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***National Nanotechnology Coordinated  
Infrastructure (NNCI)***

***Research and Education Highlights***

***Year 7 (October 2021 – September 2022)***



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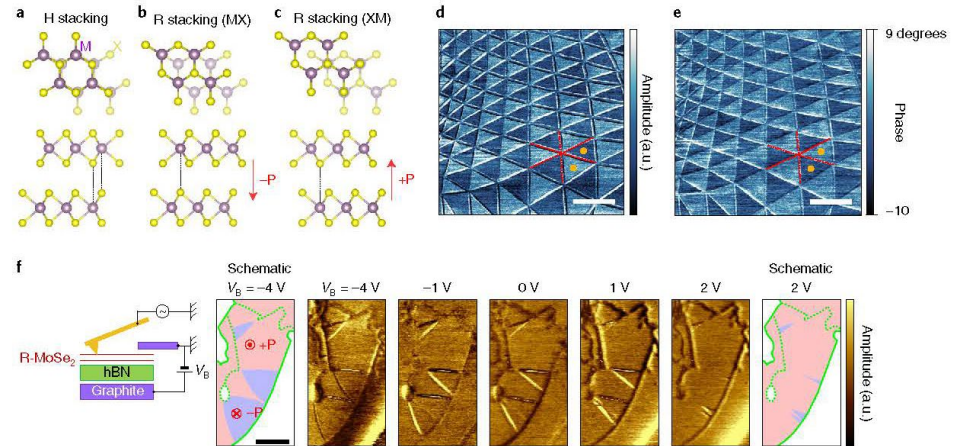
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# ***Center for Nanoscale Systems (CNS)***

# Interfacial ferroelectricity in rhombohedral stacked bilayer transition metal dichalcogenides

2D van der Waals materials have greatly expanded our design space of heterostructures by allowing individual layers to be stacked at non-equilibrium configurations, for example via control of the twist angle. Such heterostructures not only combine characteristics of the individual building blocks but can also exhibit physical properties absent in the parent compounds through interlayer interactions. Here reported is a new family of nanometre-thick, two-dimensional (2D) ferroelectric semiconductors, where the individual constituents are well-studied non-ferroelectric monolayer transition metal dichalcogenides (TMDs), namely WSe<sub>2</sub>, MoSe<sub>2</sub>, WS<sub>2</sub> and MoS<sub>2</sub>. By stacking two identical monolayer TMDs in parallel, they obtained electrically switchable rhombohedral-stacking configurations, with out-of-plane polarization that is flipped by in-plane sliding motion. Fabricating nearly parallel-stacked bilayers enables the visualization of moiré ferroelectric domains as well as electric field-induced domain wall motion with piezoelectric force microscopy. Furthermore, they were able to quantify the ferroelectric built-in interlayer potential, in good agreement with first-principles calculations. The new semiconducting ferroelectric properties of these four new TMDs opens up the possibility of studying the interplay between ferroelectricity and their rich electric and optical properties.



Xirui Wang, Kenji Yasuda, Yang Zhang, Song Liu<sup>2</sup> Kenji Watanabe, Takashi Taniguchi, James Hone, Liang Fu, and Pablo Jarillo-Herrero; Massachusetts Institute of Technology. This work was performed in part at the Center for Nanoscale Systems.

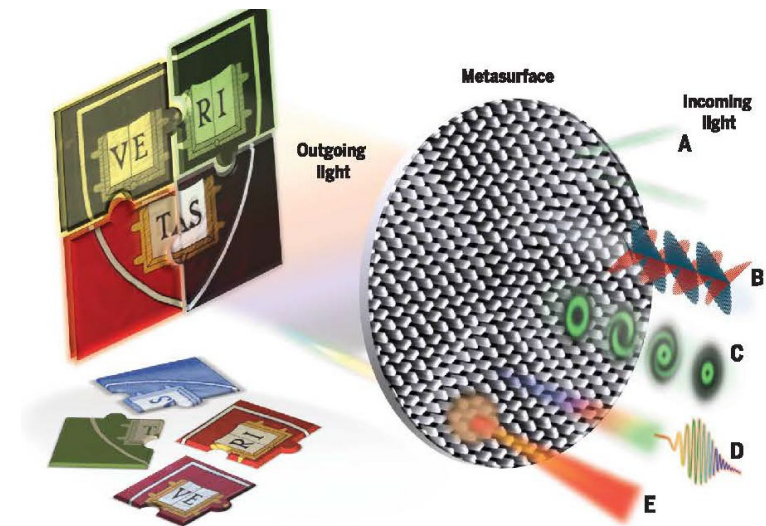
This work was supported by NSF NNCI Award ECCS-1541959. *Nature Nanotechnology* 17, 367–371, 2022.

National Research Priority: NSF–Quantum Leap



# Tunable structured light with flat optics

Flat optics has emerged as a key player in the area of structured light and its applications, owing to its subwavelength resolution, ease of integration, and compact footprint. Although its first generation has revolutionized conventional lenses and enabled anomalous refraction, new classes of meta-optics can now shape light and dark features of an optical field with an unprecedented level of complexity and multifunctionality. Here, we review these efforts with a focus on metasurfaces that use different properties of input light—angle of incidence and direction, polarization, phase distribution, wavelength, and nonlinear behavior—as optical knobs for tuning the output response. We discuss ongoing advances in this area as well as future challenges and prospects. These recent developments indicate that optically tunable flat optics is poised to advance adaptive camera systems, microscopes, holograms, and portable and wearable devices and may suggest new possibilities in optical communications and sensing.



**Tunable structured light with static meta-optics.** Different properties of input light may act as control knobs for tuning the optical response of the metasurface. These degrees of freedom include the angle of incidence (A), polarization state (B), orbital angular momentum (or spatial structure in general) (C), wavelength (D), and intensity level (manifested in a nonlinear interaction) (E). By changing one or more of these properties at the input of the metasurface, one can obtain a different light pattern at the output (depicted by the different puzzle pieces of Harvard University's logo). This tunability relies on an intricate light-matter interaction at the level of the meta-atom, which often cannot be replicated by other conventional wavefront shaping platforms.

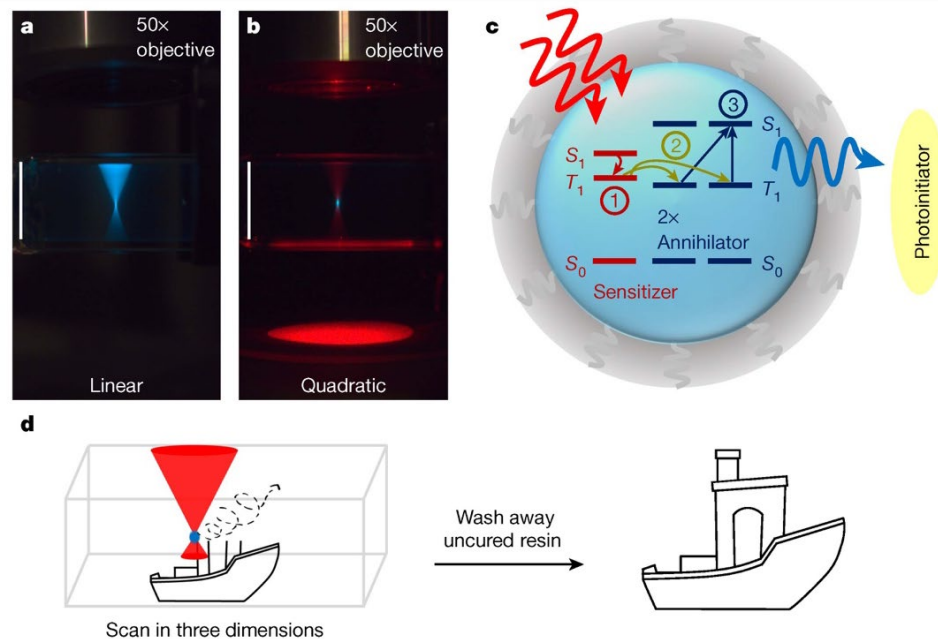
Ahmed Dorrah and Federico Capasso; Harvard University. This work was performed in part at the Center for Nanoscale Systems.

This work was supported by NSF NNCI Award ECCS-2025158. *Science* 376, eabi6860 (2022)

*National Research Priority: NSF–Quantum Leap*

# Triplet fusion upconversion nanocapsules for volumetric 3D printing

Three-dimensional (3D) printing has exploded in interest as new technologies have opened up a multitude of applications, with stereolithography a particularly successful approach. However, owing to the linear absorption of light, this technique requires photopolymerization to occur at the surface of the printing volume, imparting fundamental limitations on resin choice and shape gamut. One promising way to circumvent this interfacial paradigm is to move beyond linear processes, with many groups using two-photon absorption to print in a truly volumetric fashion. Using two-photon absorption, many groups and companies have been able to create remarkable nanoscale structures, but the laser power required to drive this process has limited print size and speed, preventing widespread application beyond the nanoscale. Here triplet fusion upconversion was developed to print volumetrically with less than 4 milliwatt continuous-wave excitation. Upconversion is introduced to the resin by means of encapsulation with a silica shell and solubilizing ligands. They further introduce an excitonic strategy to systematically control the upconversion threshold to support either monovoxel or parallelized printing schemes, enable printing at power densities several orders of magnitude lower than the power densities required for two-photon-based 3D printing.

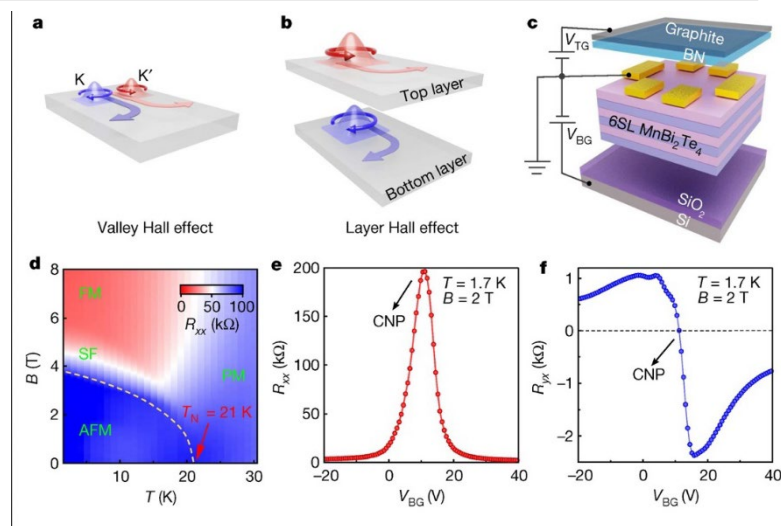


Samuel N. Sanders, Tracy H. Schloemer, Mahesh K. Gangishetty, Daniel Anderson, Michael Seitz, Aryn O. Gallegos, R. Christopher Stokes & Daniel N. Congreve; Rowland Institute at Harvard University. This work was performed in part at the Center for Nanoscale Systems.

This work was supported by NSF NNCI Award ECCS-2025158. *Nature* Vol 604, pg 474-478; (2022).

# Layer Hall effect in a 2D topological axion antiferromagnet

Whereas ferromagnets have been known and used for millennia, antiferromagnets were only discovered in the 1930s<sup>1</sup>. At large scale, because of the absence of global magnetization, antiferromagnets may seem to behave like any non-magnetic material. At the microscopic level, however, the opposite alignment of spins forms a rich internal structure. In topological antiferromagnets, this internal structure leads to the possibility that the property known as the Berry phase can acquire distinct spatial textures<sup>2,3</sup>. Here we study this possibility in an antiferromagnetic axion insulator—even-layered, two-dimensional MnBi<sub>2</sub>Te<sub>4</sub>—in which spatial degrees of freedom correspond to different layers. We observe a type of Hall effect—the layer Hall effect—in which electrons from the top and bottom layers spontaneously deflect in opposite directions. Specifically, under zero electric field, even-layered MnBi<sub>2</sub>Te<sub>4</sub> shows no anomalous Hall effect. However, applying an electric field leads to the emergence of a large, layer-polarized anomalous Hall effect of about  $0.5e^2/h$  (where  $e$  is the electron charge and  $h$  is Planck's constant). This layer Hall effect uncovers an unusual layer-locked Berry curvature, which serves to characterize the axion insulator state. Moreover, we find that the layer-locked Berry curvature can be manipulated by the axion field formed from the dot product of the electric and magnetic field vectors. Our results offer new pathways to detect and manipulate the internal spatial structure of fully compensated topological antiferromagnets<sup>4–9</sup>. The layer-locked Berry curvature represents a first step towards spatial engineering of the Berry phase through effects such as layer-specific moiré potential structured light.



Anyuan Gao, Yu-Fei Liu, Chaowei Hu, Jian-Xiang Qiu, Christian Tzschaschel, Barun Ghosh, et al. Harvard University. This work was performed in part at the Center for Nanoscale Systems.

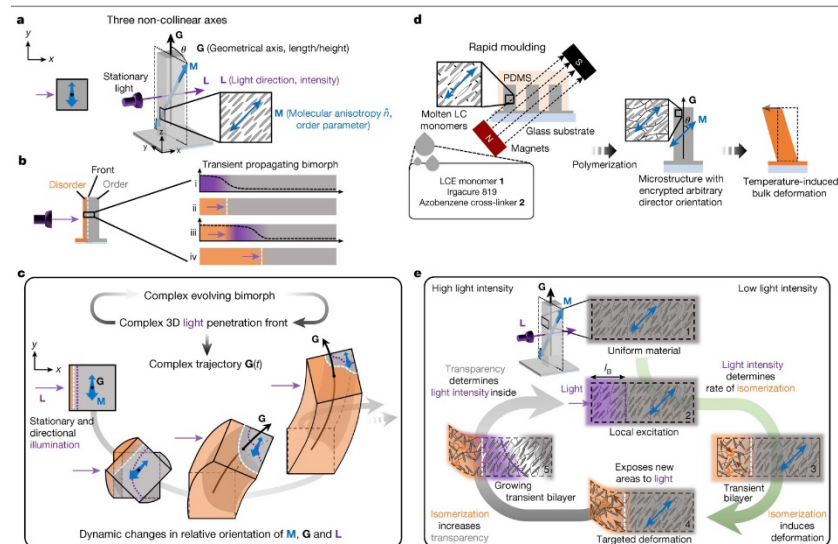
This work was supported by NSF NNCI Award ECCS-2025158; *Nature*, Vol 595 (2021), pg 521

National Research Priority: NSF–Quantum Leap



# Self-regulated non-reciprocal motions in single-material microstructures

Living cilia stir, sweep and steer via swirling strokes of complex bending and twisting, paired with distinct reverse arcs. Efforts to mimic such dynamics synthetically rely on multimaterial designs but face limits to programming arbitrary motions or diverse behaviours in one structure. Here shown is how diverse, complex, non-reciprocal, stroke-like trajectories emerge in a single-material system through self-regulation. When a micropost composed of photoresponsive liquid crystal elastomer with mesogens aligned oblique to the structure axis is exposed to a static light source, dynamic dances evolve as light initiates a travelling order-to-disorder transition front, transiently turning the structure into a complex evolving bimorph that twists and bends via multilevel opto-chemo-mechanical feedback. As captured by the theoretical model, the travelling front continuously reorients the molecular, geometric and illumination axes relative to each other, yielding pathways composed from series of twisting, bending, photophobic and phototropic motions. Guided by the model, here they choreograph a wide range of trajectories by tailoring parameters, including illumination angle, light intensity, molecular anisotropy, microstructure geometry, temperature and irradiation intervals and duration. They further show how this opto-chemo-mechanical self-regulation serves as a foundation for creating self-organizing deformation patterns in closely spaced microstructure arrays via light-mediated interpost communication, as well as complex motions of jointed microstructures, with broad implications for autonomous multimodal actuators in areas such as soft robotics, biomedical devices and energy transduction materials, and for fundamental understanding of self-regulated systems.



Shucong Li, Michael M. Lerch, James T. Waters, Bolei Deng, Reese S. Martens, Yuxing Yao, Do Yoon Kim, Katia Bertoldi, Alison Grinthal, Anna C. Balazs & Joanna Aizenberg; Harvard University. This work was performed in part at the Center for Nanoscale Systems.

This work was supported by NSF NNCI Award ECCS-2025158. *Nature*, Vol 605 (2022), pg 76

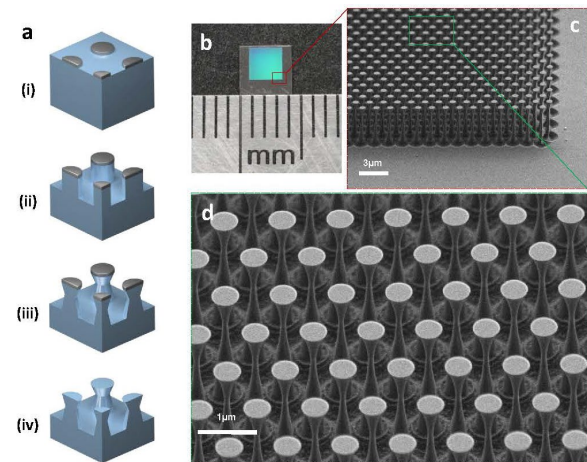
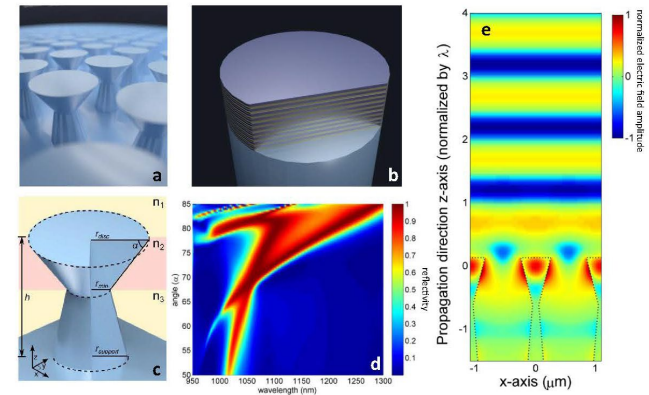
National Research Priority: NSF–Understanding the Rules of Life

# Diamond mirrors for high-power continuous-wave lasers

High-power continuous-wave (CW) lasers are used in a variety of areas including industry, medicine, communications, and defense. Yet, conventional optics, which are based on multi-layer coatings, are damaged when illuminated by high-power CW laser light, primarily due to thermal loading. This hampers the effectiveness, restricts the scope and utility, and raises the cost and complexity of high-power CW laser applications. Here we demonstrate monolithic and highly reflective mirrors that operate under high-power CW laser irradiation without damage. In contrast to conventional mirrors, ours are realized by etching nanostructures into the surface of single-crystal diamond, a material with exceptional optical and thermal properties. We measure reflectivities of greater than 98% and demonstrate damage-free operation using 10 kW of CW laser light at 1070 nm, focused to a spot of 750  $\mu\text{m}$  diameter. In contrast, we observe damage to a conventional dielectric mirror when illuminated by the same beam. Our results initiate a new category of optics that operate under extreme conditions, which has potential to improve or create new applications of high-power lasers.

Haig A. Atikian, Neil Sinclair, Pawel Latawiec, Xiao Xiong, Srujan Meesala, Scarlett Gauthier, Daniel Wintz, Joseph Randi, David Bernot, Sage DeFrances, Jeffrey Thomas, Michael Roman, Sean Durrant, Federico Capasso & Marko Lončar; Harvard University. This work was performed in part at the Center for Nanoscale Systems.

This work was supported by NSF NNCI Award ECCS-2025158; *Nature Communications* 13 : 2010 (2022)

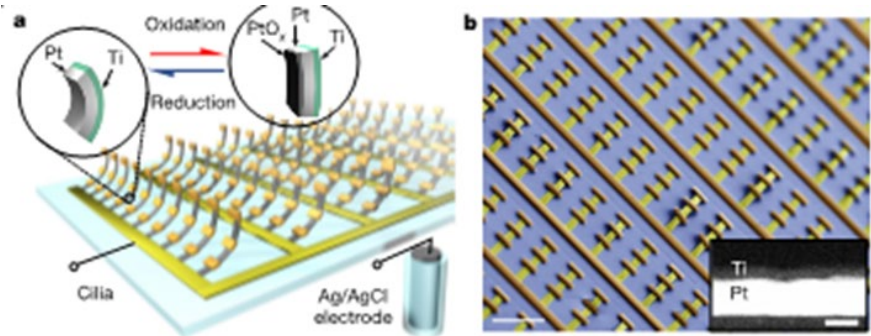


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# ***Cornell Nanoscale Science and Engineering Facility (CNF)***

# Cilia Metasurfaces for Electronically Programmable Microfluidic Manipulation

Researchers from Cornell and UPenn created active metasurfaces of electronically actuated artificial cilia that can create arbitrary flow patterns in liquids near a surface. Cilia pumping is a powerful strategy used by biological organisms to control and manipulate fluids at the microscale. However, despite numerous recent advances in optically, magnetically and electrically driven actuation, development of an engineered cilia platform with the potential for applications has remained difficult to realize. This team first created voltage-actuated cilia that generate non-reciprocal motions to drive surface flows at tens of microns per second at actuation voltages of 1 volt. They then show that a cilia unit cell can locally create a range of elemental flow geometries. By combining these unit cells, they create an active cilia metasurface that can generate and switch between any desired surface flow pattern. Finally, they integrate the cilia with a light-powered complementary metal–oxide–semiconductor (CMOS) clock circuit to demonstrate wireless operation. As a proof of concept, they use this circuit to output voltage pulses with various phase delays to demonstrate improved pumping efficiency using metachronal waves. These powerful results, demonstrated experimentally and confirmed using theoretical computations, illustrate a pathway towards fine-scale microfluidic manipulation, with applications from microfluidic pumping to microrobotic locomotion.



Microfabricated arrays of Artificial Cilia

Wei Wang, M. F. Reynolds, A. J. Cortese, M. Z. Miskin, I. Cohen et al. This work was performed, in part, at Cornell NanoScale Facility.

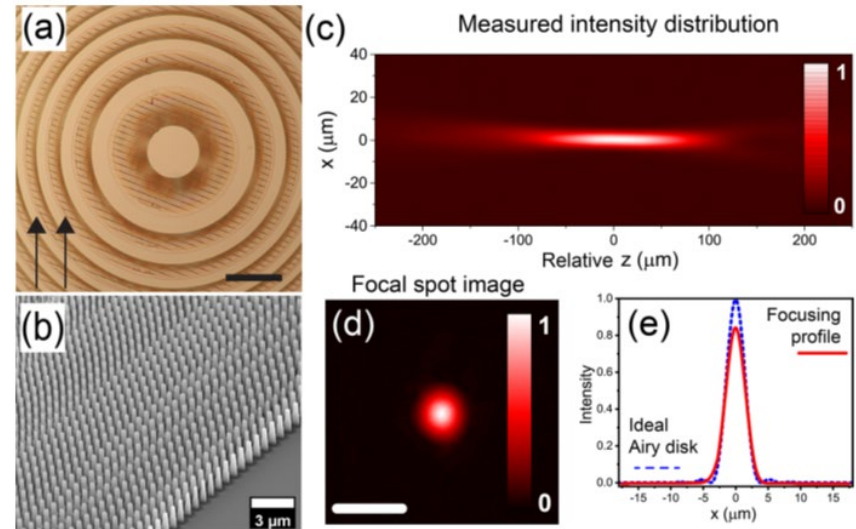
This work was supported by ARO W911NF-18-1-0032, NSF(EFMA-1935252), CCMR (DMR-171987 5), AFOSR(MURI: FA9550-16-1-0031), Kavli Institute at Cornell. *Nature* 605, 681 (2022)

*National Research Priority: NSF–Understanding the Rules of Life*



# All-Optical Tunability of Metalenses Permeated with Liquid Crystals

Researchers from Harvard used CNF to create a nanostructured planar-fused silica metalens permeated with a nematic liquid crystal (NLC) and gold nanoparticle solution. Metasurfaces have been extensively engineered to produce a wide range of optical phenomena, allowing exceptional control over the propagation of light. However, they are generally designed as single-purpose devices without a modifiable post-fabrication optical response, which can be a limitation to real-world applications. The physical properties of embedded NLCs can be manipulated with the application of external stimuli, enabling reconfigurable optical metasurfaces. They report the all-optical, dynamic control of the metalens optical response resulting from thermoplasmonic-induced changes of the NLC solution associated with the nematic–isotropic phase transition. A continuous and reversible tuning of the metalens focal length is experimentally demonstrated, with a variation of  $80\ \mu\text{m}$  (0.16% of the 5 cm nominal focal length) along the optical axis. This is achieved without direct mechanical or electrical manipulation of the device. The reconfigurable properties are compared with corroborating numerical simulations of the focal length shift and exhibit close correspondence.



G. Palermo, A. Lininger, A. Guglielmelli, L. Ricciardi, G. Nicoletta, A. De Luca, J. Park, S.W. Daniel Lim, M. L. Meretska, F. Capasso, and G. Strangi. This work was performed, in part, at Cornell NanoScale Facility.

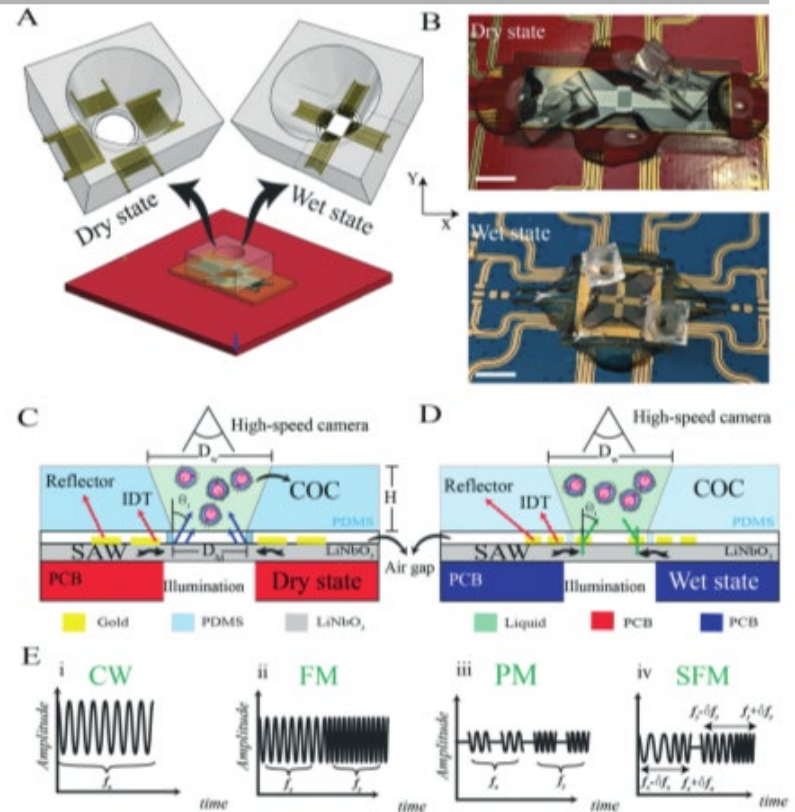
This work was supported by AFOSR MURI Grant No. FA9550-21-1-0312; NSF Grant No. 1904592. *ACS Nano* 2022, 16, 16539–16548.

*National Research Priority: Advanced Photonics*



# Non-contact Ultrasound Oocyte Denudation

Researchers from Cornell used CNF to create a chip-scale ultrasonic device consisting of four interdigitated transducers (IDT) on a lithium niobate substrate that has been engineered to deliver megahertz (MHz) range ultrasound to perform denudation. Cumulus removal (CR) is a central prerequisite step for many protocols involved in the assisted reproductive technology (ART) such as intracytoplasmic sperm injection (ICSI) and preimplantation genetic testing (PGT). The most prevalent CR technique is based upon laborious manual pipetting, which suffers from inter-operator variability and therefore a lack of standardization. Automating CR procedures would alleviate many of these challenges, improving the odds of a successful ART or PGT outcome. The acoustic streaming and acoustic radiation force agitate COCs inside a microwell placed on top of the  $\text{LiNbO}_3$  substrate to remove the cumulus cells from the oocytes. This paper demonstrates the capability and safety of the denudation procedure utilizing surface acoustic wave (SAW), achieving automation of this delicate manual procedure and paving the steps toward improved and standardized oocyte manipulation.



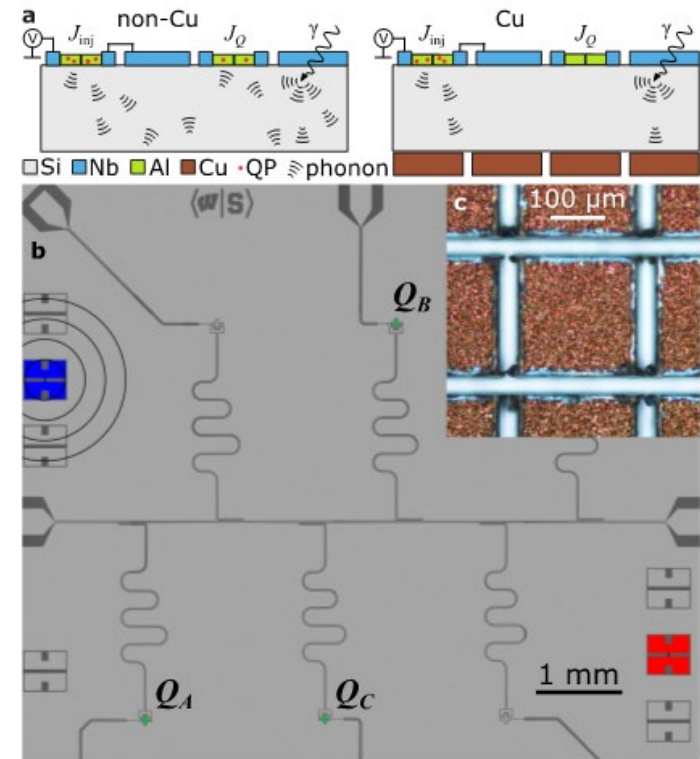
Amir Mokhtare, Benyamin Davaji, Philip Xie, Mohammad Yaghoobi, Zev Rosenwaks, Amit Lal, Gianpiero Palermo and Alireza Abbaspourra. This work was performed, in part, at Cornell NanoScale Facility.

This work was supported by DARPA PRIGM-AIMS. *LabChip*, 2022, 22, 777–792.

National Research Priority: NSF–Understanding the Rules of Life

# Phonon Downconversion To Suppress Correlated Errors In Superconducting Qubits

Researchers from Syracuse and U Wisconsin-Madison used CNF to create normal metal reservoirs on the chip back side to downconvert phonons to low energies where they can no longer poison qubits. Quantum error correction can preserve quantum information in the presence of local errors, but correlated errors are fatal. For superconducting qubits, high-energy particle impacts from background radioactivity produce energetic phonons that travel throughout the substrate and create excitations above the superconducting ground state, known as quasiparticles, which can poison all qubits on the chip. They introduce a pump-probe scheme involving controlled injection of pair-breaking phonons into the qubit chips. They examine quasiparticle poisoning on chips with and without back-side metallization and demonstrate a reduction in the flux of pair-breaking phonons by over a factor of 20. They use a Ramsey interferometer scheme to simultaneously monitor quasiparticle parity on three qubits for each chip and observe a two-order of magnitude reduction in correlated poisoning due to background radiation.



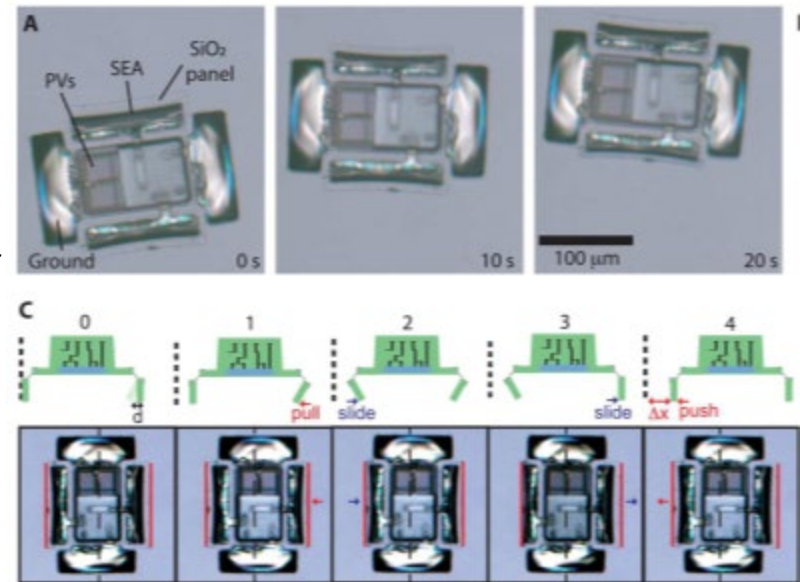
V. Iaia, J. Ku, A. Ballard, C. P. Larson, E. Yelton, C. H. Liu, S. Patel, R. McDermott & B. L. T. Plourde. This work was performed, in part, at Cornell NanoScale Facility.

This work was supported by ARO grant W911NF-18-1-0106, *Nature Communications* | (2022)13:6425.

*National Research Priority: NSF–Quantum Leap*

# Microscopic Robots With Onboard Digital Control

Researchers from Cornell and UPenn used CNF to develop an integration process and build microscopic robots controlled by onboard complementary metal oxide semiconductor electronics. Autonomous robots—systems where mechanical actuators are guided through a series of states by information processing units to perform a predefined function—are expected to revolutionize everything from health care to transportation. Microscopic robots are poised for a similar revolution in fields from medicine to environmental remediation. A key hurdle to developing these microscopic robots is the integration of information systems, particularly electronics fabricated at commercial foundries, with microactuators. The resulting autonomous, untethered robots are 100 to 250 micrometers in size, are powered by light, and walk at speeds greater than 10 micrometers per second. In addition, they demonstrate a microscopic robot that can respond to an optical command. This work paves the way for ubiquitous autonomous microscopic robots that perform complex functions, respond to their environments, and communicate with the outside world.



Michael F. Reynolds, Alejandro J. Cortese, Qingkun Liu, Zhangqi Zheng, Wei Wang, Samantha L. Norris, Sunwoo Lee, Marc Z. Miskin, Alyosha C. Molnar, Itai Cohen, Paul L. McEuen. This work was performed, in part, at Cornell NanoScale Facility.

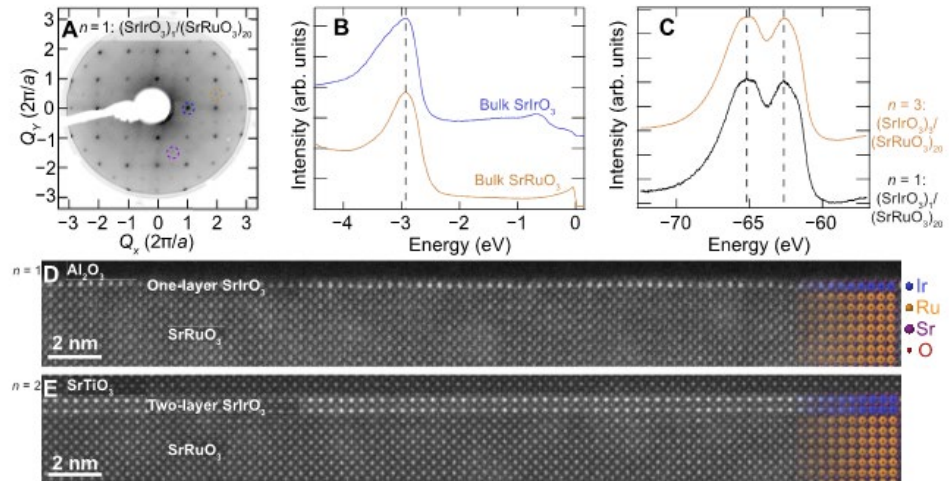
This work was supported by the Cornell Center for Materials Research (DMR-1719875), AFOSR MURI: FA9550-16-1-0031; ARO W911NF-18-1-0032; NSF (EFRI-1935252); Kavli Institute at Cornell for Nanoscale Science. *Sci. Robot.* 7, eabq2296 (2022)

National Research Priority: NSF–Growing Convergence Research



# Interfacial Charge Transfer And Persistent Metallicity Of Ultrathin $\text{SrIrO}_3/\text{SrRuO}_3$ Heterostructures

Researchers from Cornell, Columbia, the Collège de France and the Leibniz-Institut für Kristallzüchtung created substrates for angle-resolved photoemission spectroscopy and molecular beam epitaxy to reveal the electronic structure, charge transfer, doping profile, and carrier effective masses in a layer-by-layer fashion for the interface between the Dirac nodal-line semimetal  $\text{SrIrO}_3$  and the correlated metallic Weyl ferromagnet  $\text{SrRuO}_3$ . Interface quantum materials have yielded a plethora of previously unknown phenomena, including unconventional superconductivity, topological phases, and possible Majorana fermions. Typically, such states are detected at the interface between two insulating constituents by electrical transport, but whether either material is conducting, transport techniques become insensitive to interfacial properties. We find that electrons are transferred from the  $\text{SrIrO}_3$  to  $\text{SrRuO}_3$ , with an estimated screening length of  $l = 3.2 \pm 0.1 \text{ \AA}$ . In addition, we find that metallicity is preserved even down to a single  $\text{SrIrO}_3$  layer, where the dimensionality-driven metal-insulator transition typically observed in  $\text{SrIrO}_3$  is avoided because of strong hybridization of the Ir and Ru  $t_{2g}$  states.



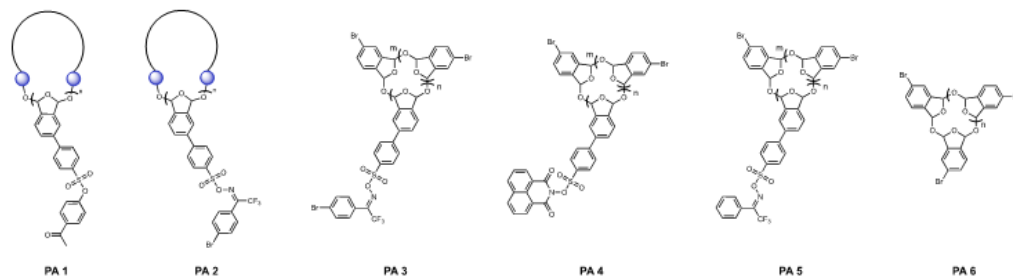
J.N. Nelson, N.J. Schreiber, A.B. Georgescu, B.H. Goodge, B.D. Faeth, C.T. Parzyck, C. Zeledon, L.F. Kourkoutis, A. J. Millis, A. Georges, D.G. Schlom, K.M. Shen. This work was performed, in part, at Cornell NanoScale Facility.

This work was primarily supported through NSF MIP PARADIM DMR-2039380 as well as grants DMR-1709255; DMR-2104427; AFOSR grants FA9550-15-1-0474; FA9550-21-1-0168. *Sci. Adv.* **8**, eabj0481 (2022)

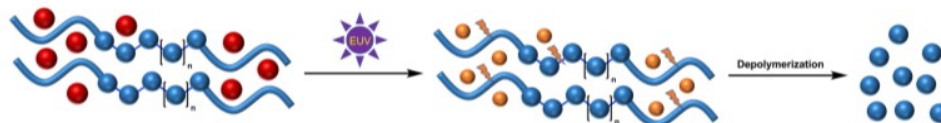
National Research Priority: DoD–Microelectronics

# Modular Synthesis of Phthalaldehyde Derivatives Enabling Access to Photoacid Generator-Bound Self-Immolative Polymer Resists with Next-Generation Photolithographic Properties

Researchers from Cornell University used CNF to investigate the first PAG-tethered self-immolative resists in an architecture that simultaneously displays high contrast, high sensitivity, and low roughness under EUV exposure. The resolution, line edge roughness, and sensitivity (RLS) trade-off has fundamentally limited the lithographic performance of chemically amplified resists. Production of next-generation transistors using extreme ultraviolet (EUV) lithography depends on a solution to this problem. A resist that simultaneously increases the effective reaction radius of its photogenerated acids while limiting their diffusion radius should provide an elegant solution to the RLS barrier. Here, they describe a generalized synthetic approach to phthalaldehyde derivatives using sulfur(VI) fluoride exchange click chemistry that dramatically expands usable chemical space by enabling non-ionic photoacid generators (PAG) to be tethered to phthalaldehyde. They believe this class of resists will ultimately enable technologists to overcome the RLS trade-off.



Previous work



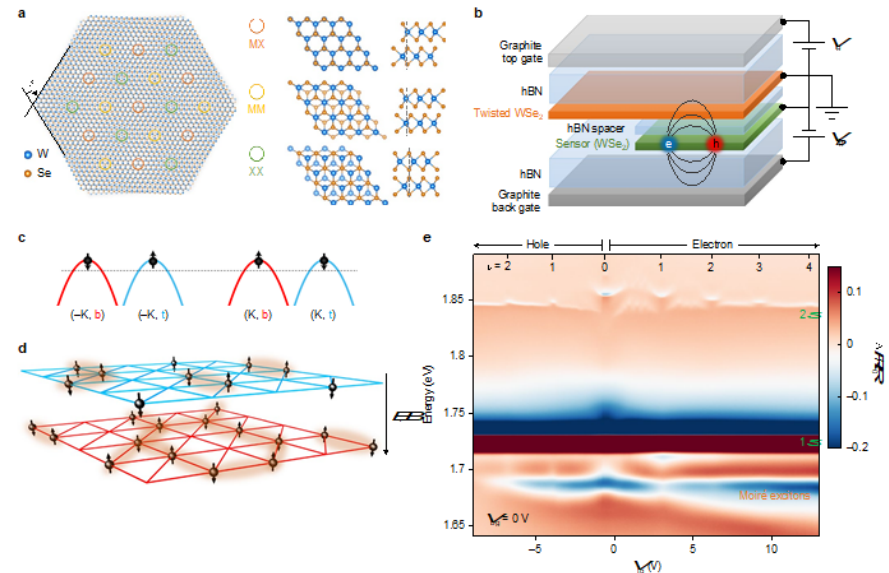
J. Deng, S. Bailey, S. Jiang, C.K. Ober. This work was performed, in part, at Cornell NanoScale Facility.

This research was supported by Intel Corporation through the SRC research project (task ID: 2885.001). *J. Am. Chem. Soc.* 2022, 144, 19508–19520

National Research Priority: DoD–Microelectronics

# A Tunable Bilayer Hubbard Model In Twisted $WSe_2$

Researchers from Cornell, China and Japan created competing electronic states in twisted AB-homobilayer  $WSe_2$ , which realizes a bilayer Hubbard model in the weak interlayer hopping limit for holes. Moiré materials with flat electronic bands provide a highly controllable quantum system for studies of strong-correlation physics and topology. In particular, angle-aligned heterobilayers of semiconducting transition metal dichalcogenides with large band offset realize the single-band Hubbard model. Introduction of a new layer degree of freedom is expected to foster richer interactions, enabling Hund's physics, interlayer exciton condensation and new superconducting pairing mechanisms to name a few. By layer-polarizing holes via a perpendicular electric field, they observe a crossover from an excitonic insulator to a charge-transfer insulator at a hole density of  $\nu = 1$  (in units of moiré density), a transition from a paramagnetic to an antiferromagnetic charge-transfer insulator at  $\nu = 2$  and evidence for a layer-selective Mott insulator at  $1 < \nu < 2$ . The unique coupling of charge and spin to external electric and magnetic fields also manifests a giant magnetoelectric response. Their results establish a new solid-state simulator for the bilayer Hubbard model Hamiltonian.



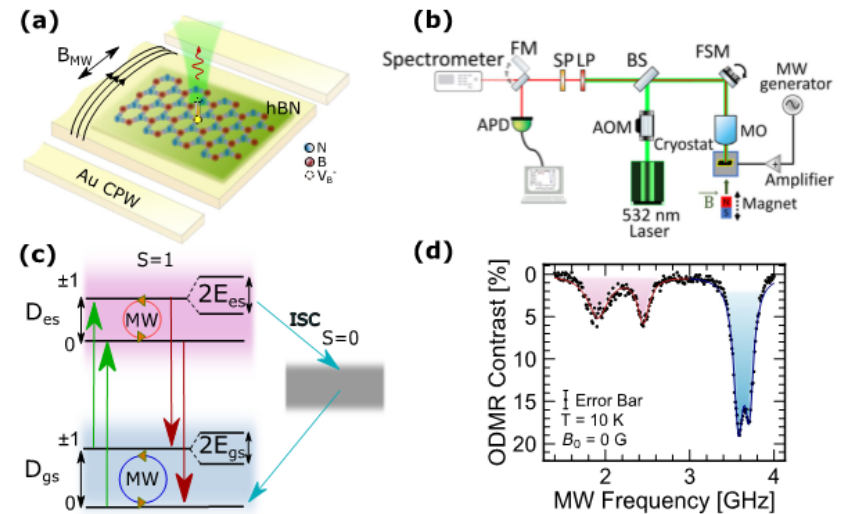
Y.Xu, K.Kang, K.Watanabe, T.Taniguchi, K.F.Mak, J. Shan. This work was performed, in part, at Cornell NanoScale Facility.

This research was supported by DOE-BES DE-SC0022058 and DE-SC0019481, NSF DMR-2114535 and ONR N00014-21-1-2471. *Nature Nanotechnology* **17**, 934 (2022)

National Research Priority: NSF–Quantum Leap

# Excited-state Spin-resonance Spectroscopy Of VB Defect Centers In Hexagonal Boron Nitride

Researchers from Cornell, U Rochester and Purdue used CNF to create devices to determine excited-state spin Hamiltonians of hexagonal boron nitride (hBN), including a room-temperature zero-field splitting of 2.1 GHz and a g-factor similar to that of the ground-state. The recently discovered spin-active boron vacancy (VB) defect center in hBN has high contrast optically-detected magnetic resonance (ODMR) at room temperature, with a spin-triplet ground-state that shows promise as a quantum sensor. Here they report temperature-dependent ODMR spectroscopy to probe spin within the orbital excited-state. They confirm that the resonance is associated with spin rotation in the excited-state using pulsed ODMR measurements and observe Zeeman-mediated level anti-crossings in both the orbital ground- and excited-state. Their observation of a single set of excited-state spin-triplet resonance from 10 to 300 K is suggestive of symmetry-lowering of the defect system from  $D_{3h}$  to  $C_{2v}$ . Additionally, the excited-state ODMR has strong temperature dependence of both contrast and transverse anisotropy splitting, enabling promising avenues



N. Mathur, A. Mukherjee, X. Gao, J. Luo, B. A. McCullian, T. Li, A. N. Vamivakas & G. D. Fuchs. This work was performed, in part, at Cornell NanoScale Facility.

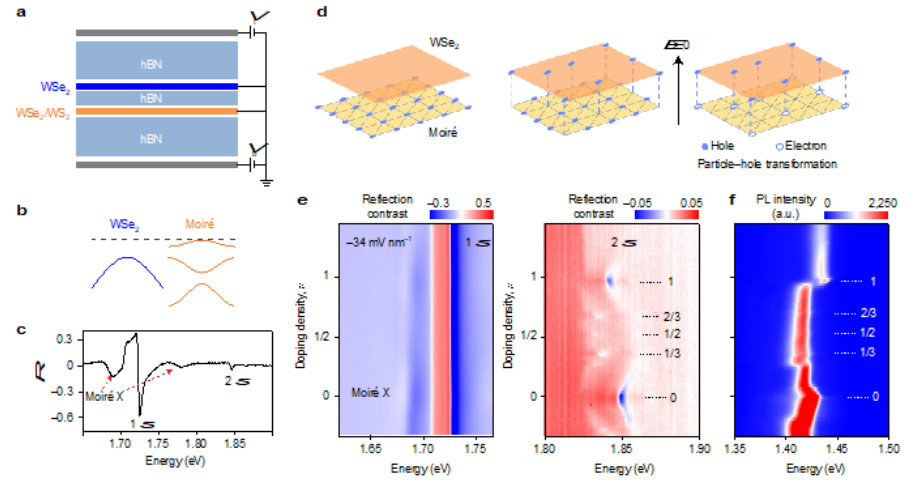
This work was supported by the Cornell Center for Materials Research (DMR-1719875); AFOSR (FA9550-18-1-0480); NSF (ECCS-1839196); AFOSR (FA9550-19-1-0074). *Nature Communications* (2022)13:3233

National Research Priority: NSF–Quantum Leap



# Dipolar Excitonic Insulator In A Moiré Lattice

Researchers from Cornell and NIMS, Japan used CNF to create a device where a  $\text{WSe}_2$  monolayer and  $\text{WSe}_2/\text{WS}_2$  moiré bilayer are coupled via Coulomb interactions. Two-dimensional moiré materials provide a highly controllable solid-state platform for studies of quantum phenomena. To date, experimental studies have focused on correlated electronic states, whereas correlated bosonic states in moiré materials have received less attention. Here they report the observation of a correlated dipolar excitonic insulator—a charge-insulating state driven by exciton formation. The system is a Mott insulator when all the holes reside in the moiré layer. Under an out-of-plane electric field, the holes can be continuously transferred to the  $\text{WSe}_2$  monolayer, but remain strongly bound to the empty moiré sites, effectively forming an inter-layer exciton fluid in the moiré lattice. They further observe the emergence of local magnetic moments in the  $\text{WSe}_2$  monolayer induced by the strong interlayer Coulomb correlation. Their result provides a platform for realizing correlated quantum phenomena described by bosonic lattice models in a solid-state system, complementary to cold-atom setups.



J.Gu , L.Ma, Song Liu, K.Watanabe, J.Shan and K.F.Mak. This work was performed, in part, at Cornell NanoScale Facility. *Nature Physics* 18, 395 (2022)

Research supported by ONR N00014- 21-1-2471; AFOSR FA9550-18-1-0480; NSF DMR-2004451; DOE-BES DE-SC0022058 and DE-SC001948. *Nature Physics* 18, 395 (2022).

*National Research Priority: NSF–Quantum Leap*



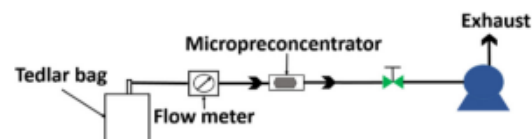
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# ***Kentucky Multi-Scale Manufacturing and Nano Integration Node (KY Multiscale)***

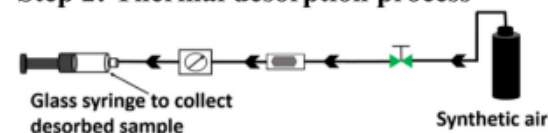
# Integration of micropreconcentrators with SPME for analysis of toxic VOCs in environmental air

The goal of this project is to develop a novel approach for analysis of toxic volatile organic compounds (VOCs) in superfund site environmental air. Chronic exposure to toxic VOCs induces adverse effects on human health such as cancers and cardiovascular diseases. Thus, there is a need to develop accurate, and rapid analytical methods for monitoring toxic VOCs in environmental air. We designed and fabricated a micropreconcentrator with micropillars in the microfluidic channel to support adsorbents and to enhance heat transfer for thermal desorption of VOCs. The innovative development of both chemical and physical adsorption enables to capture and measure a wide range of both polar and unipolar VOCs in air. The integration of micropreconcentrator with solid phase microextraction (SPME) enable analysis of VOCs by GC-MS without expensive concentration instrument and time-consuming concentration process of VOCs.

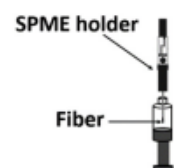
## Step 1: Preconcentration of VOCs



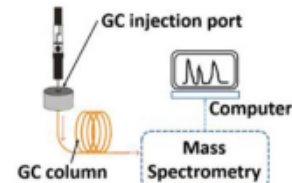
## Step 2: Thermal desorption process



## Step 3: SPME extraction



## Step 4: GCMS analysis



Schematic illustration of integration of micropreconcentrators with SPME for analysis of trace VOCs in environmental air by GC-MS.

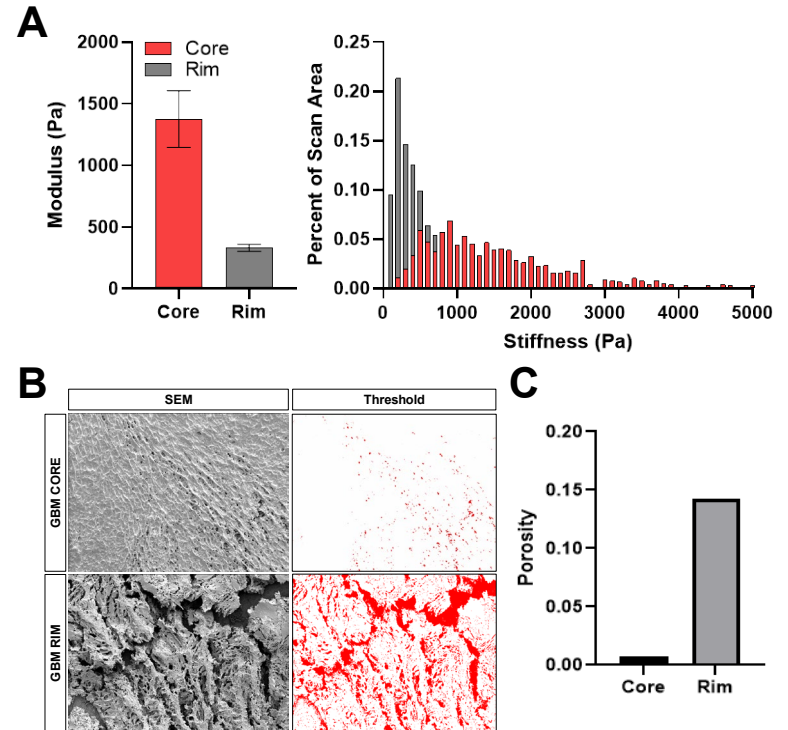
Xiao-An Fu, Department of Chemical Engineering, University of Louisville. This research was performed at the University of Louisville MicroNanoTechnology Center (MNTC).

This work was supported by NIH NIEHS Superfund center award 1P42ES023716. *J. Chromatography A*. 2022, 1673, 463083.

National Research Priority: NSF–Understanding the Rules of Life

# Interrogation of Tumor Biophysical and Ultrastructural Signatures via AFM and SEM

Glioblastoma (GBM) is a highly aggressive and invasive brain cancer, carrying a median survival of 15 months. This poor prognosis is due, in part, to its resistance to therapy through tumor adaptations mediated by the tumor microenvironment (TME). Efforts to describe the tumor-protective cues in the TME may aid in the development of strategies to combat resistance. Emerging work has indicated that tumor biophysical properties are significant modulators of tumor evolution; however, these parameters are poorly described in GBM. Here, we investigate the mechanical and ultrastructural characteristics of the tumor in patient-matched GBM core and rim tissue. Using AFM and SEM techniques, we find that GBM core is abnormally stiff and dense while rim tissue possess characteristics that resemble normal brain and are soft and more porous. These data suggest that the stiff core participates in tumor adaptations through pro-malignant mechanotransduction while the rim provides more permissive routes for GBM invasion. These novel insights support the development of TME targeting therapies that alter tumor mechanics.



*GBM core and rim exhibit unique mechanical and ultrastructural signatures. (A-C) GBM core displays a median stiffness of ~ 1.3 kPa and possess low porosity while GBM rim is soft (~ 400 Pa) and porous.*

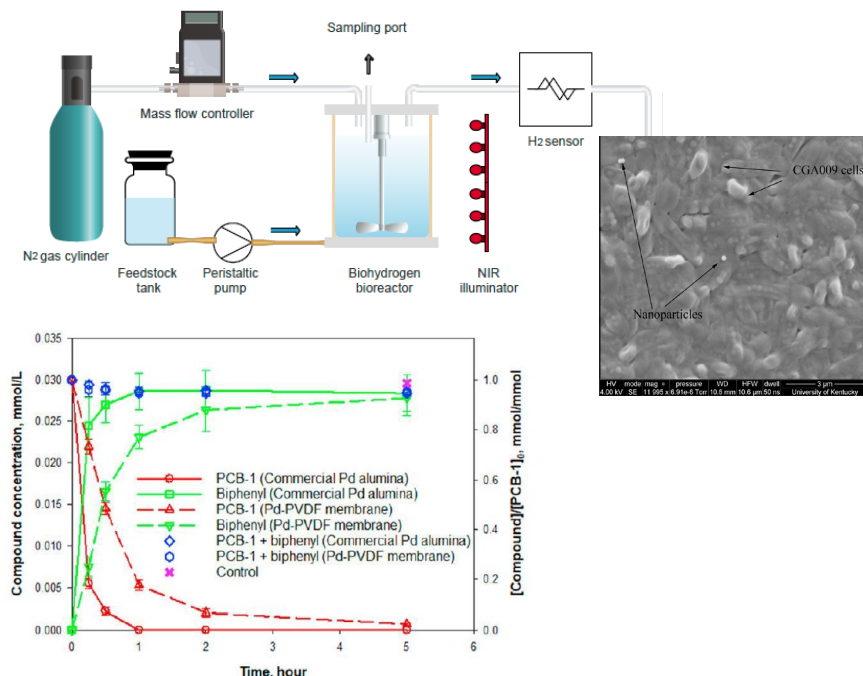
Joseph Chen, Department of Bioengineering, University of Louisville. Work was performed at University of Louisville MicroNano Technology Center (MNTC).

BMES 2022, San Antonio, TX

*National Research Priority: NSF–Understanding the Rules of Life*

# Nanoparticle Enhanced Biohydrogen Production and Pollutant Hydrogenation

A photoinduced fermentation process using *Rhodospseudomonas palustris* provides an alternative to traditional hydrogen production. In this study, biohydrogen production was investigated in the near IR region using near-field enhancement by silica-core gold-shell nanoparticles (NPs). Maximum increases in H<sub>2</sub> and CO<sub>2</sub> productions from NPs were 115% and 113% without affecting the bacterial growth rates. Model simulations showed that the energy conversion efficiency increased with NPs concentration but decreased with light intensity. In addition, the application of this biohydrogen generation process to environmental remediation was investigated by treating toxic 2-chlorobiphenyl (PCB-1) by hydrogenation using Pd catalysts.



(a) Near-infrared illuminated biohydrogen reactor containing *Rhodospseudomonas palustris* and silica-core gold-shell nanoparticles. (b) Electron micrograph of nanoparticles and cells. (c) Graph showing the remediation of toxic 2-chlorobiphenyl.

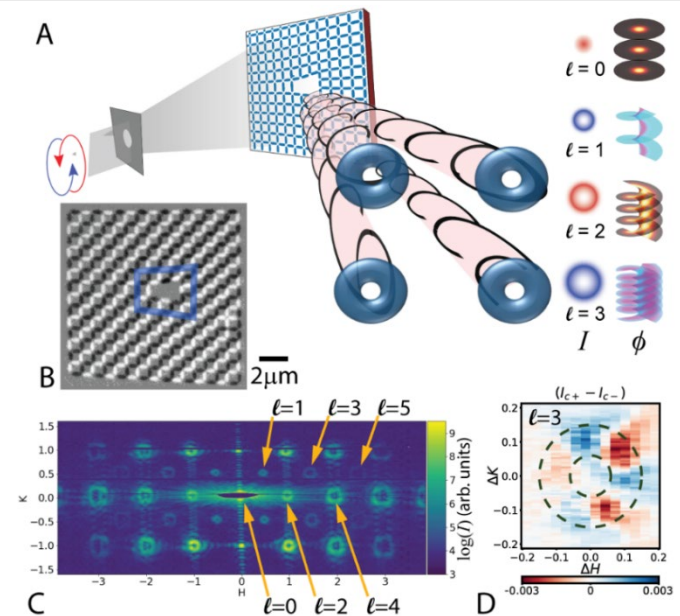
A collaboration between U. of Kentucky and Southern Company led by D. Bhattacharyya (UK Chem. Engr.). Work performed in part at KY Multiscale EMC.

Funded by CBET-1700091, NIH-NIEHS SRP (P42ES007380), and Southern Company. *International Journal of Hydrogen Energy*, 2021.

National Research Priority: NSF–Growing Convergence Research

# Switchable X-ray Orbital Angular Momentum from an Artificial Spin Ice

The orbital angular momentum (OAM) of visible photons has found application in fields as diverse as quantum cryptography, optical tweezers, and telecommunications. There is rapidly growing interest in X-ray OAM for imaging and probing materials at the nanoscale. This research showed that X-ray diffraction from an artificial spin ice (ASI) yields X-rays carrying OAM. ASIs are patterned arrays of nanomagnets that share some characteristics with water ice. The researchers confirmed that a particular ASI structure orders antiferromagnetically near room temperature and that X-ray photons have even- and odd- OAM quantum numbers depending on whether they scatter from the structure itself or from its magnetic texture. The researchers also showed that magnetically scattered OAM beams could be turned on and off by modest variations of temperature and applied magnetic field. These results imply reconfigurable X-ray optics could be designed using ASIs, and these structures may enable selective probing of electronic and magnetic states in materials.



(A) X-ray photons scattered from an artificial spin ice (ASI) acquire orbital angular momentum. (B) The ASI's antiferromagnetic ground state. (C) Odd- and even- order OAM is generated at the magnetic and structural Bragg conditions. (D) Difference between left- and right- circular polarizations confirms

A collaboration between the Univ. of Kentucky, Lawrence Berkeley NL, Argonne NL, and Brookhaven NL. Work performed at KY Multiscale CeNSE, EMC, and CAM.

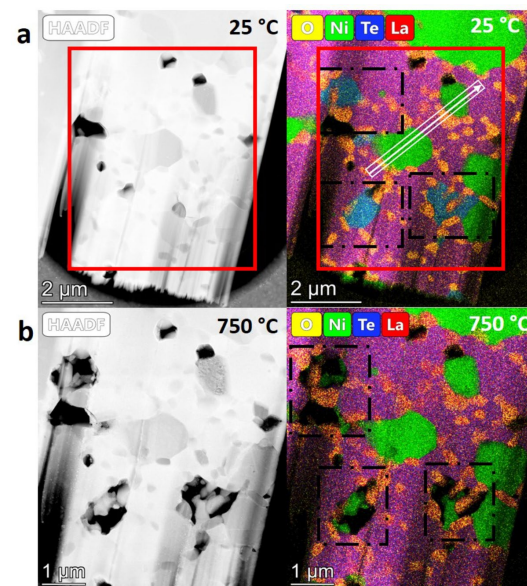
Funding provided by DE-SC0016519. *Phys. Rev. Lett.* 126, 117201 (2021).

National Research Priority: NSF–Quantum Leap



# Structure and Stability of Thermoelectric $\text{La}_{3-x}\text{Te}_4\text{-Ni}$ Composites

Thermoelectric materials convert thermal to electrical energy. Lanthanum telluride is a high-performance thermoelectric material that is well suited for use in radioisotope thermoelectric generators (RTG) powering long duration space missions. Introduction of nickel nanoparticles into lanthanum telluride systems can increase their figure of merit by 30%, but the characteristics and stability of the  $\text{La}_{3-x}\text{Te}_4\text{-Ni}$  interfaces are not well understood. This research effort used *in situ* transmission electron microscopy techniques to mimic the working environment of an RTG in deep space while studying of the smallest structural details of the  $\text{La}_{3-x}\text{Te}_4\text{-Ni}$  interfaces. The results of this study provide design guidance for next-generation thermoelectric materials with the potential for longer life span in RTG applications.



***In situ* TEM study of a partially oxidized  $\text{La}_{3-x}\text{Te}_4\text{-Ni}$  sample.**  
(a, b) Micrographs and elemental maps collected at 25 and 750 °C, respectively. The interfaces in oxidized areas (dashed squares) have degraded while nonoxidized areas (green) remained intact.

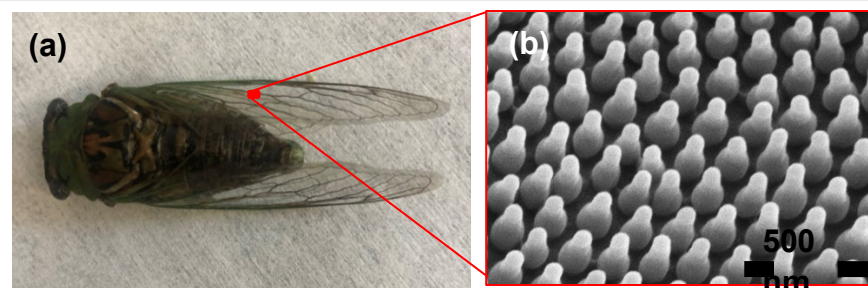
A collaboration between the Univ. of Kentucky and JPL/Caltech led by Prof. Beth Guiton (UK Chemistry). Work performed at KY Multiscale EMC.

Funding provided by DMR 1455154Y, OIA 1355438, NN15AK28A. Scialog Award 26329. *J. Phys. Chem. C* 125, 21131 (2021).

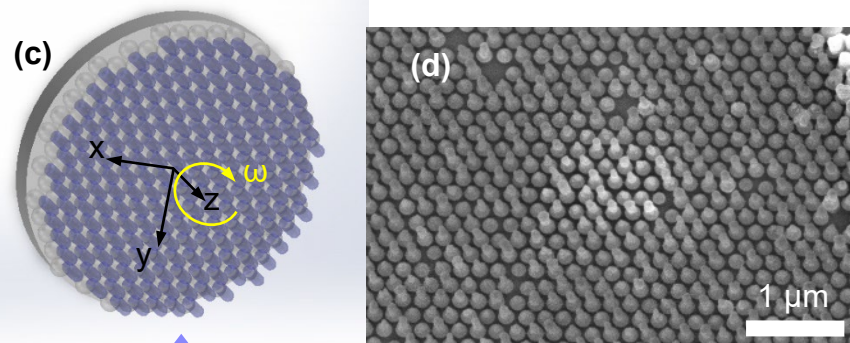
*National Research Priority: NSF–Growing Convergence Research and Windows on the Universe*

# Bio-Inspired Antibacterial Metasurfaces Fabricated by Glancing Angle Deposition (GLAD)

The goal of this research is to propose an easy and cost-effective fabrication approach of Glancing Angle Deposition (GLAD) for creating antibacterial surfaces, which are inspired by cicada wings. Antibacterial surfaces are known to be applied in a variety of specific interfaces, such as medical implants and food packaging. Nanopillar cones semi-hexagonally distributed on the cicada wings with  $\sim 170$  nm between the neighboring pillars (Fig. b). The nanopillar cones puncture into bio cells falling on top and keep the surface bacteria free. Given the nano-level three-dimensional features, it is extremely difficult to replicate these naturally occurred nanostructure using conventional top-down nanofabrication processes. GLAD is versatile bottom-up process which uses physical vapor deposition while maneuvering with incident angle and rotation of the substrate, and the cicada wing replica surfaces are created by GLAD (Fig. d). Gram negative bacteria (E Coli) are applied on top of the surface for antibacterial testing. The preliminary results show the effectiveness of the antibacterial property of our synthetic nanostructured film.



(a) Annual cicada (also known as dog-day cicada) (b) SEM image of the nanostructures on the wings of annual cicadas



(c) GLAD scheme for nanopillar cone structures on cicada wings (d) SEM image of the nanopillar cones created by GLAD

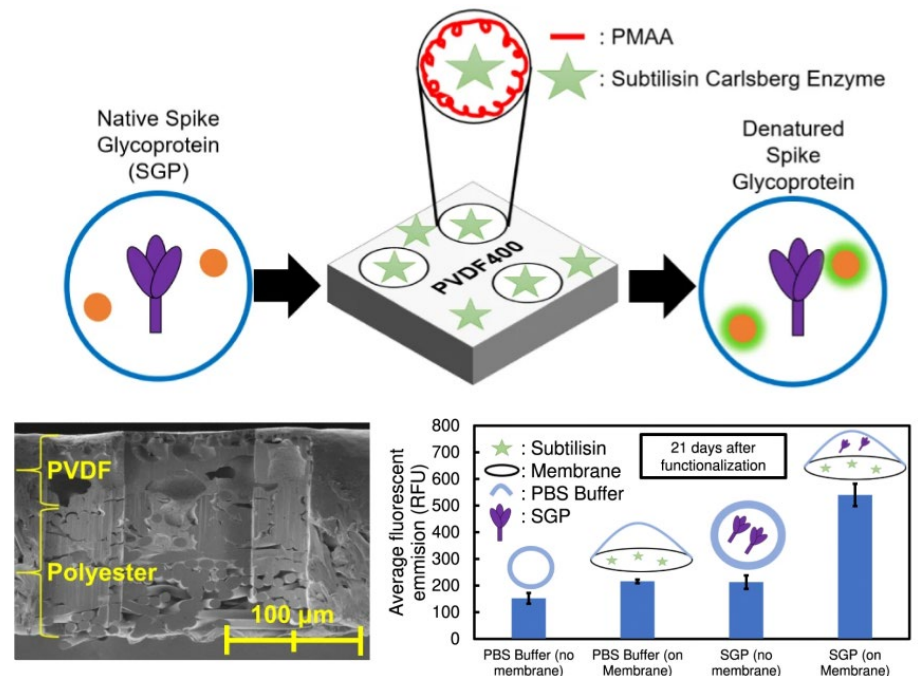
Qu, C., Rozsa J., Jung, H J., Running, M., McNamara, S., and Walsh, K. Research was performed at the University of Louisville MicroNanoTechnology Center (MNTC).

Work was supported by NSF NNCI Award #2025075. 48<sup>th</sup> ICMCTF Conference, San Diego, 2022.

National Research Priority: NSF–Understanding the Rules of Life

# Aerosol capture and coronavirus spike protein deactivation by enzyme functionalized antiviral membranes

The CoViD-19 pandemic emphasized the need for advanced mask and air filtration technologies. Professor Dibakar Bhattacharyya and co-workers at the University of Kentucky, with assistance from Solecta Membranes in California, recently developed a new antiviral membrane for filtration. The membrane captures coronavirus-sized aerosols and denatures the SARS-CoV-2 spike protein. The membrane consists of asymmetric pores functionalized with enzymes that can attack spike proteins even in low-humidity environments. The membranes showed improved protection factors and longer lifetimes without fouling compared to N95 mask materials.



**Enzyme functionalized membranes capture and deactivate SARS-CoV-2 spike proteins.** (a) Schematic of antiviral membrane. (b) Cross-sectional electron micrograph of membrane. (c) Deactivation spike protein confirmed with increased Sypro-Orange fluorescence.

Effort led by Prof. Dibakar Bhattacharyya (UK Chemical Engineering) with assistance from Solecta Membranes (Oceanside, CA). Work performed at KY Multiscale.

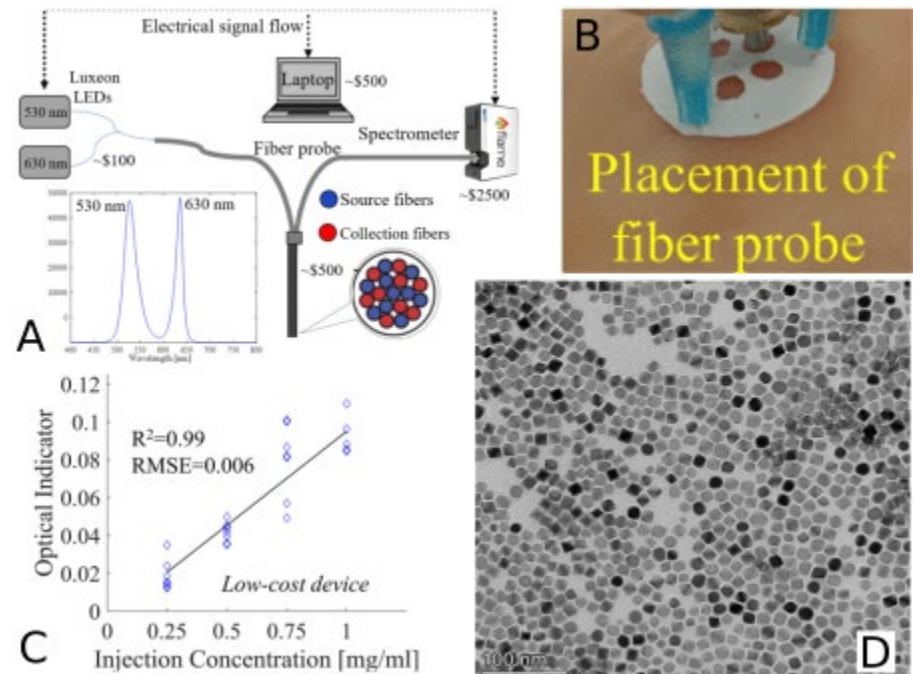
Support from NSF CBET 2030217, EMC and CeNSE. *Communications Materials* 3, 34 (2022).

*National Research Priority: NSF–Growing Convergence Research and Understanding the Rules of Life*



# Point-of-Care Real-Time Monitoring of Nanoparticle Delivery in Biological Tissue Models

Nanoparticles are being used for a wide variety of health-related applications including drug delivery and thermal cancer therapy. However, there are few approaches to point-of-care monitoring of nanoparticle concentrations in tissue. Researchers at the University of Kentucky recently developed a compact, low-cost system for monitoring nanoparticle concentrations using diffuse reflectance spectroscopy. The technique demonstrated accurate measurement of nanoparticle concentrations in ex-vivo tissue using a non-invasive, optical probe. The technique holds the potential to advance translational cancer research and nanomedicine more generally.



## Point of Care Monitor for Therapeutic Nanoparticle Delivery.

A. Diffuse reflectance spectroscopy platform. B. Fiber probe placed on chicken tissue. C. Measurement of nanoparticle concentration in chicken tissue. D. TEM of iron oxide nanoparticles.

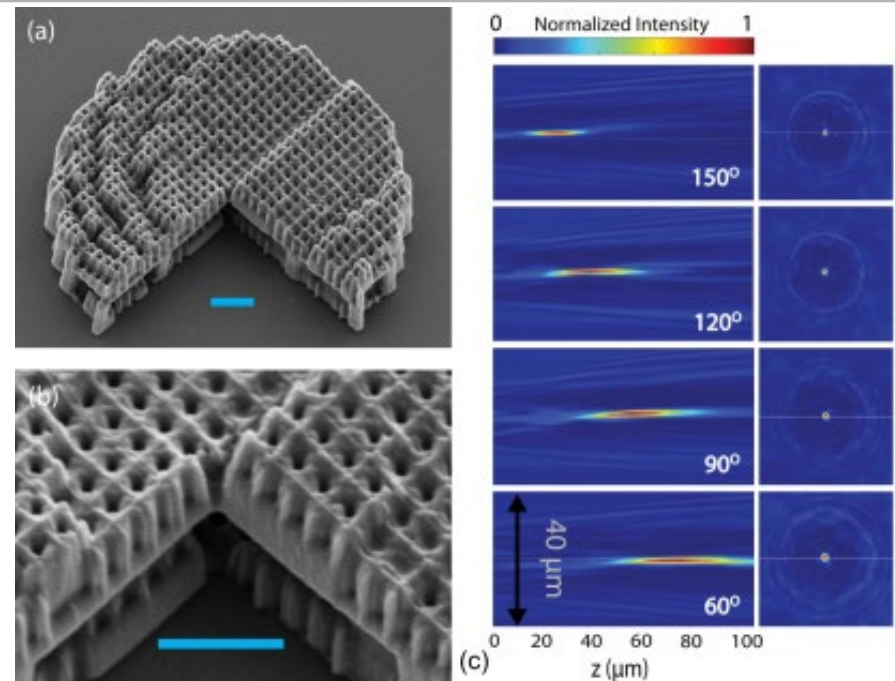
Effort led by professors Sheng Tong and Caigang Zhu (UK Biomedical Engineering). Work performed at KY Multiscale EMC.

*IEEE J. Sel. Top. Quantum Electron.* **29**, 4 (2023).

National Research Priority: NSF–Growing Convergence Research and Understanding the Rules of Life

# Rotationally tunable varifocal 3D metalens

Metalenses are nearly flat optics that employ nanostructured surfaces to manipulate light. The compact size, light weight, and mass manufacturability of metalenses make them ideal for mobile, airborne, and *in-vivo* imaging. Researchers from the University of Kentucky recently developed a metalens whose focal length can be changed by rotating one of two nanostructured elements. The nanostructures consisted of square unit cells of varying height with central holes of varying depth. The metalenses were 3D printed using two-photon lithography and characterized in the short-wave infrared spectral region. Multielement metalenses hold the potential to enhance machine vision, medical imaging, microscopy, and augmented reality.



**Metalens with variable focal length.** (a) Two-element metalens printed using two-photon lithography. (b) Enlarged view showing regions with varying thickness and hole depth. Scale bars are 4 μm. (c) Mutual rotation of the elements focuses light to different distances. ( $\lambda = 1.5 \mu\text{m}$ ).

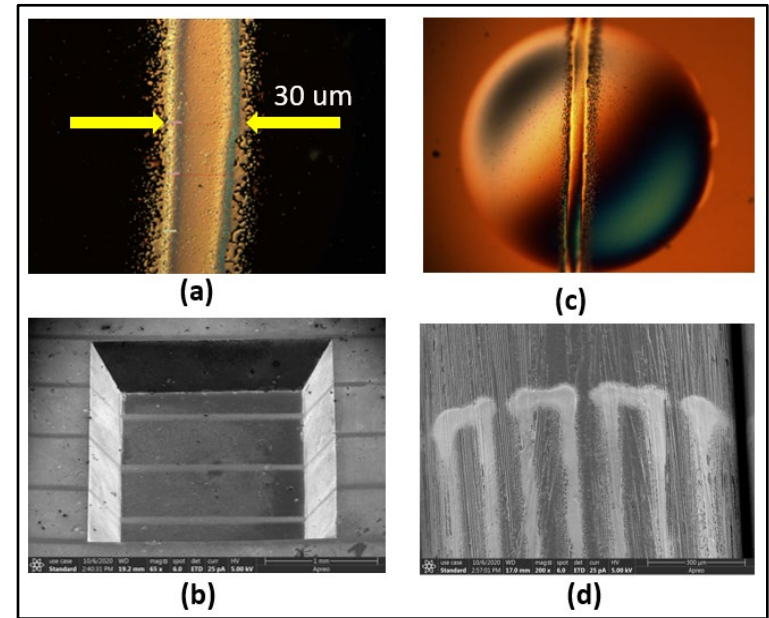
F. Balli, M. Sultan, and J. T. Hastings, University of Kentucky (ECE and Physics). Work performed at KY Multiscale CeNSE and EMC.

Supported by Intel Corp. *Optics Letters* 46(15):3548–3551 (2021).

National Research Priority: NSF–Growing Convergence Research

# Optomec Aerosol Jet Printing

Aerosol Jet printing is a maskless additive manufacturing solution that reduces the overall size of electronic systems by using nanomaterials to produce fine features and embedded components. The resulting functional electronics can have line widths and pattern features from 10 microns to several millimeters. The system can directly deposit a wide range of electronic materials, including conductive and nonconductive onto any substrate. Also, this system provides a wide range of possible applications that include flexible electronics and sensors which can be manipulated to meet the user's specific requirements. Figure shows some examples of narrow prints on different surface features. NovaCentrix silver ink was used throughout all printing and the process recipes were optimized/tuned by changing the sheath flow rate, atomizer flow rate, substrate material, stand-off distance and stage speed to change the morphology of the printed lines.



*30  $\mu\text{m}$  silver lines printed on (a) a glass slide, (b) a 300  $\mu\text{m}$  deep silicon micromachined cavity, (c) A 30  $\mu\text{m}$  tall fragile spherical buckled polyimide diaphragm, (d) A 1 mm diameter carbon fiber rod.*

Dilan Ratnayake, Alexander Thomas Curry and Kevin Walsh, Electrical and Computer Engineering, Univ. Louisville. Work was performed at KY Multiscale.

This research was supported by NSF NNCI and MRI grants (Award # 2025075 and 1828355). *IEEE Journal on Flexible Electronics (JFLEX)*, 2022. (pending).

*National Research Priority: NSF–Growing Convergence Research*

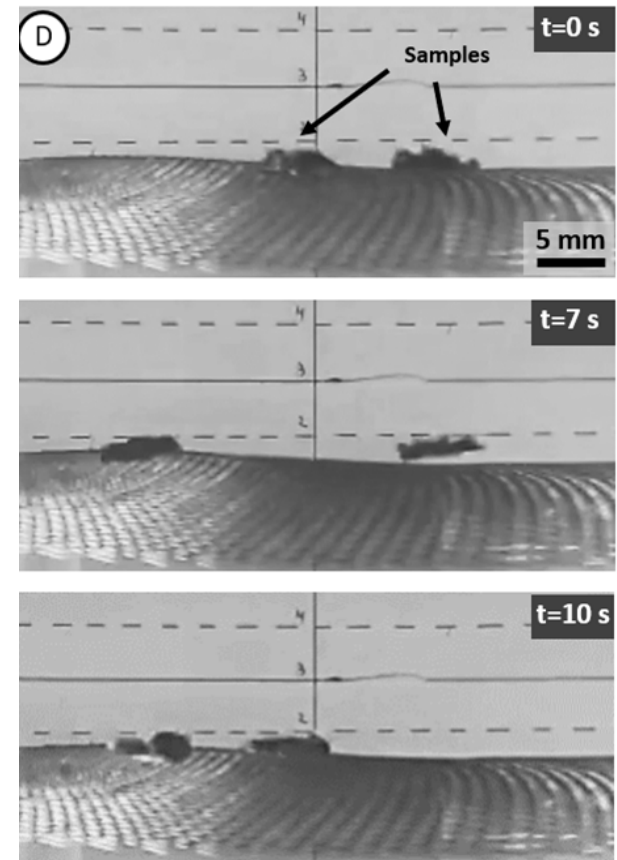
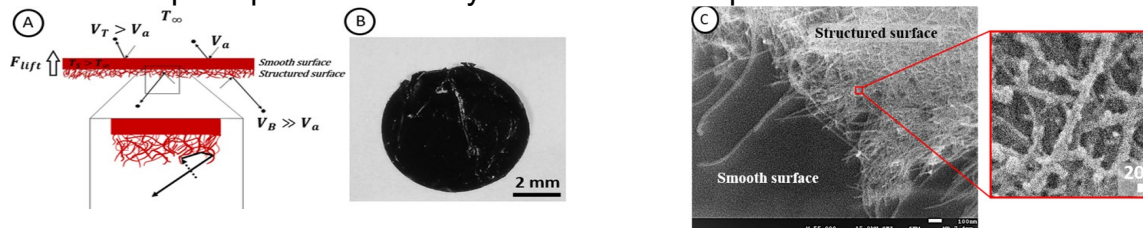
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# ***Mid-Atlantic Nanotechnology Hub (MANTH)***



# Levitation of Nanostructured Thin Films for Sun-Powered Near-Space Flight

- Rockets and aircraft cannot be used to achieve sustained flight in Earth's mesosphere—the upper layer of the atmosphere located at altitudes between ~50 and ~80 km. Photophoresis or light-driven motion is an alternative, no-moving-parts mechanism for sustained flight in near-space.
- MANTH researchers reported light-driven levitation of macroscopic polymer films with nanostructured surface as a candidate for long-duration near-space flight. They levitated centimeter-scale disks made of commercial 0.5-micron-thick mylar film coated with carbon nanotubes on one side.
- When illuminated with light intensity comparable to natural sunlight, the polymer disk heats up and interacts with incident gas molecules differently on the top and bottom sides, producing a net recoil force. They observed the levitation of 6-mm-diameter disks in a vacuum chamber at pressures between 10 and 30 Pa. The lift forces can be many times the weight of the films, allowing payloads of up to 10 milligrams for sunlight-powered low-cost microflyers at altitudes of 50-100 km.
- This work, published in *Science Advances* demonstrated a new approach to photophoretic levitation of macroscopic structures that does not require a temperature gradient within the object, offering a path to the development of affordable photophoretic microflyers for the mesosphere.



Igor Bargatin Group, Mechanical Engineering and Applied Mechanics, Univ. Pennsylvania, and in collaboration with Prof. Howard Hu. Work performed at MANTH.

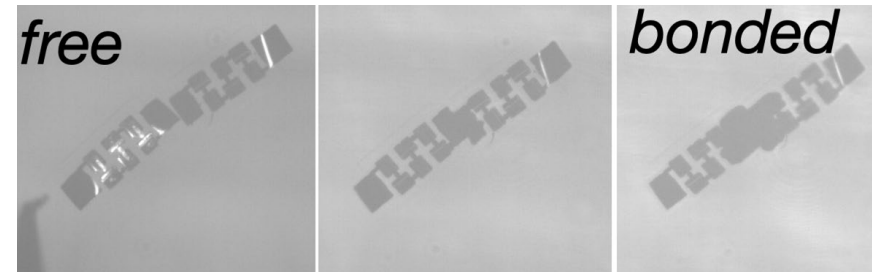
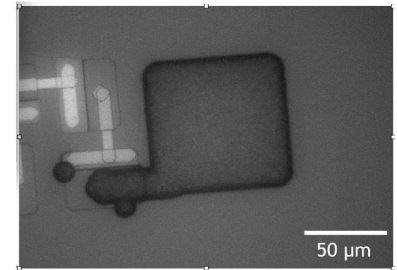
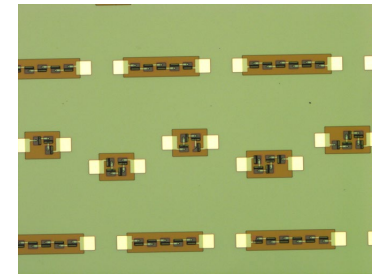
# Microrobots for Multifunctional Metallic Metamaterials

- Materials are rarely strong *and* adaptable. For example, engineered materials offer impressive figures of merit, but cannot heal or reconfigure. Conversely biological materials offer robustness and reconfigurability, but at lower performance.
- This work attempts to bridge this gap by creating a new material; one filled with microscopic robots capable of electroplating metal. The robots, in solution, electrodeposit nickel on their bodies with local application of energy. By changing the volume of nickel plated, the material could actively adapt its structural properties.
- Currently, they have demonstrated that electrodeposition of nickel on microscale electrodes using light energy harvested by onboard photovoltaics is achievable, and that the metal grown on individual robots can be merged to form bonds between robots and the surrounding matrix.
- Each robot is made massively in parallel using fully top-down lithographic techniques at the MANTH Singh Center for Nanotechnology: nearly 1 million robots can be made on a single 4-inch silicon wafer.
- These results invite studies on a new breed of metamaterial that combines some of the best features of metallic composites with the adaptability of biological systems.

*Right: An array of electroplating microrobots.*

*Center: robot plating metal on its electrode. Note the power transfer here is totally wireless: each robot converts energy from light into metal by way of the on-board photovoltaic cells.*

*Bottom: Nearby robots can bond together when plating, forming a solid block of interconnected metal.*

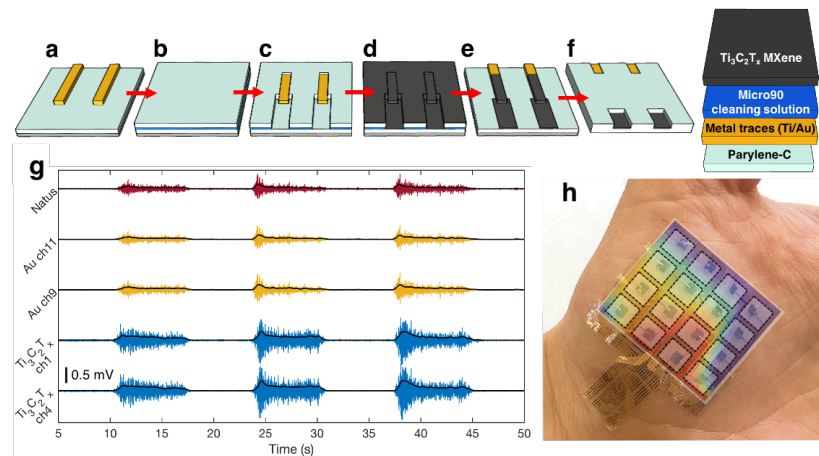


Marc Miskin group at Univ. Penn. Work performed at MANTH.

Funded by the AFOSR, grant FA9550-21-1-0313. Presented at APS, March 2022

# $Ti_3C_2T_x$ High-Density, High-Resolution Electromyograph Arrays

- High-density arrays of high-resolution electrodes are a valuable tool for recording the electromyogram (EMG), a biopotential representing the activation and coordination of various muscles groups within the human body. To achieve a higher spatial resolution and channel count, smaller electrodes are required, and it becomes challenging to maintain a low interface impedance and a high signal-to-noise ratio.
- Two-dimensional titanium carbide MXene ( $Ti_3C_2T_x$ ) possesses remarkably high volumetric capacitance and electrical conductivity, excellent mechanical properties, and a high degree of surface functionality, and it is easy to process in aqueous solutions. Leveraging these many advantages, devices were made by a novel microfabrication process, realizing high-density, thin, and flexible  $Ti_3C_2T_x$  arrays for surface EMG recording.
- They demonstrate superior performance in EMG recordings on healthy human volunteers. These results establish high-density  $Ti_3C_2T_x$  MXene arrays for recording high-fidelity, low-noise EMG, with applications in rehabilitation and sports medicine, and assistive technologies.



*The process flow used to microfabricate high-density  $Ti_3C_2T_x$  MXene EMG arrays at MANTH; sample traces of the EMG signals recorded using the MXene array; and demonstration of the high-density  $Ti_3C_2T_x$  MXene EMG array for millimeter-resolution recording of muscle activation.*

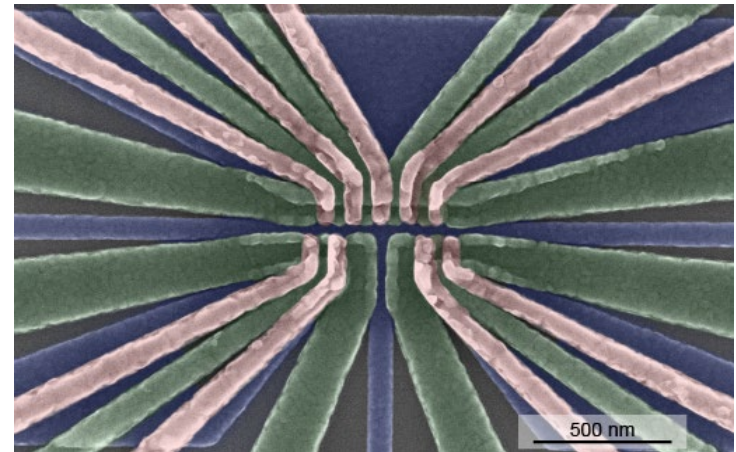
Flavia Vitale, Penn Department of Neurology, Bioengineering and Physical Medicine and Rehabilitation, with Brendan B. Murphy, Patrick J. Mulcahey, Nicolette Driscoll, Andrew G. Richardson, Gregory T. Robbins, Nicholas V. Apollo, Kathleen Maleski, Timothy H. Lucas, Yury Gogotsi, and Timothy Dillingham, Penn Department of Bioengineering, Department of Neurosurgery, Penn Medicine, and the Materials Science and Engineering Department at Drexel University. Work performed at MANTH.

National Research Priority: NSF–Understanding the Rules of Life



# Scalable Quantum Architectures in Silicon

- The group led by Prof. Anthony Sigillito at Penn is developing new scalable architectures for quantum dot spin qubits in Si/SiGe. These devices can be thought of as arrays of nanoscale transistors, each with a single electron accumulated under its gate. Quantum information is encoded in the magnetic moment of each electron.
- Hughes Research Laboratories provides the isotopically enriched Si/SiGe quantum wells and fabrication is done at MANTH. The process requires 10 layers of aligned optical lithography followed by four layers of aligned ebeam lithography (now done at UDel). The tight overlay accuracy requirements are to be met with the **new Raith EBPG system** which will be installed in the QNF next year.
- The group recently demonstrated high-fidelity multi-qubit operation in small scale processors and the hope is that once a few outstanding control challenges are overcome, they can leverage mature silicon processing technologies to scale up these systems.
- The false-color SEM image of a device (fabricated at Princeton University) on the right is similar to the devices now fabricated at MANTH.



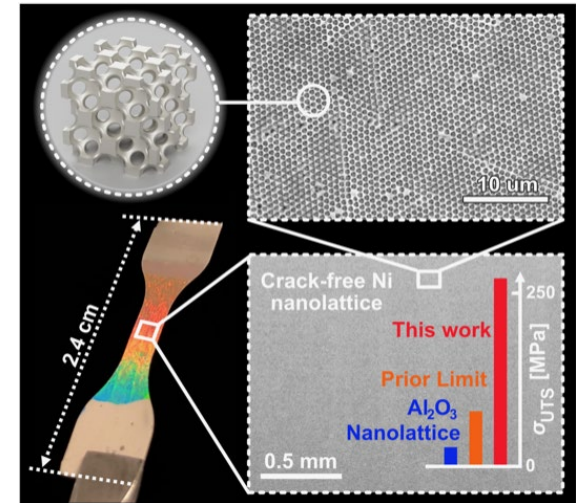
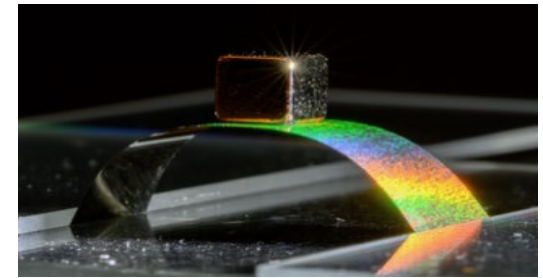
Students of Prof. Anthony Sigitto, in collaboration with researchers from HRL and using EBL equipment from U Delaware. Work partially performed at MANTH.

*National Research Priority: NSF–Quantum Leap*



# Crack-free Self-assembly for Ultra-high Tensile Strength Metallic Nanolattices

- Strong and lightweight porous materials are commonly used in industry, but the difficulties in controlling their physical and chemical structures during fabrication have limited their mechanical properties. Nanolattices are porous materials with nanoscale features that promise to overcome these limitations using size-based effects
- Dense cracks in self-assembled lattice templates, limiting characterization of these materials' properties to be under sub-millimeter scale.
- To eliminate template cracks and precisely control metallic nanostructure, researchers at Penn have leveraged the capabilities at MANTH to develop a self-assembly method for fabricating centimeter-scale nickel nanolattices.
- The large-area nickel nanolattices had an ultra-high tensile strength 2.6 times that of prior porous metals and achieved a combination of strength and relative density that may enhance the performance of many applications such as sensing, high-power-density batteries, efficient heat and mass exchangers and selective infiltration membranes.



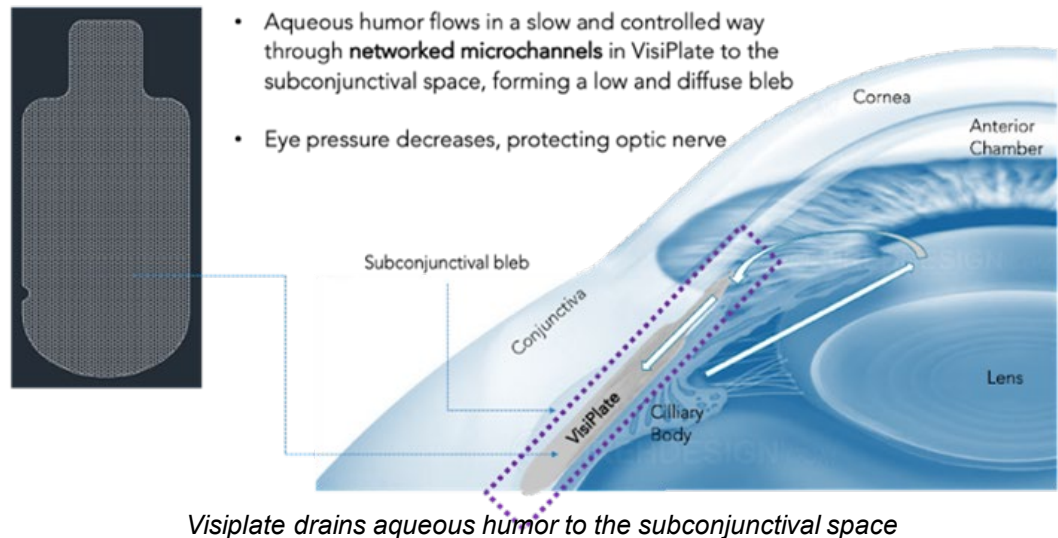
Zhimin Jiang and James H. Pikul. Work performed in part at MANTH.

Funding from the pilot grant Program from the Center for Innovation and Precision Dentistry at the University of Pennsylvania, NSF CAREER Grant, ASME Applied Mechanics Division Haythornthwaite Foundation Research Initiation Grant, and NSF NNCI.

*National Research Priority: DoD Critical Technology Area—Advanced Materials*

# Industry User Research

- Avisi Technologies, Inc., founded in March 2017, is an early-stage, medical device company developing VisiPlate, a nanoscale ocular implant for treating glaucoma. Open-angle glaucoma (OAG) is the second leading cause of blindness in the world and leads to vision loss that cannot be cured or reversed. OAG will affect 3.4m people in the U.S. and 80m people globally.
- In OAG patients, excess fluid builds up inside the eye, putting pressure on and permanently damaging the optic nerve, leading to blindness.
- VisiPlate is a nanotechnology-enabled ultra-thin drainage implant that creates a secondary drainage pathway for this excess fluid. VisiPlate leverages a shape-recovering, mechanically robust nanotechnology invented by the University of Pennsylvania and licensed to Avisi with an exclusive field of use in ophthalmology.
- Avisi has developed VisiPlate prototypes at MANTH for all early feasibility work. This development has led to \$1.7M in Phase 1 and Phase 2 SBIR grants from the National Science Foundation and \$1.6M in additional seed investment.



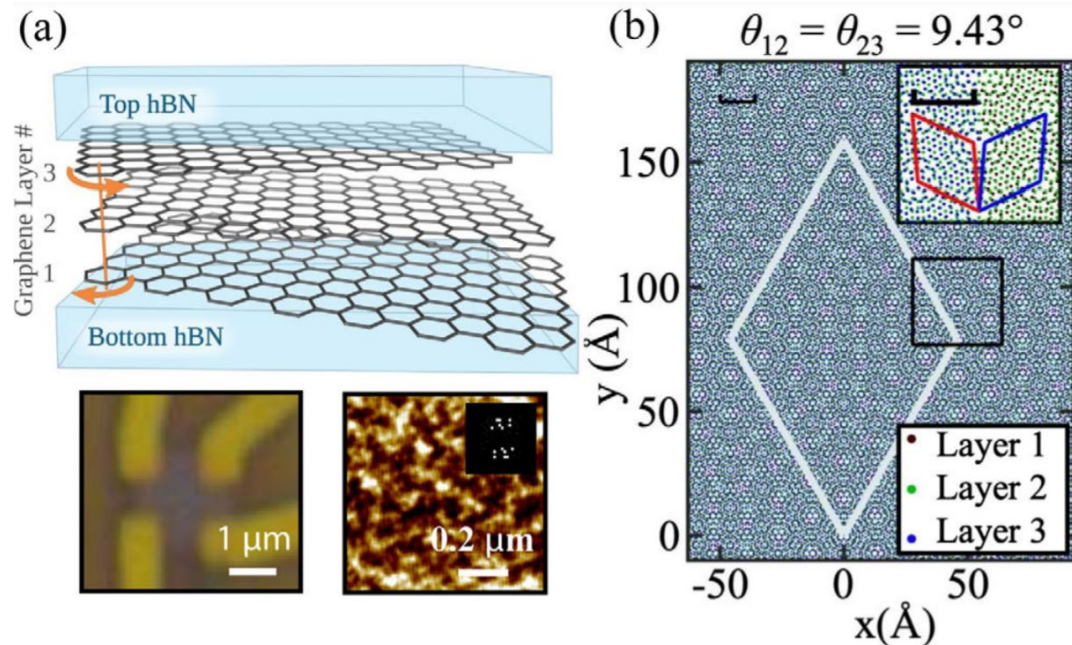
*National Research Priority: NSF–Understanding the Rules of Life*

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# ***Midwest Nanotechnology Infrastructure Corridor (MiNIC)***

# Correlated Insulating States in Twisted Graphene Multilayers

Two-dimensional (2D) materials stacked with a small twist angle give rise to beating periodic patterns on a scale much larger than the original lattice, referred to as a moire superlattice. In this work, transport in twisted trilayer graphene with two consecutive small twist angles is studied. Correlated insulating states are observed at an extremely low carrier density ( $\sim 10^{10} \text{ cm}^{-2}$ ), near which a zero-resistance transport behavior is observed, indicative of a possible superconducting state.



Twisted graphene multi-layer structure and resulting moire pattern at a twist angle of  $9.43^\circ$ .

Ke Wang group at Minnesota, with contributors from Minnesota and Harvard. Fabrication performed in the MNC as part of the Midwest Nano Infrastructure Corridor (MiNIC).

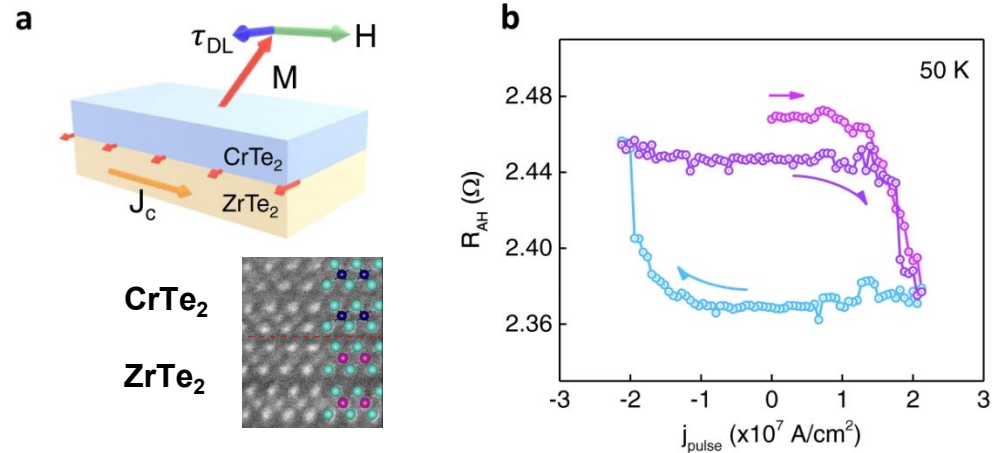
Supported by an ARO MURI and other awards. *Phys. Rev. Lett.* **127**, 166802 (2021).

National Research Priority: NSF–Quantum Leap



# Epitaxial van der Waals Spintronics Platform

Synthesis of van der Waals (vdW) heterostructures with well-controlled interfaces is an attractive route towards novel device concepts. In this work, molecular-beam epitaxy (MBE) was used to synthesize a vdW heterostructure which merged a 2D ferromagnet ( $\text{CrTe}_2$ ) with a topological semi-metal ( $\text{ZrTe}_2$ ). It was found that one unit-cell thick 1T- $\text{CrTe}_2$  grown by MBE on  $\text{ZrTe}_2$  is a 2D ferromagnet, while ultrathin  $\text{CrTe}_2$  can show current-driven magnetization switching. These results are encouraging for realizing novel spintronics devices in a wafer-scale VdW platform.



Left: Schematic of the spin-orbit torque assisted magnetization switching in the  $\text{ZrTe}_2/\text{CrTe}_2$  heterostructure and XTEM of the structure. Right: Pulse current-induced magnetization switching of a  $\text{ZrTe}_2/\text{CrTe}_2$  heterostructure at 50 K.

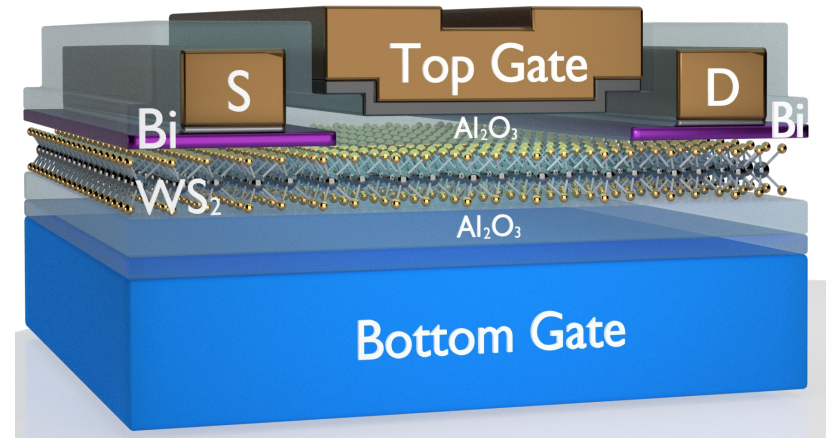
Samarth group at Penn State. TEM performed in the CharFac as part of the Midwest Nano Infrastructure Corridor (MiNIC).

Work supported by multiple awards, including SMART, one of seven centers of nCORE, a Semiconductor Research Corporation program supported by several industrial partners and NIST. *Nat. Commun.* **13**, 2972 (2022).

National Research Priority: Supporting Large Multi-Institution Centers

# $WS_2$ MOSFETs with Semi-Metallic Bi Contacts

Two-dimensional (2D) materials such as  $WS_2$  are promising candidates for future applications in sub-1-nm-node CMOS technology because their thickness can be reduced to a single atomic layer. However, formation of low-resistance contacts to these materials has been an ongoing problem. In this work, semi-metallic bismuth (Bi) contacts were used to realize dual-gated, single-layer  $WS_2$  MOSFETs with very high drive current and low contact resistance. The results are particularly promising since the  $WS_2$  was grown using chemical vapor deposition (CVD), a technique compatible with synthesis on large-area substrates.



Schematic 3D cross-sectional diagram of dual-gated  $WS_2$  MOSFET with Bi contacts. The channel consisted of single-layer  $WS_2$  with thickness  $< 1$  nm, and was grown by chemical vapor deposition (CVD).

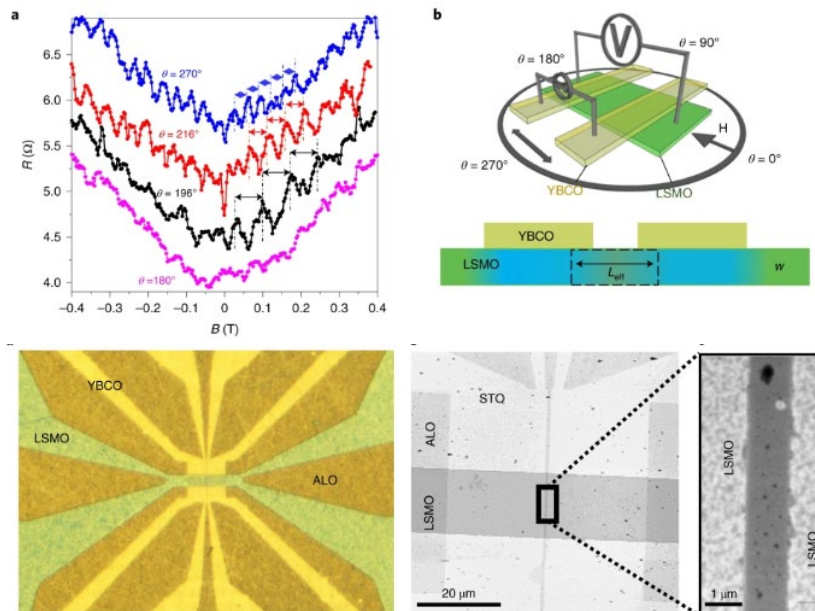
Koester group at the University of Minnesota. Fabrication performed in the MNC as part of the Midwest Nano Infrastructure Corridor (MiNIC).

Work supported by Intel Corporation. *IEEE Elect. Dev. Lett.* **43**, 639-642 (2022).

*National Research Priority: CHIPS+Science Act Research*

# Extremely Long-Range Josephson Coupling Across a Half-Metallic Ferromagnet

Superconducting coupling between two regions separated by a one micron wide ferromagnetic material was shown to exist at high temperature (tens of Kelvins). Such a long-range macroscopic quantum interaction, called the Josephson effect, in junctions with ferromagnetic spacers had remained elusive until this work. The results pave the way for low-power superconducting / spintronic applications where spin-polarized currents can be protected by quantum coherence. This highlight provides an excellent example of how the expert CharFac staff facilitate research from international collaborators.



Top: Field modulation of Josephson current depending on the relative direction between the current and the magnetic field.

Bottom: Micrograph of the planar Josephson junctions.

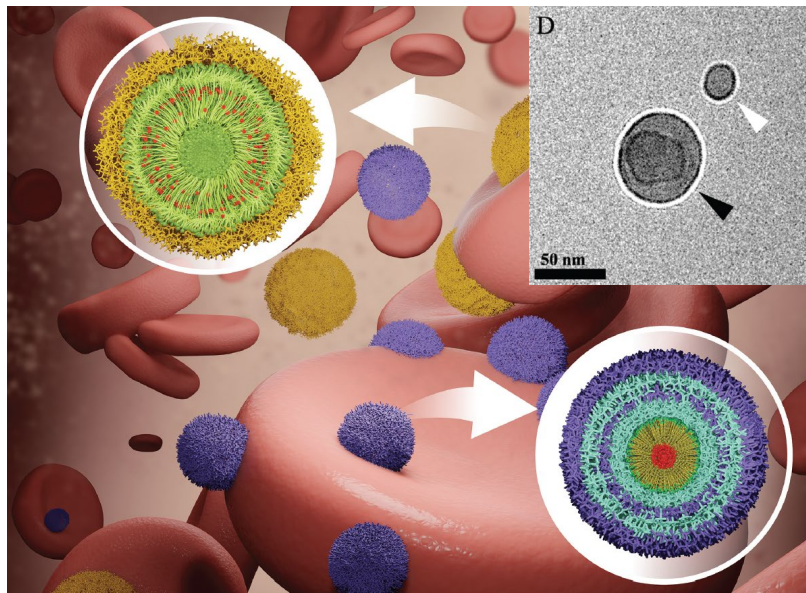
Collaboration between CharFac scientist Dr. Javier Garcia-Barriocanal and the research group of Dr. Jacobo Santamaria Complutense University of Madrid. Work performed in part at MiNIC.

*Nat. Mater.* **21**, 188-194 (2022).

National Research Priority: International Collaboration

# Bio-Functionalization of Block Copolymers with Biocompatible Ionic Liquids

Linear-dendritic block copolymers (LDBC)s are promising for using as encapsulants in both hydrophobic and hydrophilic dyes for bioimaging, cancer therapeutics, and small biomolecules. However, LDBC)s also can exhibit high dispersities, poor shelf-life, and high cytotoxicity to non-target blood cells. This work reports the use of ionic liquids to modify linear and linear-dendritic block copolymer nanoaggregates for use in drug delivery and improve their performance. Cryo-TEM analysis suggests this improvement is due to formation of a nanoparticle surface coating, which protects against red blood cell hemolysis. The results show that by controlling the nanoscale physical chemistry, biological function can be more precisely tailored.



*Schematic diagram of poly(lactic-co-glycolic acid) (PLGA) particles. Inset: Black arrowhead shows a 50-nm PLGA particle surrounded by an ionic liquid shell. The white arrowhead points to a 20-nm ionic liquid nanodroplet.*

Tanner Lab at the University of Mississippi. Cryo-TEM performed in the CharFac as part of the Midwest Nano Infrastructure Corridor (MiNIC).

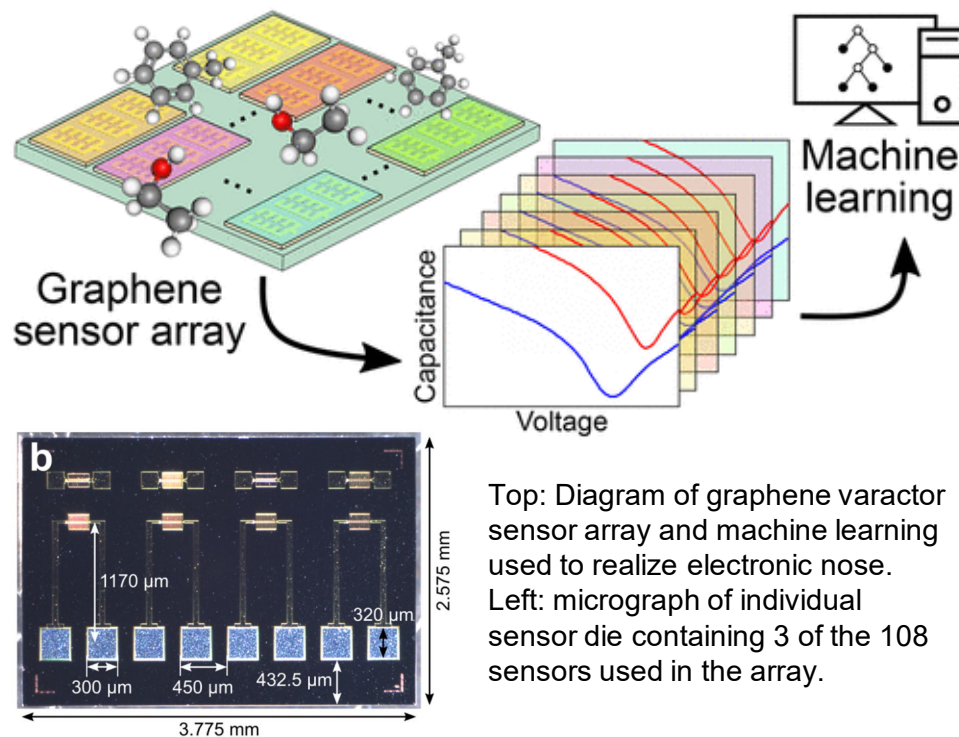
Work supported by the College of Liberal Arts at the University of Mississippi. *Nanoscale* **14**, 6021-6036 (2022).

*National Research Priority: NSF–Understanding the Rules of Life*



# Rapid Detection of Volatile Organic Compounds in a Graphene Electronic Nose

Electronic noses are important vehicles for rapid detection of volatile organic compounds (VOCs) for detection of a variety of disease states in human breath. In this work, a large array of graphene sensors is used to selectively and rapidly detect multiple VOCs. Each array contains 108 sensors functionalized with 36 chemical receptors for cross-selectivity. A supervised machine learning algorithm shows excellent results of 98% accuracy between 5 analytes at four concentrations each. This is an important step toward fully utilizing graphene-based sensor arrays for rapid gas sensing for disease detection in human breath.



Collaboration between groups at the University of Minnesota and Boston Scientific Corporation. Fabrication partially performed in the MNC as part of the Midwest Nano Infrastructure Corridor (MiNIC).

ACS Nano **16**, 19567-19583 (2022).

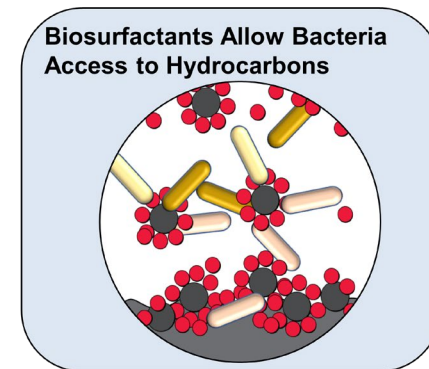
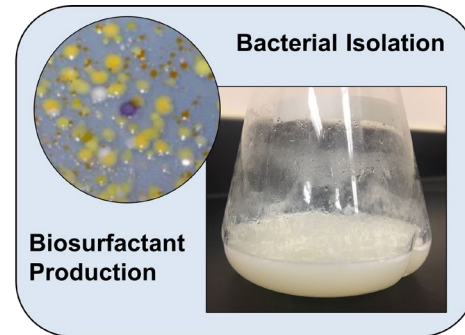
National Research Priority: NSF–Understanding the Rules of Life

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# ***Montana Nanotechnology Facility (MONT)***

# Determination of Biosurfactant Producing Microbes for Biotechnological Applications

- Surfactants have many valuable industrial and remediation applications. Unlike synthetic surfactants, biosurfactants are naturally produced by microorganisms, low in toxicity, and more eco-friendly and biodegradable. To date, many natural environments, such as those in cold climates, have been largely overlooked in the search for biotechnologically relevant organisms. To fill this gap, cold temperature microorganisms previously gathered by the Foreman Research Group in Antarctica were screened for their biosurfactant producing ability.
- We identified 16 prominent biosurfactant producing organisms with strong oil displacement and emulsification properties. Using mass spectrometry these were putatively identified as surfactins, sophorolipids, and di- & mono-rhamnolipids.



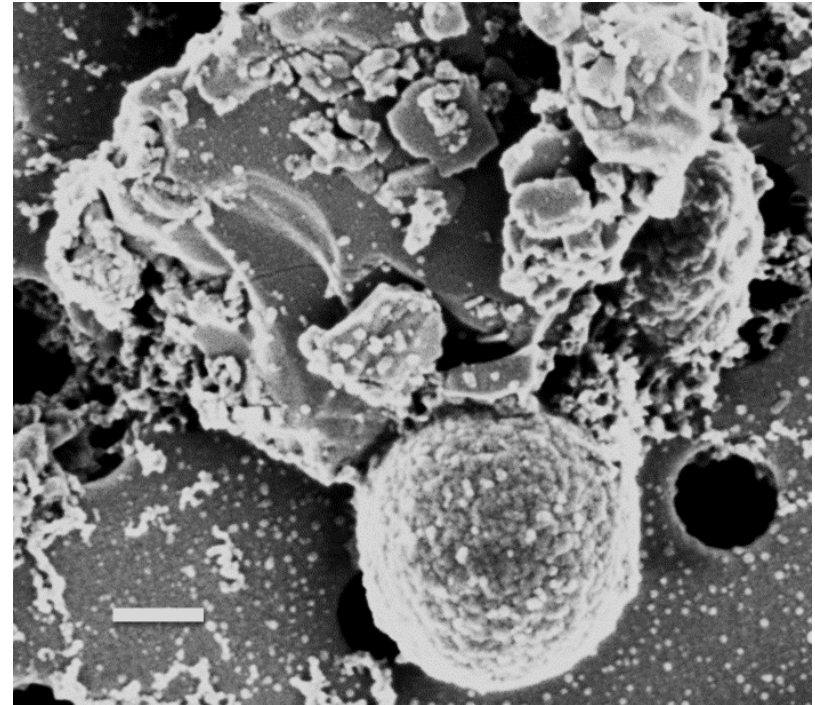
Mass Spectrometry putatively Identified these as surfactins, sophorolipids, dirhamnolipids and monorhamnolipids

Aspen Burke, Markus Dieser, Donald Smith and Christine M. Foreman, Montana State University. Work performed at Montana State University, Center for Biofilm Engineering and Proteomics, Metabolomics and Mass Spectrometry Facility.

*National Research Priority: NSF–Navigating the New Arctic*

# Reductive Dissolution of Pyrite by Methanogenic Archaea

Pyrite ( $\text{FeS}_2$ ) is the most abundant sulfide mineral in Earth's crust and hosts a number of trace metals (nickel, cobalt) of economic importance. Traditional methods to oxidatively mine pyrite ores can lead to acid mine drainage, which can negatively impact ecosystems. Here, we investigate anaerobic reductive dissolution of  $\text{FeS}_2$  by methanogenic archaea.  $\text{FeS}_2$  reduction requires direct contact with  $\text{FeS}_2$  grains, as depicted in the electron micrograph. Reductive dissolution of  $\text{FeS}_2$  yields aqueous metal clusters that are assimilated to meet biosynthetic demands. Further, methanogens growing on  $\text{FeS}_2$  hyperaccumulate Fe as intracellular Fe-S containing nanoparticles. As such, biological reduction of  $\text{FeS}_2$  is an emerging technology for concentrating elements in mining operations and natural waters and is currently being optimized for this purpose.



Electron micrograph of *Methanococcus voltae* attached to grain of specimen pyrite. Scale bar = 200 nm.

Devon Payne, Rachel L. Spietz and Eric S. Boyd. Montana State University. Work performed at Montana State University, Imaging and Chemical Analysis Laboratory.

Funded by the DOE Geosciences and Physical Biosciences programs through EPSCoR. DE-SC0020246. *ISME J*, **2021**, 10.1038/s41396-021-01028-3; *J Bac*, **2021**, 10.1128/jb.00146-21; *Front. Microb.*, **2022**, 10.3389/fmicb.

National Research Priority: NSF–Growing Convergence Research



# Correlative Microscopy

The Hatzenpichler lab in the Department of Chemistry and Biochemistry at Montana State University has developed a new correlative microscopy workflow that combines fluorescence microscopy, electron microscopy, Raman microspectroscopy, and nano-scale secondary ion mass spectrometry imaging of the same cell.

A correlative microscopy image created by graduate student George Schaible graces the cover of the 2022 volume of The ISME Journal, the premier journal in microbial ecology. Impact factor: 12.28 (2020).

Another image of George Schaible was selected to cover the 7<sup>th</sup> edition of the microbiology textbook *Microbiology: an evolving science*.

*Correlative microscopy image generated at ICAL graces the 2022 volume of The ISME Journal.*

The **ISME** Journal  
Multidisciplinary Journal of Microbial Ecology



SPRINGER NATURE

ISME

Schaible G, Kohtz AJ, Cliff J, Hatzenpichler R, Montana State University and Pacific Northwest National Laboratory. Work performed at Montana State University, ICAL

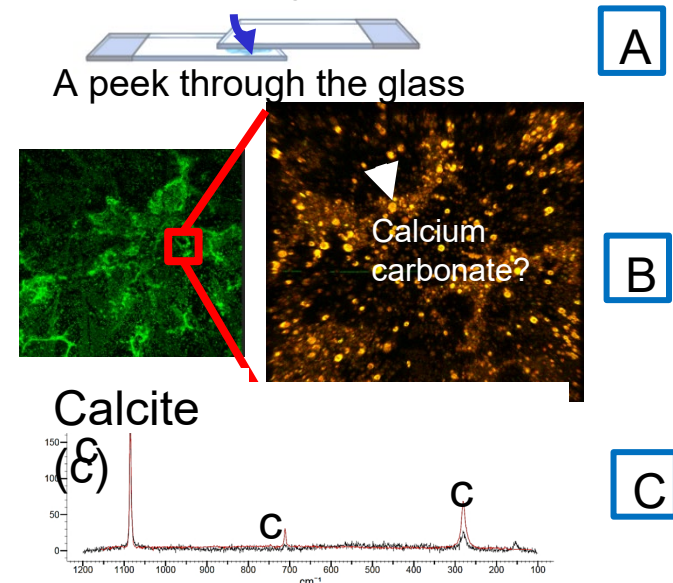
Funding provided by NASA Exobiology (NNX17AK85G), FINESST (80NSSC20K1365 ), Gordon and Betty Moore Foundation (5999). *ISME Commun.* **2**, 52 (2022).

*National Research Priority: NSF–Growing Convergence Research*

# Microbially Induced Calcium Carbonate Composite (MICC) Adhesives

Bio-adhesives are currently being explored as an alternative to currently available volatile organic compound-based adhesives, which can be harmful to the environment and human health. The microbially induced calcium carbonate based composite adhesive being developed in the MSU-Center for Biofilm Engineering Bioprocess laboratory is one such alternative. The bacterial cells used here are *Sporosarcina pasteurii* that carry out ureolysis, which generates carbonate ions, thus inducing calcium carbonate precipitation. The precipitates consist of calcium carbonate, bacterial cells, their extracellular matrix, and organics added to the composite mixture. This composite is being explored as an adhesive and construction binder, and its components are being characterized using instruments available in the MONT facility.

Calcium carbonate-based bioadhesive applied between two glass slides



An MICC adhesive is applied between two glass slides (A). Calcium carbonate precipitates are visualized through their autofluorescence using confocal laser scanning microscopy (B). The presence of calcium carbonate is confirmed using Raman microspectroscopy (C).

Sobia Anjum, Kendall Parks, Kaylin Clark, Robin Gerlach, Montana State University. Work performed at Montana State University, MONT facility, and Bioprocess Laboratory at the Center for Biofilm Engineering.

This work was supported by NSF grants #2036867, #1736255, and NNCI.

National Research Priority: NSF–Understanding the Rules of Life

NSF 2026–Engineered Living Materials and Unlocking the Future of Infrastructure

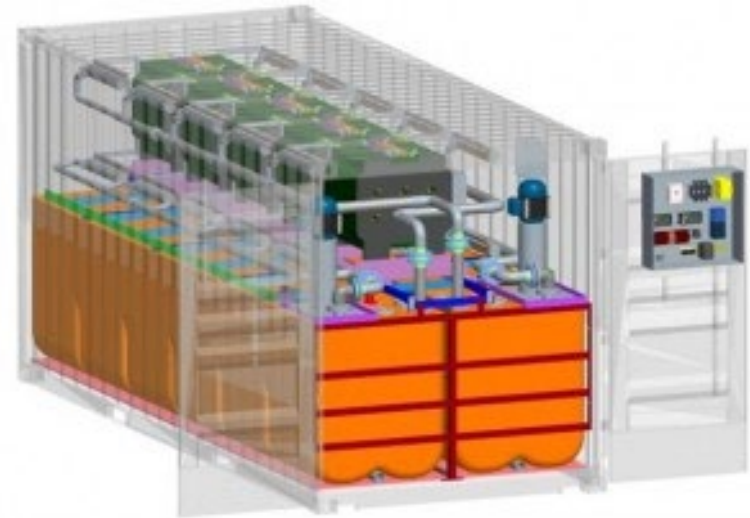
# Hybrid Flow Battery Technology Optimization

ViZn Energy Systems, Inc. utilizes hybrid flow battery technology to store electrical energy for grid-scale applications.

Thanks to MSU's ICAL laboratory, ViZn has utilized SEM and EDS analysis to better optimize the performance and efficiency of our batteries.

Furthermore, useful discoveries made in ICAL's analysis on specific ViZn components have allowed for preemptive planning on future production designs which may have been otherwise overlooked.

ViZn is grateful for the work performed at ICAL and plans to regularly utilize their services in the future.



*An ViZn energy-storage unit (ESU) outlining the flow-battery components like electrolyte tanks, plumbing, controls, and reaction-site stacks.*

David Buckingham, ViZn Energy Systems, Inc. Work performed at Montana State University, Imaging and Chemical Analysis Laboratory.

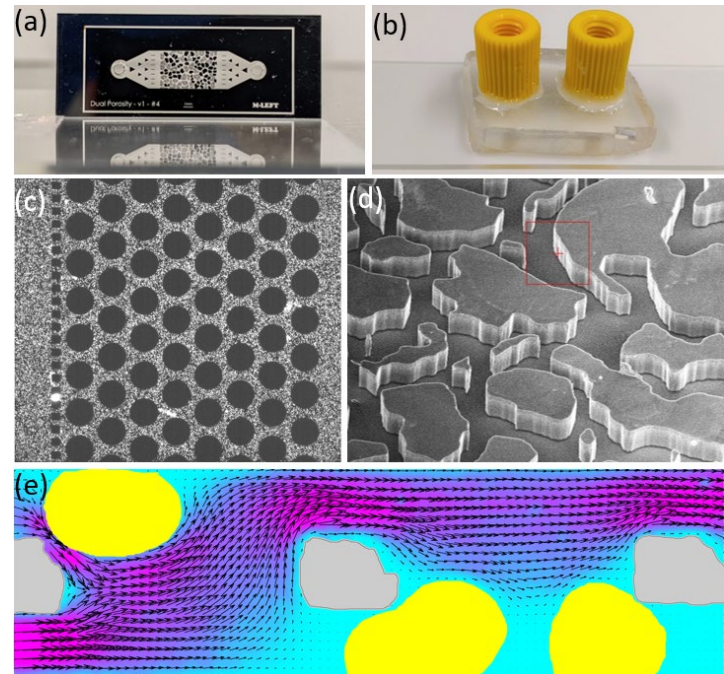
*National Research Priority: NAE Grand Challenge—Make Solar Energy Economical*

# Fabrication of Novel Microfluidic Devices

Goal: our goal is to fabricate novel microfluidic devices that allow us to study and sense fluid and energy transport at microscales.

Results: With Montana Microfabrication Facility (MONT) we are able to design and fabricate a series of novel devices, including silicon-based microchannels, three-layer glass-silicon-glass porous micromodels, calcite-based microchannel and PDMS-based pressure sensors (see Figure 1 on right). The processes used include photolithography, soft lithography, etching, etc.

Impact: These projects are relevant to a broad range of applications such as heat pipes, coating, inkjet printing, thermal management of electronics, oil recovery and CO<sub>2</sub> sequestration



Various microfluidic devices fabricated at MONT (a, b, d), and some representative flow measurements (e, d).

Razin Molla, Rafid Rahman, Elliott Niemus, Kshithij Nandishwara, and Diego Armstrong, Montana State University. Work performed at Montana State University, Montana Microfabrication Facility

Funded by PRF 62687-DNI9; NSF ECCS-1542210. Frontiers in Water 3 (2021); ISPIV, Chicago, IL (2021)

National Research Priority: NSF–Growing Convergence Research



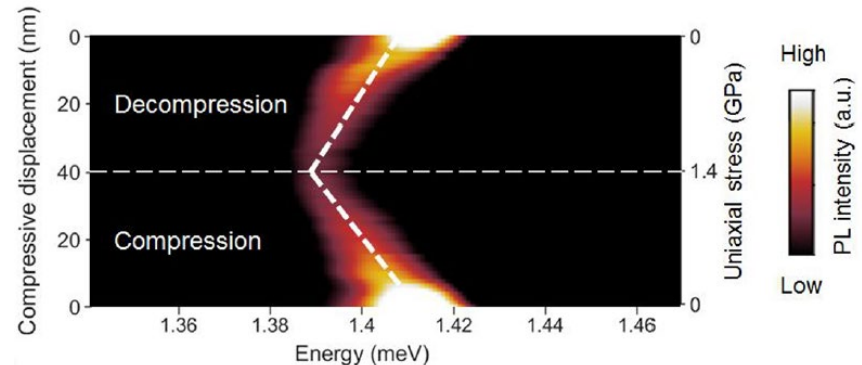
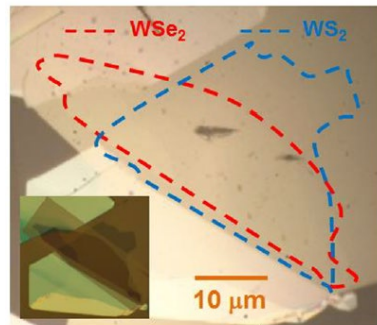
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# ***Nanotechnology Collaborative Infrastructure Southwest (NCI-SW)***

# Tuning of Moiré Excitons in a WSe<sub>2</sub>/WS<sub>2</sub> Heterostructure via Mechanical Deformation

We demonstrate a novel way to dynamically tune Moiré excitons in a WSe<sub>2</sub>/WS<sub>2</sub> heterostructure. The sharp tip of a cryogenic scanning near-field optical microscope allows us to apply mechanical stress and perform background-free near-field spectroscopy simultaneously. We observe a red shift of both the intralayer and interlayer excitons which corresponds to a deformation induced modulation of the Moiré potential coefficient by 7 meV. Our study of Moiré excitons under deformation can be extended to other 2D materials such as twisted graphene and graphene-hBN superlattices, opening the door to electronic and optical studies of deformed 2D materials.

Transmission electron microscopy detected protein particles recovered from crosslinked HEK293T/17 cells by IP of EWS-FLI1 (anti-FLI1 antibody, left) and RNA Pol II (CTD4H8, left), but not of YS37 (anti-FLI1 antibody, center). Scale bar inset represents 50 nm.



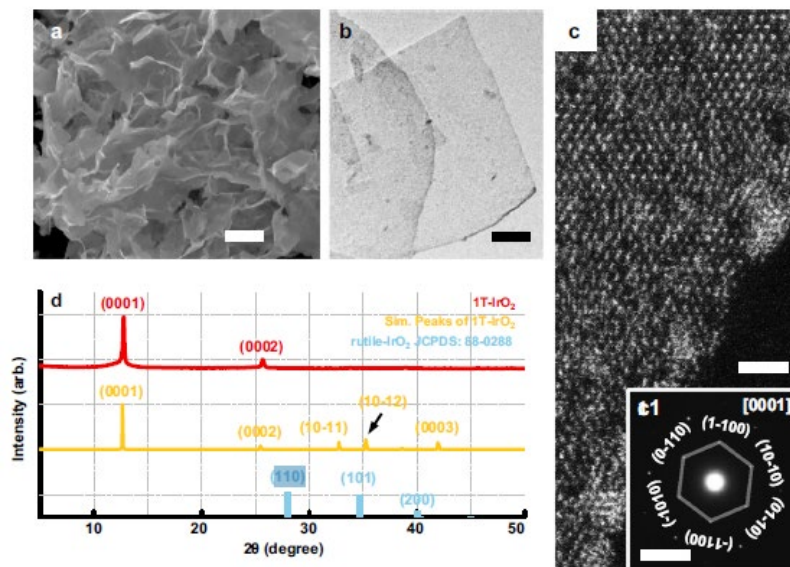
Feng Wang, Department of Physics, University of California at Berkeley. Work was performed at NCI-SW.

This work was supported by the U.S. Department of Energy under contract number DE-AC02-05CH11231. *Nano Letters*, vol.21, pp. 8910 -8916 (2021)

National Research Priority: NSF-Quantum Leap

# Iridium metallene oxide for acidic oxygen evolution catalysis

Exploring new materials is essential in the field of material science. Especially, searching for optimal materials with utmost atomic utilization, ideal activities and desirable stability for catalytic applications requires smart design of materials' structures. Herein, we report iridium metallene oxide by a synthetic strategy combining mechanochemistry and thermal treatment in a strong alkaline medium. This material demonstrates high activity for oxygen evolution reaction with a low overpotential. Theoretical calculations reveal that the active site of Ir IrO<sub>2</sub> provides an optimal free energy uphill in \*OH formation, leading to the enhanced performance. The discovery of this new metallene oxide material will provide new opportunities for oxygen evolution catalysis.



Structural characterizations of IrO<sub>2</sub>. a) The SEM and b) TEM images of IrO<sub>2</sub>, representing ultrathin morphology. c) The aberration-corrected STEM image of monolayer IrO<sub>2</sub>. The hexagonal pattern is indicative of the [0001] projection

Dang et al., College of Chemistry, Soochow University, Jiangsu, P. R. China. Work was performed at NCI-SW.

This work was financially supported by Development Program of China contract number 2017YFA0204800. *Nature Communications*, vol.12, article 6007 (2021)

National Research Priority: DOE Energy Storage Grand Challenge

# ***Fusion protein EWS-FLI1 is incorporated into a protein granule in cells***

Ewing sarcoma is a rare type of cancer that occurs in bones or in the soft tissue around the bones. Its is driven by fusion proteins containing a low-complexity domain that is intrinsically disordered and a powerful transcriptional regulator. The most common fusion protein found in Ewing sarcoma is EWS-FLI1. We find that EWS-FLI1 can raise transcript levels closer to that observed in Ewing sarcoma cells, where transcript levels can be further increased by removal of EWS-FLI1. This may indicate more than one mechanism can drive and moderate expression of EWS-FLI1 targets in Ewing sarcoma. Further investigations of cells that thrive without EWSR1 may reveal which critical functions must be restored for EWS-FLI1 to continue to sustain tumor growth and help identify new factors involved.

*Transmission electron microscopy detected protein particles recovered from crosslinked HEK293T/17 cells by IP of EWS-FLI1 (anti-FLI1 antibody, left) and RNA Pol II (CTD4H8, left), but not of YS37 (anti-FLI1 antibody, center). Scale bar inset represents 50 nm.*



Jacob Schwartz, Department of Chemistry and Biochemistry, The University of Arizona. Work was performed at NCI-SW.

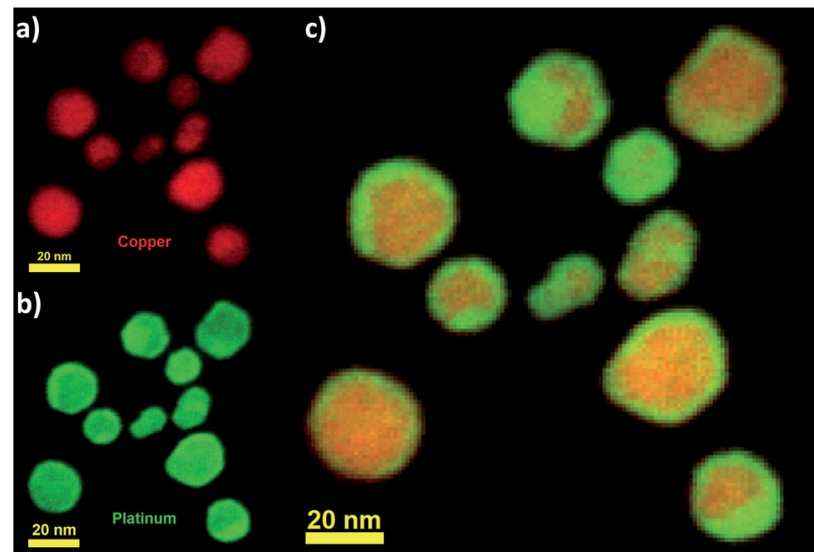
This work was supported by funding from the National Institutes of Health award no. R21CA238499. *RNA.*, vol.27, pp. 920 - 932 (2021)

*National Research Priority: NAE Grand Challenge—Engineer Better Medicines*



# Lowering the Cost of Platinum Catalysis Using Cu-Pt Nano-Alloys

Platinum is an important catalyst used to produce nitric acid, benzene, and to improve the efficiency of fuel cells. However, its low abundance in the Earth's crust makes its price high and accessibility at risk. Therefore, strategies to reduce the amount of platinum in applications are welcome. In this work, we demonstrate that it is possible to preserve the catalytic properties of platinum with a copper–platinum alloy by converting the substitutional alloy into an interstitial one. This conversion occurs when the size of the copper–platinum system is reduced to the nanoscale. Experimentally, the electron microscopy characterization of copper–platinum alloyed nanoparticles synthesized by wet chemistry supports the predicted structural transition.



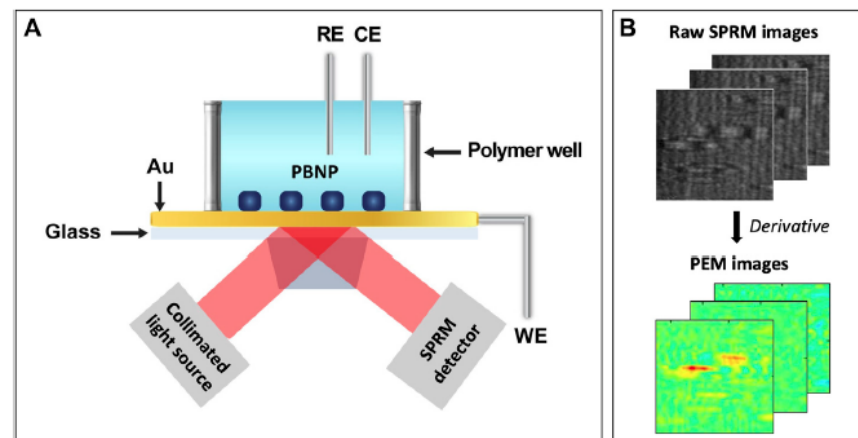
*Chemical mapping of Cu–Pt nanoparticles acquired using electron energy loss spectroscopy (EELS).*

Gregory Guisbiers, Dept. of Physics & Astronomy, University of Arkansas at Little Rock. Work was performed at NCI-SW.

This work was supported by NSF Award # 2030488. *Nanoscale Adv.*, vol. 3, pp. 3746-3751 (2021).

# Plasmonic Imaging of Electrochemical Reactions at Prussian Blue Nanoparticles

Prussian blue is an iron-cyanide-based pigment used as an electrochemical sensor in detecting hydrogen peroxide at low concentration levels. Prussian blue nanoparticles (PBNPs) have been studied using traditional ensemble methods, which only provide averaged information. Investigating PBNPs at a single entity level is paramount for correlating the electrochemical activities to particle structures and will shed light on the major factors governing the catalyst activity of these nanoparticles. Here we use plasmonic electrochemical microscopy to study the effect of particle structures on the electrochemistry of PBNPs and the catalytic reduction of hydrogen peroxide at individual PBNPs. A microfabricated gold sensing chip is used to detect the surface plasmon resonance. We show that the sensing capacity is highly dependent on the individual nano-particles and the electrochemical measurement conditions.



A) Prussian blue nanoparticles (PBNP) deposited on a gold sensing chip, functioning as both the surface plasmon resonance sensing chip and the working electrode for a three-electrode electrochemical cell. B) representative raw SPRM images collected from a PBNPs-containing sample, in which each NP generated a parabolic tail that is the summation of the partially reflected light and scattered plasmonic waves.

Yixian Wang, California State University, Los Angeles. Work was performed at NCI-SW.

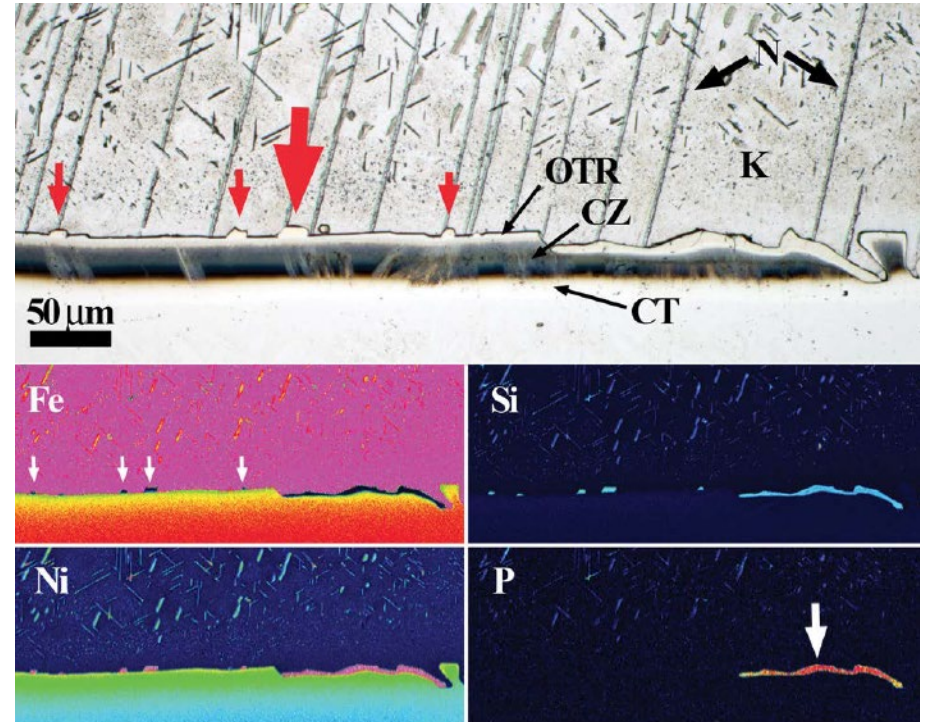
This work was supported by NSF MRI grant CHE 1828334, and NSF CAREER award CHE 2045839. *Frontiers in Chemistry*, vol. 9, article 718666 (2021).

# Carletonmooreite, $Ni_3Si$ , a New Silicide from the Norton County Aubrite Meteorite

Carletonmooreite,  $Ni_3Si$ , is a new nickel silicide mineral that occurs in metal nodules from the Norton County aubrite meteorite. The chemical composition of the holotype carletonmooreite has been determined by wavelength-dispersive electron-microprobe analysis. This new mineral is named in honor of Carleton B. Moore, chemist and geologist, and founding director of the Center for Meteorite Studies at Arizona State University, for his many contributions to cosmochemistry and meteoritics.

*Top panel: Optical, reflected-light image of the polished section through the Norton County nodule.*

*Bottom panel: Four false color images showing element maps for Fe, Ni, Si and P.*



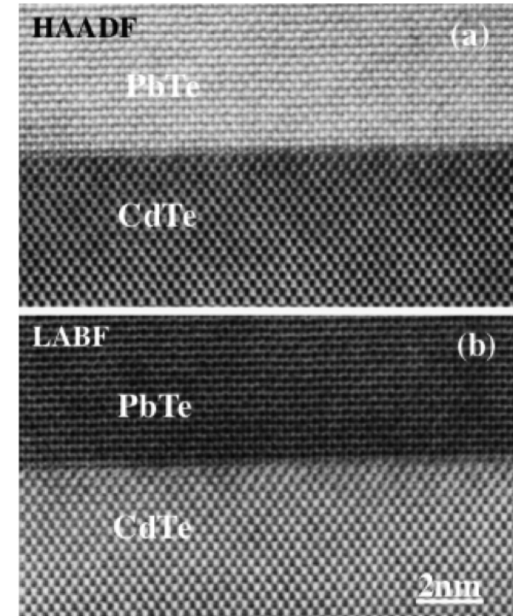
Laurence Garvie, School of Earth and Space Exploration, Arizona State University. Work was performed at NCI-SW.

This work was supported in part by NASA Emerging Worlds grant NNX17AE56G. *American Mineralogist*, vol. 106, pp. 1828–1834 (2021).

*National Research Priority: NSF–Windows on the Universe*

# Heterovalent semiconductor structures and devices grown by molecular beam epitaxy

The rock salt chalcogenides are generating much current attention, especially because of their potential applications as quantum materials. Rock salt PbTe has a unit-cell lattice parameter that is very close to those of zincblende CdTe and InSb. The closely coincident nature of these three lattice constants has motivated our recent interest in exploring the use of CdTe/InSb heterostructures as composite substrates for PbTe growth. Experiments with different flux conditions led to a smooth transition between the zincblende CdTe and the rock salt PbTe. Streaky RHEED patterns indicated the layer-by-layer PbTe growth. Cross-sectional TEM observations showed the high crystalline quality of the PbTe epilayer and the absence of interfacial dislocations, while the aberration-corrected dark-field and bright-field images confirm the abruptness of the PbTe/CdTe interface and clearly show the [001] || [001] vertical lattice-plane alignment



*Aberration-corrected atomic-structure images showing an abrupt PbTe/CdTe interface, and absence of interfacial dislocations.*

Yong-Hang Zhang, School of ECEE, ASU, and David Smith, Dept. of Physics, ASU. Work was performed at NCI-SW.

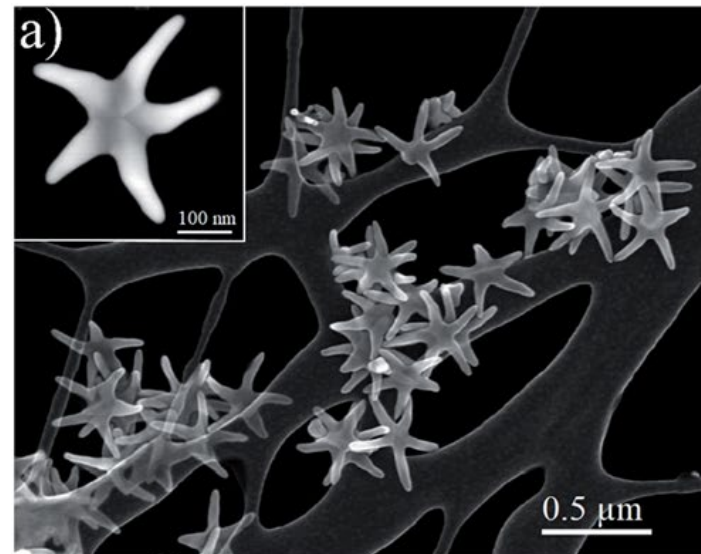
The work was partially supported by AFOSR grants FA9550-10-1-0129 and FA9550-15-1-0196. *J. Vac. Sci. Technol. A*, vol. 39, article 030803 (2021).

*National Research Priority: NSF–Quantum Leap*



# Detection of COVID-19 and its S and N proteins using surface enhanced Raman spectroscopy

The COVID-19 pandemic demonstrated the critical need for accurate and rapid testing for virus detection. This need has generated a high number of new testing methods aimed at replacing RT-PCR, which is the golden standard for testing. Most of the testing techniques are based on biochemistry methods and require chemicals that are often expensive, and the supply might become scarce in a large crisis. In the present paper we suggest the use of methods based on physics that leverage novel nanomaterials. We demonstrate that using Surface Enhanced Raman Spectroscopy (SERS) of virion particles a very distinct spectroscopic signature of the SARS-CoV-2 virus can be obtained. We demonstrate that the spectra are mainly composed by signals from the spike and nucleocapsid proteins. It is believed that a clinical test using SERS can be developed. The test will be fast, inexpensive, and reliable.



*SEM image of the Au–Cu nanostars used as SERS substrate. The image shows very sharp peaks which act as plasmonic antennas.*

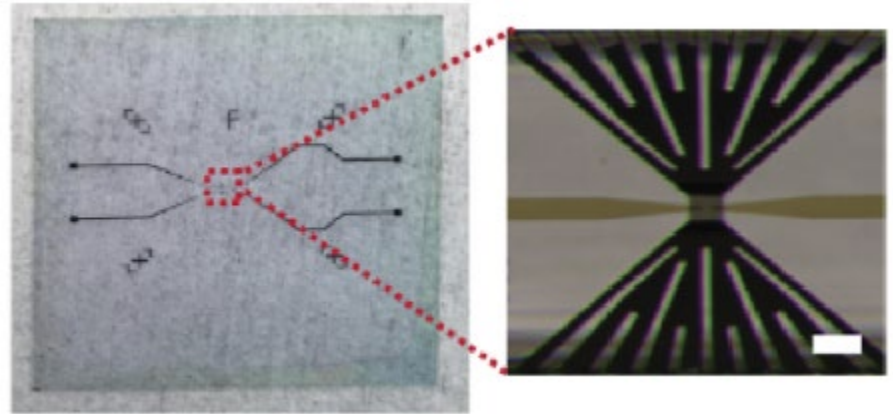
Miguel Jose Yacaman, Applied Physics and Materials Science Department, Northern Arizona University. Work was performed at NCI-SW.

The work was supported by NSF Rapid Grant # 2030488. *RSC Adv.*, vol. 11, article 25788 (2021).

*National Research Priority: NAE Grand Challenge—Advanced Health Informatics*

# Nanopore chip with self-aligned transverse tunneling junction for DNA detection

We have demonstrated a new strategy to produce nanopore chips that integrate precisely aligned transverse electrodes tuned to detect DNA translocation. Recordings of high-yield correlated signals from both the ionic current through the nanopore and between the transverse electrodes correspond to the same translocation events. Our approach can serve as a first step to provide an accessible, flexible and reproducible platform that can be further optimized for quantum-tunneling-based DNA detection, and potentially sequencing.



Picture of the bare nanopore chip and magnified image of one initial device. Scale bar: 10  $\mu\text{m}$ .

Quan Qing, Dept. of Physics, Arizona State University, Tempe. Work performed at NCI-SW.

This work was supported in part by the Air Force Office of Scientific Research under award number FA9550-16-1-0052. *Biosensors and Bioelectronics*, vol. 193, article no. 113552 (2021).

*National Research Priority: NAE Grand Challenge—Advanced Health Informatics*

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# ***Nebraska Nanoscale Facility (NNF)***

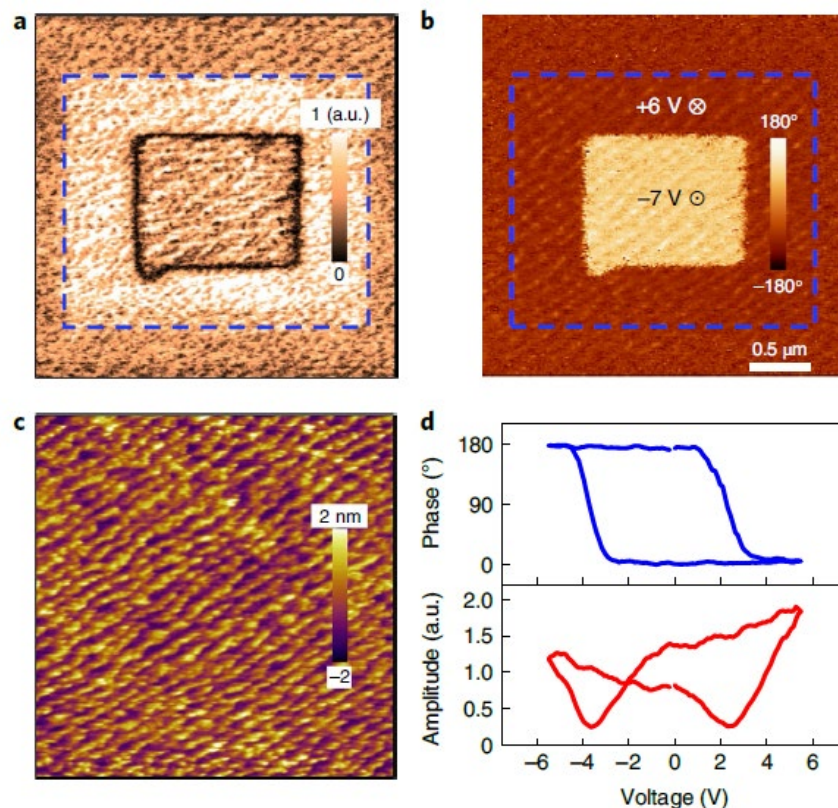
# Intrinsic ferroelectricity in Y-doped HfO<sub>2</sub> thin films

Xiaoshan Xu and coworkers at UNL used NNF facilities to demonstrate that stable and enhanced polarization can be achieved in epitaxial HfO<sub>2</sub> films with a high degree of structural order (crystallinity). An out-of-plane polarization value of 50  $\mu\text{C cm}^{-2}$  has been observed at room temperature in Y-doped HfO<sub>2</sub>(111) epitaxial thin films, with an estimated full value of intrinsic polarization of 64  $\mu\text{C cm}^{-2}$ , which is in close agreement with density functional theory calculations. The crystal structure of films reveals the *Pca*2<sub>1</sub> orthorhombic phase with small rhombohedral distortion, underlining the role of the structural constraint in stabilizing the ferroelectric phase. Our results suggest that it could be possible to exploit the intrinsic ferroelectricity of HfO<sub>2</sub>-based materials, optimizing their performance in device applications.

Yu Yun, Pratyush Buragohain, Ming Li, Zahra Ahmadi, Yizhi Zhang, Xin Li, Haohan Wang, Jing Li, Ping Lu, Lingling Tao, Haiyan Wang, Jeffrey E. Shield, Evgeny Y. Tsybmal, Alexei Gruverman & Xiaoshan Xu, Dept. of Phy. & Astron, UNL, Nebraska Center for Materials and Nanoscience (NCMN), MME UNL, SME Purdue Univ., Sandia National Lab. Work performed at Nebraska Nanoscale Facility (NNF).

*Nature Materials*, 2022, 21, 903.

National Research Priority: NSF–Harnessing the Data Revolution



*a,b*, Amplitude (a) and phase (b) of the PFM image after poling with +6 and -7 V, demonstrating stable, bipolar, remanent polarization states. *c*, Atomic force microscopy image of the surface of the YHO(111)/LSMO(001) film, displaying the atomic step-and-terrace morphology. *d*, Phase- and amplitude-switching spectroscopy loops, demonstrating ferroelectric-like hysteresis on bare surface.

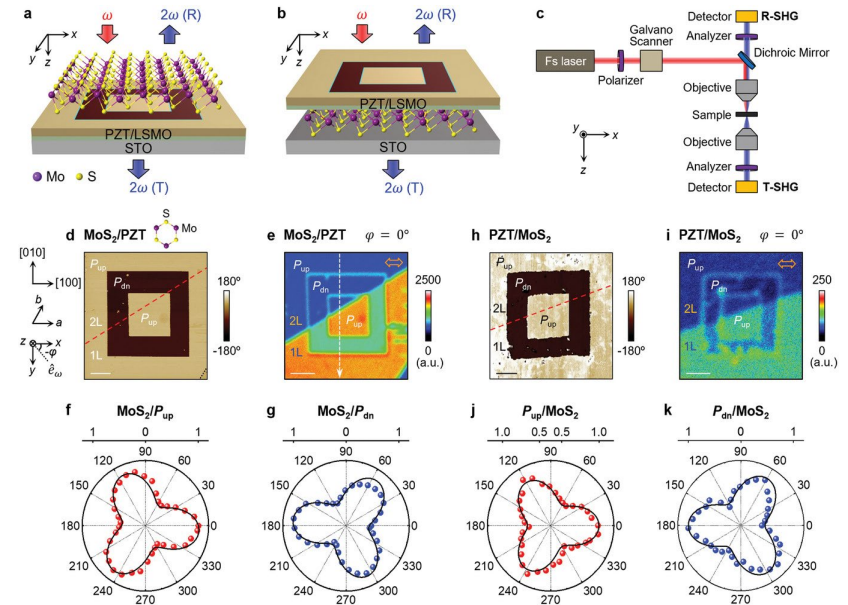


# Ferroelectric Domain Control of Nonlinear Light Polarization in $\text{MoS}_2$ via $\text{PbZr}_{0.2}\text{Ti}_{0.8}\text{O}_3$ Thin Films and Free-Standing Membranes

NNF researchers Xia Hong and coworkers demonstrated that by interfacing monolayer  $\text{MoS}_2$  with epitaxial  $\text{PbZr}_{0.2}\text{Ti}_{0.8}\text{O}_3$  (PZT) thin films and free-standing PZT membranes, the amplitude and polarization of the second harmonic generation (SHG) signal are modulated via ferroelectric domain patterning, which demonstrates that PZT membranes can lead to in-operando programming of nonlinear light polarization. The interfacial coupling of the  $\text{MoS}_2$  polar axis with either the out-of-plane polar domains of PZT or the in-plane polarization of domain walls tailors the SHG light polarization into different patterns with distinct symmetries, which are modeled via nonlinear electromagnetic theory. This study provides a new material platform that enables reconfigurable design of light polarization at the nanoscale, paving the path for developing novel optical information processing, smart light modulators, and integrated photonic circuits.

Dawei Li, Xi Huang, Qiuchen Wu, Le Zhang, Yongfeng Lu, Xia Hong, Nebraska Center for Materials and Nanoscience (NCMN), Dept. of Elect. and Comp. Eng., Dept. of Physics and Astronomy, University of Nebraska – Lincoln. Work performed at NNF.

This work was supported by the NSF DMR-2118828, DMR-1710461, and EPSCoR RII Track-1: Emergent Quantum Materials and Technologies (EQUATE), Award No. OIA-2044049. *Advanced Materials* 2022, 2208825 (in press).

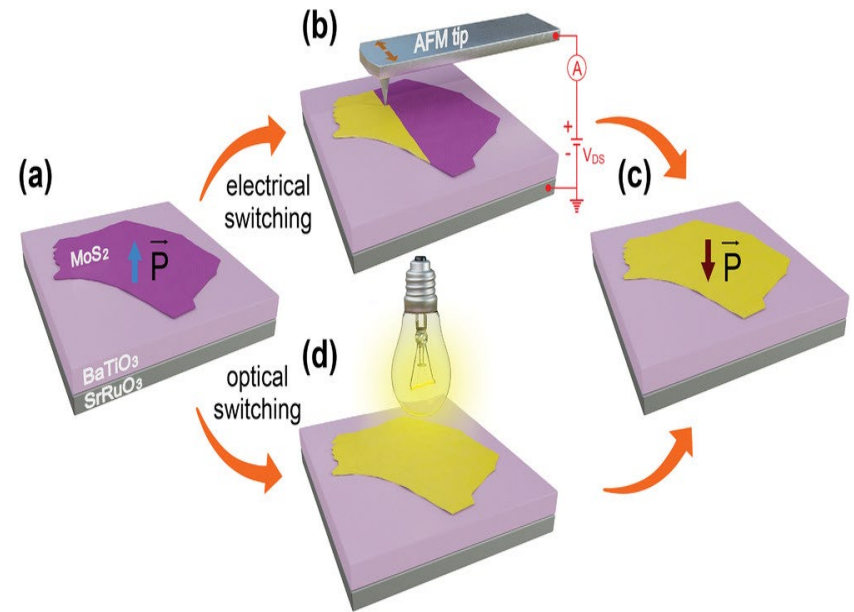


**Ferroelectric domain control of SHG signals.** a, b) Schematics of a)  $\text{MoS}_2/\text{PZT}$  and b)  $\text{PZT}/\text{MoS}_2$  samples. c) Schematic of experimental setup. d) PFM phase image and e) T-SHG mapping of  $\text{MoS}_2/\text{PZT}$ . Inset: The laboratory coordinate system and the crystalline orientations of PZT and  $\text{MoS}_2$ . The dashed and dotted lines in (d) mark the 1L/2L boundary and the edge of  $\text{MoS}_2$ , respectively. The excitation laser power is 30 mW. Scale bars: 3  $\mu\text{m}$ . f, g) Polar plots of normalized T-SHG intensity versus  $\varphi$  taken on f)  $P_{\text{up}}$  and g)  $P_{\text{dn}}$  domains in (e) with fits (solid lines). h) PFM phase image and i) R-SHG mapping of  $\text{PZT}/\text{MoS}_2$ . The dashed line in (h) marks the 1L/2L boundary of  $\text{MoS}_2$ . The excitation laser power is 7 mW. Scale bars: 2  $\mu\text{m}$ . j, k) Polar plots of normalized R-SHG intensity versus  $\varphi$  taken on j)  $P_{\text{up}}$  and k)  $P_{\text{dn}}$  domains in (i) with fits (solid lines). The open arrows in (e) and (i) show the incident light polarization direction.

National Research Priority: NSF–Harnessing the Data Revolution

# Using Light for Better Programming of Ferroelectric Devices

The recent discoveries of light–matter interactions in heterostructures based on 2D semiconductors and FE materials open new opportunities for using light as an additional tool for device programming. Sinitskii and coworkers utilized NNF Facilities and demonstrated that the combined use of an electrical field and visible light improves the nonvolatile ON/OFF ratios in MoS<sub>2</sub>-PZT memories by several orders of magnitude compared to their purely electrical operation. The memories are read at zero gate voltage ( $V_G$ ) in darkness, but their ON and OFF currents, which routinely varied for different devices by over  $10^5$ , are achieved by programming at the same  $V_G = -6$  V with (ON state) and without (OFF state) light illumination, demonstrating its crucial importance. The light can likely serve as an important tool for better programming of a large variety of other semiconductor-FE devices.



*Scheme of electrical and optical switching of FE polarization of BTO under a MoS<sub>2</sub> flake in a MoS<sub>2</sub>-FE heterostructure; see text for details. The purple and yellow colors and the vertical arrows represent the polarization of BTO under the MoS<sub>2</sub> flake.*

Alexi Lipatove, Natalia S. Vorobeva, Tao Li, Alexei Gruverman, Alexander Sinitskii, Dept. of Chemistry, Nebraska Center for Materials and Nanoscience (NCMN) and Dept. of Physics and Astronomy, University of Nebraska – Lincoln. Work performed at Nebraska Nanoscale Facility (NNF).

This work was supported by NSF Award # DMR-1420645. *Adv. Elect. Mater.* 2021, 7, 2001223.

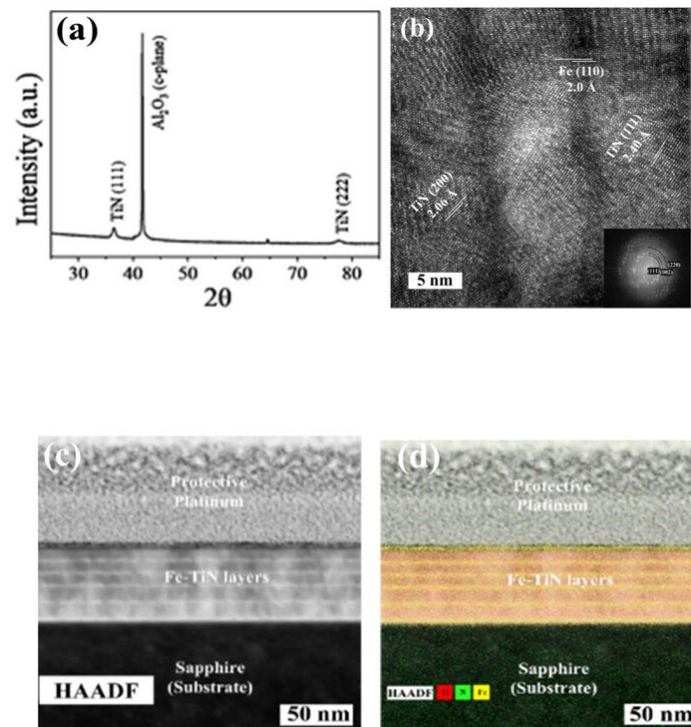
*National Research Priority: NSF–Growing Convergence Research*

# Large refrigerant capacity in superparamagnetic iron nanoparticles embedded in a thin film matrix

Magnetic refrigeration is a promising alternative in the modern-day quest for sustainable, energy-efficient, and environmentally friendly cooling technology. It is based on a physical phenomenon called the magnetocaloric effect (MCE). NNF-UNL & North Carolina A & T Univ. researchers reported a magnetocaloric effect (MCE) with sizable isothermal entropy change ( $\Delta S$ ) for a rare earth-free superparamagnetic nanoparticle system comprising of Fe–TiN heterostructure. Superparamagnetic iron (Fe) particles were embedded in a titanium nitride (TiN) thin film matrix in a TiN/Fe/TiN multilayered pattern using a pulsed laser deposition method. HAADF images in conjunction with EDX, recorded using scanning TEM, show a clear presence of alternating layers of Fe and TiN with a distinct atomic number contrast between Fe particles and TiN. With the absence of a dynamic magnetic hysteresis above the blocking temperature, the negative  $\Delta S$  as high as  $4.18 \times 10^3 \text{ J/Km}^3$  (normal or forward MCE) is obtained at 3 T at 300 K.

Kaushik Sarkar, Surabhi Shaji, Suchit Sarin, Jeffrey E. Shield, Christian Binek and Dhananjay Kumar, Dept. of ME, North Carolina A & T State Univ., & Nebraska Center for Materials and Nanoscience (NCMN), Dept. of MME, University of Nebraska – Lincoln. Work performed at Nebraska Nanoscale Facility (NNF).

Work was supported by NSF through DMR-2122067, DMR-1420645, DMR-1719875, & OIA-2044049. *J. of Appl. Phys.* 2022, 132, 193906.



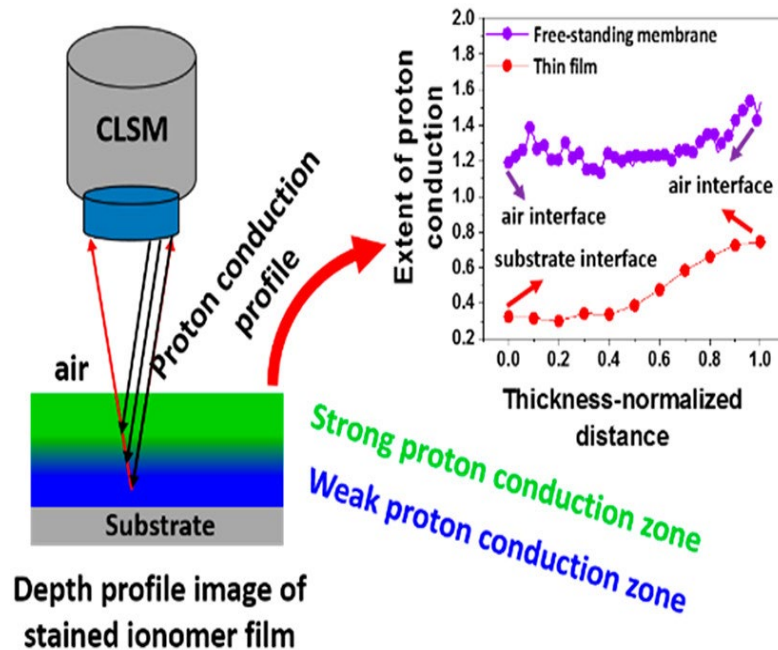
(a) X-ray diffraction pattern of Fe–TiN multilayer sample grown on a *c*- $\text{Al}_2\text{O}_3$  substrate at  $500^\circ \text{C}$ . (b) HRTEM image of the same sample showing the inclusion of Fe nanoparticles in the TiN thin film matrix. The corresponding FFT pattern (inset) corroborates the TiN polycrystallinity. Lattice spacings are noted on the image for different planes of TiN and Fe. (c) High angle annular dark-field (HAADF) image and (d) elemental map showing the alternating layers of Fe and TiN.

National Research Priority: NSF–Growing Convergence Research



# Unraveling Depth-Specific Ionic Conduction and Stiffness Behavior across Ionomer Thin Films and Bulk Membranes

Interfacial behavior of submicron thick polymer films critically controls the performance of electrochemical devices. Shudipto Dishari and coworkers developed a robust, everyday-accessible, fluorescence confocal laser scanning microscopy (CLSM)-based strategy that can probe the distribution of mobility, ion conduction, and other properties across ionomer samples. When fluorescent photoacid probe 8-hydroxypyrene-1,3,6-trisulfonic acid trisodium salt (HPTS) was incorporated into  $<1\ \mu\text{m}$  thick Nafion films on substrates, the depth-profile images showed thickness- and interface-dependent proton conduction behavior. In these films, proton conduction was weak over a region next to substrate interface, then gradually increased until air interface at 88% RH. The CLSM-based strategy now allows us to reveal how the ionic conductivity is at different depths and how far the interfacial effects propagate inside very thin films. Such information will guide us to design catalyst layers for electrochemical devices.



Seefat Farzin, Ehsan Zamani, and Shudipto K. Dishari, Dept. of Chem. and Biomolecular Eng., University of Nebraska – Lincoln. Part of the work performed at Nebraska Nanoscale Facility (NNF).

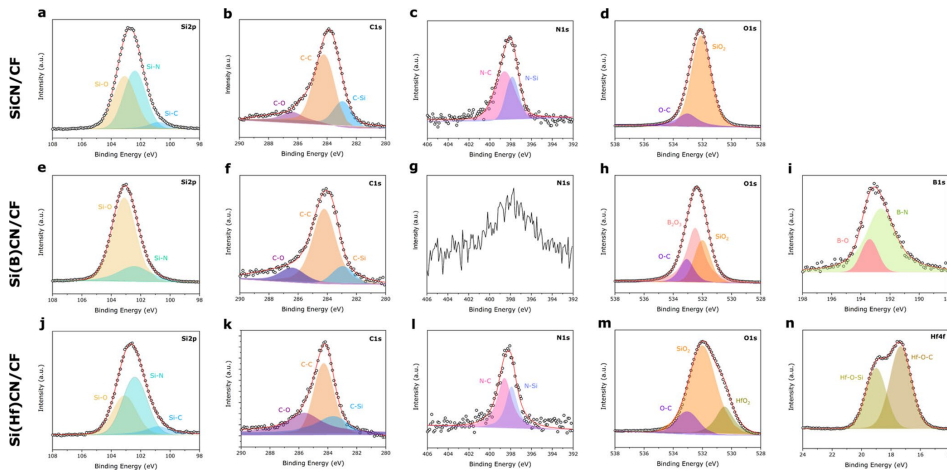
This work was supported by NSF Award # 1750040. *ACS Macro Letters*, 2021, 10, 791.

National Research Priority: NSF–Growing Convergence Research



# Evaluating Use of Boron- and Hafnium-Modified Polysilazanes for Ceramic Matrix Minicomposites

Ceramic matrix composites (CMCs) are suitable candidates for extreme environment applications due to their low density, high limit of damage tolerance, and higher use temperature capability. Gurpreet Singh and coworkers at the Kansas State University used NNF facilities to study the potential of polymer-derived CMCs by the addition of thin ceramic coatings on carbon fiber (CF) bundles. Boron- and hafnium-modified polysilazane liquid precursors were synthesized and used to infiltrate the fiber bundles of CF to fabricate lab-scale Si(B)CN/CF and Si(Hf)CN/CF CMC minicomposites, respectively by crosslinking and then pyrolysis at 800 ° C. The Si(B)CN/CF contained Si–N and B–N bonds, while Si–N and Hf–O–Si bonds were observed for the Si(Hf)CN/CF sample with uniform and dense surfaces. Room-temperature tensile tests showed that the Si(Hf)CN/CF sample could reach a tensile strength of ~790 MPa and an elastic modulus of 66.88 GPa among the composites. An oxidation study of the Si(Hf)CN/CF minicomposites showed higher stability compared to SiCN/CF and Si(B)CN/CF minicomposites up to 1500 ° C.



High-resolution XPS spectra of the CMC samples for Si 2p, C 1s, N 1s, O 1s, B 1s, and Hf 4f and (a–d) SiCN/CF, (e–i) Si(B)CN/CF, and (j–n) Si(Hf)CN/CF.

Shakir Bin Mujib, Mohammed Rasheed, Gurpreet Singh, Department of Mechanical and Nuclear Engineering, Kansas State University, Kansas. Part of the work performed at Nebraska Nanoscale Facility (NNF).

ACS Omega 2022, 7, 49, 45325.

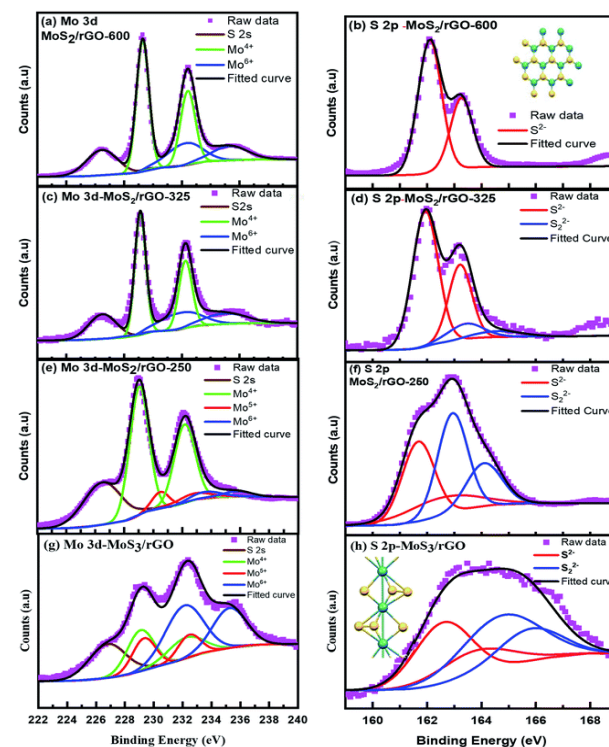
National Research Priority: NSF–Growing Convergence Research

# Tuning the defects in MoS<sub>2</sub>/reduced graphene oxide 2D hybrid materials for optimizing battery performance

Jun Li and coworkers from Kansas State University utilized NNF Facilities to demonstrate that defect engineering is critical for improving Zn-ion storage. The study reports the preparation of a set of hybrid materials consisting of molybdenum disulfide (MoS<sub>2</sub>) nanopatches on reduced graphene oxide (rGO) nanosheets by microwave specific heating of graphene oxide and molecular molybdenum precursors followed by thermal annealing in 3% H<sub>2</sub> and 97% Ar. The unique defect-engineered hybrid material of MoS<sub>2</sub> on reduced graphene oxide opens a new road to enhance monovalent and divalent ion storage. The few-layered MoS<sub>2</sub> nanopatches stacked on the rGO nanosheets significantly improve the intercalation of Li<sup>+</sup> ions while the high-density Mo-deficient defects enhance the Zn-ion storage at the defective S-edges. The highly defective MoS<sub>2</sub>/rGO hybrid prepared by annealing at 250 °C shows the highest initial Zn-ion storage capacity (~300 mA h g<sub>MoS<sub>x</sub></sub><sup>-1</sup>) and close to 100% coulombic efficiency.

Kamalambika Muthukumar, Levon Leban, II Archana Sekar, Ayyappan Elangovan, Nandini Sarkar & Jun Li, Dept. of Chemistry, Kansas State University, Part of the work performed at Nebraska Nanoscale Facility (NNF).

*Sustainable Energy Fuels*, 2021, 5, 4002.



The Mo 3d and S 2p XPS spectra of (a, b) MoS<sub>2</sub>/rGO-600, (c, d) MoS<sub>2</sub>/rGO-325, (e, f) MoS<sub>2</sub>/rGO-250 and (g, h) the MoS<sub>3</sub>/rGO-intermediate product. The inset of panel (b) shows the schematic structure of the hexagonal MoS<sub>2</sub> nanopatches. The inset of panel (h) shows the schematic structure of the MoS<sub>3</sub> chain.

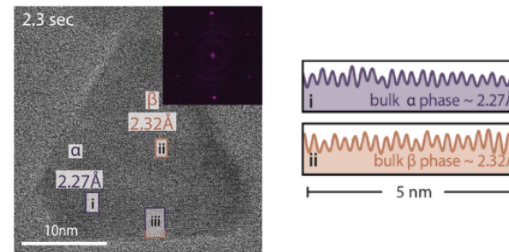
National Research Priority: NSF–Growing Convergence Research

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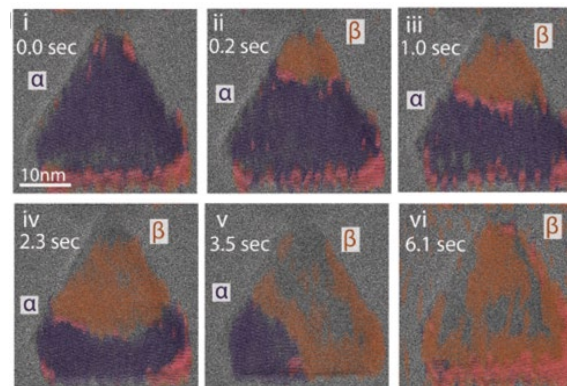
# ***NNCI Site @ Stanford (nano@stanford)***

# Hydrogenation-Induced Lattice Expansion Within AgPd Nanoparticle

Palladium alloys are critical for hydrogen-based technologies such as catalysis, fuel cells, and sensors. Alloying palladium can help tailor and optimize its properties but, hydrogen gas diffusion has not been well studied in palladium alloys or at a single nanoparticle level. This work, **by Prof. Jennifer Dionne's group at Stanford University**, studied the hydrogenation, nucleation, and phase transition of AgPd nanoparticles by observing hydrogen gas diffusion into the nanoparticles at an atomic scale and in real time. During the experiments, a high hydrogen gas pressure was maintained in a FEI Titan environmental TEM. In a single AgPd nanoparticle, both  $\alpha$  and  $\beta$  phases could be distinguished by their different lattice spacings, as measured by using line profiles through the TEM images. Bigger lattice spacing correlated with a higher hydrogen content. By inverse FFT processing the time-lapsed TEM images, it was possible to follow the progression of the phase change over time. Observations included surface-limited  $\beta$  phase growth, reorientation of the  $\alpha/\beta$  interface, and no preferential nucleation at the sharpest nanoprism corners.



*Real space pixel intensity line profiles taken from boxes i & ii marked in TEM. “Box i” has a 2.27 Å lattice spacing and is  $\alpha$  phase. “Box ii” has a spacing of 2.32 Å and is  $\beta$  phase*



*Inverse FFT TEM images, at different time points, filtered for  $\alpha$  and  $\beta$  phases. The time lapse images show the progression of the phase change over time.*

Daniel K. Angell, Briley Bourgeois, Michal Vadai, and Jennifer A. Dionne, School of Engineering, Stanford University. Work was performed at the Stanford Nano Shared Facilities (SNSF)

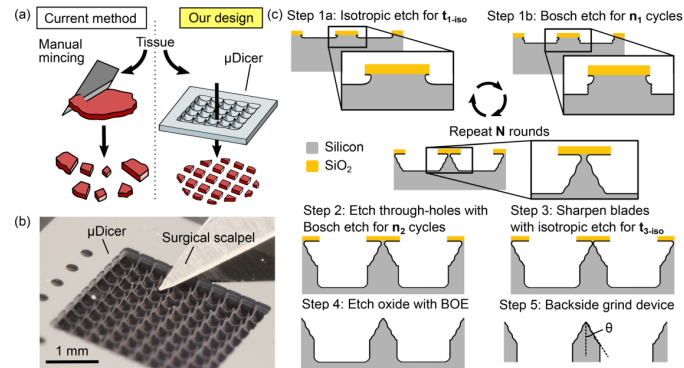
This work was supported by NSF Award # ECCS-2026822. *ACS Nano*, 16(2), 1781–1790.

*National Research Priority: DoD Critical Technology Area–Advanced Materials*



# Silicon-based, Microfabricated $\mu$ Dicer Dissection Tool

Previously tissue sectioning for disease diagnostics and drug discovery was performed by manual mincing, which resulted in uneven pieces that could result in inconsistencies in drug screening and disease diagnostics. This paper presented the invention of a novel  $\mu$ Dicer tool for cutting uniformly-sized tissues slices. The  $\mu$ Dicer was a silicon-based hollow array of evenly spaced blades. To fabricate the  $\mu$ Dicer, the authors utilized isotropic and anisotropic etching and only a single silicon oxide mask, which was made for a faster and more efficient fabrication process. The invention also included designs with variable numbers of serrations, which will likely affect the needed cutting force, and an open-access simulation model to guide design decisions. The graduate students in **Prof. Sindy Tang's group at Stanford University**, had never used a fabrication facility before this project. They collaborated with staff at the Stanford Nanofabrication Facility (SNF) to become proficient facility users and to make their idea into a product. The publication of this paper led to a grant to further fund work in this area.



(a) Diagram comparing manual mincing and cutting with a  $\mu$ Dicer. (b) Photograph of the  $\mu$ dicer with a scalpel blade for size reference. (c) The process flow for etching the blades and through-holes.

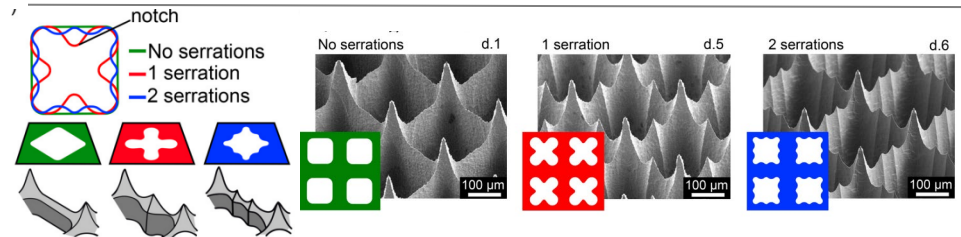


Diagram showing different mask designs (for a single cell) and the corresponding serration geometries. SEM images of  $\mu$ Dicers with 0, 1, and 2 serrations.

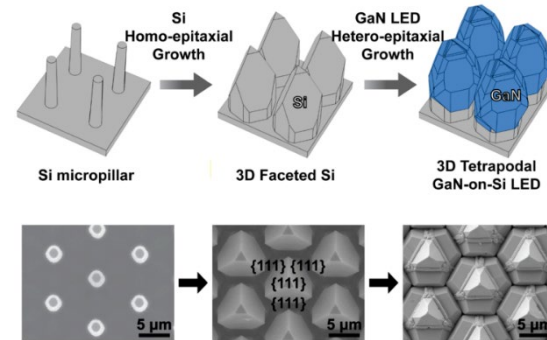
Seth C. Cordts, Nicholas Castaño, Saisneha Koppka, and Sindy K. Y. Tang, School of Engineering, Stanford University. Work was performed at the Stanford Nanofabrication Facility (SNF) and Stanford Nano Shared Facilities (SNSF)

This work was supported by NSF Award # ECCS-2026822. *Appl. Phys. Lett.*, 119(1), 011904.

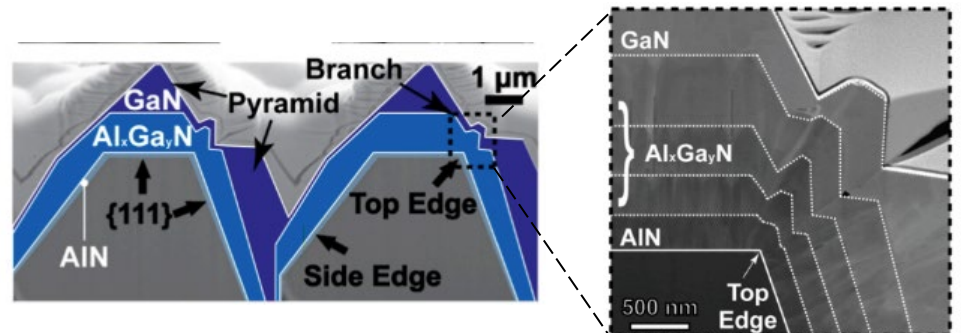
National Research Priority: NSF–Growing Convergence Research

# GaN-on-Si LED Fabrication (3D tetrapodal over 3D faceted Si)

In this publication, by **Prof. Dong Rip Kim's group at Hanyang University**, a fabrication process was developed for building GaN LEDs onto silicon (instead of sapphire). Using silicon is appealing from a cost perspective and it also has better thermal conductivity and optical properties than sapphire. However, traditional planar deposition methods of GaN onto silicon result in electronics with poor performance because of a mismatch of physical properties and stress/strain issues. To mitigate these issues, Professor Kim deposited GaN onto 3D silicon structures. The fabrication process used photolithography to pattern micropillars onto a Si(111) surface. The 3D faceted structures then were created with silicon epitaxial growth onto the micropillar array. An AlN nucleation layer and three-graded AlGa<sub>y</sub>N buffer layers were introduced. Finally, MOCVD was used to deposit GaN onto the 3D silicon. The 3D tetrapodal over 3D faceted Si structure reduced threading dislocation densities, resulting in better performance and enabling multi-color emission. Overcoming the stress/strain interface issues of GaN on Si will enable a more cost-effective substrate for LEDs.



*Deposition schematic and top-down SEM images: growing GaN on 3D faceted Si reduced threading dislocation densities, resulting in better performance*



*Cross-section SEM & TEM images show a AlN nucleation layer, a Al<sub>x</sub>Ga<sub>y</sub>N buffer layer, & growth contours in the formation of branch GaN structures*

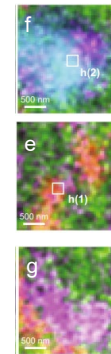
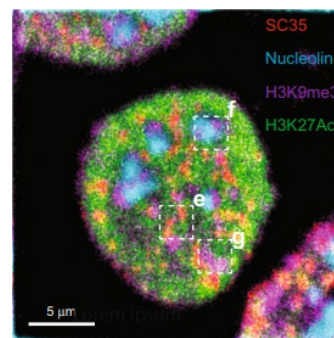
Youngshik Cho, Min Soo Jeon, Hanmin Jang, Heung Soo Lee, and Dong Rip Kim, School of Mechanical Engineering, Hanyang University. Work was performed at the Stanford Nanofabrication Facility (SNF).

This work was supported by NSF Award # ECCS-2026822. *Appl. Surf. Sci.*, 565(1), 150584.

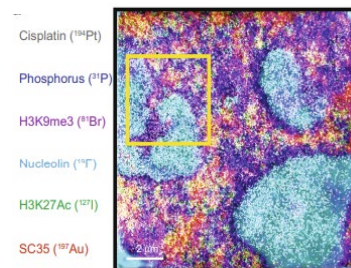
*National Research Priority: CHIPS+Science Act Research*

# Subcellular Chemical Imaging of Biomolecules and Drugs

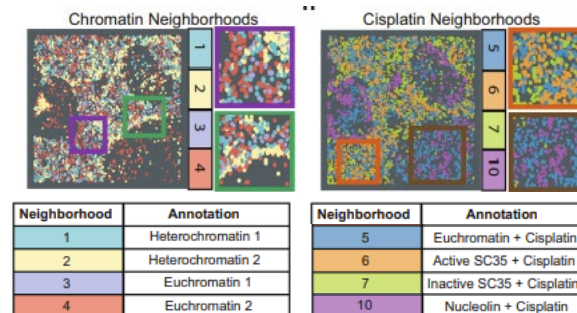
Chemical imaging of biomolecules will provide an improved understanding of biological processes and drug mechanisms. This paper, by **Prof. Gary Nolan at Stanford's School of Medicine**, demonstrated multiplexed, high-resolution, direct chemical imaging of subcellular species using nanoSIMS. As a proof-of-concept, HeLa cells were treated with mass-oligonucleotide conjugated antibodies (MoC-Ab) that targeted specific subcellular bodies/species (e.g., nucleoli, inactive chromatin, active chromatin, and a pre-mRNA splicing protein, SC35) and were tagged with different isotopes or gold nanoparticles. It was possible to observe multiple isotopes simultaneously and, by processing the images with dimensionality reduction techniques, to resolve discrete subnuclear structures. Another experiment used nanoSIMS to examine TYK-nu nuclei that were treated with the same isotopic markers, along with the chemotherapy drug, cisplatin. Regions were identified where cisplatin was colocalized with specific nuclear species (e.g., nucleolin, active chromatin, and SC35). The results supported a role for cisplatin in the active regions of chromatin. The results further showcase how nanoSIMS can be used to better understand drug interactions.



nanoSIMS images of a HeLa cell nucleus treated with antibodies (i.e., MoC-Ab) isotopically-tagged to highlight specific subcellular bodies/species: **nucleoli**, **inactive chromatin**, **active chromatin**, **alternative splicing protein SC35**



nanoSIMS images of a TYK-nu cell nucleus additionally treated with cisplatin (cancer drug) and isotopically tagged DNA.



After denoising and extensive data processing, “nuclear neighborhoods” with cisplatin-protein interactions (e.g., 5, 6, 7, 10) were identified. Note that this false color scale is different than in the nanoSIMS images.

Xavier Rovira-Clave, Sizun Jiang, Yunhao Bai, Bokai Zhu, Graham Barlow, Salil Bhate, Ahmet F. Coskun, Guojun Han, Chin-Min Kimmy Ho, Chuck Hitzman, Shih-Yu Chen, Felice-Alessio Bava, and Gary P. Nolan, School of Medicine, Stanford University. Work was performed at the Stanford Nano Shared Facility (SNSF).

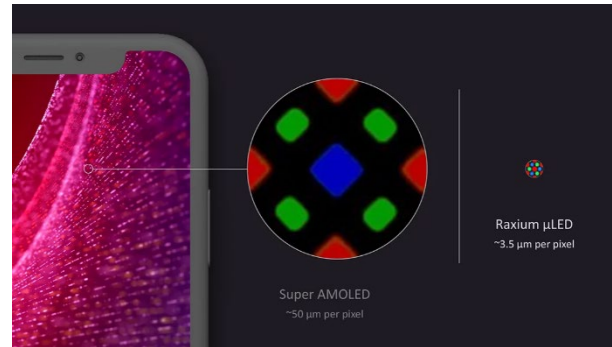
This work was supported by NSF Award # ECCS-2026822. *Nature Commun.*, 12, 4628.

National Research Priority: NSF–Understanding the Rules of Life



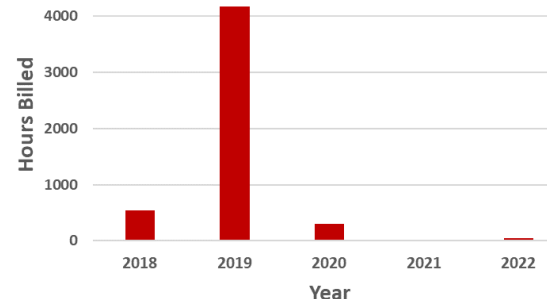
# Raxium: A Start-Up Success Story

Shortly after launching their company in 2017, Raxium contacted nano@stanford to jump-start its process development while building its own fabrication facility. Raxium only recently revealed its product, said to be the most vibrant and smallest micro-LED commercially produced. The micro-LED device boasts an impressively small pixel pitch of  $3.5\mu\text{m}$ ; whereas the industry standard for Super AMOLED screens on phones has a pitch of  $50\mu\text{m}$ .



*Relative sizes of the industry standard LED and Raxium's microLED.*

Raxium's activity in our facilities peaked in 2019, with its team of 22 process engineers and technicians all trained and using the facilities. Raxium's choices in building their own fab were strongly informed by their experience with the nano@stanford processes and equipment, including their purchase of an ASML stepper. As their own facility came online, Raxium's use of nano@stanford declined but the company does still occasionally use our facilities. **In May 2022, Raxium was acquired by Google for a reported ~\$1B.**



*Annual equipment & training hours billed to Raxium*

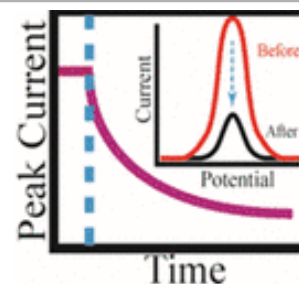
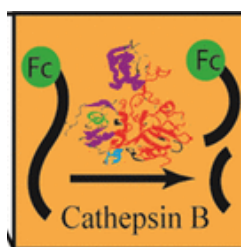
Raxium. Work was performed at the Stanford Nanofabrication Facility (SNF) and the Stanford Nano Shared Facility (SNSF).

This work was supported by NSF Award # ECCS-2026822.

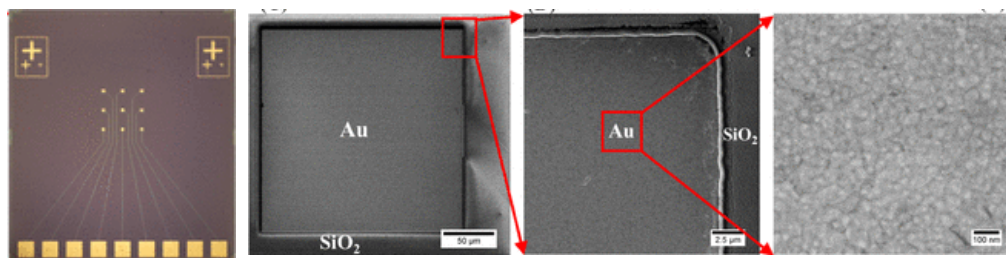


# Peptide-functionalized, gold microelectrode arrays for multiplexed analysis of human serum proteases

Proteases are essential for enabling protein degradation in metabolic functions and have become a small molecule drug target for cancer therapeutics. Proteases are present in extremely small quantities, requiring highly sensitive detection methods, and the importance of detecting only active proteases adds analytical complexity. In their latest paper, **Prof. Jun Li and his group from Kansas State University** developed an electrochemical method to detect active proteases with parallel detection for increased throughput and multiplexing. The method uses gold patterned microelectrode arrays (MEA, fabricated at nano@stanford) functionalized with an electrochemically active peptide (peptide-Fc). The peptides produce a strong signal by alternative current voltammetry (ACV) when they are attached to the gold. Once the peptides are cleaved by proteases, the total electrochemical signal decreases. In this paper, the authors optimized the buffer conditions to allow for simultaneous detection of a greater diversity of proteases with greater sensitivity (LOD of 57.1pM for rhCB) and they demonstrated detection in human serum.



(Left) Schematic describing the detection method. A gold microelectrode, functionalized with peptide-Fc, produces a strong ACV signal. When a peptide is cleaved by an active protease, it diffuses away from the surface and its ACV signal disappears. (Right) Therefore, the total current signal decreases.



Optical image of a MEA chip with nine microelectrodes (gold contact pads along the bottom are 1 mm x 1 mm). SEM images of a single microelectrode with increasing magnification to show the microstructure of the gold electrode surface.

Song Yang, Jestin Gage Wright, Morgan Anderson, Sabari Rajendran, Zhaoyang Ren, Duy Hua, Jessica Koehne, M. Meyyappan, Jun Li, Department of Chemistry, Kansas State University; NASA Ames Research Center. Work was performed at the Stanford Nanofabrication Facility (SNF).

This work was supported by NSF Award # ECCS-2026822. *ACS Sensors.*, 6, 3621.

National Research Priority: NSF–Understanding the Rules of Life

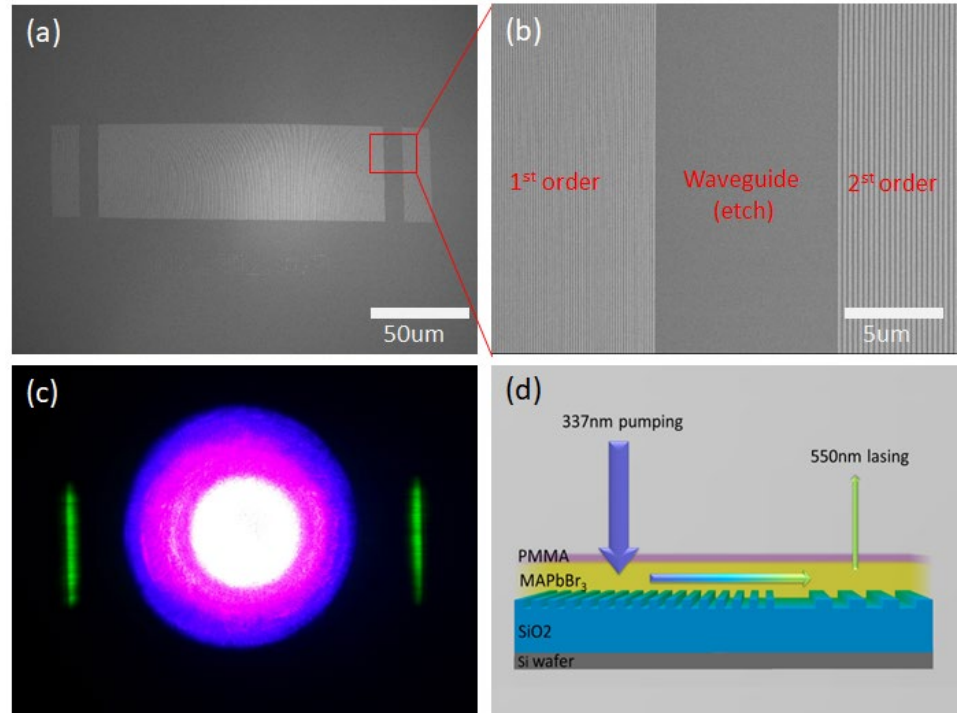
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# ***Northwest Nanotechnology Infrastructure (NNI)***

# Perovskite 1<sup>st</sup>-order DFB Laser

First-order DFB lasers are key components for integrated photonics. In this work, we demonstrated a green-emitting perovskite 1<sup>st</sup>-order DFB laser with low lasing threshold, narrow emission linewidth, high resistance to moisture and long-term stability. The laser was fabricated on a silicon wafer with a CMOS-compatible process.

(a) SEM image of the 1<sup>st</sup>-order DFB grating for the perovskite laser with (b) a 2<sup>nd</sup>-order DFB grating area for output coupling. (c) Dark-field optical microscope image of the 1<sup>st</sup>-order perovskite DFB laser under operation. (d) Schematic of the perovskite 1<sup>st</sup>-order DFB laser.



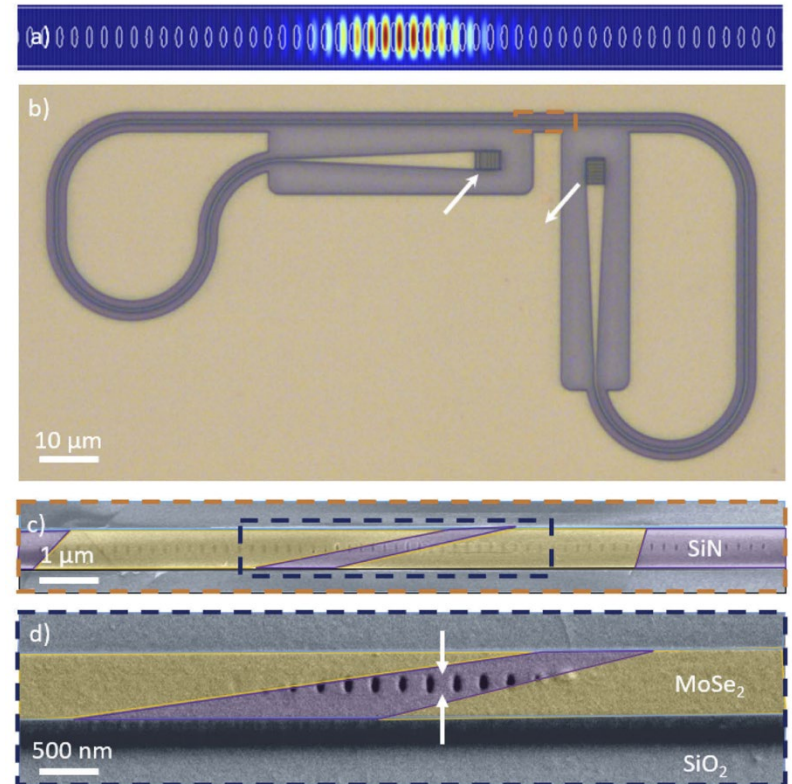
Cheng Chang and Lih Y. Lin, Department of Electrical and Computer Engineering, University of Washington. Work performed at the Washington Nanofabrication Facility.

This work was supported by NSF Award # ECCS-1807397 and UW RRF. *Advanced Photonics Research* 2022, 2200071.

*National Research Priority: NSF–Harnessing the Data Revolution*

# Dispersive Coupling Between $\text{MoSe}_2$ and an Integrated Zero-dimensional Nanocavity

Establishing a coherent interaction between a material resonance and an optical cavity is a necessary first step to study semiconductor quantum optics. Here we report on the signature of a coherent interaction between a two-dimensional excitonic transition in monolayer  $\text{MoSe}_2$  and a zero-dimensional, ultra-low mode volume ( $V_m \sim 2(\lambda/n)^3$ ) on-chip photonic crystal nanocavity. This coherent interaction manifests as a dispersive shift of the cavity transmission spectrum, when the exciton-cavity detuning is decreased via temperature tuning. The exciton-cavity coupling is estimated to be  $\approx 6.5$  meV, with a cooperativity of  $\approx 4.0$  at 80 K, showing our material system is on the verge of strong coupling. The small mode-volume of the resonator is instrumental in reaching the strongly nonlinear regime, while on-chip cavities will help create a scalable quantum photonic platform.



*Fabricated nanobeam cavity with integrated 2D material*

David Rosser, Yueyang Chen, Arka Majumdar, Physics, ECE, UW. Work performed in WNF, MAF.

Funding Source: NSF-1845009 and NSF-ECCS-1708579

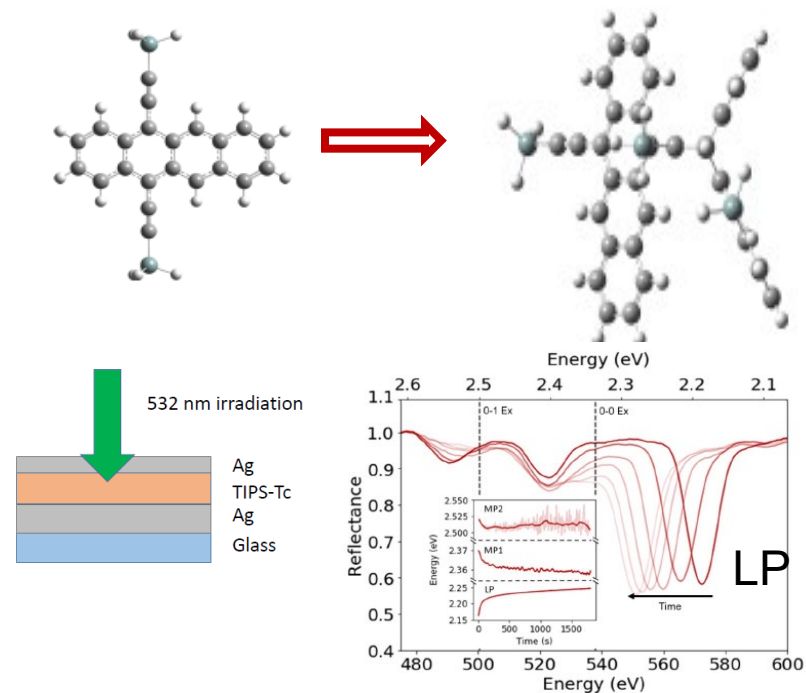
National Research Priority: NSF–Quantum Leap



# Strong Coupling in Microcavities for Enhancing Photostability of High-performance Organic Semiconductors

The project investigates how the properties of hybrid light-matter states (created by strong coupling of molecules to the cavity) and of the states resulting from molecules not coupled to the cavity control the rates of chemical reactions responsible for photodegradation and recovery of organic semiconductors.

Benchmark organic semiconductors such as TIPS-Tc under continuous photoexcitation experience photodimerization as the dominant anaerobic photodegradation mechanism (top Figure). When TIPS-Tc molecules are strongly coupled to the cavity photon, polaritons form and add relaxation pathways that alter populations of excited states. These populations can be manipulated by cavity design and detuning, resulting in tunable photodimerization rates (bottom Figure).



Example of photodimerization reaction in functionalized tetracene (TIPS-Tc) films which can be modified by strong coupling of tetracene molecules to the cavity photon.

O. Ostroverkhova (OSU Physics) and Winston Goldthwaite, Roshell Lamug. Work performed at ATAMI and MaSC.

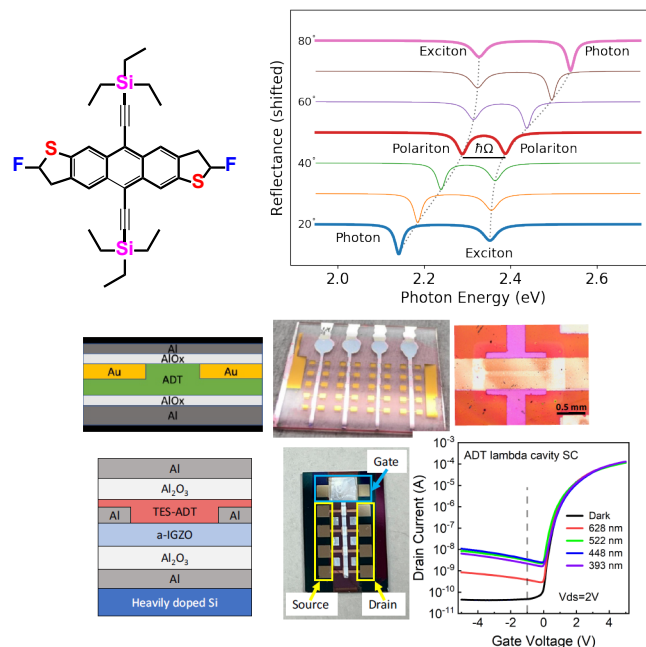
This work was supported by CHE-1956431. *Journal of Physical Chemistry C* 125 , 27072-27083 (2021)

National Research Priority: NSF–Quantum Leap

# Designing Light-matter Hybrid States for High-performance Organic (Opto)electronics

The project investigates optical and electronic properties of hybrid light-matter quasiparticles (polaritons) and their ability to enhance optoelectronic properties of model organic semiconductors.

When organic molecules (such as ADT shown in Figure or Pentacene) are strongly coupled to a microcavity, hybrid exciton-photon (polariton) states form and manifest into optical properties (top right Figure). Optical and optoelectronic properties of strongly-coupled benchmark organic semiconductor films are studied in cavity transistor structures (bottom Figure) to establish how photoresponsivity and charge transfer can be manipulated by polaritons.



Examples of organic and organic/inorganic transistors incorporated in microcavities to establish effects of polariton formation on device performance.

O. Ostroverkhova (OSU Physics), L.-J. Cheng (OSU EECS), Roshell Lamug, Winston Goldthwaite, Ahasan Ullah. Work performed at ATAMI and MaSC.

This work was supported by DMR-1808258. *Journal of Physical Chemistry C* 125 , 27381-27393 (2021)

National Research Priority: NSF-Quantum Leap

# Out-of-oven Rapid Synthesis of Entropy Stabilized Oxides Using Electromagnetic Fields

Entropy stabilized oxides (ESOs) are a new class of stable hybrids and single-phase metal oxides made from multiple ions with material properties somewhere between the constituent oxides synthesized in large ovens that require very high temperatures ( $\sim 1000^\circ\text{C}$ ) over extended time. We developed a new energy-efficient ESO synthesis method that uses the ability of carbonaceous materials to heat rapidly in response to radio frequency (RF) fields in 1-200 MHz range. Using carbon fibers and graphene as RF susceptors, synthesis of  $(\text{MgCoNiCuZn})_{0.2}\text{O}$  is achieved through RF-initiated combustion synthesis with heating rates of  $203^\circ\text{C/s}$  at 20 W of input power. This method reduces the formation time of ESOs down to less than a minute, allowing for much more efficient fabrication. This work opens an exciting frontier for the rapid synthesis of ESOs using RF heating as a non-contact, rapid manufacturing process.

Jared Rapp, Ankush Nandi, and Aniruddh Vashisth, Department of Mechanical Engineering, University of Washington.  
Work performed at MAF, UW

Partial support from an NSF-NNCI seed grant.

National Research Priority: DoD Critical Technology Area—Advanced Materials

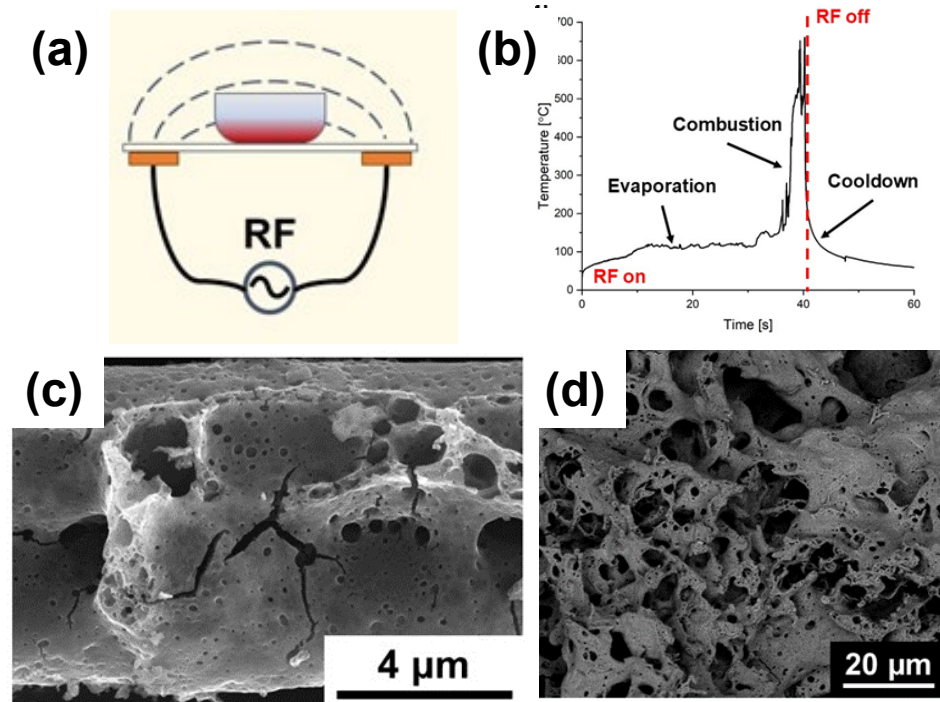
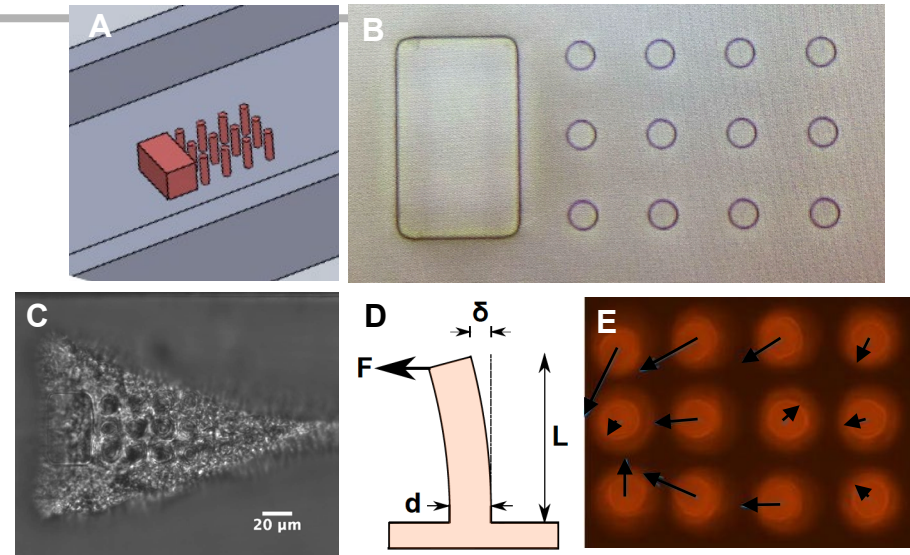


Fig: (a) ESO synthesis setup using RF fields in 1-200 MHz (1-20 W); (b) Temperature vs time profile for rapid synthesis; SEM image of ESO synthesized using RF fields on (c) carbon fibers and (d) graphene nanoparticles.

# A Microfluidic Device to Measure the Contractile Force of Platelet Aggregates Formed Under Shear Flow

Platelets aggregate, activate, and contract at the site of vascular injury to stop bleeding; however, disruptions to hemostasis can cause life-threatening bleeding or thrombi. There is a growing need for rapid diagnostic tests that assess platelet function to predict bleeding or thrombotic risk in clinical settings, or assays for drug assessment.

We have developed a microfluidic device, with force sensors embedded in the channel (Fig. 1A-B), that induces the adhesion and aggregation of platelets under shear using a rigid block in a manner akin to the formation of platelet-rich thrombi in arterial flow (Fig. 1C). Platelet force is determined from the deflection of the posts using Hooke's Law (Fig. 1D-E). Our findings indicate that platelet forces may be a useful metric for assessing platelet function to monitor thrombotic or bleeding risk.



**Figure 1.** Force sensor in a microfluidic channel (A) isometric view (B) top view. (C) A platelet-plug formed 120 secs after blood entered the channel. (D) The force that aggregated platelets produced was calculated using Hooke's law,  $F = k\delta$  where  $F$  is force,  $\delta$  is post deflection,  $k = 3\pi Ed^4/64L^3$ ,  $E$  is the modulus of elasticity,  $d$  is the diameter, and  $L$  is the length of the post. (E) High spatial resolution contractile force of the platelet-plug at 120 secs. The yellow arrows are force vectors where  $\sum|F| = 478.6 \text{ nN}$ .

Ava Obenaus and Nate Sniadecki (Mechanical Engineering, University of Washington. Work performed at WNF.

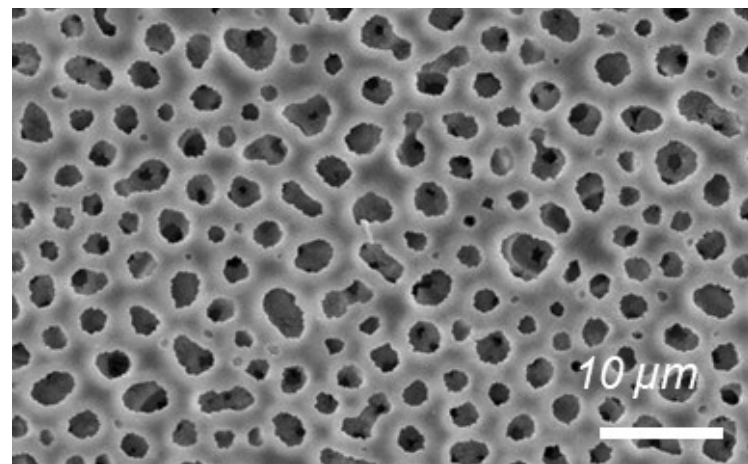
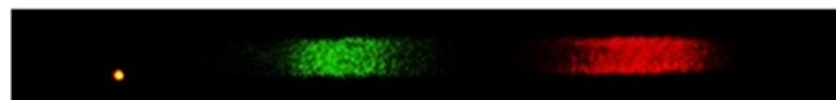
Research supported by National Heart, Lung, And Blood Institute of the National Institutes of Health under Award Number 5R35HL145262 and F31HL156697.

National Research Priority: NSF–Understanding the Rules of Life



# Picoliter Thin Layer Chromatography

A picoliter thin layer chromatography (pTLC) platform was developed for analyzing extremely miniature specimens, such as an assay of the contents of a single cell of 1 picoliter volume. The pTLC chip consisted of an array of microscale bands made from highly porous monolithic silica designed to accept picoliter-scale volume samples. pTLC bands were fabricated by combining sol-gel chemistry and microfabrication technology. Single cells loaded with fluorescent lipophilic dyes or sphingosine kinase reporter were spotted on microbands, and the single-cell contents separated by pTLC were detected from their fluorescence. pTLC has potential for applications in many areas where miniature specimens and high throughput parallel analyses are needed.



Top: Separation of fluorescent lipophilic dyes from a single cell. Bottom: SEM images of the macroporous structure of pTLC silica microbands

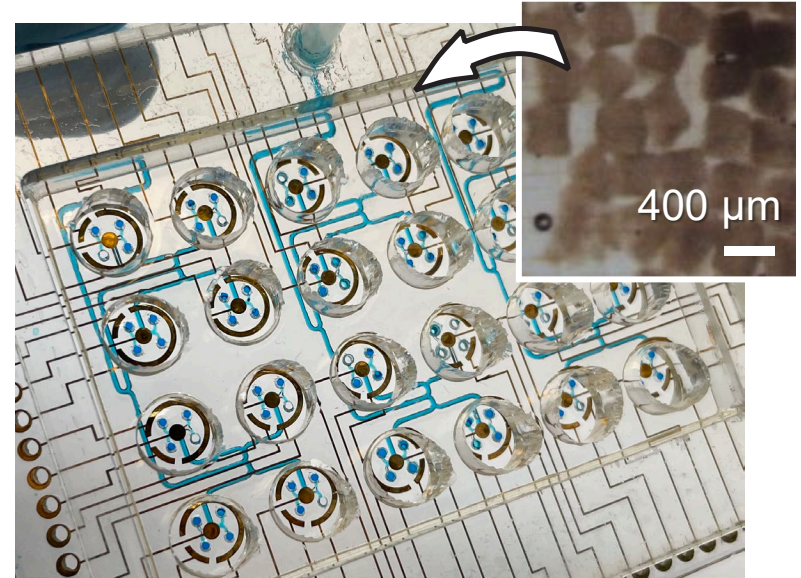
Yuli Wang, Ming Yao, Christopher E. Sims, Nancy L. Allbritton, Department of Bioengineering, University of Washington. Work performed at MAF.

This work was supported by the National Institutes of Health under Award CA233811, and the Washington Research Foundation pilot grant. *Analytical Chemistry* 2022, 94, 39, 13489–13497.

*National Research Priority: NAE Grand Challenges—Engineer Better Medicines*

# Integrated Electrochemical Aptasensors for Continuous Monitoring of Intact Tumor “Cuboids”

The integration of electrochemical biosensors with microelectronics has sparked huge interest and has paved the way for miniaturized, low-cost systems that merge biomolecular sensing with digital computing, programming, and communication. Electrochemical aptamer-based sensors (“aptasensors”) offer multiple advantages including real-time and label-free readouts (i.e. user-friendliness), ease of manufacture and CMOS integration, high sensitivity, and stability (versus antibodies). We have developed an intact-tissue microfluidic drug testing platform based on regularly-sized, cuboidal-shaped microdissected tissues (referred to as “cuboids”) mechanically cut with a tissue chopper. The gold electrodes for the aptasensors are fabricated by dry-film photolithography.



*Aptasensor array integrated in a microfluidic multi-well platform (4 cuboid traps per well). The inset shows mouse breast cancer cuboids.*

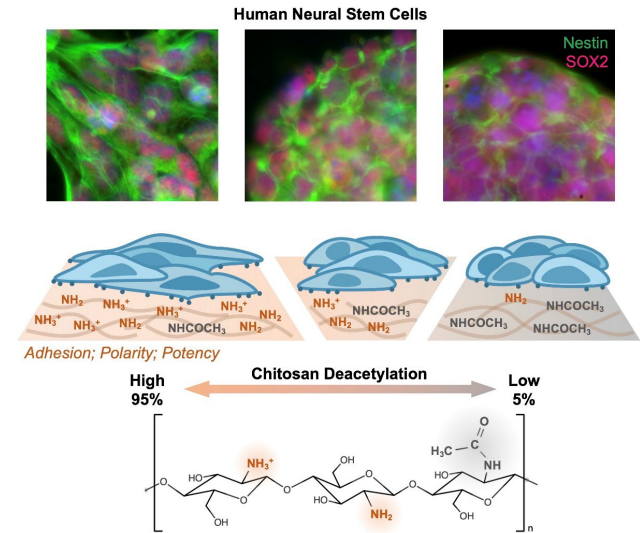
Dr. Tran Nguyen and Prof. Albert Folch, Bioengineering Dept., University of Washington. Work performed at the WNF.

This work was supported by the Brotman Baty Institute (UW).

*National Research Priority: NAE Grand Challenges—Engineer Better Medicines*

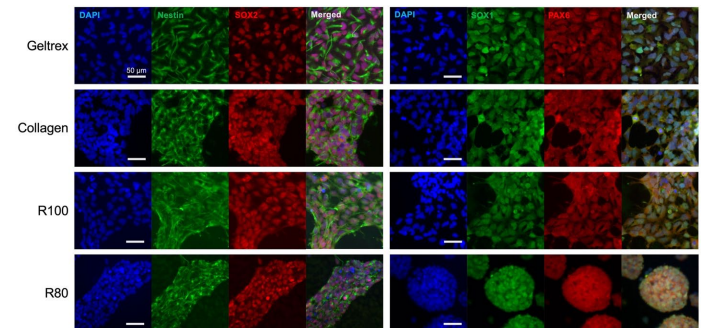
# Chitosan Film for Renewal of Human Neural Stem Cells

Stem cell therapy and research for neural diseases depends on reliable renewal of neural stem cells. Chitosan/chitin films with a wide range of degree of deacetylation (DD) were constructed and the effect of the DD on neural stem cell culture was investigated. We demonstrated that chitosan films with high DDs can serve chemically defined, xeno-free, and cost-effective substrates supporting renewal of human neural stem cells. This study demonstrate the potential of using chitosan film as a safer and cost-effective alternative to animal-source based substrates for culturing hNSCs.



Miqin Zhang, Dept of Materials Science and Engineering and Neurological Surgery. Work performed at NNI

This work was supported by Kyocera Corporation and NSF (NNCI-1542101). *Molecular Bioscience*, in press, 2022.



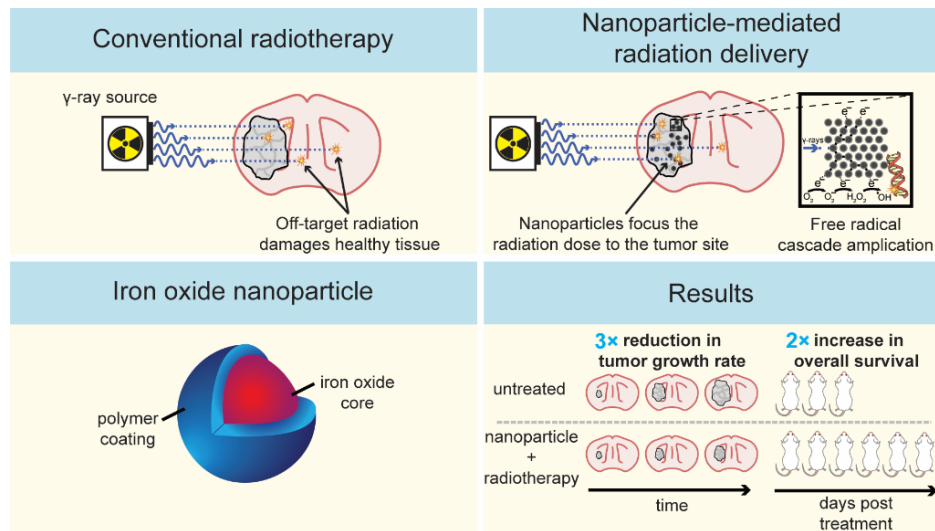
Characterization of stem cell pluripotency using immunocytochemistry staining

National Research Priority: NSF–Understanding the Rules of Life



# Iron Oxide Nanoparticle-Mediated Radiation Delivery for Glioblastoma Treatment

Radiotherapy is a mainstay adjunctive therapy for glioblastoma (GBM). Despite the outcome improvement achieved with radiation, GBM prognosis remains dismal. Here we introduce a tumor-targeted iron oxide nanoparticle (NP) that intensifies the energy transfer of conventional photon radiotherapy on a selective cellular basis. Mice bearing GBM tumors in orthotopic mouse model that received intravenous NP before irradiation demonstrated a 3-fold reduction in tumor growth and a 2-fold increase in survival. Cellular damage was investigated using *in vivo* magnetic resonance spectroscopy, which demonstrated increased therapeutic cytotoxicity specific to the tumor mass. Our work presents a viable therapeutic strategy to improve radiation therapy for GBM.



Miqin Zhang, Dept of Materials Science and Engineering and Neurological Surgery. Work performed at NNI.

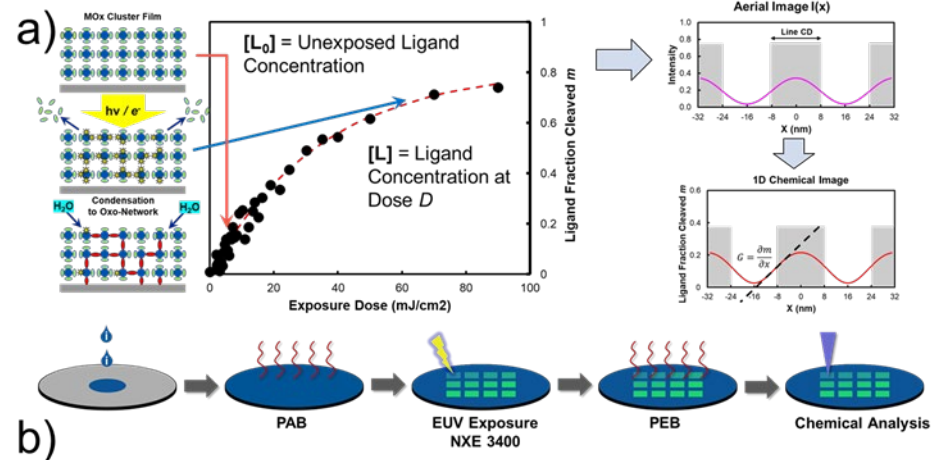
This work was supported by NIH (R01CA161953, R01EB026890), and NSF (NNCI-1542101). *Materials Today*, in press, 2022.

National Research Priority: NAE Grand Challenge—Engineer Better Medicines



# Metal Oxide EUV Resist Development

Inpria Corporation is the world leader in metal oxide (MOx) photoresists for advanced-node extreme ultraviolet (EUV) Lithography, a core technology for continued semiconductor scaling. The most important nexus of MOx resist development lies at the intersection of patterning chemistry and lithographic observables. By combining XPS, FTIR, NMR, Ellipsometry, and MS measurements of photoresists as a function of EUV exposure and process conditions, we are able to define and track chemical image formation and evolution throughout the lithographic process. Connecting this information back through resist structure and formulation allows the optimization of photoresist resolution, line-width roughness, and defectivity to meet the demands of global chipmakers at the 3-nm node and beyond.



a) Aligning chemical analyses with EUV exposures allows evaluation of resist chemical image at a given process condition.  
 b) SEM image of 9-nm MOx resist lines exposed on a 18-nm pitch using a high-NA EUV exposure tool.

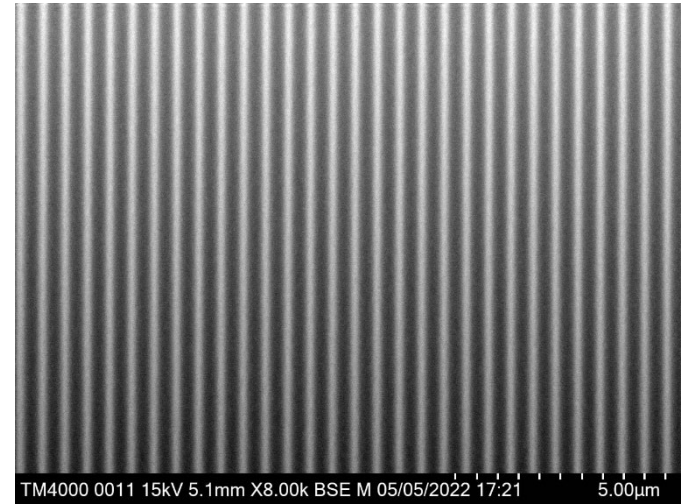
Meyers, S. T. et al. Inpria. Aspects of this work were performed at ATAMI, MaSC, & OPIC facilities in Corvallis, OR

Proc. SPIE 11609, Extreme Ultraviolet (EUV) Lithography XII, 116090K (22 February 2021).

National Research Priority: CHIPS+Science Act Research

# Transparent Inorganic Photoresists for High Index Photonics

High refractive-index transparent coatings patterned by nanoimprint lithography find wide application as engineered optics for photonic, electro-optic, data storage, imaging, and medical applications. Optics manufacturers currently deposit polymeric/organic thin films that fundamentally has low index of refraction and doesn't satisfy the market requirements. For example, augmented reality (AR) manufacturers are currently unable to manufacture high quality small form factor AR headsets because high refractive index coatings ( $n > 2.0$ ) are currently unavailable. With the support from NSF, Phosio is focusing on developing inorganic coatings with refractive indices as high as 2.4. Our method results in high-index coating solutions with low haze and high index ( $1.6 < n < 2.4$ ) that can be patterned with industry standard manufacturing equipment. Our technology provides a unique path to elevating field of view (FOV), miniaturizing AR glasses and improving image quality beyond current state-of-the-art AR headsets.



*Top-down SEM micrograph of the imprinted PhosioLux™ on glass. PhosioLux™ have ultra high indices of refraction which enable friendly, high-quality headsets for an enhanced user experience.*

Omid Sadeghi, Phosio Corp. Work performed at ATAMI, and MaSC.

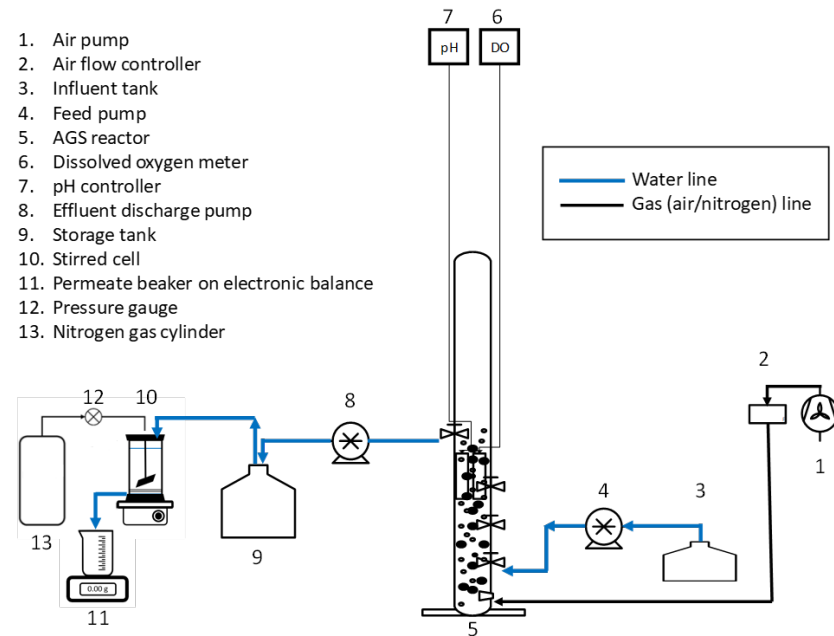
This work was supported by NSF, award number 2052401. U.S. Provisional Patent Application No. 63/347,956

*National Research Priority: CHIPS+Science Act Research*

# Ultrafiltration of Aerobic Granular Sludge Bioreactor Effluent

Tools at OPIC help the environmental engineers developing compact, higher efficiency wastewater treatment technologies.

- Aerobic granular sludge (AGS) reactor achieved excellent chemical oxygen demand (COD) and nutrient removal after complete granulation.
- Model results indicate cake layer formation is dominating fouling mechanism.
- The polyethersulfone (PES) membrane exhibited a better antifouling performance compared to polyvinylidene fluoride (PVDF) membrane.
- There appeared a threshold flux for fouling reversibility.



Schematic of side-stream aerobic granular sludge membrane bioreactor

Zhengjian Yang, Quang N. Tran, Xue Jin, School of CBEE, Oregon State University. Part of the characterization work was conducted at Oregon Process Innovation Center (OPIC).

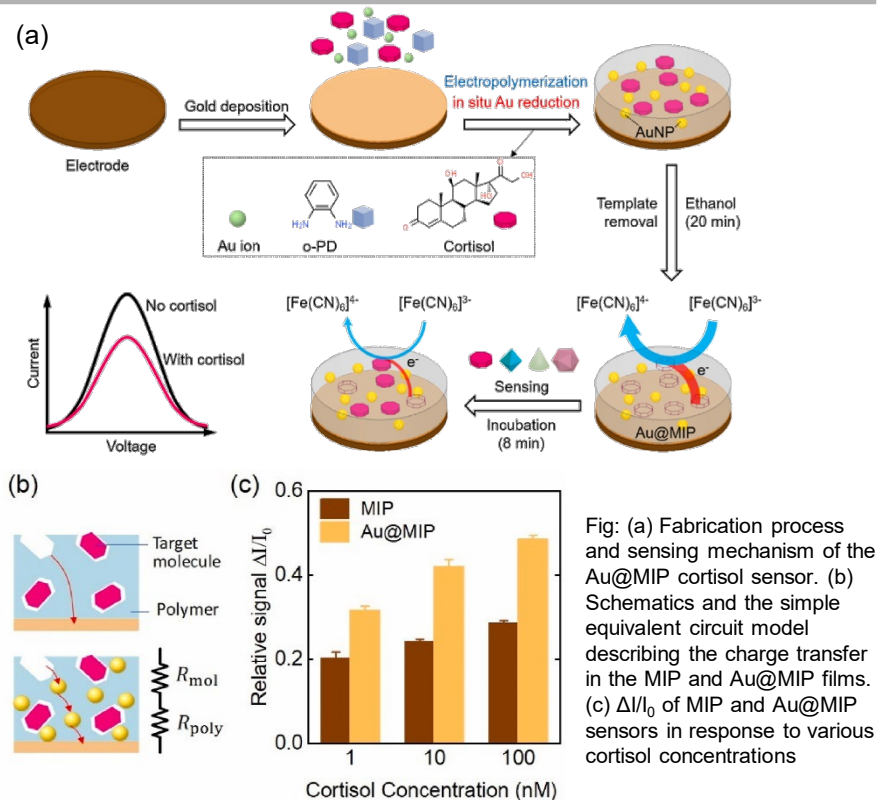
*Journal of Water Process Engineering*, 2022, 47, 102805.

National Research Priority: NAE Grand Challenges—Provide Access to Clean Water

# Nano Gold-doped Molecularly Imprinted Electrochemical Sensor for Rapid and Ultrasensitive Cortisol Detection

Tools at OPIC help the developing of rapid and sensitive electrochemical sensor for steroid hormone cortisol which can benefit the diagnosis of diseases related to adrenal gland disorders and chronic stress.

- Nano gold-doped molecularly imprinted polymer (Au@MIP) improved the transduction of cortisol binding into current change.
- In situ gold reduction upon electropolymerization of MIP increased the active surface area and conductivity of MIP.
- The co-deposition method improved detection sensitivity compared to the MIP embedded with pre-synthesized AuNPs.
- The sensor exhibited a 200 fM detection limit and selective detection against other steroid hormones.
- The sensor was successfully applied to determine cortisol in spiked saliva at normal and elevated levels.



Sanjida Yeasmin, Bo Wu, Ye Liu, Ahasan Ullah, Li-Jing Cheng, School of Electrical Engineering and Computer Science, Oregon State University. Part of the characterization work was conducted at OPIC.

Part of the research was supported by NSF-1810067 and NIH-1R21DE027170-01. *Biosensors and Bioelectronics*, 2022, 206 (15), 114142.

National Research Priority: NAE Grand Challenges—Advance Health Informatics



# Plastics To Fuel: Engineering a Novel Solution to Plastic Waste

OPIC Tools support our young undergraduate students to convert the plastic waste to an alternative diesel fuel. Fourier-transform infrared spectrometer provides the accurate identification for the fuel product.

In Skip Rochefort's polymer research lab, he and his students are testing a novel use for a well-established chemical process to tackle the challenge of plastic waste. Pyrolysis uses very high temperatures to break down plastic hydrocarbon chains in the absence of oxygen to create an alternative diesel fuel. Their goal is to install a pyrolysis reactor in remote and underserved communities, where it's difficult and expensive to dispose of plastic waste. Those plastics, converted to fuel, could then be used for boats, tractors and other diesel-powered equipment in those communities.



Photos courtesy of OSU Believe-It campaign, 2022

Willie (Skip) Rochefort, School of Chemical, Biological, and Environmental Engineering

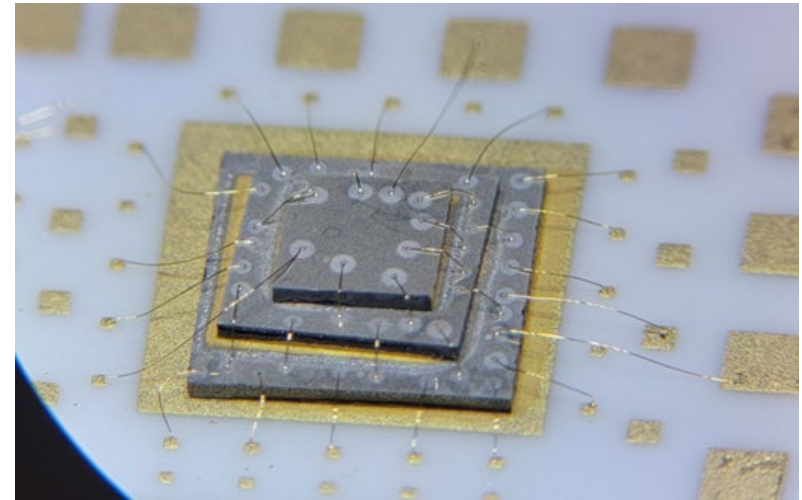
This project is supported by OSU Undergraduate Research, Scholarship and the Arts (URSA) Program

*National Research Priority: NAE Grand Challenges—Develop Carbon Sequestration Methods*

# 3D Stacking of SiC Integrated Circuit Chips with Gold Wire Bonded Interconnects for Long-Duration High-Temperature Applications

Silicon carbide integrated circuits (SiC ICs) have been demonstrated to operate at high temperatures, such as ~460 °C at the Venus' surface, for two months, and for over a year at 500 °C. At these high temperatures, the SiC integrated circuits and sensors need to be packaged in quite different ways than those below 300 °C. In addition, to integrate more devices into the limited footprint of the high-temperature circuit board, three-dimensional (3-D) packaging is a notable advantage.

In this work, 3-D stacking of SiC chips using a gold wirebonding interconnect is investigated. The gold bonding wire is used due to its mechanical robustness and chemical inertness at high temperatures. Triple-stacked SiC chips are bonded to each other and to the gold conduction pads on the alumina substrate with screen-printed gold pastes. The mechanical die shear test, wire pull tests, and interconnect electrical resistance tests are executed and analyzed before and after the 3-D SiC chip packages are subject to a 600 °C thermal aging process in the air for up to 10 days. This 3-D SiC chip packaging has promise for long-duration high-temperatures (up to 600 °C) applications and may be potential for use for applications such as Venus's surface sensing and telemetry.



Micrograph of wirebonded 3D SiC IC stack with Pt ohmic contact after 48 hours of the thermal aging process at 600°C.

Feng Li, Department of Electrical and Computer Engineering, University of Idaho. Work was partially performed at WNF.

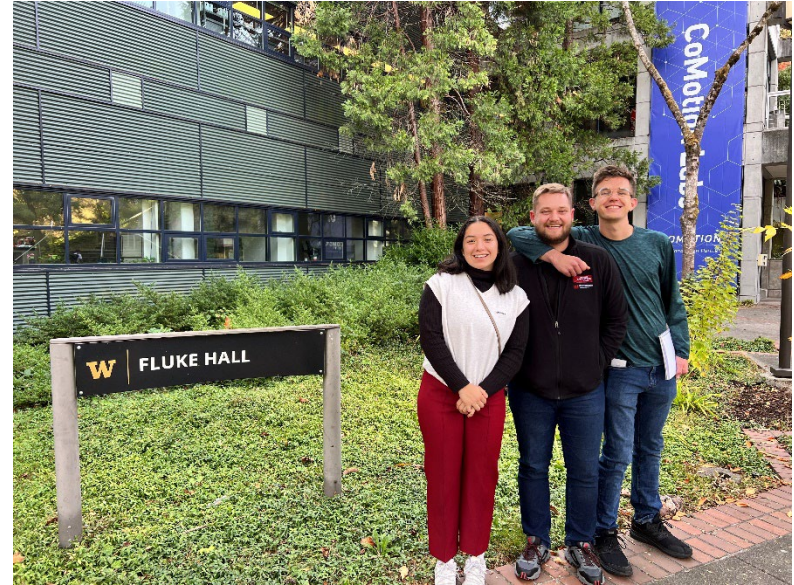
This work was supported by NASA EPSCoR. *IEEE Transactions on Components, Packaging and Manufacturing Technology*, vol. 12, no. 10, pp. 1601-1608, Oct. 2022.

*National Research Priority: NAE Grand Challenges—Engineer the Tools of Scientific Discovery*

# Resin Optical Characterization by Undergraduates

This project was designed primarily as a means for undergraduate students to gain training on materials optical metrology instruments in a cleanroom environment. This was to introduce students to the fields of materials research, semiconductor device fabrication techniques and metrology.

The project involved optical characterization of resins that are not currently reported in the literature as well as novel polymers.



*Undergraduate students that benefited by the cleanroom training. Students (left to right) are McKenzie Garcia, Helio Ramollari and Trevor Thomas.*

McKenzie Garcia, Helio Ramollari, Trevor Thomas and Dr. Philip Measor, Microdevices Lab, Engineering & Physics Department, Whitworth University. Work was performed at Washington Nanofabrication Facility (WNF).

This work was supported by the Murdock Foundation and Whitworth University. Publication expected in 2023.

*National Research Priority: NSF–NSF INCLUDES*

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# ***Research Triangle Nanotechnology Network (RTNN)***



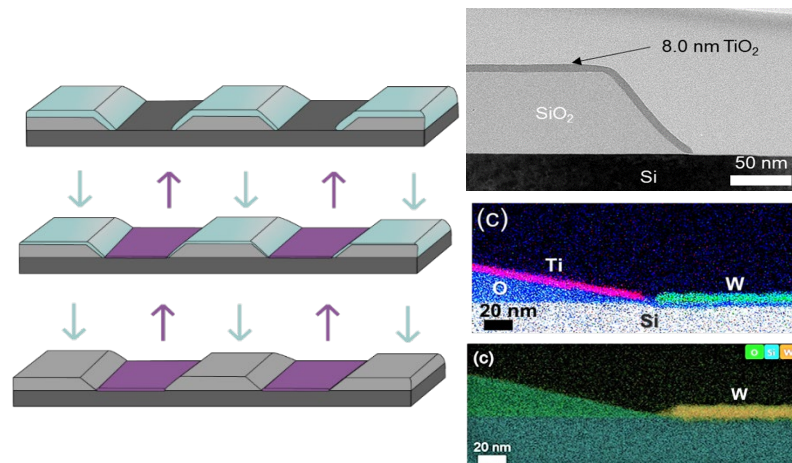
# Multimaterial Self-Aligned Nanopatterning by Simultaneous Adjacent Thin Film Deposition and Etching

One source of inefficient power use is *nano-scale pattern misalignment*, which can lead to excess resistance, open circuits, and electrical shorts

Mitigating inefficient energy use in semiconductor devices requires the development and integration of self-aligned bottom-up patterning techniques such as area selective deposition (ASD)

Combining W-ALD (Atomic Layer Deposition) and  $\text{TiO}_2$ -CVE (Chemical Vapor Etching) into a single process results in W-ASD in the regions where  $\text{TiO}_2$  is initially absent, and selectivity is maintained throughout the process

Simultaneous deposition and etching demonstrates opportunities for low-temperature bottom-up self-aligned patterning for electronic and other nanoscale systems



Initial substrates used in this study consisted of 8-16 nm of  $\text{TiO}_2$  line space patterns supported in  $\text{SiO}_2$  and  $\text{Si}$  as the growth surface. After exposing these substrates to 15  $\text{SiH}_4/\text{WF}_6$  ASD cycles,  $\text{W}$  is deposited in the  $\text{Si}$  regions and  $\text{TiO}_2$  is simultaneously removed in an etching reaction. After 20 ASD cycles, the remaining  $\text{TiO}_2$  is etched away, leaving 10 nm of selectively deposited  $\text{W}$  on  $\text{Si}$ . SEM/EDS on the right correspond to diagrams of each process step shown on the left

Seung Keun Song, Jung-sik Kim, Hannah Margavio, Gregory N Parsons; Department of Chemical and Biomolecular Engineering, NC State. Work performed at NC State's Analytical Instrumentation Facility, Duke's Shared Materials Instrumentation Facility, and UNC's Chapel Hill Analytical and Nanofabrication Laboratory

This work was supported by Semiconductor Research Corporation, Task 2729.001 and Task 3036.001 and NSF Award # 1704151. *ACS Nano* 15 (2021) <https://doi.org/10.1021/acsnano.1c04086>

National Research Priority: DoD Critical Technology Area—Advanced Materials

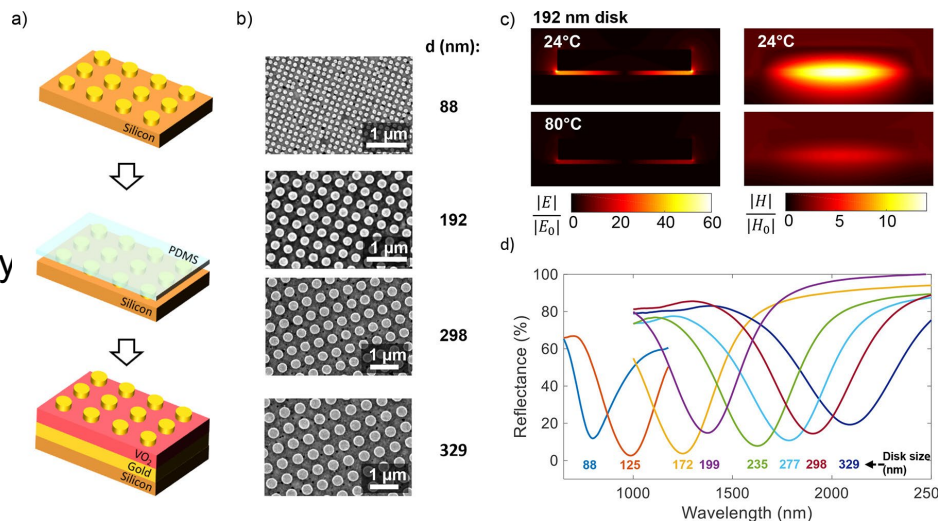
# Actively Tunable Metasurfaces via Plasmonic Nanogap Cavities with sub-10 nm VO<sub>2</sub> Films

Actively tunable optical materials with engineered subwavelength structures could enable a new generation of optoelectronic devices

This work integrates sub-10-nm-thick VO<sub>2</sub> films with plasmonic nanogap cavities made via E-Beam Lithography (EBL) to demonstrate tunable, spectrally selective absorption across 1200 nm in the near-infrared (NIR) range

Upon inducing the phase transition via heating, the absorption resonance is blue-shifted by as much as 60 nm. This process is reversible upon cooling and repeatable over more than 10 temperature cycles

Ultrathin VO<sub>2</sub> films deposited by ALD, as demonstrated here, create new potential architectures and applications to provide reconfigurability including three-dimensional, flexible, and large-area structures



- Fabrication process for transferring EBL-fabricated gold nanodisks onto the VO<sub>2</sub> film
- SEM images of gold nanodisks for a few of the fabricated sizes
- Simulated electric (left) and magnetic (right) field profiles at 24 and 80 °C for the nanogap cavity structure. An incident plane wave with wavelength matching the room-temperature resonance of the nanocavity at 1495 nm is utilized
- Room-temperature reflectance spectra for metasurfaces consisting of eight different sizes of nanodisks

Andrew M. Boyce, Qixin Shen, Jon W. Stewart, Maiken H. Mikkelsen, Department of Electrical and Computer Engineering, Department of Physics, Duke University. Work performed at NC State's Analytical Instrumentation Facility (RTNN) and Joint School of Nanoscience and Nanoengineering (SENIC).

The work was supported by ONR. N00014-17-1-2589, ARO W911NF1610471, AFOSR FA9550-18-1-0326 and FA9550-21-1-0312. *Nano Letters* (2022) doi.org/10.1021/acs.nanolett.1c04175 (in press).

National Research Priority: DoD Critical Technology Area—Advanced Materials

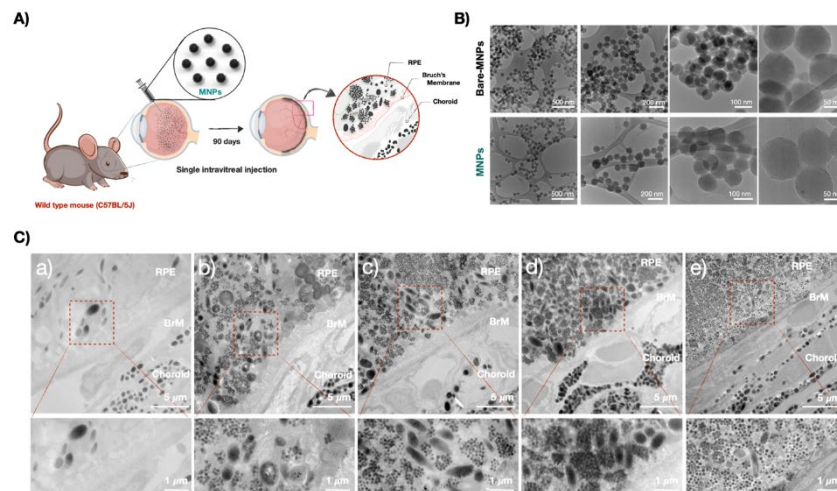
# Melanin Nanoparticles as an Alternative to Natural Melanin in Retinal Pigment Epithelium (RPE) Cells and Their Therapeutic Effects against Age-Related Macular Degeneration

In contrast to skin melanin, melanin in the RPE does not regenerate and functions as a potential radical scavenger and photoprotective agent in eyes

Protective effects of melanin against oxidative stress are known to decline with increasing age

This work shows that Melanin Nano Particles (MNPs) are promising substitutes for natural melanin in the RPE of eyes

MNPs may be promising as a natural radical scavenger against oxidative stress in Reactive Oxygen Species (ROS) related diseases, such as Age-Related Macular Degeneration (AMD)



A) Illustration of intracellular trafficking showing that MNPs reach their target and are stable in the retinal pigment epithelial (RPE) layer for up to 3 months via a single-dose intravitreal (IVT) injection. B) Transmission electron microscopy (TEM) images of bare-MNPs (no PEGylation) and MNPs (PEGylation). C) Bio-TEM images show intracellular trafficking of MNPs. a) before and after IVT administration

Yong-Su Kwon, Min Zheng, Alice Yang Zhang, and Zongchao Han, UNC Chapel-Hill. Work performed at UNC's Chapel Hill Analytical and Nanofabrication Laboratory.

This work was supported by Bright Focus Foundation (M2019063 to Z.H. and M2022001F to Y.-S.K.) and the Edward N. & Della L. Thome Memorial Foundation (138289, Z.H.). *ACS Appl Mater Interfaces* 12(43) 48845-48853 43 (2020)

National Research Priority: NSF–Understanding the Rules of Life  
and NAE Grand Challenge–Engineer Better Medicines

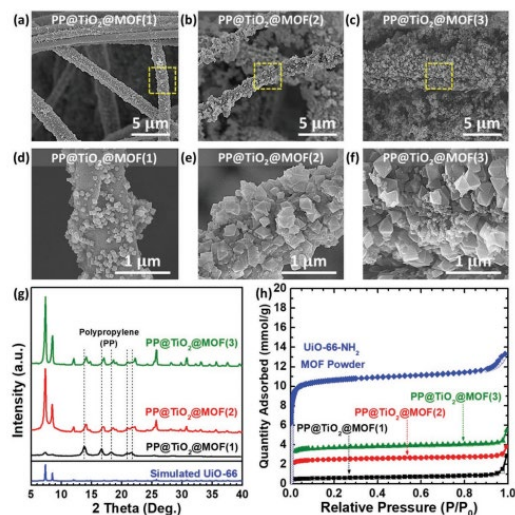


# Highly Breathable Chemically Protective MOF-Fiber Catalysts

A systematic structure–property–function analysis is provided for Zr-based metal–organic frameworks (MOFs) coated onto fibers

MOF-fiber composite shows a rapid catalytic hydrolysis rate for a chemical warfare agent simulant, p-nitrophenyl phosphate with  $t_{1/2} < 5$  min, and a significant permeation restriction of a real agent (soman nerve agent) through the composite

The observed moisture vapor transport rate of  $15,000 \text{ g m}^{-2} \text{ day}^{-1}$  for the composite is notably superior to that of other commercially available chemical-protective fabrics and overcomes the breathability/detoxification trade-off and shows promise for the materials to be deployed in a realistic field



SEM images of non-modulated UiO-66-NH<sub>2</sub> MOF coated fibrous mats: a) MOF growth with no seeds on PP@TiO<sub>2</sub>, b) subsequent MOF growth to (a), and c) succeeding MOF growth to (b). d–f) Enlarged SEM images in the dotted square area of (a–c), respectively. g) XRD patterns and h) N<sub>2</sub> isotherms at 77 K of the synthesized MOF coated textiles together with simulated UiO-66 pattern, respectively.

Dennis T Lee, Zijian Dai, Gregory W. Peterson, Morgan G. Hall, Natalie L Pomerantz, Nicole Hoffman, Gregory N Parsons, Dept. of Chemical and Biomolecular Engineering, NC State University, Aberdeen Proving Ground, US Army CCDC Chemical and Biological Center, US Army CCDC Soldier Center, Innovation Center for Textile Science and Technology, Donghua University, Dept. of Chemical and Biomolecular Engineering & Institute for NanoBiotechnology, Johns Hopkins University. Work performed at NC State's Analytical Instrumentation Facility.

This work was supported by ARO Cooperative Agreement Number W911NF-19-2-0154 and DTRA project CB3934. *Advanced Functional Materials* 32,6 (2021)



# High-Throughput Oxygen Chemical Potential Engineering of Perovskite Oxides for Chemical Looping (CL) Application

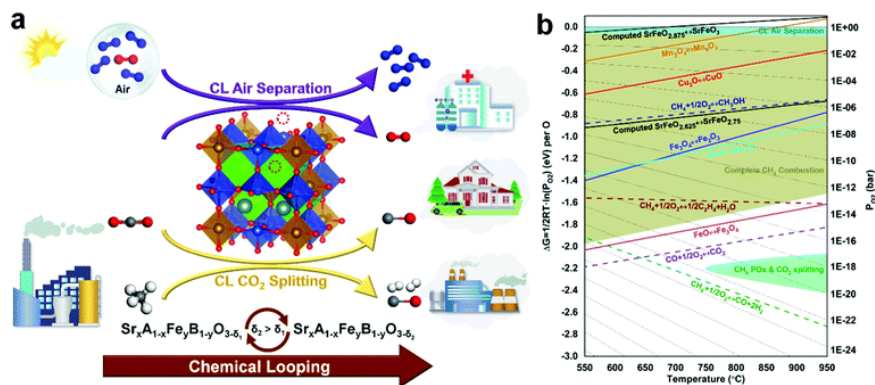
CL involves decoupling an overall reaction into multiple sub-reactions, whereby an intermediate facilitates such sub-reactions by releasing or replenishing oxygen under temperature or pressure swings

This work seeks to rationally substitute  $\text{SrFeO}_{3-\delta}$  A- and/or B-site cations to tailor the equilibrium oxygen partial pressure over 20 orders of magnitude

Over 2,400 perovskite-phase structures were investigated using high-throughput density functional theory (DFT) and high-entropy perovskites were screened via machine learning

19 previously reported oxygen carriers were correctly identified by the algorithm, while 15 new oxygen carriers with superior redox performance were identified

These results support the effectiveness of high-throughput approaches for accelerated materials discovery



Chemical looping strategy. (a) Schematic illustration and potential applications. (b) Ellingham diagram depicting the correspondence between oxygen carrier redox properties and applications

Xijun Wan, Yunfei Gao, Emily Krzystowczyk, Sherafghan Iftikhar, Jian Dou, Runxia Cai, Haiying Wang, Chongyan Ruan, Sheng Ye and Fanxing Li, Dept. of Chemical and Biomolecular Engineering, NC State University, College of Chemistry & Chemical Engineering, Northeast Petroleum University, School of Chemistry and Materials Science, University of Science and Technology of China. Work performed at NC State's Analytical Instrumentation Facility.

This work was supported by U.S. DOE (Award No. FE0031521), NSF (Grant No. CBET-1510900). *Energy and Environmental Science* (2022) <https://doi.org/10.1039/D1EE02889H>

National Research Priority: DOE Office of Science Priority—Artificial Intelligence and Machine Learning

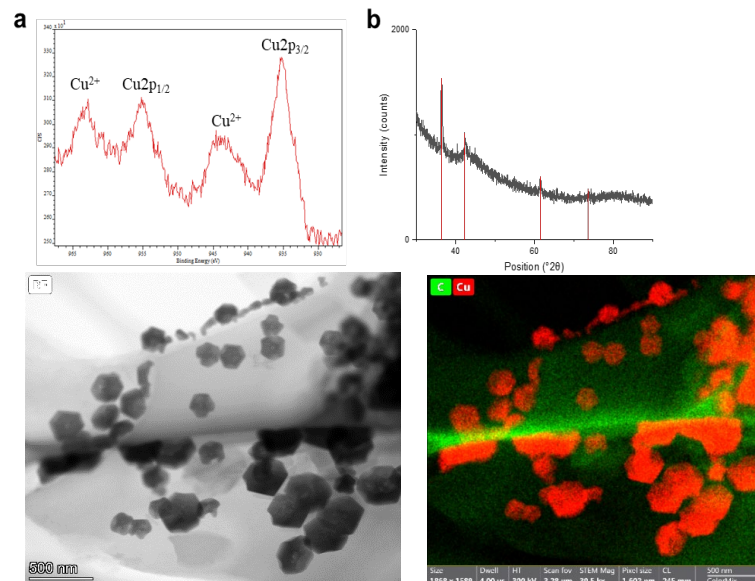
# Materials Characterization of a Sensor Based on a Carbon-Copper Composite for the Detection of Glyphosate

Glyphosate is a widely used herbicide. However, residues in the environment can cause negative effects to non-target organisms and humans

There is a need develop new testing technologies that enable the detection of the presence of pesticide residues in water sources, especially in developing countries where access to standard laboratory methods is cost prohibitive

An electrochemical sensor was developed based on a three-electrode system fabricated via UV laser-inscribing polyimide film and incorporating functionalized turbostratic graphene decorated with copper nanoparticles with an affinity for organophosphates

Sensitivity of this sensor was highest for glyphosate, showing promise for monitoring pesticides



Top Left: X-ray photoelectron spectroscopy (XPS) spectrum of LIG-Cu electrodes. Top Right: X-ray Diffraction of LIG-Cu electrodes. Red lines indicate peaks corresponding to the spectrum of copper oxide., Bottom: STEM image of particles and accompanying EDS image (right)

David Bahamon-Pinzon, Diana Vanegas; Environmental Engineering and Earth Sciences, Clemson University. Work performed at NC State's Analytical Instrumentation Facility.

*Microchimica Acta*, 189(7), 1-11 (2022)

National Research Priority: NSF–Growing Convergence Research and  
NAE Grand Challenge–Provide Access to Clean Water

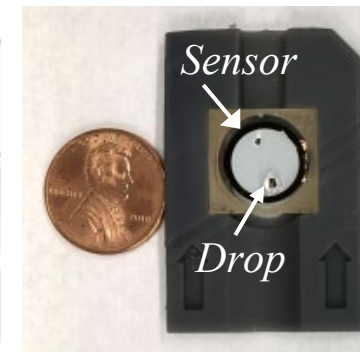
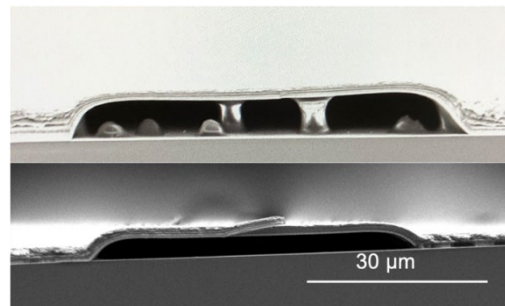
# Microfluidic Quartz Resonator Based Viscometers for Injectability Pre-screening of Protein-based Therapeutics

Protein-based therapeutics need to be formulated at high concentrations to achieve injections with effective dosing

Existing viscometers consume very high volumes of liquid sample with shear-rate ranges not adequate for detecting injectability, researchers cannot identify uninjectable molecules until manufacturing is scaled-up significantly

This work presents microfluidic quartz resonators utilized to behave as a capillary viscometer with a wide shear-rate range

The resulting sensor can measure injectability using only 5 microliters in 5 minutes, which can significantly de-risk development of protein-based therapeutics



*(Left) SEM is used for determining if the microfluidic channels (cross-sections shown) cleaned properly, (Right): shows a fabricated microfluidic quartz viscometer in a plastic holder for easy handling*

Zehra Parlak, Daniel French, Stefan Zauscher; QATCH Technologies LLC, Duke University. Quality control and imaging performed at Duke's Shared Materials Instrumentation Facility.

This work was supported by NSF SBIR/STTR contracts 2025974, 70NANB20H116, 1R43GM139438-01, 70NANB19H042, and 1721833. Patent Applications: WO2021076867A1, EP3516384A2

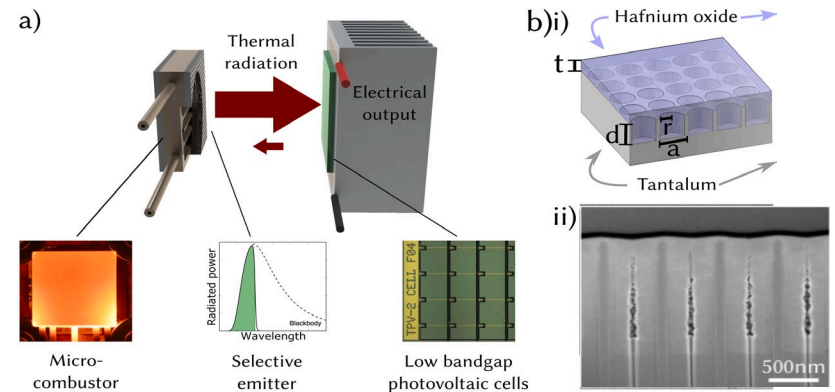
*National Research Priority: NSF–Understanding the Rules of Life and  
NAE Grand Challenge–Engineer Better Medicines*

# Hafnia-Filled Photonic Crystal Emitters for Mesoscale Thermophotovoltaic (TPV) Generators

One way to increase both TPV power and efficiency is to use a spectrally selective emitter

One promising class of TPV emitters is 2D photonic crystals (PhCs) made of tantalum, however, uniform/thin capping layers are hard to achieve

This work presents **a process of planarization and physical dry etching that results in reduced roughness and better thickness control of Ta capping layers**, paving the way toward efficient, practical, and portable mesoscale generators for off-the-grid applications



- (a) In a TPV system, a heat source heats up an emitter that emits thermal radiation that is then converted to electricity by a low bandgap photovoltaic cell. This work uses a microcombustor (for fuel combustion) and a photonic crystal (PhC) broadband selective emitter on the hot side
- (b) (i) This PhC is both filled and capped with hafnium oxide (HfO), which allows for high in-band ( $2\ \mu\text{m}$  cutoff) emittance and low out-of-band ( $2\ \mu\text{m}$  cutoff) emittance for both normal ( $0^\circ$ ) and off-normal ( $45^\circ$ ) incidence, as well as hemispherical. (ii) cross-section SEM image of capping layer

Reyu Sakakibara, Veronika Stelmakh, Walker R. Chan, Robert D. Geil, Stephan Krämer, Timothy Savas, Michael Ghebrehan, John D. Joannopoulos, Marin Soljačić, Ivan Čelanović ; Massachusetts Institute of Technology, Mesodyne, Inc., UNC-Chapel Hill, Harvard University, LumArray, Inc., U.S. Army Natick Soldier Research, Development, and Engineering Center. Work performed in part at UNC's Chapel Hill Analytical and Nanofabrication Laboratory.

This work was supported by the ARO through the Institute for Soldier Nanotechnologies under Award No. W911NF-13-D-0001 and W911NF-18-2-0048. *Solar Energy Materials and Solar Cells*, 238 (2022)

National Research Priority: DoD Critical Technology Area—Advanced Materials



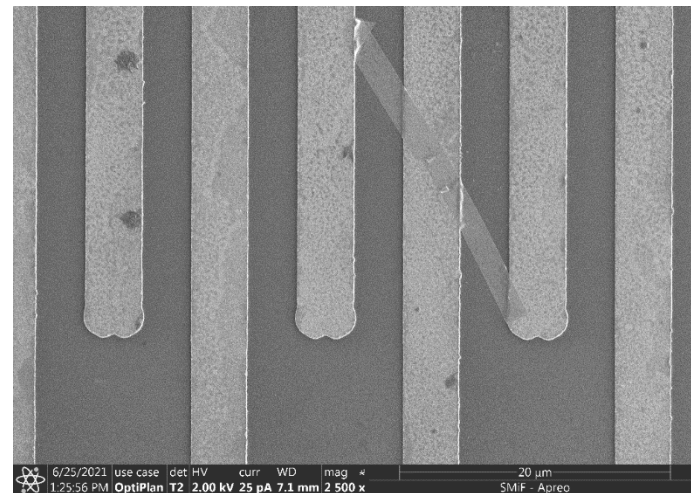
# Probing Charge Transfer in 2D MoS<sub>2</sub>/tellurene type-II p-n Heterojunctions

2D heterostructures offer new opportunities for harnessing a wider range of the solar spectrum in high-performance photovoltaic devices

This work explores type-II p-n heterojunction, by exploiting air-stable tellurene (Te) in combination with MoS<sub>2</sub>, to study its charge transfer for photovoltaic applications

The charge transfer of MoS<sub>2</sub>/Te heterojunction is confirmed by photoluminescence spectroscopy, Raman spectroscopy and Kelvin probe force microscopy. The exciton binding energy for MoS<sub>2</sub>/Te heterojunction is estimated to be around 10 meV, which is much lower than that for monolayer MoS<sub>2</sub>

This strategy can be exploited to develop next-generation intrinsically ultrathin light-harvesting devices



*SEM image of solution-processed tellurene sitting between pre-patterned gold electrodes*

Basant Chitara, Kunyan Zhang, Shengxi Huang, Fei Yan; Department of Chemistry and Biochemistry, North Carolina Central University, Department of Electrical Engineering, Pennsylvania State University. Work performed at Duke's Shared Material Instrumentation Facility.

This work was supported by NSF Award # 1831133 and #2122044, and ECCS-1943895. *MRS Communications* (2021) <https://doi.org/10.1557/s43579-021-00117-w>

*National Research Priority: NAE Grand Challenge—Make Solar Energy Economical*

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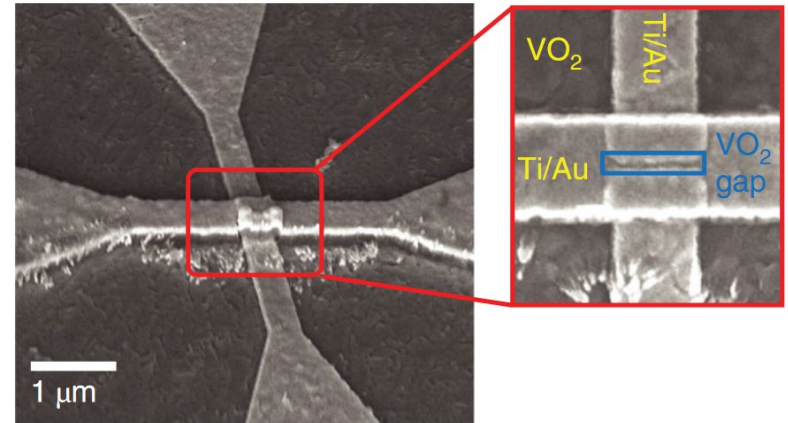
# ***San Diego Nanotechnology Infrastructure (SDNI)***

# Energy-efficient Mott activation neuron for full-hardware implementation of neural networks

Inspired by the rapid advances in neural sciences and brain functions, a new paradigm in computing and data processing system emerges, which uses the neural network architectures with deep learning and artificial intelligence (DL/AI). The work lays the foundation for large-scale, highly parallel, and energy-efficient in-memory computing systems for neural networks.

SDNI researchers in quantum materials, electrical engineering, and computer sciences have applied a wide variety of tools at SDNI to demonstrate the world's first LeNet-5 network with Mott activation neurons with >98% accuracy on the MNIST dataset. The incorporation of quantum materials for nano-scaled Mott activation neurons is key to the research success because it allows energy-efficient neuron-like activation and integration with a conductive bridge random access memory (CBRAM) crossbar array by occupying only one hundreds of the area of conventional CMOS implementation.

The research impact goes beyond the field of neural computing as the technologies involve the integration of quantum materials and CBRAM, presenting a new nanotechnology paradigm beyond conventional Si CMOS technology.



A scanning electron microscope image of the Mott device (scale bar, 1  $\mu\text{m}$ ). The inset shows the nanowire heater on the top of the 50 nm VO<sub>2</sub> gap.

Duygu Kuzum (Electrical and Computer Engineering) and Ivan K. Schuller (Physics), UCSD. The fabrication of the devices was performed at the San Diego Nanotechnology Infrastructure (SDNI) of the University of California San Diego, supported by the National Science Foundation (ECCS-1542148).

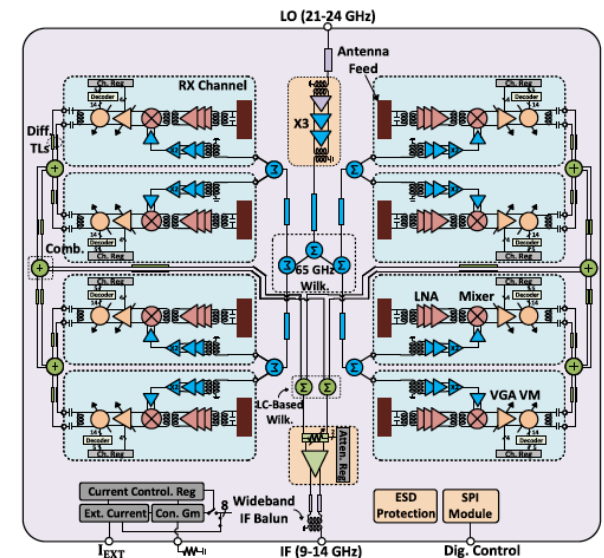
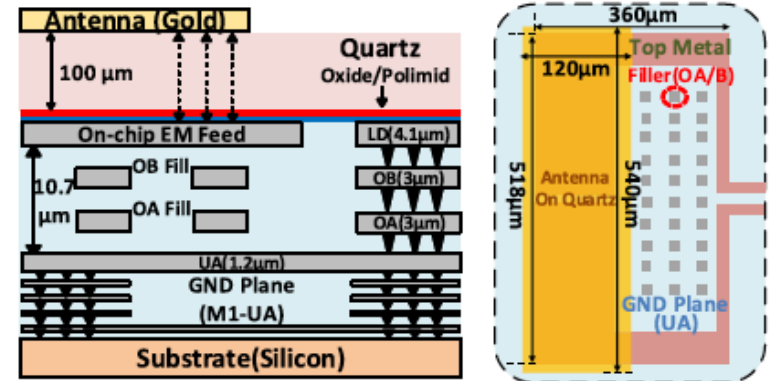
National Research Priority: NSF–Quantum Leap

# 140GHz Wafer Scale Integration of IF Beamforming Phase-Array Receiver in CMOS RFSOI

Performed pioneering work for 6G communications by demonstrating 140-GHz eight-element wafer-scale phased-array receiver based on intermediate frequency (IF bandwidth of 9.5–12.5 GHz) beamforming with 5-bit phase and 4-bit gain control. The chip contains a shared local-oscillator (LO) multiplier chain and distribution network, active combiners, LC-based combiners, digitally tuned attenuators for a near system-on-chip solution.

- The chip is designed in the GlobalFoundries 45RFSOI process (floating-body thin-oxide RF 40-nm CMOS-SOI transistor is used for RF/LO/IF circuits design).
- The antenna array was fabricated on a quartz substrate.

Gabriel Rebeiz (UCSD). The heterogeneous integration of antenna array on quartz substrate with CMOS-SOI was performed at the San Diego Nanotechnology Infrastructure (SDNI) of UCSD by SDNI technical staff.



National Research Priority: CHIPS+Science Act Research

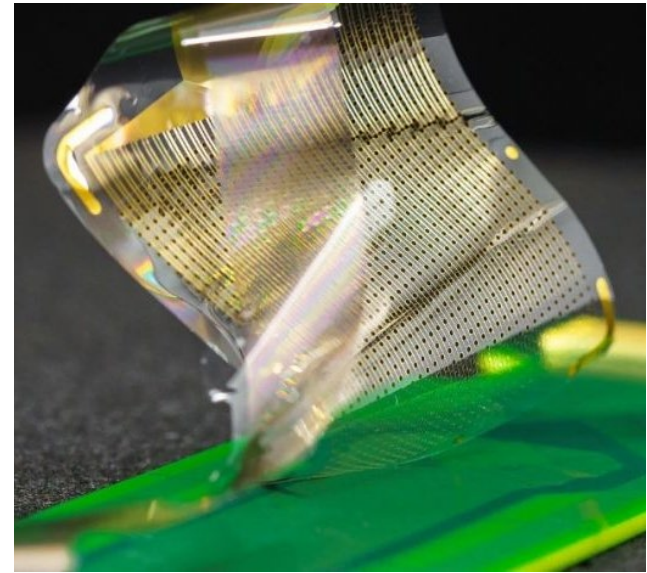


# Human brain mapping with multithousand-channel PtNRGrids resolves spatiotemporal dynamics

The multidisciplinary, multi-institute team, led by a SDNI researchers, utilized SDNI facilities to develop scalable manufacturing processes and dense connectorization to achieve reconfigurable thin-film, multi-thousand channel neurophysiological recording grids using platinum-nanorods, called PtNRGrids.

With PtNRGrids, thousands of channels of small low impedance provide unparalleled spatial and temporal resolution over a large cortical area. In the clinical setting, PtNRGrids can resolve fine, complex temporal dynamics from the cortical surface in an awake human patient and identify the spatial spread and dynamics of epileptic discharges in a patient undergoing epilepsy surgery at 1 mm spatial resolution. Besides its scientific impact, these thin, pliable grids of embedded electrocorticography sensors have significant clinical impact by offering neurosurgeons brain-signal information directly from the brain's cortex surface in 100x higher resolution than what is available today.

The high-resolution data in both time and space unlocks many new possibilities for uncovering new knowledge about how the brain works.



*Optical image of the 32 mm by 32 mm large-area fabrication of 1024-channel electrode array . Microscale features of PtNR grids include contacts, metal leads, and perfusion holes.*

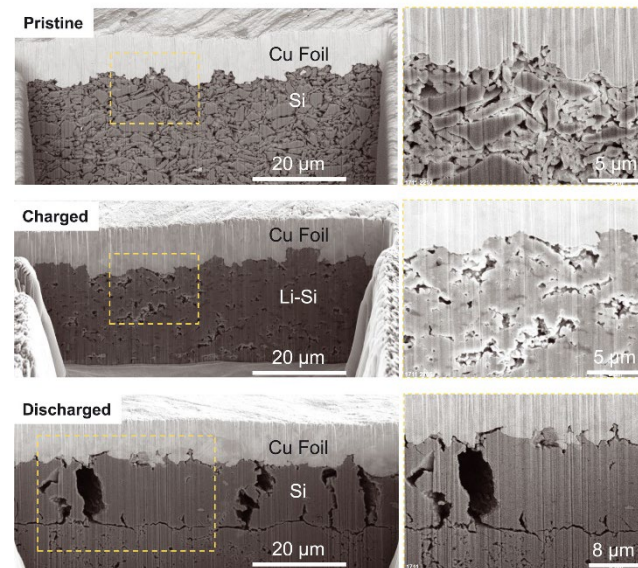
Shadi A. Dayeh, Electrical and Computer Engineering, UCSD. Research involved UCSD, Oregon Health and Science University, and Massachusetts General Hospital. PtNRGrid fabrication was conducted in the nano3 cleanroom facilities at UCSD's Qualcomm Institute.

*National Research Priority: NSF–Growing Convergence Research*

# Carbon-free high-loading silicon anodes enabled by sulfide solid electrolytes

The development of silicon anodes for lithium-ion batteries is impeded by poor interfacial stability against liquid electrolytes, which results in poor cycling and shelf life caused by continuous solid electrolyte interphase (SEI) growth between the highly reactive Li-Si alloy and organic liquid electrolytes. Two key challenges to apply Si to LIBs are: (i) stabilizing the Li-Si | electrolyte interface to prevent continuous SEI growth and trapped Li-Si accumulation, and (ii) mitigating growth of new interfaces induced by volume expansion that results in Li<sup>+</sup> consumption.

The research, led by SDNI, enables the stable operation of a  $\mu$ Si anode by using the interface passivating properties of sulfide solid electrolytes. The research produces a  $\mu$ Si electrode consisting of 99.9 wt %  $\mu$ Si in  $\mu$ Si||SSE||lithium nickel cobalt manganese oxide (NCM811) cells that overcome both the interfacial stability challenges of  $\mu$ Si and the current density limitations of all solid-state batteries (ASSBs). Microsilicon full cells demonstrated in this research achieve high areal current density, wide operating temperature range, and high areal loadings.



*A scanning electron microscope image showing the lithiation and delithiation of 99.9 wt % Si. Pristine porous microstructure of  $\mu$ Si electrode. Charged state with densified interconnected Li-Si structure. Discharged state with void formation between large dense Si particles.*

Ying Shirley Meng (Nanoengineering) and Zheng Chen (Nanoengineering), UCSD. Research involved UCSD and LG Energy Solution. This work was performed in part at the San Diego Nanotechnology Infrastructure (SDNI) of UCSD, a member of the National Nanotechnology Coordinated Infrastructure, which is supported by the National Science Foundation (grant ECCS-1542148).

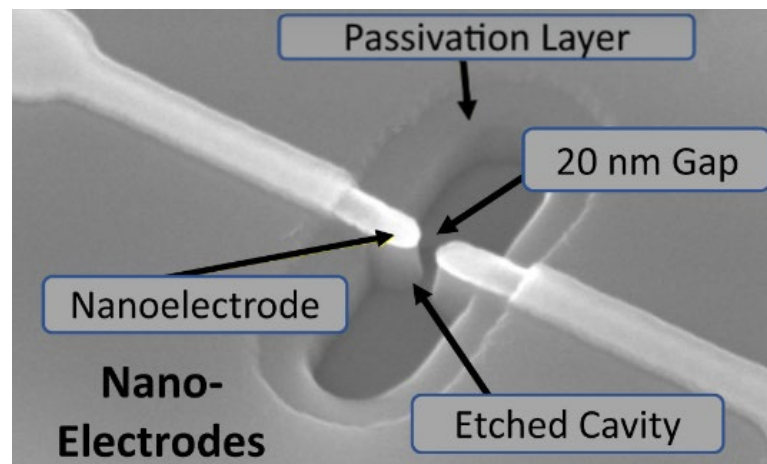
*National Research Priority: NSF–NSF 2026*

# ***Molecular electronics sensors on a scalable semiconductor chip: A platform for single-molecule measurement of binding kinetics and enzyme activity***

Roswell Biotechnologies is a San Diego biotechnology start-up and a beneficiary of SDNI's deep nano-scaled manufacturing capabilities. In collaboration with scientists and engineers from Harvard, UCSD, and Rice University, the Roswell team demonstrates molecular electronics on a semiconductor chip for single-molecule measurement of binding kinetics & enzyme activity.

The chip uses single molecules as universal sensor elements in a circuit to create a programmable biosensor with real-time, single-molecule sensitivity and nearly unlimited scalability in sensor pixel density. The sensor is programmed by attaching a probe molecule to the molecular wire, via a central, engineered conjugation site between nanoelectrodes. The observed current provides a direct, real-time electronic readout of molecular interactions of the probe. These picoamp-scale current-versus-time measurements are read out from the sensor array in digital form, at a rate of 1000 frames per second, to capture molecular interactions data with high resolution, precision, and throughput.

This ultra-scalable chip unlocks the possibility for highly distributed sequencing for personal health or environmental monitoring, and for future ultra-high throughput applications such as Exabyte-scale DNA data storage.



*SEM image of sensor nanoelectrodes, showing the 20 nm gap for the molecular bridge. Nanoelectrodes shown are fabricated using electron beam lithography at SDNI and other CMOS foundry-compatible processes.*

The chip was developed by Roswell Biotechnologies. Research also involved UCSD, Rice, and Harvard Medical School. This work was performed in part at the San Diego Nanotechnology Infrastructure (SDNI) of UCSD, a member of the National Nanotechnology Coordinated Infrastructure, which is supported by the National Science Foundation (grant ECCS-1542148).

*National Research Priority: NSF–Understanding the Rules of Life*

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# ***Soft and Hybrid Nanotechnology Experimental (SHyNE) Resource***

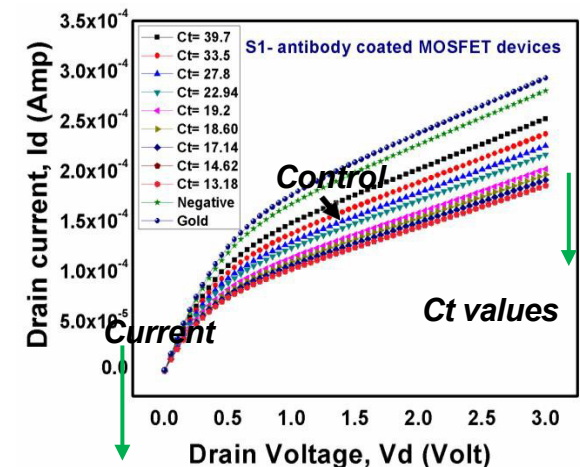
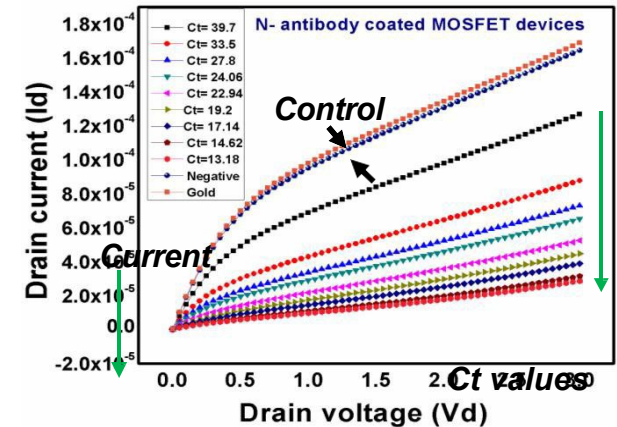


# Nanomechanical Cantilevers as a Multiplexed Diagnostics System

MOSFET embedded microcantilever devices can be used for multiplexed detection of COVID proteins. We first conducted optimized MOSFET detection of SARS-CoV-2 through patients' nasopharyngeal swab. Nine patients' samples with a Ct value varying from 13.8 to 39.7 were employed here. The MOSFET drain current measurements were performed by using two different sets of MOSFET devices coated with antibodies for SARS-CoV-2 N and S1 proteins to compare the current change. Separation of different curves for different Ct values were significantly large in anti-N-antibody coated devices compared to anti-S1- antibody coated MOSFET devices, resulting less change in the current in latter's case. There was no significant change in the current for the COVID negative and control samples in both cases within error bars. Here, the data collected from the gold coated cantilevers with conjugated antibodies (with no antibody-antigen interaction) is used as the control sample. MOSFET sensor can be developed as a switch for POC applications which shows the positive detection signal above a certain threshold of viral load (red zone) and below which it exhibits a linear regime (green zone).

D. Agarwal, Shekhawat, G.S. and Vinayak Dravid et al. Northwestern University. Work performed at SHyNE Resource.

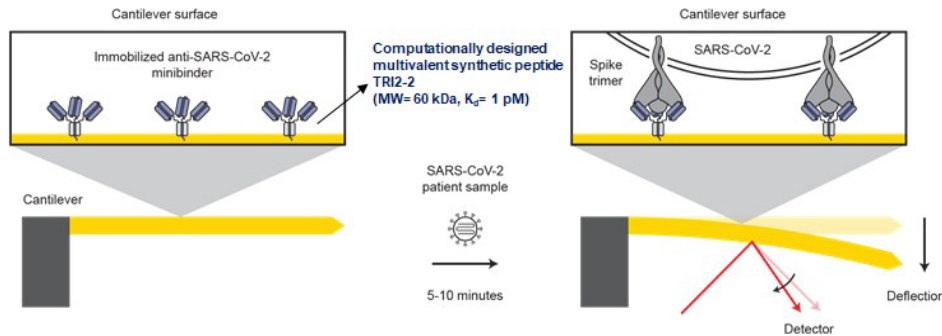
This project received support from the SHyNE Resource (NSF ECCS-2025633). *Biosensors and Bioelectronics* 195, 113647 (2022).



National Research Priority: NSF–Understanding the Rules of Life

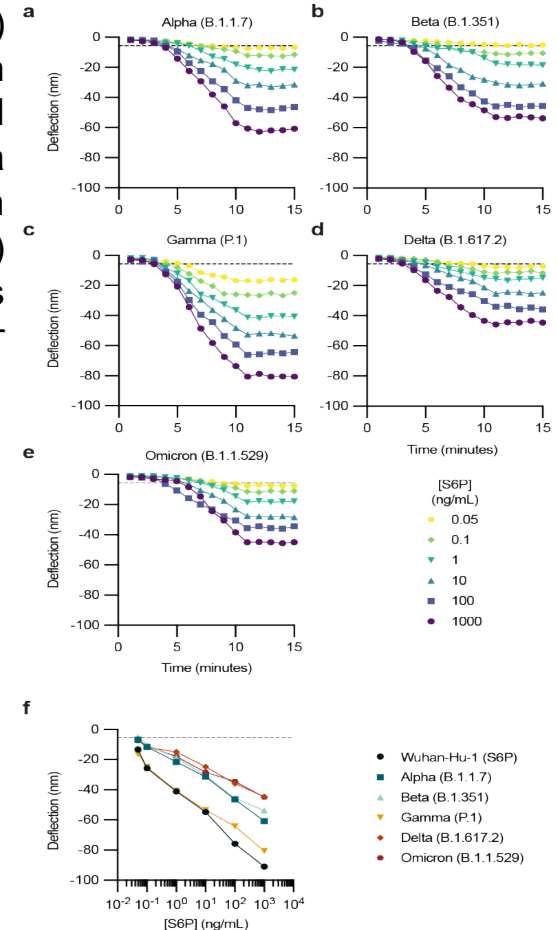
# SARS-CoV-2 Variant of Concerns (VOC) Detection using the Synthesized Peptides

We employed computationally designed multivalent minibinders (TRI2-2) immobilized on a microcantilever surface using synthetic biology approach for COVID-19 variant of concerns (VOC) detection. We demonstrated the ability of cantilevers to sense S trimer corresponding to the Alpha (B.1.1.7), Beta (B.1.351), Gamma (P.1), Delta (B.1.617.2), and Omicron (B.1.1.529) variants of concern along with the wild type (Wuhan) strain. The sensor exhibits rapid (< 5 min) detection of the target antigens down to concentrations of 0.05 ng/mL (362 fM) and is more than an order of magnitude more sensitive than an antibody-based cantilever sensor.



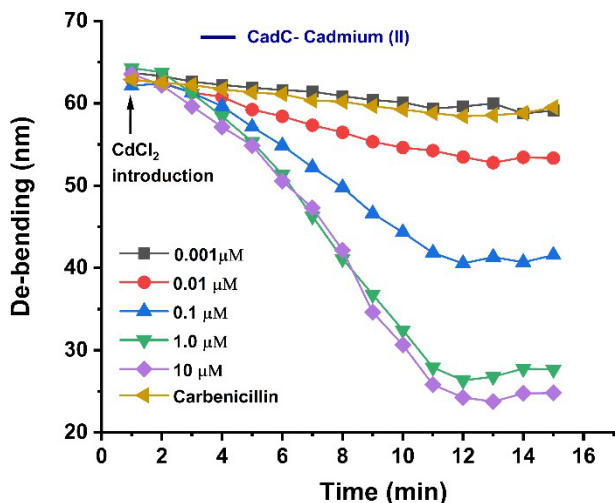
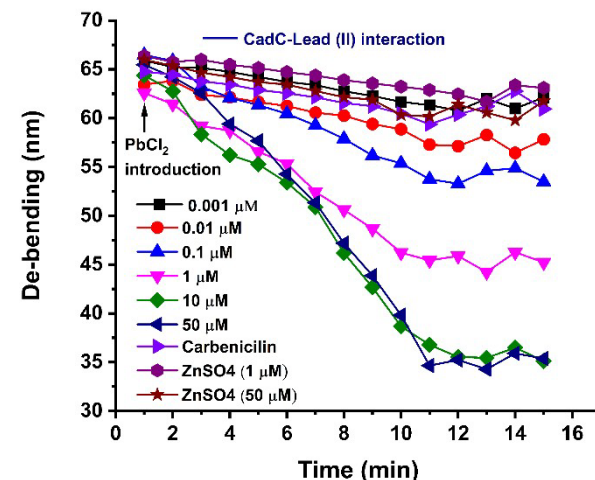
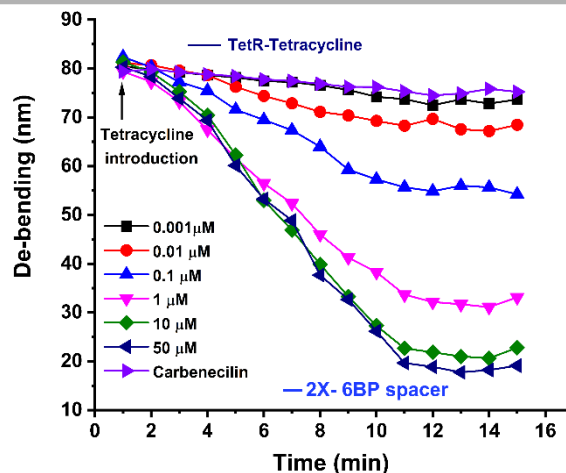
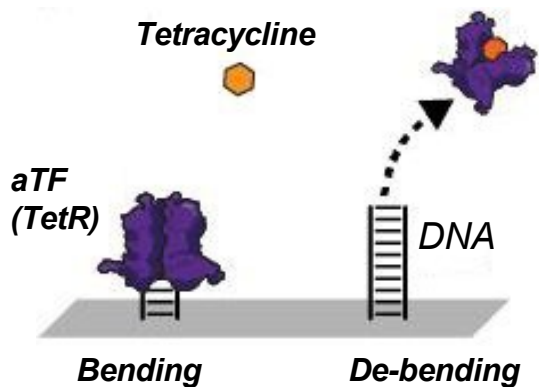
D. Agarwal et al. Work performed at SHyNE Resource.

This project received support from the SHyNE Resource (NSF ECCS-2025633).  
*Analytical Chemistry* 94, 23 (2022).



National Research Priority: NSF–Understanding the Rules of Life

# Detection of Tetracycline in Spike Water Sample using Synthetic Biology Approach



- Detection of harmful water contaminants (Tetracycline, Lead, Cadmium) through cantilever bending-debending reactions
- Limit of detection in line with EPA (Environment protection agency) limit of safe drinking water
- This approach can be used to monitor the quality of water used in hospitals and other healthcare settings
- The detection is also compatible with MOSFET (Point-of-care) devices

Julius Lucks, Gajendra Shekhawat, and Vinayak David.

This project received support from the SHyNE Resource (NSF ECCS-2025633).

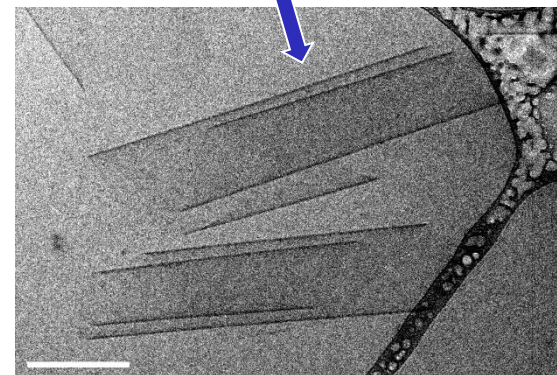
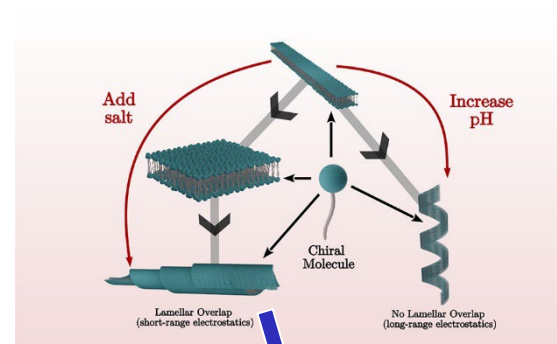
National Research Priority: NSF-Understanding the Rules of Life

# Electrostatic Control of Shape Selection and Nanoscale Structure in Chiral Molecular Assemblies

How molecular chirality manifests at the nano- to macroscale has been a scientific puzzle since Louis Pasteur discovered biochirality. Chiral molecules assemble into meso-shapes such as helical ribbons, nanotubes, twisted ribbons, helicoidal scrolls (cochleates), and möbius strips. We analyzed self-assembly for a series of amphiphiles, consisting of an ionizable amino acid coupled to alkyl tails with carbons. This simple system allowed us to probe the effects of electrostatic and van der Waals interactions in chiral assemblies.

Soft chiral assemblies have potential nanotechnological applications. The following examples illustrate the need for developing control over shape selection, internal architecture of chiral assemblies, and interconversion mechanisms.

- Helicoidal scrolls are being explored as drug/macromolecular delivery platforms due to their ability to encapsulate nanoscale objects within the bilayers and in the aqueous phase between adjacent bilayers.
- Helical ribbons and nanotubes are recognized as possible templates for nano- and meso-electronic components such as nanowires and solenoids.



CryoTEM of cochleate structure, scalebar = 200 nm

Joseph M. McCourt, Sumit Kewalramani, Changrui Gao, Eric W. Roth, Steven J. Weigand, Monica Olvera de la Cruz and Michael J. Bedzyk. Work performed in part at SHyNE Resource.

This project received support from the SHyNE Resource (NSF ECCS-2025633). *ACS Central Science* 2022, 8, 8, 1169–1181.

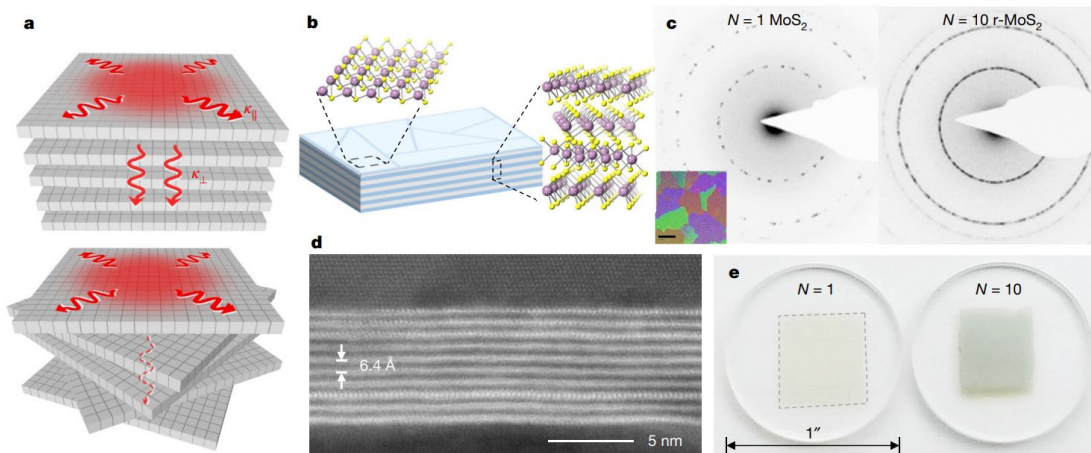
*National Research Priority: NSF–Understanding the Rules of Life*



# Extremely anisotropic van der Waals thermal conductors

The densification of integrated circuits requires thermal management strategies and high thermal conductivity materials<sup>1–3</sup>. Recent innovations include the development of materials with thermal conduction anisotropy, which can remove hotspots along the fast-axis direction and provide thermal insulation along the slow axis. However, most artificially engineered thermal conductors have anisotropy ratios much smaller than those seen in naturally anisotropic materials. Here we report extremely anisotropic thermal conductors based on large-area van der Waals thin films with random interlayer rotations, which produce a room-temperature thermal

anisotropy ratio close to 900 in MoS<sub>2</sub>, one of the highest ever reported. Our work establishes interlayer rotation in crystalline layered materials as a new degree of freedom for engineering-directed heat transport in solid-state systems.



**Fig. 1 | Structure of r-TMD films.** **a**, Conceptual strategy for engineering thermal anisotropy in a single material system, using random interlayer rotation in polycrystalline vdW layered materials. **b**, Schematic of an r-MoS<sub>2</sub> film with random crystalline orientation. **c**, Greyscale-inverted TEM electron diffraction patterns probed from a 500 nm × 500 nm area of a monolayer and

an  $N = 10$  r-MoS<sub>2</sub> film. Inset: darkfield TEM image of a monolayer; the scale bar denotes 400 nm and the colours denote different domain orientations from different crystal domains. **d**, HAADF-STEM image of a cross-section of an  $N = 10$  r-MoS<sub>2</sub> film on AlO<sub>3</sub>, coated with Al, with an interlayer spacing of 6.4 Å. **e**, Large-area MoS<sub>2</sub> films transferred onto 1-inch diameter fused silica substrates.

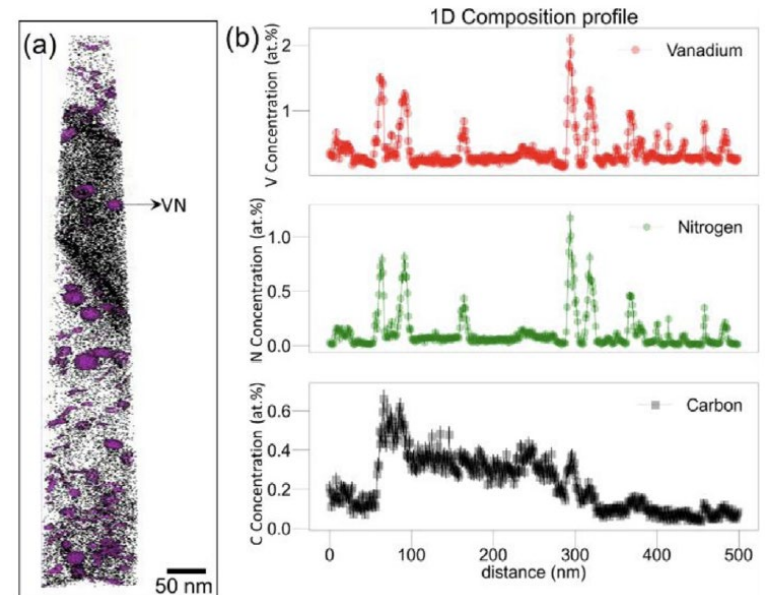
Kim, S.E., Mujid, F., Rai, A. et al. Work performed in part at the SHyNE Resource.

This project received support from the SHyNE Resource (NSF ECCS-2025633). *Nature* 597, 660–665 (2021).

*National Research Priority: NSF–Harnessing the Data Revolution*

# Microstructures and mechanical properties of a modified 9Cr ferritic-martensitic steel in the as-built condition after additive manufacturing

An additive-manufactured nanostructured alloy, ANA, was newly developed to generate a nanostructured high-Mn 9Cr ferritic-martensitic steel with tensile strength and Charpy impact toughness comparable to oxide-dispersion-strengthened (ODS) alloys. Microstructural characterization revealed an extremely high dislocation density and a high density of ultrafine nano-structured precipitates as the dominant strengthening elements. The ultrafine precipitates also act as the primary contribution to estimated irradiation defect sink density. This work demonstrates the ability to exploit the unique consolidation properties of additive manufacturing to fabricate steels which marry the benefits of both ODS alloys and conventionally processed ferritic-martensitic steels.



(a) 3D APT reconstruction of the tip showing C atom positions (marked with black dots) overlaid with 8% V iso-concentration surfaces to highlight the ultrafine precipitates. (b) 1-D concentration profiles along the length of the APT reconstruction.

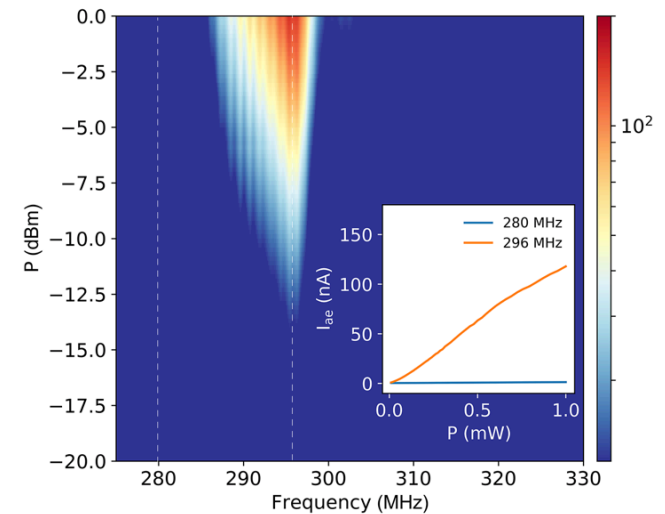
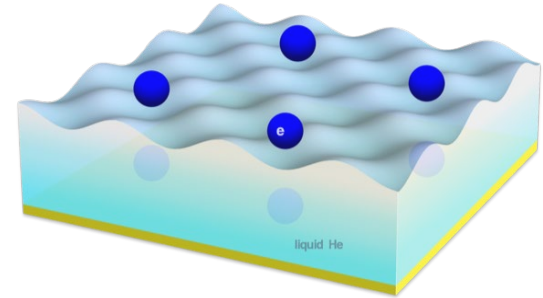
W. C. Zhong et al. Work performed at Northwestern University Center for Atom-Probe Tomography.

This project received support from the SHyNE Resource (NSF ECCS-2025633). *Journal of Nuclear Materials* 545, 152742, 2021.

National Research Priority: NSF–Growing Convergence Research

# Electrons on helium coupled to high-frequency surface acoustic waves

Piezoelectric surface acoustic waves (SAWs) are powerful for investigating and controlling elementary and collective excitations in condensed matter. In semiconductor two-dimensional electron systems SAWs have been used to reveal the spatial and temporal structure of electronic states, produce quantized charge pumping, and transfer quantum information. In contrast to semiconductors, electrons trapped above the surface of superfluid helium form an ultra-high mobility, two-dimensional electron system home to strongly-interacting Coulomb liquid and solid states, which exhibit non-trivial spatial structure and temporal dynamics prime for SAW-based experiments. Here we report on the coupling of electrons on helium to an evanescent piezoelectric SAW. We demonstrate precision acoustoelectric transport of as little as  $\sim 0.01\%$  of the electrons, opening the door to future quantized charge pumping experiments. We also show SAWs are a route to investigating the high-frequency dynamical response, and relaxational processes, of collective excitations of the electronic liquid and solid phases of electrons on helium.



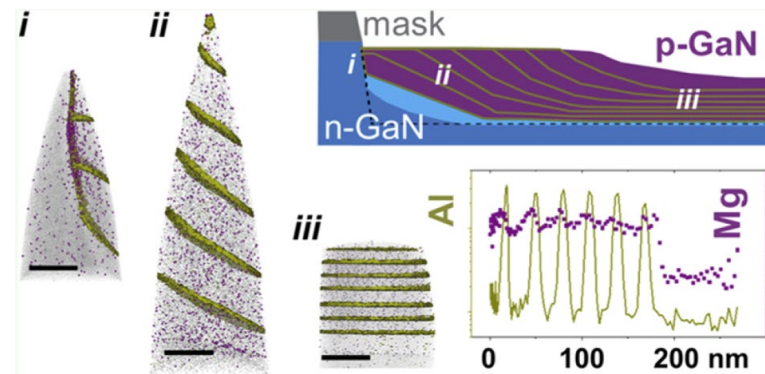
Byeon, H., Nasyedkin, K., Lane, J.R. et al. Work performed in part at SHyNE Resource.

This project received support from the SHyNE Resource (NSF ECCS-2025633). *Nature Communications* 12, 4150 (2021).

*National Research Priority: NSF–Growing Convergence Research*

# Selective Area Regrowth Produces Nonuniform Mg Doping Profiles in Nonplanar GaN p-n Junctions

Nonplanar GaN p-n junctions formed by selective area regrowth were analyzed using pulsed laser atom probe tomography. Dilute Al marker layers were used to map the evolution of the p-GaN growth interface, enabling extraction of time-varying growth rates for nonpolar, semipolar, and polar surfaces from the trench edge to the center, respectively. The Mg dopant concentration is facet dependent and varies inversely with the growth rate for the semipolar facets that grow rapidly away from the trench sidewalls. The negligible growth on the vertical sidewall of the trench coincides with an order of magnitude higher Mg concentration and substantial clustering of likely inactive dopants. A high Mg concentration is also observed near the regrowth interface of polar and semipolar planes, which we attribute to etching damage. We conclude that device fabrication processes employing selective area regrowth on nonplanar interfaces should consider both the spatial and temporal dependencies of growth rate that lead to nonuniform doping and explore growth conditions that could reduce variations in growth rate when nonuniform doping would adversely affect device performance.



APT reconstructions from selected regions (i), (ii), and (iii) in nonplanar GaN p-n junctions. Ga atoms are shown as gray dots, and Mg atoms are shown as purple spheres. Regions in which  $x_{Al} > 0.4$  atom % are delineated by dark-yellow isosurfaces. Scale bars are 50 nm.

S. Chang et al., Work performed at Northwestern University Center for Atom-Probe Tomography.

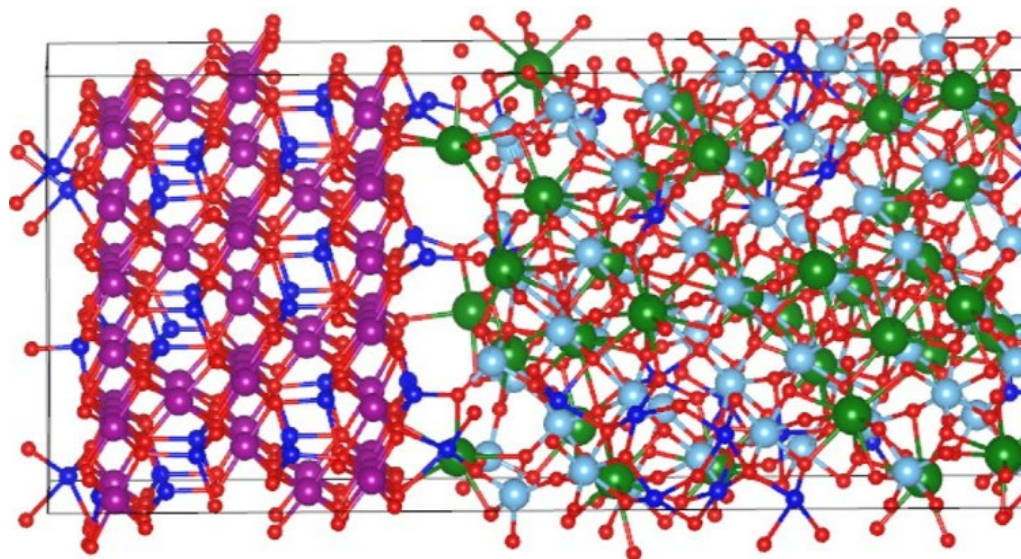
This project received support from the SHyNE Resource (NSF ECCS-2025633). *ACS Appl. Electron. Mater.* 2021, 3, 704–710.

National Research Priority: NSF–Growing Convergence Research



# Understanding the Solid-State Electrode-Electrolyte Interface of a Model System Using First-Principles Statistical Mechanics and Thin-Film X-ray Characterization

For optimizing the synthesis and performance of fully solid-state rechargeable batteries, thin films of Lithium Manganese Oxide (LMO) and Lithium Lanthanum Titanate (LLTO) were grown on strontium titanate (1 1 1) [STO (111)] substrates in the pulsed laser deposition facility. LMO acts as the cathode and the LLTO as the solid electrolyte. Based on X-ray reflectivity/X-ray diffraction studies in the XRD facility, it was found that the performance of these rechargeable batteries is optimized when this half-cell is synthesized below 600 C.



*Relaxed interface model of the LMO and LLTO interface. In this figure, purple spheres are manganese, red spheres are oxygen, dark blue spheres are lithium, light blue spheres are titanium, and green spheres are lanthanum*

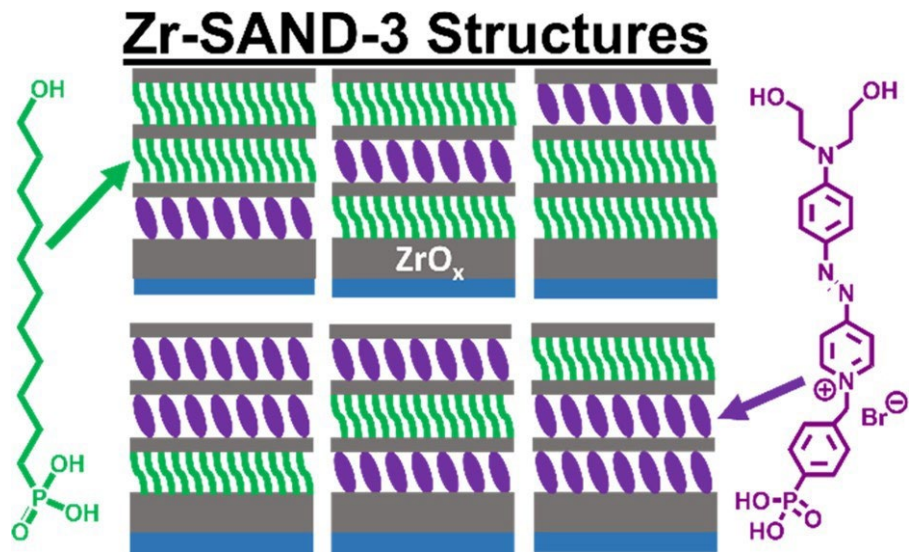
Jason D. Howard, Guennadi Evmenenko, Jae Jin Kim, Robert E. Warburton, Shane Patel, Timothy T. Fister, D. Bruce Buchholz, Jeffrey Greeley, Larry A. Curtiss, and Paul Fenter. Work performed at SHyNE Resource.

This project received support from the SHyNE Resource (NSF ECCS-2025633). *ACS Appl. Mater. Interfaces* 2022, 14, 7428–7439.

*National Research Priority: NSF–Windows on the Universe*

# Systematic Analysis of Self-Assembled Nanodielectric Architecture and Organization Effects on Organic Transistor Switching

Self-assembled nanodielectric (SAND) hybrid inorganic-organic architecture is composed of alternating inorganic (e.g.,  $ZrO_x$  and  $HfO_x$ ) and  $\pi$ -organic nanolayers (e.g., stilbazolium). As gate dielectrics, SANDs are compatible with a wide variety of organic and inorganic semiconductors and often impart superior thin-film transistor (TFT) performance in comparison to analogous inorganic-only dielectrics. To probe the role of the highly polarizable stilbazolium (Chr) organic layer in SAND and dielectric response, a saturated hydrocarbon chain-based self-assembling building block (Alk) was incorporated in SAND structures. By using Chr and Alk in the different SAND organic layers, the effects of the Chr built-in dipole on bulk SAND structural and dielectric characteristics can be evaluated. XRD facility was utilized to characterize Zr-SAND structures. Based on these and electronic measurements, the layer identity and arrangement of the organic layers within the Zr-SAND structure were found to have a significant impact on the capacitor leakage current and pentacene transistor threshold voltage/turn-on voltage characteristics.



Self-assembled *Nanodielectric* architecture.

Katie Stallings, Riccardo Turrisi, Yao Chen, Li Zeng, Binghao Wang, Jeremy Smith, Michael J. Bedzyk, Luca Beverina, Antonio Facchetti, and Tobin J. Marks. Work performed at SHyNE Resource.

This project received support from the SHyNE Resource (NSF ECCS-2025633). *ACS Applied Electronic Materials* 2022 4 (4), 2015-2025.

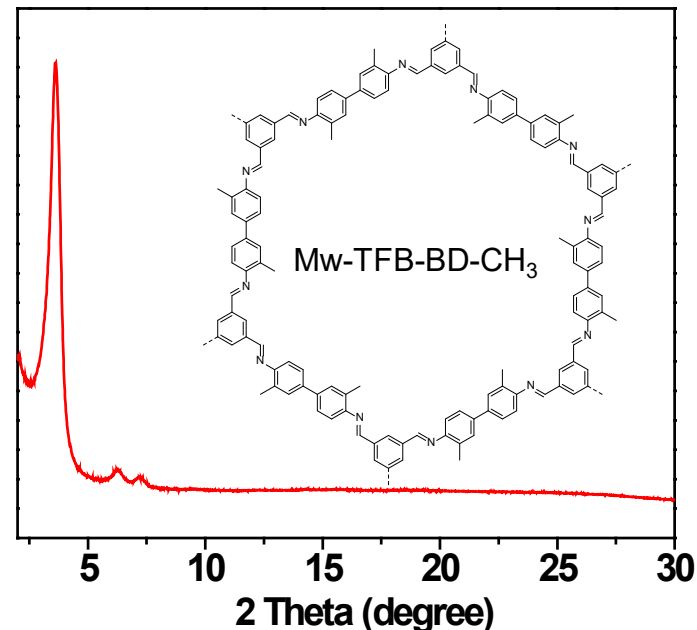
National Research Priority: NSF–Growing Convergence Research

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# ***Southeastern Nanotechnology Infrastructure Corridor (SENIC)***

# Imine-Linked Covalent Organic Frameworks for Exceptional Iodine Capture

This work delineates a facile microwave-assisted synthesis of 2D imine-linked covalent organic frameworks (COFs), Mw-TFB-BD-X, (X = -CH<sub>3</sub> and -OCH<sub>3</sub>) under air within merely 1 hour. The resultant COFs possessed higher crystallinity, better yields, and more uniform morphology than those of their solvothermal counterparts. Remarkably, Mw-TFB-BD-CH<sub>3</sub> exhibited exceptional iodine adsorption capacities of 7.83 g g<sup>-1</sup>, placing it among the best-performing COF adsorbents for static iodine capture to date. Moreover, Mw-TFB-BD-CH<sub>3</sub> can be reused 5 times with no apparent loss in the adsorption capacity. This work establishes a benchmark for developing advanced iodine adsorbents that combine fast kinetics, high capacity, excellent reusability, and facile synthesis.



Powder X-ray diffraction (PXRD) pattern of COF, Mw-TFB-BD-CH<sub>3</sub>, showing highly crystallinity.

Ziad Alsudairy, Normanda Brown, and Xinle Li, Department of Chemistry, Clark Atlanta University. PXRD analysis was performed at Materials Characterization Facility at Georgia Institute of Technology.

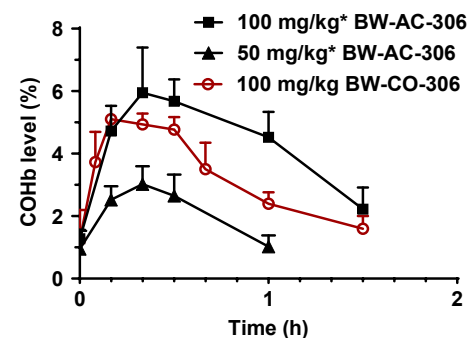
This work was supported by NSF Award # 2100360.

*National Research Priority: DoD Critical Technology Area—Advanced Materials*

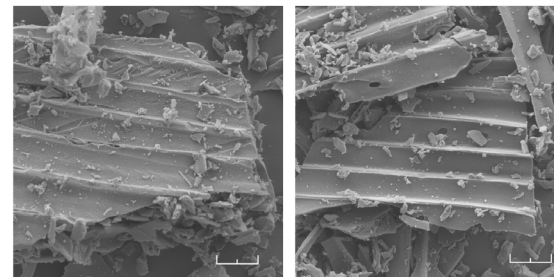


# Activated Charcoal Dispersion of Carbon Monoxide Prodrugs for Oral Delivery of CO in a Pill

A novel orally bioavailable solid formulation to deliver a gaseous signaling molecule, carbon monoxide (CO), was developed by adsorbing oxalyl saccharin, a newly developed organic CO prodrug, in the activated charcoal (AC). The resulting solid dispersion formulation addresses key developability issues of this CO prodrug, including the paradoxical problem of low water solubility of the prodrug and the requirement of hydrolysis to release CO, the need for an organic cosolvent, and systemic exposure to the CO prodrug and release byproduct. This formulation allows encapsulation in normal and enteric-coated gel capsules, which enables controllable CO delivery to the upper or lower GI system. Through in-vivo pharmacokinetic studies in mice, the AC formulation showed better CO delivery efficiency of delivering CO through oral administration compared to the prodrug dosed with an organic cosolvent. We envision the wide applicability of this formulation in facilitating the future development of CO-based therapeutics



COHb level in blood following p.o. administration of **BW-AC-306**



SEM image of (A) AC, and (B) **BW-AC-306**. (Scale bar: 20  $\mu$ m)

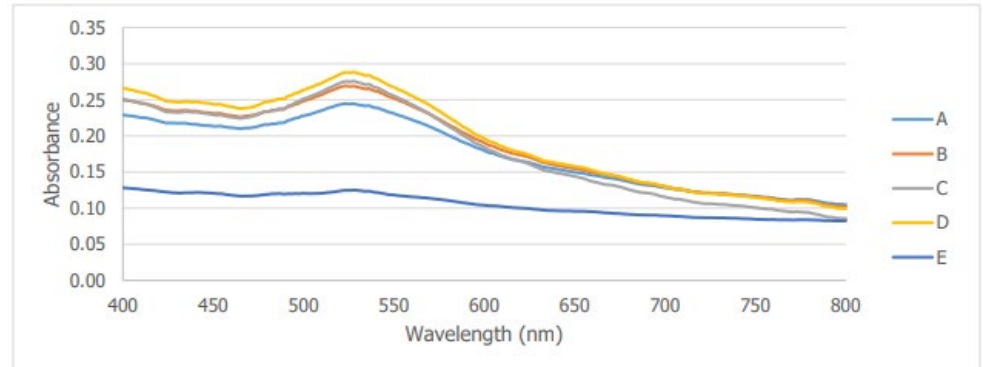
Xiaoxiao Yang, Wen Lu, Ladie Kimberly De La Cruz, and Binghe Wang, Dept. of Chemistry, Georgia State Univ., and Minjia Wang and Chalet Tan, Univ. of Mississippi. DSC/TGA were performed at Georgia Tech's IEN.

Partially supported by a SENIC Catalyst grant. Research funded by National Institutes of Health (R01DK119202). International Journal of Pharmaceutics, 2022, 618, 121650.

*National Research Priority: NAE Grand Challenge—Engineer Better Medicines*

# Sensitive Detection of Aflatoxin B1 in Peanuts Using Gold Nanoparticles

This aim of this research was to develop a colorimetric immunoassay for the detection of aflatoxin B1 (AFB1), a dangerous fungal toxin that contaminates a variety of crops. The two major components of the assay are monoclonal anti-AFB1 antibody immobilized on experimentally synthesized gold nanoparticles, and magnetic iron beads covalently bonded to AFB1 antigen. UV-Vis detection of toxin was able to rapidly and sensitively detect AFB1 in peanuts at concentrations of 0.2 ppb. The commercial minimum AFB1 allowable for the US is reported to be 20 ppb, which means the assay can detect 1000x times lower than the allowable limit of consumption]



Average of UV-Vis absorption spectra of supernatant after magnetic separation with different concentrations of AFB (from sample A to E: 800, 500, 200, 100, 50, 20 ng/L).

Amber D. Davenport, and Hari P. Singh, Nanotechnology Laboratory, Agricultural Research Station, Department of Agricultural Sciences, College of Agriculture, Family Sciences & Technology (CAFST), Fort Valley State University. Georgia Tech's Institute for Electronics and Nanotechnology facility was utilized for sample analysis.

This work was supported by USDA-ARS grant 335149 "Reducing Aflatoxin Contamination in United States Peanuts" and a SENIC Catalyst grant.

National Research Priority: NSF–Growing Convergence Research

# Biological Benefits of Collective Swimming of Sperm in a Viscoelastic Fluid

This work uses a microfluidic device to study motility related biological benefits for sperm to swim collectively when in a viscoelastic fluid. We found that under no flow, collective sperm trajectories are more linear, or sperm swim in a more directed manner. As the flow rate increases, sperm in groups are better aligned against the flow than sperm swimming individually. Finally, under a strong flow that is capable of carrying sperm downstream, we found that sperm swimming close to each other are less likely to move in the flow direction, suggesting the collective swimming protect them from removal by a strong flow.

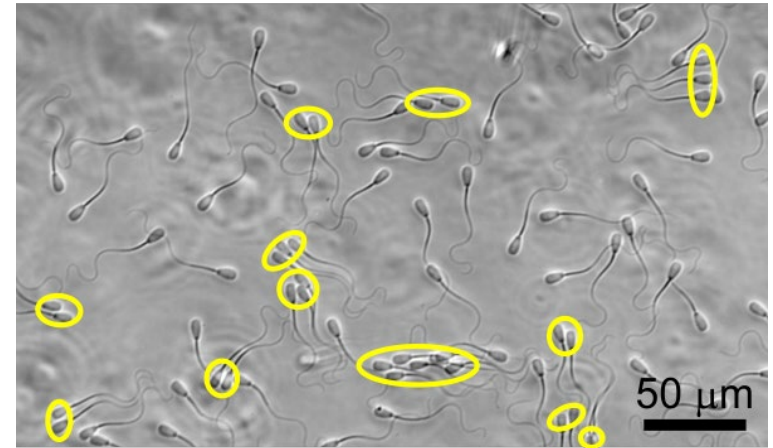


Image of sperm swim collectively in a microfluidic device (shown below) filled with viscoelastic fluid.

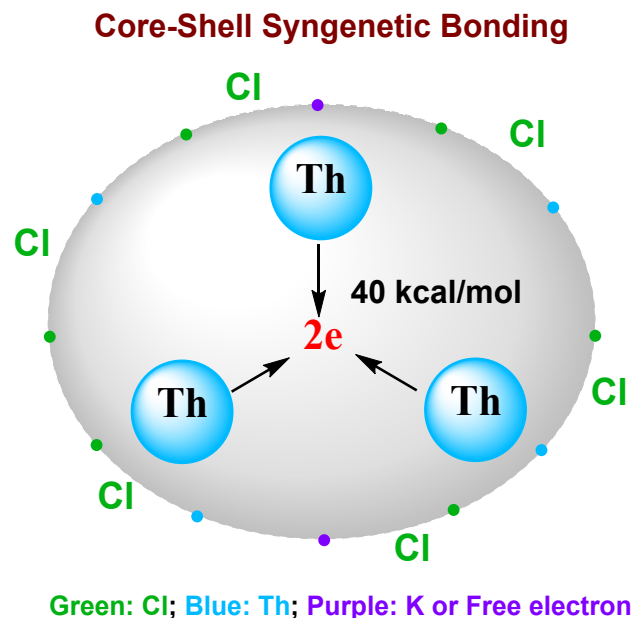
Shiva Phuyal and Chih Kuan Tung, Department of Physics, North Carolina A&T State University. Work performed at Joint School of Nanoscience and Nanotechnology.

This work was supported by NIH Award R15HD095411. *Front. Cell Dev. Biol.* 10:961623 (2022).

*National Research Priority: NSF–Understanding the Rules of Life*

# Bonding Nature in the Crystalline Tri-Thorium Cluster: Core-Shell Syngenetic $\sigma$ -Aromaticity

The aromaticity in transition metal clusters has been fascinating chemists. Recently, Boronski et al. reported a crystalline tri-thorium cluster at the mild experimental condition. In this work, we explore the bonding nature of the tri-thorium cluster with modern ab initio valence bond (VB) theory in order to understand its stability. We propose a new type of core-shell syngenetic bonding model which involves a three-center two electron bond in the  $\text{Th}_3$  core and a multicentered  $(\text{ThCl}_2)_3$  charge-shift bond with 12 electrons scattering along the outer shell. Contributions from both bonds to the stability of the crystalline tri-thorium cluster were differentiated and quantified and compared with the well-regarded  $\sigma$ -aromatic  $\text{H}_3^+$  and non-aromatic  $\text{Li}_3^+$ . We conclude that there is considerable  $\sigma$ -aromaticity in the tri-thorium cluster.



A core-shell syngenetic bonding model is proposed to describe the chemical bond in a stable crystalline tri-thorium cluster.

Xuhui Lin, Southwest Jiaotong University, China; Yirong Mo, Department of Nanoscience, University of North Carolina at Greensboro. Work performed in part at JSNN.

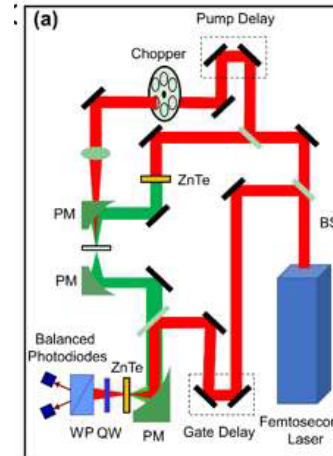
Angewandte Chemie International Edition, 61(37), e202209658 (2022).

*National Research Priority: DoD Critical Technology Area—Advanced Materials*

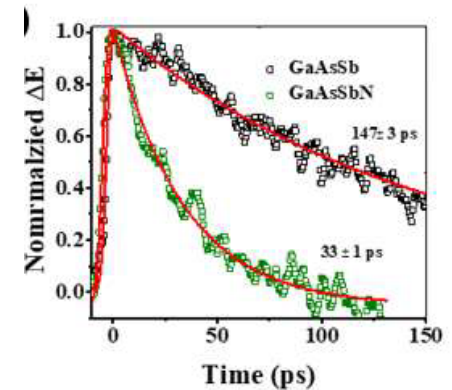


# Carrier Dynamics and Transport in Te-doped GaAsSb and GaAsSbN NWs by Correlating Ultrafast Terahertz Spectroscopy

It is established that a small amount of nitrogen (N) incorporation in III-V semiconductor NWs can effectively redshift their wavelength of operation and tailor their electronic properties for specific applications. In this work, ultrafast optical pump-terahertz probe spectroscopy has been used to study non-equilibrium carrier dynamics and transport in Tedoped GaAsSb and dilute nitride GaAsSbN NWs, with the goal of correlating these results with electrical characterization of their equilibrium photo-response under bias and low-frequency noise characteristics. Nitrogen incorporation in GaAsSb NWs led to a significant increase in the carrier scattering rate, resulting in a severe reduction in carrier mobility. Finally, we observed a very fast rise time of  $\sim 2$  ps for both NW materials, directly impacting their potential use as highspeed photodetectors



Schematic of the OPTP experiment



Transient photoinduced change in the THz electric (E) field for GaAsSb and GaAsSbN NWs probed by OPTP spectroscopy

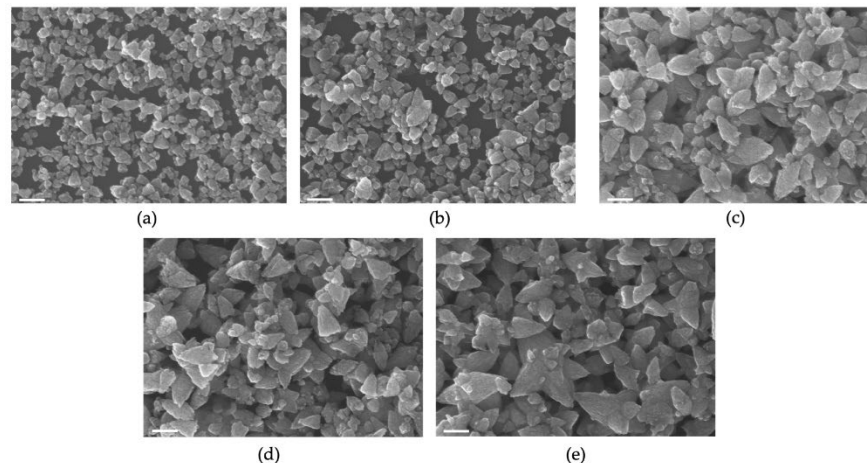
Shanthi Iyer and Rohit Prasankumar, NC A&T State University and Los Alamos National Laboratory. Work was partly performed at JSNN.

Supported by U.S. Army Grant Number W911NF-19-1-0002, Air Force Office of Scientific Research; partly funded by the National Science Foundation (NSF) (Grant: EECS-18322117). Nanotechnology, 33, 425702 (8 pp) (2022).

National Research Priority: NSF–Quantum Leap

# Mg-Doped ZnO Nanoparticles with Tunable Band Gaps for Surface-Enhanced Raman Scattering (SERS)-Based Sensing

This work aims at improving the signal enhancement effect of ZnO when employed as substrate in surface-enhanced Raman scattering (SERS) based applications. We doped ZnO with different concentrations of Mg by co-precipitation to synthesize Mg-doped ZnO nanoparticles with bandgap observed to be modified with the dopant amount. An enhanced Raman signal was observed on molecules using these substrates in colloidal form, which is appropriate for cellular applications and in point-of-care environments. In addition, stability tests and dose-dependent doping for toxicity responses in live cells were carried out to determine the suitability of the substrates in a biological environment. Substrates showed high stability in the first hour of dissolution in a cell media, and it was observed that there was no significant cytotoxic effect resulting from doping Mg with ZnO. Overall, this study provides evidence for the tunability of ZnO substrates and may serve as a platform for applications in molecular biosensing.



FE-SEM images (scale bar: 200 nm) of (a) pure ZnO nanoparticles; (b) 2% Mg-doped nanoparticles; (c) 5% Mg-doped nanoparticles; (d) 7% Mg-doped nanoparticles; (e) 10% Mg-doped nanoparticles.

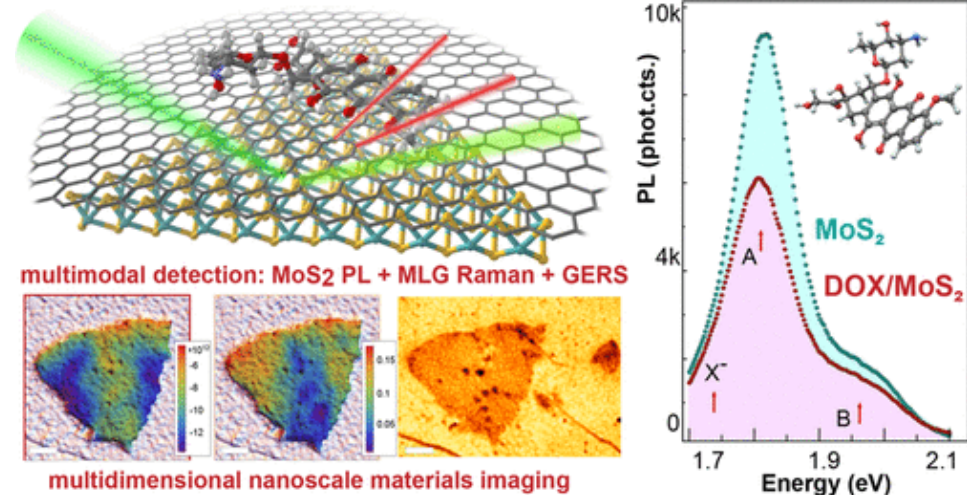
Adesoye, Samuel, Saqer Al Abdullah, Kyle Nowlin, and Kristen Dellinger, Nanoengineering, NC A&T State University. Work performed at Joint School of Nanoscience and Nanotechnology

This research was funded by KL2 Scholar Award from the National Center for Advancing Translational Sciences, NIH, Grant KL2TR002490. *Nanomaterials* 12, no. 20: 3564.

*National Research Priority: DoD Critical Technology Area—Advanced Materials*

# Multidimensional Imaging for Multimodal Label-free Biosensing

Researchers at Penn State University, University of North Carolina-Greensboro, and North Carolina A&T State University developed a multidimensional optical imaging technique to map subdiffractional distributions for doping and strain and understand the role of those for modulation of the electronic properties of the material. This group reported on the optical label-free detection of doxorubicin via three independent optical detection channels (photoluminescence shift, Raman shift, and graphene enhanced Raman scattering). Multidimensional nanoscale imaging allows one to reveal the physical origin for local responses and best strategy for the mitigation of materials variability to enable multiplexed biosensing



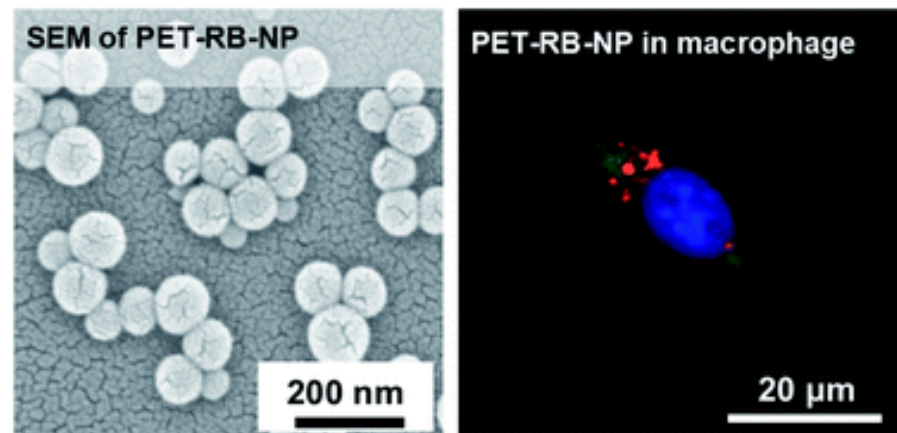
Tetyana Ignatova and Slava Rotkin, Penn State and UNC Greensboro. Part of work performed at Joint School of Nanoscience and Nanotechnology

Work partially supported by NSF CHE-2032582) and CHE-2032601. ACS Nano, 16, 2598–2607, 2022

*National Research Priority: NSF–Growing Convergence Research*

# Polyethylene terephthalate (PET) nanoparticles for studies in mammalian cells

Environmental presence of fragmented plastics, derived from high-commodity polymers, is an emerging concern with unknown consequences for human health. As a crucial high-commodity polymer and contributor of plastic waste, PET has infiltrated drinking water, food, and beverages in the form of small-scale debris (i.e., microplastics). Fluorescent nanoparticles (NPs) comprising polyethylene terephthalate (PET) with a hydrodynamic diameter of  $158 \pm 2$  nm were synthesized in a bottom-up approach. Concentration-dependent uptake and cytotoxicity of PET NPs in macrophages are shown. The fabrication of well-characterized NPs, derived from high-commodity polymers, will support future studies to assess effects on biological systems.



FE-SEM images of for PET-RB NPs; fluorescence microscopy of RAW 264.7 cells exposed to PET-RB nanoparticles

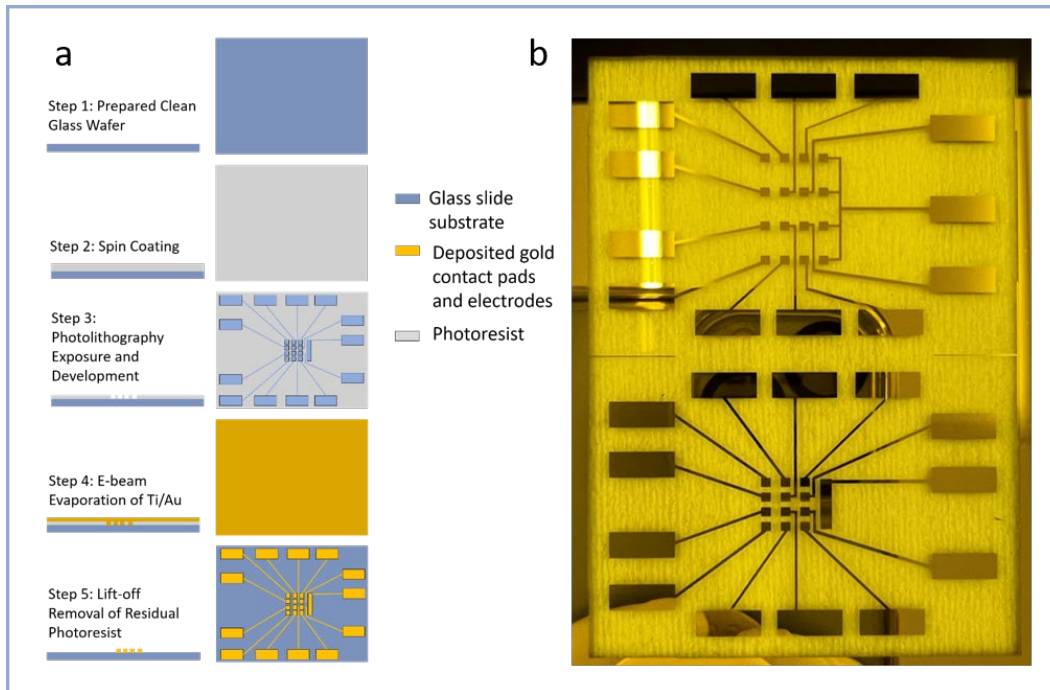
Leah Johnson and Ninell Mortensen, RTI International. Part of work performed at Joint School of Nanoscience and Nanotechnology

Nanoscale Advances, 3(2), 2021, 339-346

*National Research Priority: NSF–Understanding the Rules of Life*



# Development of Lab-on-a-Chip Platform for the Study of Extracellular Electron Transfer



(a) Developed fabrication process and (b) digital image of electrode chips with Ti/Au deposition.

This project aimed to develop a lab on a chip (LOAC) platform monitoring the microbial attachment and extracellular electron transfer processes of microorganisms in situ and in real time. The step-by-step process flow of the electrode chip design was completed to ensure consistent fabrication using photolithography and lift off methods shown in Figure 1a. Multiple electrode and flow configurations were also developed with examples shown in Figure 1b. Future work involves finalizing the device with enclosure to enable flow and perform preliminary testing for extracellular electron transfer processes between microorganisms and the deposited solid electrodes on the chip surface.

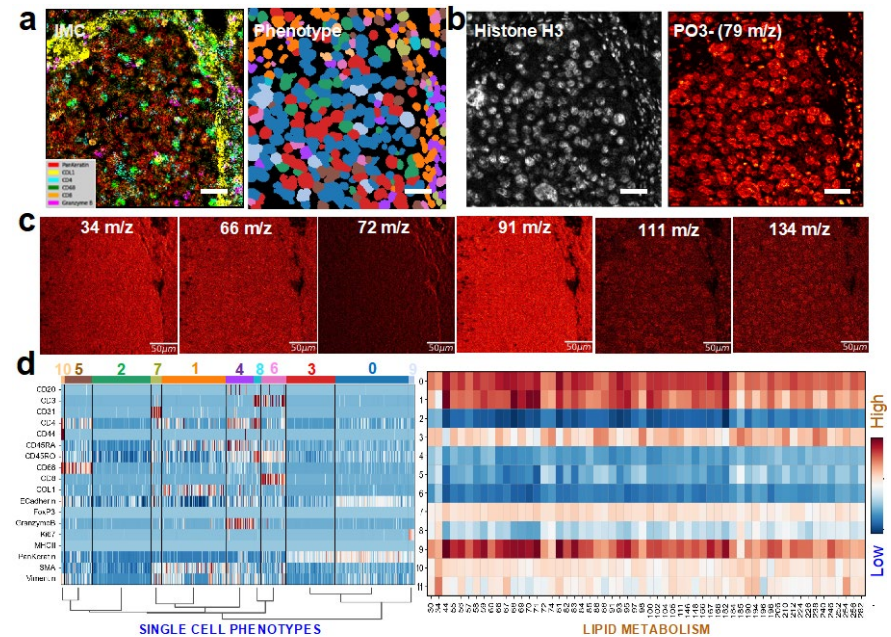
Mourin Jarin, Ting Wang, Xing Xie; School of Civil and Environmental Engineering, Georgia Institute of Technology  
Work performed at Georgia Tech's Institute for Electronics and Nanotechnology.

This work was partially supported by an IEN Core Facility Seed Grant.

*National Research Priority: NSF–Understanding the Rules of Life*

# Correlative Spatial Metabolomics And Protein In Situ Imaging

This work aims to decipher spatially resolved metabolic profile at the single cell level by incorporating untargeted spatial metabolomics using (TOF-SIMS) and targeted multiplexed protein imaging (IMC). Human lung adenocarcinoma tissue were labeled by a 20-plex isotope-barcoded antibody library one-time to enhance the contrast of specific immune and cancer cell types then imaged using TOF-SIMS (IONTOF 5 GmbH, Münster, Germany) with a doubly charged 50-kV clustered (Bi3++) bismuth liquid metal ion gun to generate secondary ions from the sample surface, followed by identification of m/z of secondary ions by a TOF analyze. For depth profiling, a Cesium Ion gun (Cs+ ions, 2-kV energy, and microampere current) is used to iteratively sputter away very thin layers of tissue and image across 30-40 depth slices. IMC was used for multiplexed protein imaging for unsupervised single-cell phenotyping. Spatial protein and metabolites imaging modalities were registered using single-cell nuclei features with a cross-correlation metric



(a) IMC multiplex protein imaging (left) with corresponding unsupervised single-cell phenotypes (right) (b) Single cell nuclei registration by cross-correlation using Histone H3 protein marker (left) and 3D-SMF PO3- 79 m/z (right) (c) Spatial metabolomic profiling examples at different m/z channels (d) Comparison of unsupervised clustering of protein phenotype (left) with lipids expression level (right) at single-cell resolution

Thomas Hu, School of Electrical and Computer Engineering, Georgia Institute of Technology. Ahmet F. Coskun, Georgia Institute of Technology and Emory University. Work performed at Georgia Tech's Institute for Electronics and Nanotechnology.

This work was partially supported by an IEN Core Facility Seed Grant.

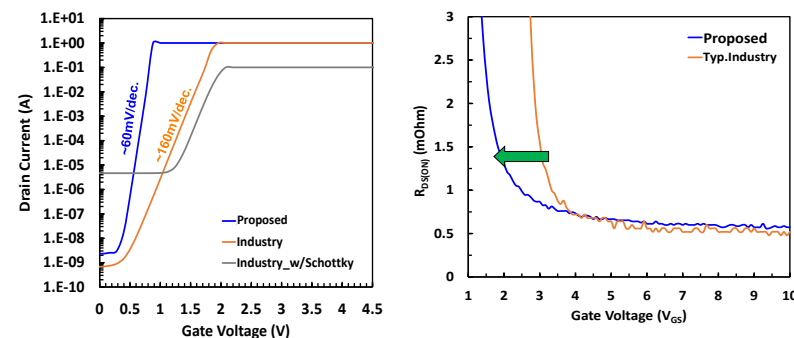
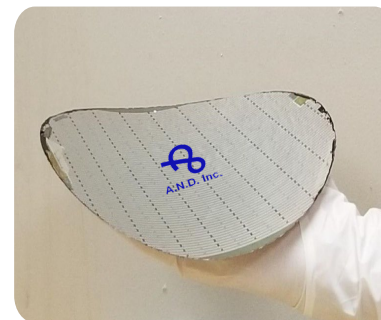
National Research Priority: NSF–Understanding the Rules of Life

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# ***Texas Nanofabrication Facility (TNF)***

# Power FETs with low subthreshold slope and ON resistance

A new class of Si power MOSFET technology with sub-30 $\mu\text{m}$  substrate was developed. The technology enables 2x lower output charge ( $Q_{\text{OSS}}$ ) and superior specific on-resistance ( $\text{SR}_{\text{DS(ON)}}$ ) at gate drive as low as 2.5V compared to the state-of-the-art low-voltage (<60V) MOSFETs. Near-zero reverse-recovery charge/losses for all voltage applications is also inherent to the new power MOSFET. Manufacturing with self-aligned and low-complexity process has been established in a high-volume 8-inch Si foundry. Thin-Crystalline Technology is utilized to yield mechanically handleable sub-30 $\mu\text{m}$  substrate. Finally, the novel device architecture is well-suited for SiC and GaN-like wide-bandgap (WBG) devices as it inherently yields enhancement-mode WBG MOSFETs.



Power FETs on sub-30 micron Si

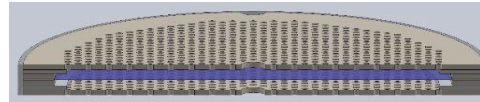
Leo Mathew, Rajesh Rao, Dan Fine, Applied Novel Devices. Work performed at Texas Nanofabrication Facility. This work led to productization at Skywater Foundry.

National Research Priority: NSF–Growing Convergence Research



# CVD graphene membrane based microphones and speakers

GraphAudio has utilized the cost-effective cleanroom resources, processing equipment and test instrumentation available at the MRC and NmFab facilities. Initial work successfully demonstrated the feasibility of using large-area, graphene-based transducers for applications including acoustic sensing, micro-speakers and microphones targeted for mobile, consumer and enterprise electronics. Recent work has optimized the process flow for defect reduction, surface smoothness, improved reliability and enhanced transducer performance. A graphene-based MEMS microphone compatible with high-volume manufacturing is now under development.



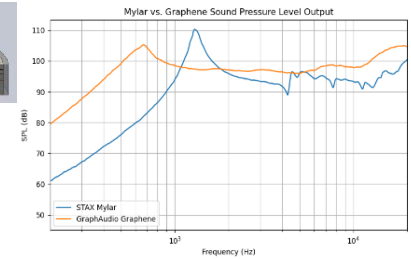
Coin Cross-Sectional Drawing



Graphene Diaphragm



FA Test Enclosure



Free-Air Test Plots of Graphene and Mylar



16mm Coin Transducer



Amplifier



Earbud Pair

Graphene Diaphragm Microphone

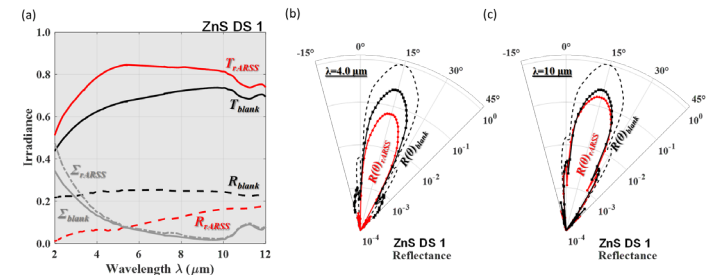
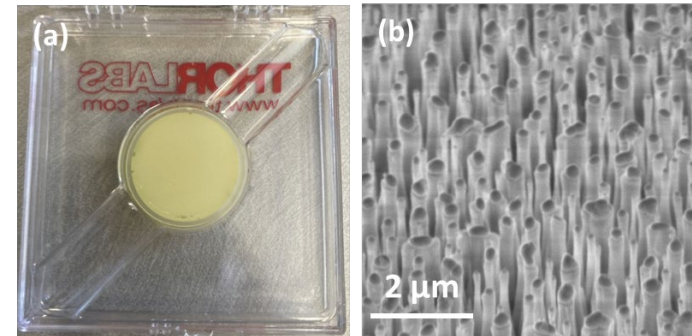
Burt Fowler., Lonnie Wilson, GraphAudio. Work performed at Texas Nanofabrication Facility. This work has led to product sampling with several companies such as Apple and Sony.

*National Research Priority: NSF–Future of Work at the Human-Technology Frontier*

# Broadband infrared anti-reflective nanostructures for 3<sup>rd</sup> generation IR sensors

Nanohmics Inc. developed broadband anti-reflective nanostructures to reduce the reflection losses at optical interfaces in the mid-wave and long-wave infrared spectrum on a variety of IR window materials. A lithography-free manufacturing process was developed to fabricate high-aspect ratio sub-wavelength random anti-reflective surface structures (rARSS) on ZnSe, ZnS, GaAs, Ge, Si, and AMTIR. The key innovations in the process were generating a random metal nanoparticle etch mask and developing reactive ion etch recipes to produce structures with the desired geometry and height. The fabrication process does not depend on lithography and was designed to be applied to optical elements with a wide range in size, and surfaces with varying degrees of curvature. A portion of the work was performed at the UT Austin Microelectronic Research cleanroom facilities, including the dry etching, rapid thermal anneal, and scanning electron microscopy.

Example: AR nanostructures on ZnS



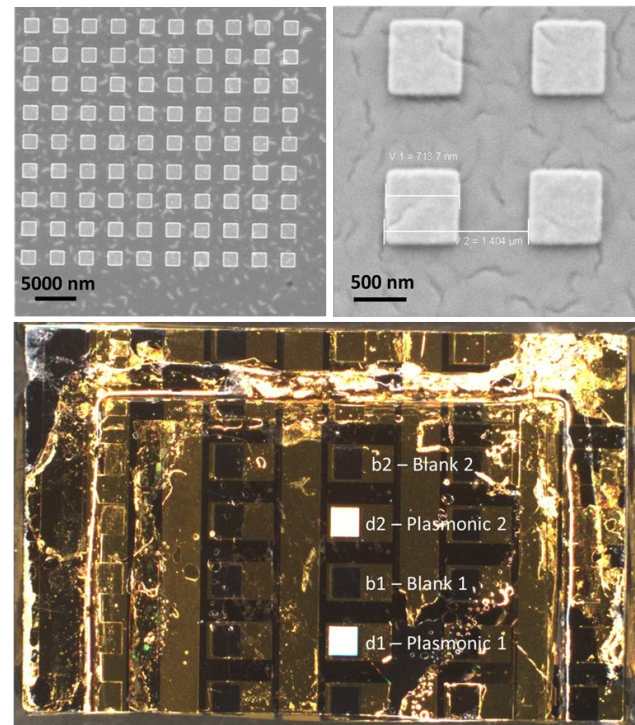
Karun Vijay, Nanohmics. Work performed at Texas Nanofabrication Facility.

This work was supported by Army SBIR grant W909MY-18-C-0018.

National Research Priority: NSF–Quantum Leap

# Metasurface-enhanced mid-infrared light emitting diodes

Mid-infrared light emitting diodes arrays are the workhorses in infrared scene projector systems used for hardware in the loop testing. However, the wall plug efficiency of devices is less than 3%, with a large percentage of photons trapped inside the device due to total internal reflection. Nanohmics Inc. is exploring methods of enhancing the internal and external quantum efficiency of LEDs by designing pixels with a metasurface cavity, comprising of the active region embedded between a ground plane and sub-wavelength array of patterned metal. The enhanced field confinement enables thinner active regions and increases spontaneous emission due to a Purcell enhancement, and the metasurface is designed to outcouple photons over wider range of incident angles. Key steps in the fabrication process include forming the ground plane with a metal-metal thermocompression bond, and patterning the metasurface using electron beam lithography. Both steps are performed at the UT Austin Microelectronics Research Center cleanroom.



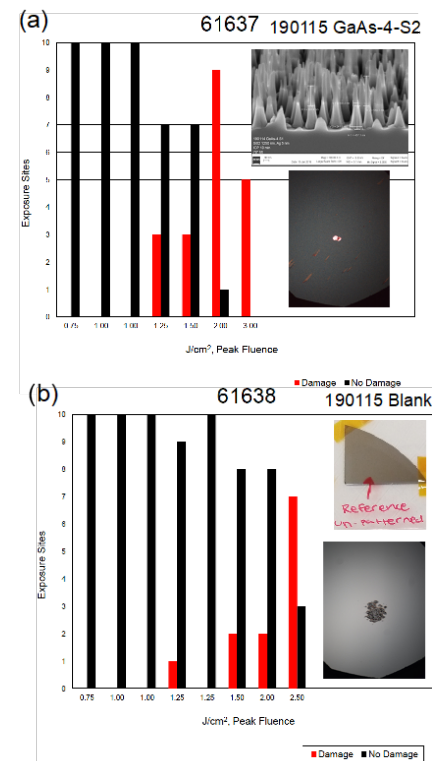
Karun Vijay, Nanohmics. Work performed at Texas Nanofabrication Facility.

This work was supported by Navy SBIR N6893621C0053.

*National Research Priority: NSF-Quantum Leap*

# High-damage threshold anti-reflective nanostructures for nonlinear optics

Future infrared counter-measure systems may use optical parametric oscillators to generate high-power and broadly tunable mid-infrared radiation. Anti-reflective (AR) treatments applied to the facets of the nonlinear crystals are required to maximize device conversion efficiency. Traditional AR thin films have prohibitively small laser induced damage threshold, limited bandwidth, and may have issues with adhesion. Nanohmics Inc. is developing monolithic broadband anti-reflective nanostructures applied to the facets of oriented patterned GaAs crystals. The company has developed a method to pattern and etch nanostructures using a self-assembled masking process that is well-suited to apply on facets as small as  $1 \times 5 \text{ mm}^2$ . The key steps in the process is generating a random nanoparticle metal etch mask using rapid thermal anneal process and forming nanostructures in a subsequent reactive ion etch step. Both processes are performed at the UT Austin Microelectronics Research Center cleanroom facilities.



Karun Vijay, Nanohmics. Work performed at Texas Nanofabrication Facility.

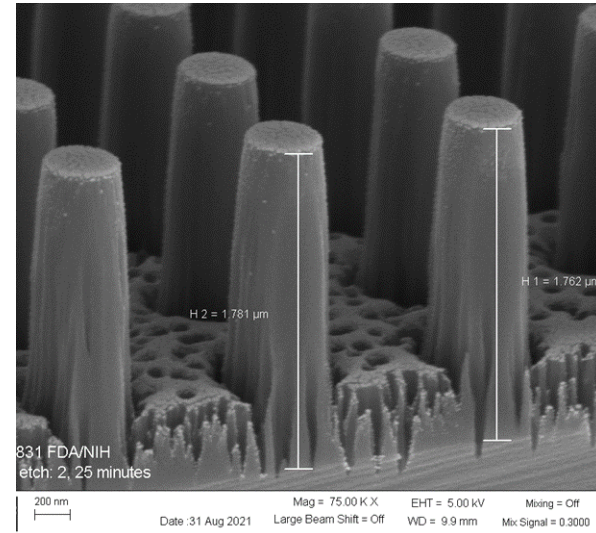
This work was supported by Air Force STTR FA8650-19-C-1947.

National Research Priority: NSF–Quantum Leap



# Polarization Metasurface Detection Device for Food Safety

The ongoing effort in food safety diagnostics is in constant need of tests, which are more deployable (i.e. lower-cost and more compact), with the utmost accuracy. Metasurface-based optics offer an opportunity to meet these goals. Nanohmics seeks to develop a polarization metasurface-based biosensor, which is robust against vibrational and background noise, in order to develop a highly sensitive washless “bind and detect” assay with rapid testing (within minutes), high-throughput multiplexing, and portability to ports of entry and food processing plants.



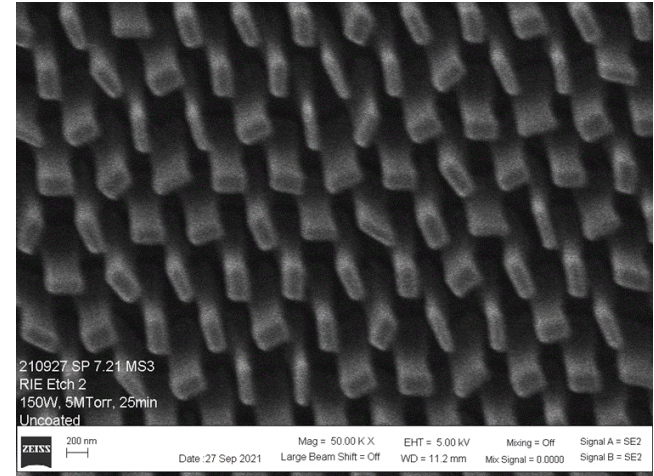
Mark Lucente, Nanohmics. Work performed at Texas Nanofabrication Facility.

This work was supported by FDA SBIR 1 R43 FD006910-01.

*National Research Priority: NSF–Growing Convergence Research*

# Compact Imaging Spectropolarimeter Based On Multifunction Meta-optic

The overall goal of this program is to develop a low-SWaP imaging spectropolarimeter using an ultrathin, light-weight, microfabricated multifunction meta-optic. Because of their extremely low mass and thickness, these low-aberration optics are ideal for sensors and imagers in SWaP-constrained vehicles. Both orbital and cost-effective suborbital Earth science measurements can benefit from instruments with small size, weight and power consumption (SWaP). Low-SWaP spectral imagers accelerate data collection such as atmospheric aerosol absorption and scattering. Metamaterial optics provide dramatic reductions in mass and thickness compared with traditional optics, which often require bulky supporting structures. A multifunction meta-optic combines the optical functions of multiple conventional optics, further reducing cost, volume, and mass while enhancing sensor performance and robustness.



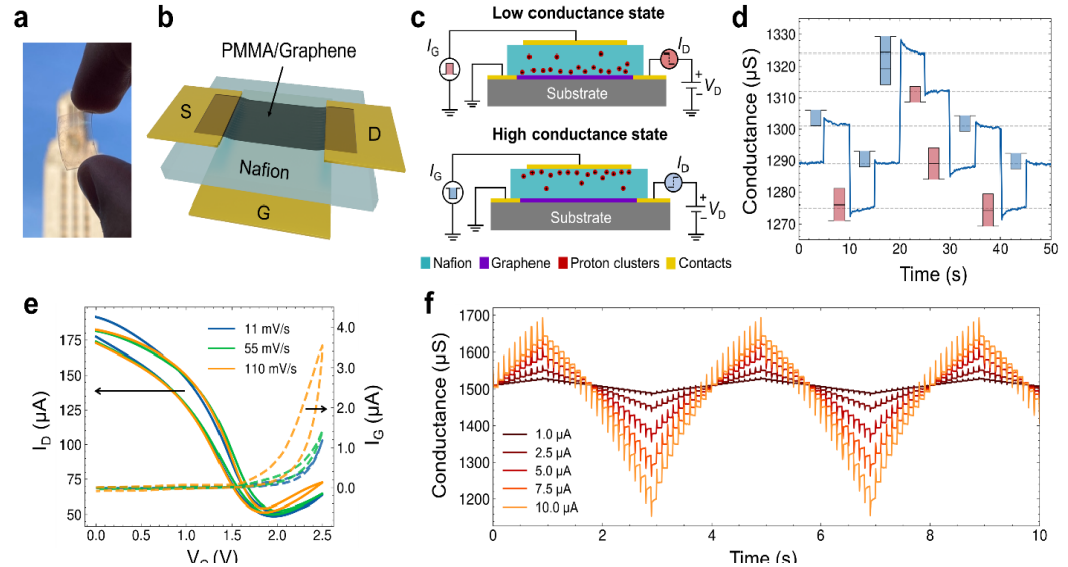
Mark Lucente, Nanohmics. Work performed at Texas Nanofabrication Facility.

This work was supported by NASA SBIR 80NSSC21C0302

*National Research Priority: NSF–Quantum Leap*

# Graphene neuromorphic device

CMOS-based computing systems that employ the von Neumann architecture are relatively limited when it comes to parallel data storage and processing. In contrast, the human brain is a living computational signal processing unit that operates with extreme parallelism and energy efficiency. Although numerous neuromorphic electronic devices have emerged in the last decade, most of them are rigid or contain materials that are toxic to biological systems. In this work, we report on biocompatible bilayer graphene-based artificial synaptic transistors (BLAST) capable of mimicking synaptic behavior.



Neuromorphic device based on graphene

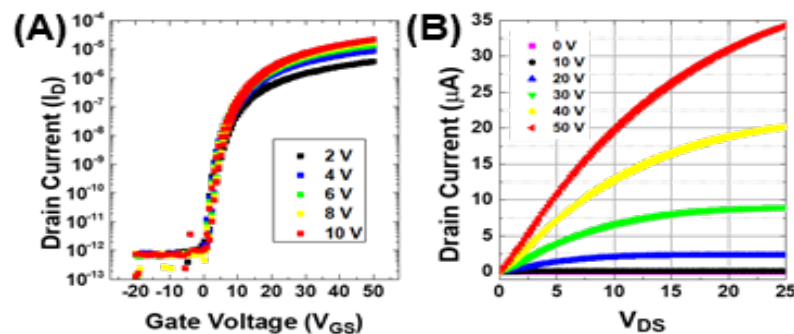
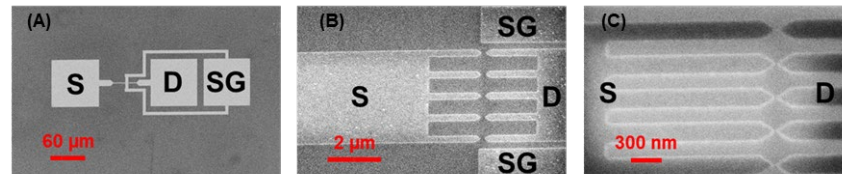
C.Bennett, Sandia with Prof. Incorvia (UT). Work performed at Texas Nanofabrication Facility.

This work was supported by NSF MRSEC grant DMR-1720595. Sandia supported by DOE grant DE-NA0003525.

National Research Priority: NSF-Quantum Leap

# Indium gallium zinc oxide (IGZO) thin-film transistors

A new design for short channel length thin-film transistors (TFTs) was demonstrated with indium gallium zinc oxide (IGZO) [1]. The principal advantages of the new design arise from two distinct effects: (i) Nanospike array electrodes produce field-emission enhanced charge injection from the source/drain contacts to the semiconductor and lead to higher currents, current densities, and carrier velocities. (ii) Quasi-three-dimensional gate control at low gate voltages leading to improved turn-off characteristics, better sub-threshold swing and reduced drain voltage dependence of sub-threshold behavior. These advantages are very beneficial for back-end-of-the-line (BEOL) TFTs and will also apply to most other semiconductor material TFTs with Schottky contacts.



IGZO TFTs with channel lengths 50-200 nm

Kelly Liang, Carla McCully, Ananth Dodabalapur, Dept. of Elec. Comp. Eng. Univ. of Texas. Work performed at TNF.

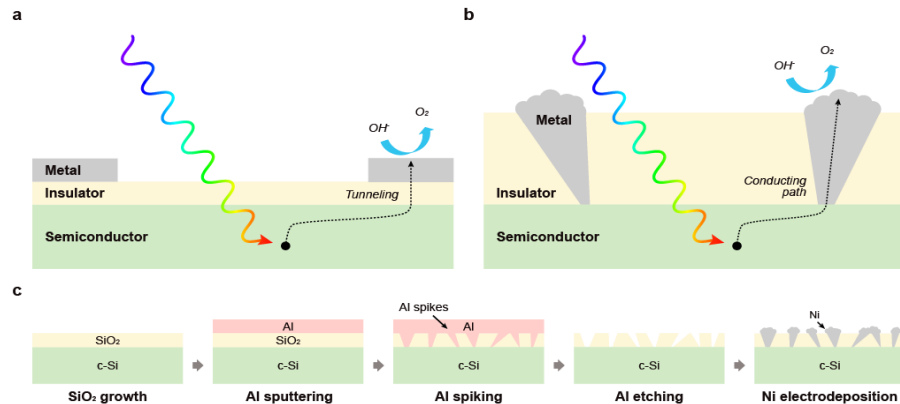
*Sci. Adv.* 8, 28 January 2022; *ACS Applied Electronic Materials*, October 2021.

National Research Priority: NSF–Quantum Leap



# High-Performance Metal-Insulator-Silicon Photoanodes for Solar Powered Water Oxidation

In work supported by CBET and DMR, the latter through the Center for Dynamics and Control of Materials: an NSF MRSEC, researchers in Edward Yu's laboratory have demonstrated a low-cost, scalable approach for fabrication of high-performance, extremely stable photoanodes for solar-powered water oxidation. Such devices are a key element in systems for using the power in solar illumination to split water molecules into hydrogen and oxygen. A fundamental issue plaguing conventional semiconductor photoelectrodes is that semiconductor materials that are efficient absorbers of solar illumination, e.g., silicon, are easily corroded in the liquid environment in which solar-driven photoelectrochemical reactions typically must occur. Incorporation of a wide-bandgap electrically insulating protective layer atop the semiconductor to separate the semiconductor from the solution has been explored quite extensively as an approach to improve stability, resulting in the development of metal-insulator-semiconductor (MIS) photoelectrodes. However, MIS photoelectrodes must contend with the challenge of providing efficient transport of photogenerated carriers across the insulator to the catalyst (typically metal) at the device-liquid interface.



*MIS photoanode for solar water oxidation*

Soonil Lee, Ed Yu, Electrical Eng., Univ. of Texas at Austin. Work performed at Texas Nanofabrication Facility.

This work was supported by NSF MRSEC at UT Austin.

*National Research Priority: NSF–Growing Convergence Research*

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***Virginia Tech National Center for Earth and  
Environmental Nanotechnology  
Infrastructure (NanoEarth)***

# Developing commercial pigments from nanoparticles recovered from mine waste

Acid mine drainage (AMD) is a result of coal and mineral mining that negatively impacts ecosystem health and soil and water quality at thousands of sites worldwide. Hoover Color in Hiwassee, VA is using metals recovered from coal AMD waste in a line of pigments called EnvironOxide™. Their properties meet those of conventional pigments, and they have the added benefit of being the result of an environmental restoration effort.

NanoEarth demonstrated that the pigments sourced from AMD consist of nanosized iron hydroxide or oxide particles. The extreme small sizes of the particles may explain why they exhibit better transparency, dispersibility, and tinting strength compared with their conventional counterparts.



Pigment

BG-836 BG-832 BR-834 BR-833

1. AMD discharging from abandoned coal mine
2. Engineered AMD neutralization and settling ponds
3. Iron hydroxide precipitates ready to be harvested

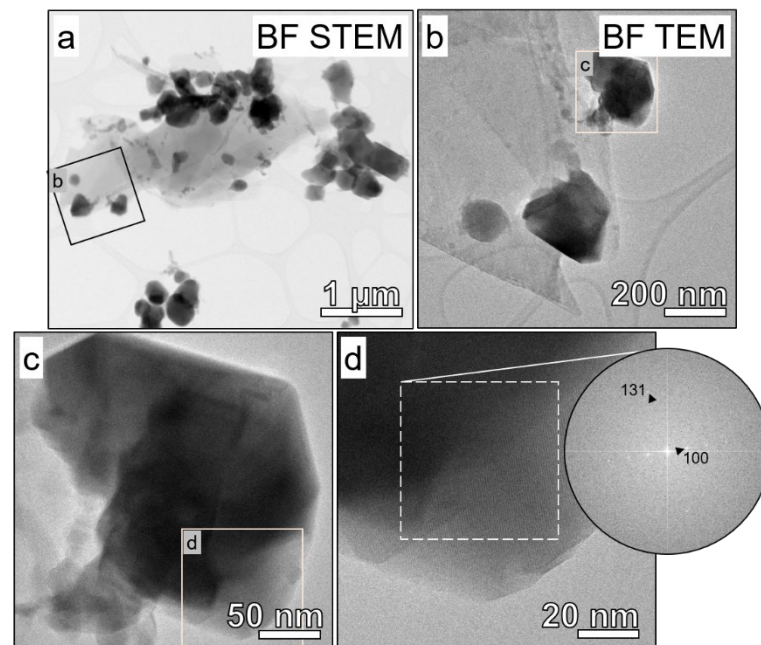
Hoover Color is a division of Cathay Industries. Work performed at Virginia Tech National Center for Earth and Environmental Nanotechnology Infrastructure (NanoEarth).

*National Research Priority: NSF–Growing Convergence Research and  
USDA–Sustainable Use of Natural Resources*

# Nanomaterials from wildland-urban fires

Fires generate numerous combustion products; however, the nature of the material consumed can have important implications for the environment as well as human health. The focus of this research is the identification and characterization of metal-based incidental nanomaterials present in fire ash from the wildland-urban interface.

Transmission electron microscopy (TEM) at NanoEarth demonstrated the presence of numerous types of metallic nanomaterials, including Fe-, Ti-, Zn-, and Cr, Cu, As-bearing nanoparticles (NPs) in field samples. Exposure to some of these phases may have implications for human health and climate change.



Transmission electron microscopy (TEM) images of an ash aggregate containing Fe-bearing nanoparticles identified as the iron oxide mineral magnetite (darker particles in a–d).

Mohammad Baalousha, University of South Carolina. Work performed in part at NanoEarth.

This research is funded by NSF CBET 2101983 Rapid Collaboration Proposal: Characterization, Quantification, and Transport of Incidental Nanomaterials from Wildland-Urban Fires in Surface Waters. It also received funding support under NanoEarth's (ECCS 1542100 and ECCS 2025151) Multicultural and Underserved Nanoscience Initiative (MUNI).

*National Research Priority: NSF–Growing Convergence Research and  
EPA–Children's Environmental Health*



# Practical method of detection for humic substances

The study aims to discover a practical analytical procedure for the detection of humified materials. Humic substances (HS) are the active components of soil humus and dissolved natural organic matter in the aqueous environment, which provide profound positive effects on agricultural production and the overall health of the environment. HS are the most widespread natural biogenic organic substances in all terrestrial and aquatic environments, representing the major portion of organic carbon in the global carbon cycle. Various commercial humic products are used as agriculture fertilizer products, livestock feeds, and human nutraceuticals to increase the efficacy of nutrients and other benefits. Despite the fact that there are numerous definitions for HS, there are no standards to determine if the materials used for research or the manufacture of humic products are genuine.

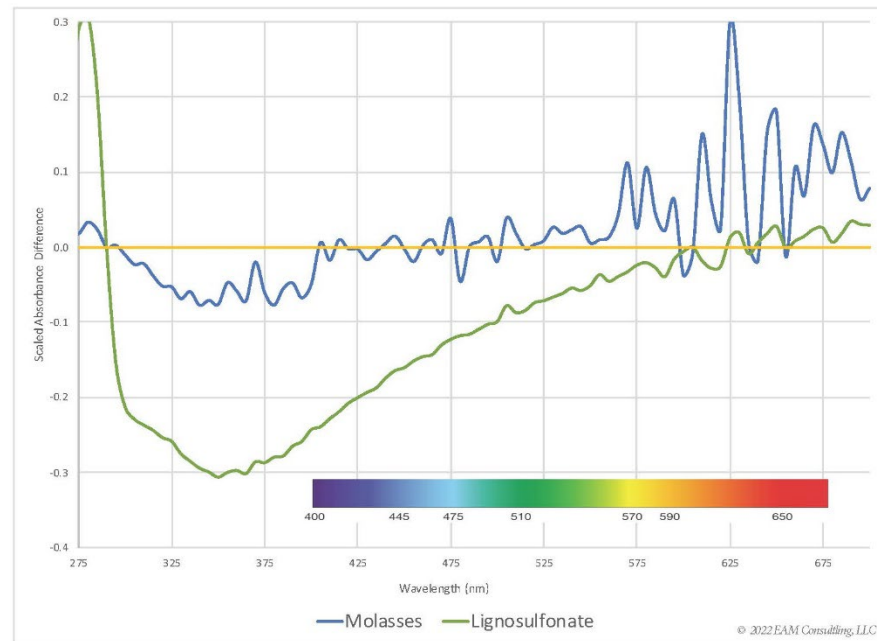
This project involves testing an analytical technique based on ISO 19822:2018 (E) extraction protocols using common laboratory equipment followed by UV-visible spectroscopy to determine if a material is humified.

Lawrence Mayhew, EAM Consulting, LLC. Work performed at Virginia Tech National Center for Earth and Environmental Nanotechnology Infrastructure (NanoEarth).

Supported by NSF Award # ECCS-1542100 & ECCS 2025151.

National Research Priority: NSF–Understanding the Rules of Life and

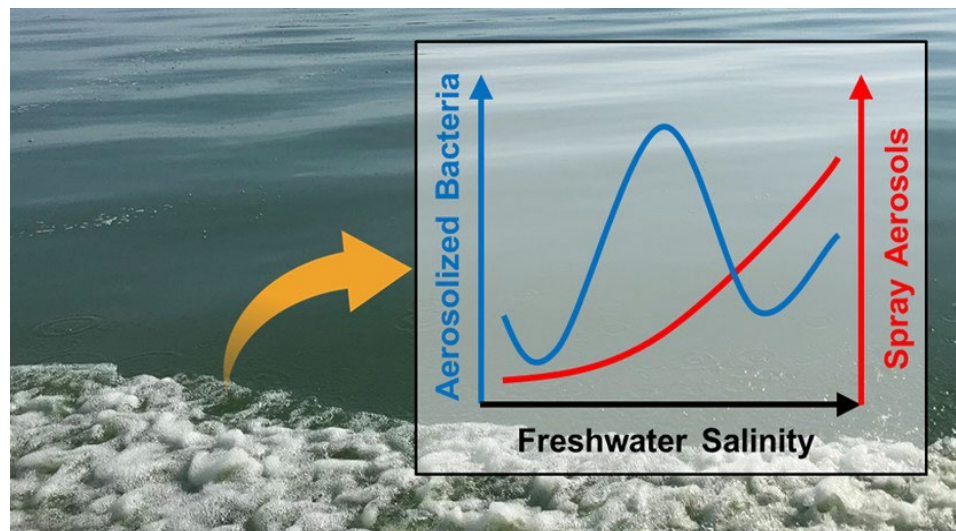
USDA–Global Food Supply and Security



*Scaled Absorbance Difference of two well know humic product adulterants compared to humic substances. 0.0 = xAsc*

# Increasing freshwater salinity impacts aerosolized bacteria

Increases in the salt concentration of freshwater result in detrimental impacts on water quality and ecosystem biodiversity. Biodiversity effects include freshwater microbiota, as increasing salinity can induce shifts in the structure of native freshwater bacterial communities, which could disturb their role in mediating basal ecosystem services. Moreover, salinity affects the wave breaking and bubble-bursting mechanisms via which water-to-air dispersal of bacteria occurs. This project demonstrates that increases in freshwater salinity are likely to influence the abundance and diversity of aerosolized bacteria. These shifts in aerosolized bacterial communities might have broader implications on public health by increasing exposure to airborne pathogens via inhalation. Impacts on regional climate, related to changes in biological ice-nucleating particles (INPs) emission from freshwater, are also expected.



*Effects of freshwater salinity on aerosols and aerosolized bacteria*

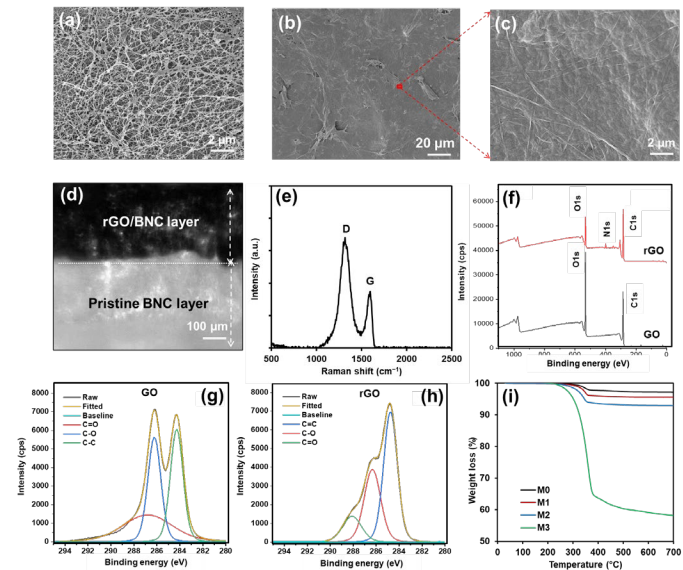
Harb, C., Pan, J., DeVilbiss, S., Badgley, B., Marr, L., Schmale, D., & Foroutan, H. Work performed at Virginia Tech National Center for Earth and Environmental Nanotechnology Infrastructure (NanoEarth).

Supported by NSF Award # ECCS-1542100 & ECCS 2025151 and the Virginia Tech Fralin Life Science Institute.  
*Environmental Science & Technology*, 55(9), 5731–5741, 2022.

*National Research Priority: NSF–Growing Convergence Research*

# One-step biosynthesis of bilayered graphene oxide embedded bacterial nanocellulose hydrogel for versatile photothermal membrane applications

The sustainability capacity of the developing world is being stressed by the ever-increasing demand for clean water and energy resources. Efficient utilization of abundant solar energy to harvest clean water is the most viable solution to the world's problem at the water-energy nexus. Developing nano-enabled photothermal membranes is a way towards developing sustainable water purification through filtration, photothermal disinfection, and distillation. Layered integration of desired functions of photothermal materials onto suitable support material without compromising its chemical, thermal, and mechanical properties remains challenging. In this project, a one-step green approach to biosynthesize the bilayer structured hydrogel composite of graphene oxide (GO) and bacterial nanocellulose (BNC) through modifying the growth rate of BNC producing bacteria *Gluconacetobacter xylinus* was demonstrated. The in situ integration of GO layers onto the BNC fiber network was controlled via amending the corn steep liquor as a bacterial growth enhancer. The multipurpose nature of the biosynthesized photothermal membranes was also explored.



Scanning electron microscopy (SEM) images showing the surface morphologies of (a) BNC, (b) rGO/BNC:BNC membrane (low magnification), (c) rGO/BNC:BNC (high magnification) membrane as well as (d) cross sectional image of bilayer rGO/BNC:BNC membrane, (e) Raman spectrum of GO, (f) XPS survey scans of pristine GO and rGO/BNC:BNC membrane, (g) C1s high resolution spectra of GO, (h) C1s high resolution spectra of rGO, and (i) TGA analysis of membranes M0 – M3

Divyapriya, G., Rahman, A., Leng, W., Wang, W., & Vikesland, P. J. Work performed at Virginia Tech National Center for Earth and Environmental Nanotechnology Infrastructure (NanoEarth).

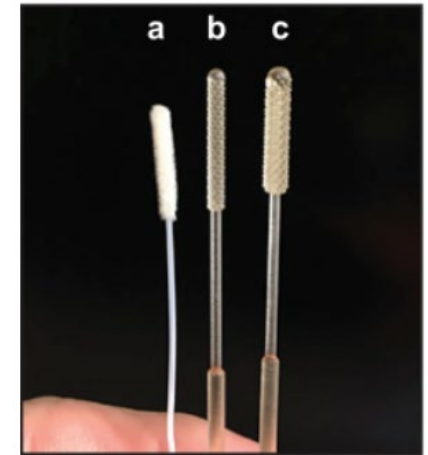
This research was supported by the Fulbright Program, United States-India Educational Foundation, India, and Institute of International Education, USA (2471/FNPDR/2019) and NSF PIRE grant (1545756). *Environmental Science: Nano*, 5(9), 1639-1650, 2022.

National Research Priority: DOE–Advanced and Sustainable Energy and

USDA–Climate and Energy Needs

# Development and implementation of a scalable and versatile test for COVID-19 diagnostics in rural communities

Rapid and widespread testing of severe acute respiratory coronavirus 2 (SARS-CoV-2) is essential for an effective public health response aimed at containing and mitigating the coronavirus disease 2019 (COVID-19) pandemic. Successful health policy implementation relies on early identification of infected individuals and extensive contact tracing. However, rural communities, where resources for testing are sparse or simply absent, face distinctive challenges to achieving this success. Accordingly, we report the development of an academic, public land grant University laboratory-based detection assay for the identification of SARS-CoV-2 in samples from various clinical specimens that can be readily deployed in areas where access to testing is limited. The test, which is a quantitative reverse transcription polymerase chain reaction (RT-qPCR)-based procedure, was validated on samples provided by the state laboratory and submitted for FDA Emergency Use Authorization. Our test exhibits comparable sensitivity and exceeds specificity and inclusivity values compared to other molecular assays. Additionally, this test can be re-configured to meet supply chain shortages, modified for scale up demands, and is amenable to several clinical specimens. Test development also involved 3D engineering critical supplies and formulating a stable collection media that allowed samples to be transported for hours over a dispersed rural region without the need for a cold-chain. These two elements were critical when shortages impacted testing and when personnel needed to reach areas that were geographically isolated from the testing center. Overall, using a robust, easy-to-adapt methodology, we show that an academic laboratory can supplement COVID-19 testing needs and help local health departments assess and manage outbreaks. This additional testing capacity is particularly germane for smaller cities and rural regions that would otherwise be unable to meet the testing demand.



*Top Left: Completed print of 324 adult swabs using Formlabs Form2 with Surgical Guide resin. Top Right: PurFlock (Puritan Medical Products, LLC) test swab (a) compared to 3D-printed pediatric (b) and adult (c) swabs.*

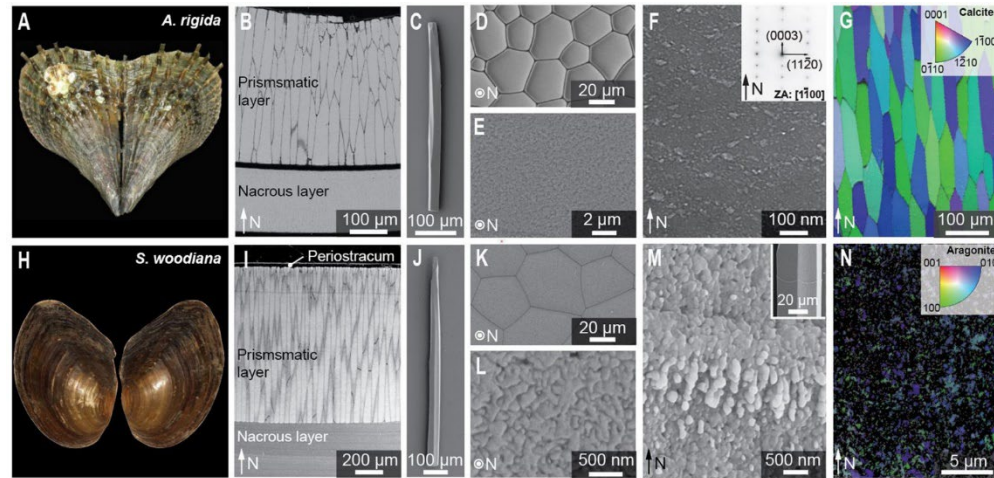
Ceci, A., Munoz-Ballester, C., Tegge, A., et al. Work performed at Virginia Tech National Center for Earth and Environmental Nanotechnology Infrastructure (NanoEarth).

This project was supported by a Go Virginia Economic Resilience and Recovery Grant (20-GOVA-ERR-02A and 20-GOVA-ERR-02D), the Department of General Services of the Commonwealth of Virginia (DGS-201020-UVT), and funds from Virginia Tech, the Fralin Biomedical Research Institute at VTC, and the Fralin Life Sciences Institute. *Nature Communications*, 12, 4400, 2021.



# Intrinsic mechanical properties of individual biogenic mineral units in biomineralized skeletons

A bioinspired study on replicating the superior damage tolerance of bioceramic composites requires a detailed understanding of the intrinsic properties of biogenic mineral units. Here, we investigate and compare the intrinsic properties of biogenic calcite (*Atrina rigida*) and aragonite (*Sinanodonta woodiana*) by conducting microbending experiments on the separated prismatic building blocks. Analyzed bending results indicate that the biogenic calcite has a higher modulus ( $36.24 \pm 14.4$  GPa for *A. rigida* vs.  $29.9 \pm 10.5$  GPa for *S. woodiana*) and strength ( $446.5 \pm 141.5$  MPa for *A. rigida* vs.  $338.6 \pm 63.2$  MPa for *S. woodiana*) than the biogenic aragonite, while the nanoindentation results indicate the opposite trend. Furthermore, systematic fractographic analysis suggests that the biogenic calcite fractures like amorphous glass, while the biogenic aragonite resembles polycrystalline ceramics. These contradictory behaviors of biogenic calcite and aragonite under tension-dominated (microbending) and indentation loading conditions are attributed to their different intrinsic structures, that is, intracrystalline organic inclusions in single-crystal calcite versus interlocked nanograins in polycrystalline aragonite.



Microstructure of prismatic building blocks in mollusk shells of (A–G) *Atrina rigida* and (H–N) *Sinanodonta woodiana*. (A,H) Photos of the (A) *A. rigida* and (H) *S. woodiana* shells, respectively. (B,I) Scanning electron microscopy (SEM) images of the vertical cross sections showing the prismatic layer and the nacreous layer in the (B) *A. rigida* and (I) *S. woodiana* shell, respectively. The arrow (“N”) indicates the normal direction of the shells. (C,J) SEM images of isolated prismatic building blocks from the (C) *A. rigida* and (J) *S. woodiana* shells, respectively. (D,E,K,L) SEM images of horizontally polished prismatic layers to compare the (D,K) cross-sectional geometry and (E,L) surface features from the (D,E) *A. rigida* and (K,L) *S. woodiana* shells, respectively. (F) Transmission electron microscopy (TEM) image of a vertical cross section from *A. rigida* prism, where the brighter parts highlighting the intracrystalline organic inclusions elongated along the transversal direction. The inset shows the corresponding selected area electron diffraction (SEAD) pattern taken from a section parallel to the “N” direction (zone axis (ZA): [1100]). (M) High-magnification SEM image on the *S. woodiana* prism surface, showing the nanograins. The inset shows the bowed line features on the prism surface. (G,N) EBSD results of the two prisms, confirming the crystalline nature of (G) single-crystal calcite in *A. rigida* and (N) polycrystalline aragonite in *S. woodiana*, respectively.

Deng, Z. & Li, L. Work performed at Virginia Tech National Center for Earth and Environmental Nanotechnology Infrastructure (NanoEarth).

This project was supported by Virginia Tech through the Department of Mechanical Engineering and the Institute for Critical Technology and Applied Science. *American Chemical Society Biomaterials Science and Engineering* (2021).

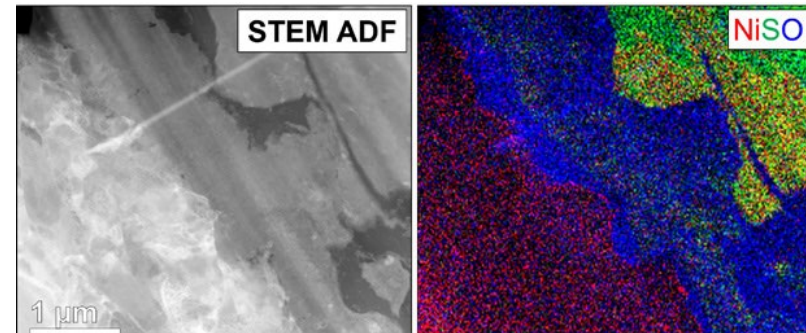
National Research Priority: NSF–Growing Convergence Research

# Nanoscience in the Earth & Environmental Sciences Research Community Virtual Workshop

- Designed for nano-novices in Earth, environmental, agricultural, water, geoscience, or related fields
- 2+ days focused on Case Studies:
  - 1) nanoparticles formed in a drinking water reservoir
  - 2) nanoscale structures in a meteorite sample
- Emphasis on collecting, preserving, and preparing environmental nano samples, instrumental data acquisition, analysis, and integration/interpretation
- 144 registrants and 65 workshop participants – very positive feedback
- All workshop materials available [online](#)



Falling Creek Reservoir (Vinton, VA), the sampling site for one of the case study examples



TEM image and compositional map of the meteorite case study sample, which illustrates boundaries between iron metal (left), magnetite (middle), and iron sulfides (right).

National Research Priority: NNCO–Nano4EARTH Challenge