Functional Coatings of 3D Nanoarchitectures, ACS Applied Nanomaterials, Article ASAP, (2022). DOI: 10.1021/acsanm.2c02182.

- 8. Kang, S. *et al.* Highly enhanced ferroelectricity in HfO₂-based ferroelectric thin film by light ion bombardment. *Science* **376**, 731–738 (2022).
- Jungjohann, K., Merrill, L., Gannon, R., Long, D., Randolph, S., & Harrison, K. (2022). Relevance of In-Situ Electrochemical STEM Observations to Li-Metal Batteries for Evaluating Performance. *Microscopy and Microanalysis*, 28(S1), 826-827.
- Echlin, M.P., Polonsky, A.T., Lamb, J., Geurts, R., Randolph, S.J., Botman, A., Pollock, T.M., Recent Developments in Femtosecond Laser-Enabled TriBeam Systems, *J. Minerals, Metals, & Materials Society (TMS)*, https://doi.org/10.1007/s11837-021-04919-0 (2021).
- Katharine L. Harrison, Laura C Merrill, Daniel Martin Long, Steven J. Randolph, Subrahmanyam Goriparti, Joseph Christian, Benjamin Warren, Scott A. Roberts, Stephen J. Harris, and Katherine L. Jungjohann, Cryogenic Electron Microscopy Reveals that Applied Pressure Promotes Short Circuits in Li Batteries, iScience, 2021, 103394, ISSN 2589-0042, https://doi.org/10.1016/j.isci.2021.103394.
- Jungjohann, K., Gannon, R., Goriparti, S., Randolph, S.J., Johnson, D., Zavadil, K., Harris, S., Harrison, K. Cryogenic Laser Ablation Reveals Short Circuit Mechanism in Lithium Metal Batteries, ACS Energy Lett. 2021, 6, 2138-2144.
- 13. Wang, J., Randolph, S., Wu, Q. *et al.* Reactive oxygen FIB spin milling enables correlative workflow for 3D super-resolution light microscopy and serial FIB/SEM of cultured cells. *Sci Rep* **11**, 13162 (2021).
- 14. Martin, A. A., et al. (2017). Radiation-Induced Damage and Recovery of Ultra-Nanocrystalline Diamond: Toward Applications in Harsh Environments, American Chemical Society. **9:** 39790-39794.
- 15. Kianinia, M. *et al.* Robust, directed assembly of fluorescent nanodiamonds. *Nanoscale* **8**, (2016).
- 16. Echlin, M. P., Straw, M., Randolph, S., Filevich, J. & Pollock, T. M. The TriBeam system: Femtosecond laser ablation in situ SEM. *Mater. Charact.* **100**, 1–12 (2015).
- 17. Martin, A. A., Randolph, S., Botman, A., Toth, M. & Aharonovich, I. Maskless milling of diamond by a focused oxygen ion beam. *Sci. Rep.* **5**, (2015).(Liang, Zhang et al. 2020)
- 18. Straw, M. & Randolph, S. Direct spatiotemporal analysis of femtosecond laser-induced plasma-mediated chemical reactions. *Laser Phys. Lett.* **11**, 035601 (2014).
- 19. Bresin, M., Botman, A., Randolph, S. J., Straw, M. & Hastings, J. T. Liquid Phase Electron-Beam-Induced Deposition on Bulk Substrates Using Environmental Scanning Electron Microscopy. *Microsc. Microanal.* **20**, 376–384 (2014).
- Randolph, S. J., Botman, A. & Toth, M. Deposition of Highly Porous Nanocrystalline Platinum on Functionalized Substrates Through Fluorine-Induced Decomposition of Pt(PF3)4 Adsorbates. *Part. Part. Syst. Charact.* **30**, 201300036 (2013).
- Botman, A., Bahm, A., Randolph, S., Straw, M. & Toth, M. Spontaneous Growth of Gallium-Filled Microcapillaries on Ion-Bombarded GaN. *Phys. Rev. Lett.* **111**, 135503 (2013).
- 22. Randolph, S. J. S., Botman, A. & Toth, M. Capsule-free fluid delivery and beam-induced electrodeposition in a scanning electron microscope. *RSC Adv.* **3**, 20016–20023 (2013).
- 23. Randolph, S., Toth, M., Cullen, J., Chandler, C. & Lobo, C. Kinetics of gas mediated electron beam induced etching. *Appl. Phys. Lett.* **99**, 213103 (2011).
- Young, R. *et al.* A comparison of xenon plasma FIB technology with conventional gallium LMIS FIB: imaging, milling, and gas-assisted applications. *Microsc. Microanal.* **17**, 652–653 (2011).
- 25. Botman, A. *et al.* Electron postgrowth irradiation of platinum-containing nanostructures grown by electron-beam-induced deposition from Pt (PF3) 4. *J. Vac. Sci. Technol. B* **27**, 2759–2763 (2009).

- 26. Li, J. *et al.* Electron postgrowth irradiation of platinum-containing nanostructures grown by electron-beam-induced deposition from Pt (PF3) 4. *J. Vac. Sci. Technol. B Microelectron. Nanom. Struct.* **27**, 2759–2763 (2009).
- 27. Klein, K. L. *et al.* Single crystal nanowires grown via electron-beam-induced deposition. *Nanotechnology* **19**, 345705 (2008).
- 28. Rahman, T. *et al.* Integration of a dose control circuit with a vertically aligned nanofiber field emission device. *J. Vac. Sci. Technol. B* **25**, (2007).
- 29. Rack, P. D., Fowlkes, J. D. & Randolph, S. J. In situ probing of the growth and morphology in electron-beam-induced deposited nanostructures. *Nanotechnology* **18**, 465602 (2007).
- 30. Randolph, S. J. *et al.* Controlling thin film structure for the dewetting of catalyst nanoparticle arrays for subsequent carbon nanofiber growth. *Nanotechnology* **18**, 465304 (2007).
- 31. Randolph, S. J., Fowlkes, J. D. & Rack, P. D. Focused, nanoscale electron-bearninduced deposition and etching. *Crit. Rev. Solid State Mater. Sci.* **31**, 55–89 (2006).
- Choi, Y. R., Rack, P. D., Randolph, S. J., Smith, D. A. & Joy, D. C. Pressure effect of growing with electron beam-induced deposition with tungsten hexafluoride and tetraethylorthosilicate precursor. *Scanning* 28, 311–318 (2006).
- 33. Yang, X. *et al.* Integrated tungsten nanofiber field emission cathodes selectively grown by nanoscale electron beam-induced deposition. *Appl. Phys. Lett.* **86**, 183106 (2005).
- 34. Randolph, S. J., Fowlkes, J. D. & Rack, P. D. Effects of heat generation during electronbeam-induced deposition of nanostructures. *J. Appl. Phys.* **97**, 124312 (2005).
- 35. Randolph, S. J., Fowlkes, J. D. & Rack, P. D. Focused electron-beam-induced etching of silicon dioxide. *J. Appl. Phys.* **98**, 34902 (2005).
- Fowlkes, J. D., Randolph, S. J. & Rack, P. D. Growth and Simulation of High Aspect Ratio Nanopillars by Primary and Secondary Electron – Induced Deposition. J. Vac. Sci. Technol. B 23, 2825–2832 (2005).
- Randolph, S. J., Hale, M. D., Guillorn, M. A., Rack, P. D. & Simpson, M. L. A microfabrication process for a vacuum-encapsulated microchamber. *Microelectron. Eng.* 77, 412–419 (2005).
- 38. Rack, P. D. *et al.* Nanoscale electron-beam-stimulated processing. *Appl. Phys. Lett.* **82**, 2326–2328 (2003).

Conferences: Contributed Talks and Posters

- * Indicates SJR as presenter
- 1. In Situ microscopy of laser melting processes in 316 stainless steel powders, NNCI Nano + Additive Manufacturing Summit (2023).*
- 2. Deterministic Activation of Resistive switching and Light Emission from TaOx with Nanoscale Resolution, AVS 2022.
- 3. *NRL capabilities:Helium, Neon, Gallium ion beams, Electron and pulsed laser beam chemistry,* Center for Nanophase Materials Sciences User Workshop (virtual 2022).*
- 4. Relevance of In-Situ Electrochemical STEM Observations to Li-Metal Batteries for Evaluating Performance. Microscopy and Microanalysis (2022).
- 5. Cryogenic EM Imaging of Li Me Metal Battery Aging and Failure Mechanisms, Sandia National Laboratory (2021).
- 6. Cryogenic Laser Plasma FIB Cross Sections of Coin Cell Batteries, Sandia National Laboratory (2021).

- 7. *Electrolyte Comparison for Li-Metal Anodes with Cryo-Laser PFIB.* Microscopy and Microanalysis (2021).
- 8. DualBeam Platform Applications in Lithium Battery Research, 239th Electrochemical Society (2021).
- 9. Identifying Intact Electrode Interfaces with Cryogenic Electron Microscopy, MRS (2021)
- 10. TriBeam Tomography for 3D Data Acquisition, Microscopy and Microanalysis (2020)
- 11. Cryo-EM of Li Metal Battery Aging and Failure Mechanisms, Microscopy and Microanalysis (2020).
- 12. Cross-platform Holder Kit for a Real 3D Correlative Tomography and Microscopy, Microscopy and Microanalysis (2020)
- 13. Femtosecond Laser-Enabled TriBeam as a Platform for Analysis of Thermally- and Charge-Sensitive Materials, Microscopy and Microanalysis (2019)*
- 14. Advances in Multi-beam and Multi-ion FIB-SEM for 3D Correlative Microscopy, Microscopy and Microanalysis (2019)
- 15. *Site-specific in-situ laser ablation of charge sensitive materials*, Microscopy and Microanalysis (2019)
- 16. Combined femtosecond laser and plasma DualBeam for in-situ failure and materials analysis, Failure Analysis and Material Diagnostics of Electronic Components (2018)
- 17. Evidence for surface-plasmon-mediated precursor dissociation in ultrashort-pulselaser-induced surface chemistry, American Chemical Society (2017)*
- 18. *Hydrogen-beam induced deposition of platinum and tungsten microstructures*. The 59th International Conference on Electron, Ion, and Photon Beam Technology and Nanofabrication (2015).
- 19. Spontaneous nanostructure assembly with new focused charged particle beaminduced processes. 247th ACS National Meeting (March), Dallas, TX, USA, 2014.
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