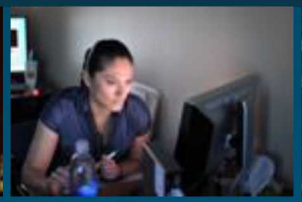
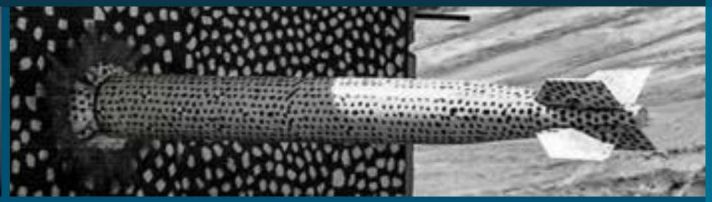
Sandia National Laboratories

SAND2018-9796C

TEM Mechanical Testing in Liquid with Temperature Control





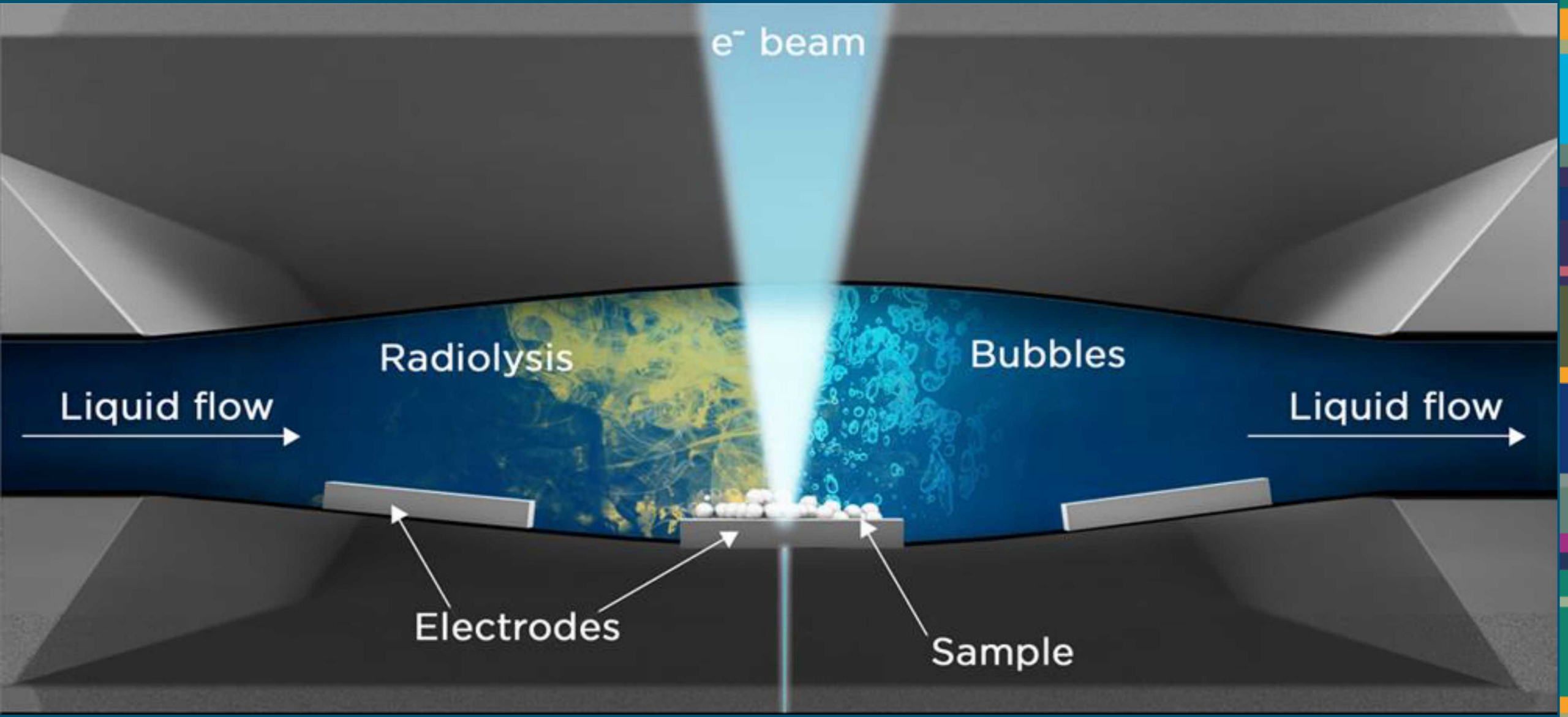
PRESENTED BY

Katherine Jungjohann

Claire Chisholm, Andrew Leenheer, William Mook, Khalid Hattar, and Michael Shaw

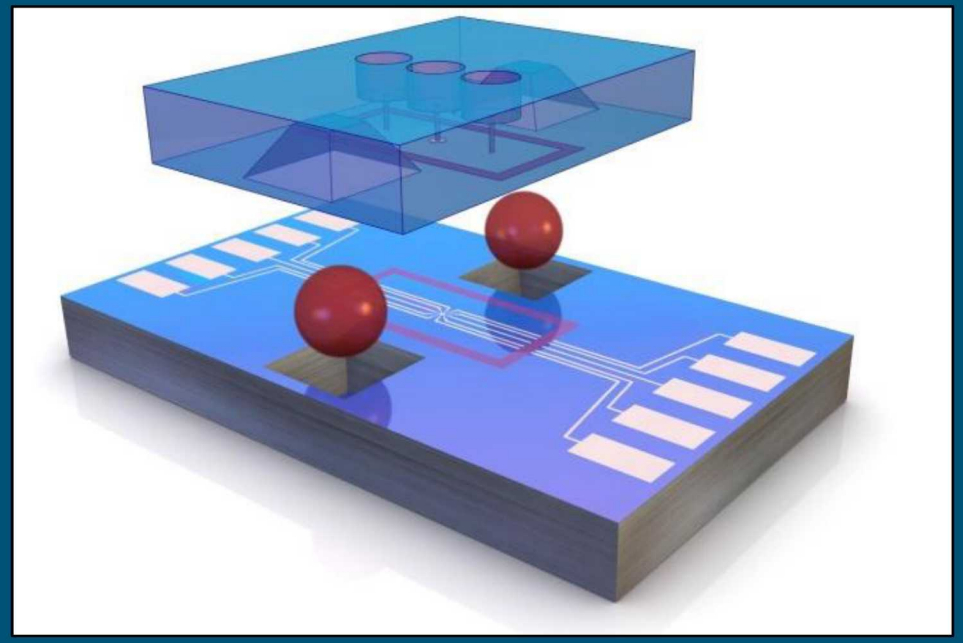
Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

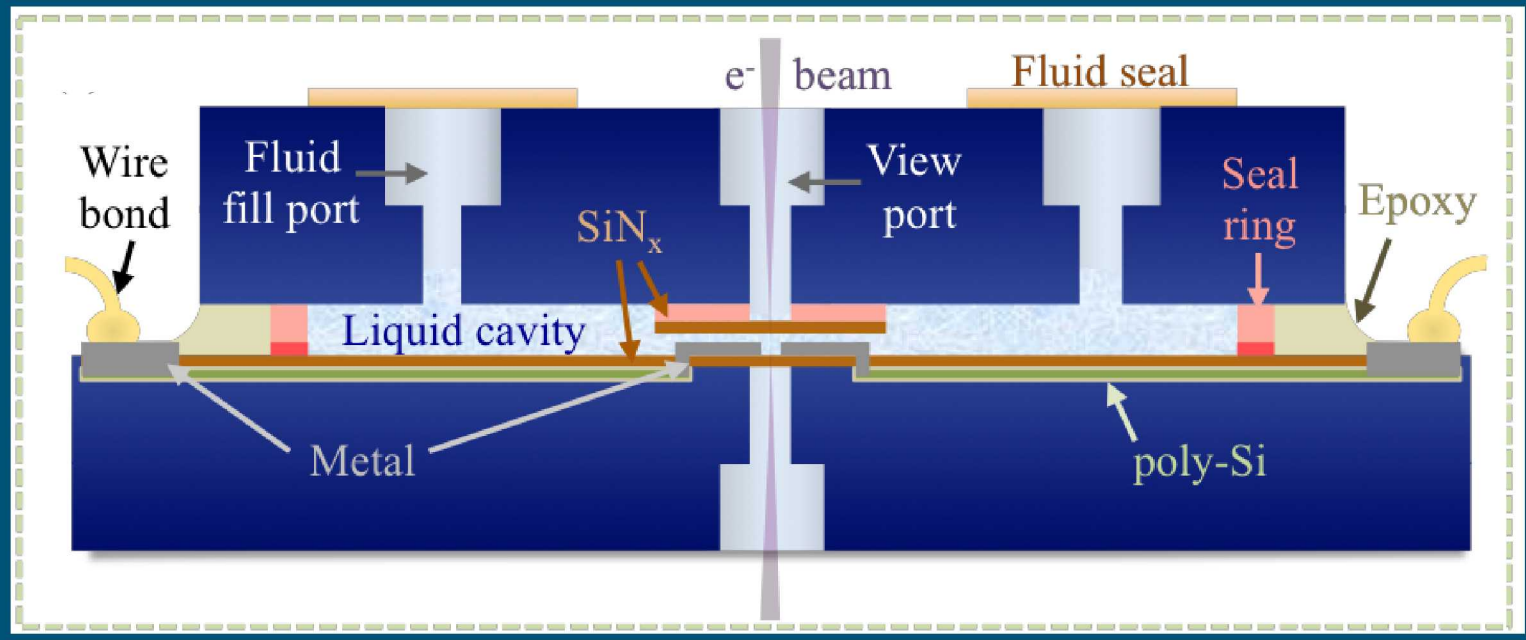
Liquid-Cell Electron Microscopy



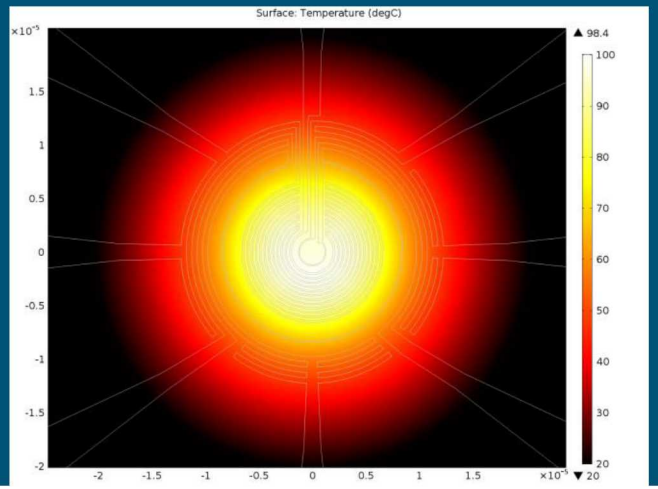
Hodnik et al., 2016 Acc. Chem. Res. **49**, 2015.

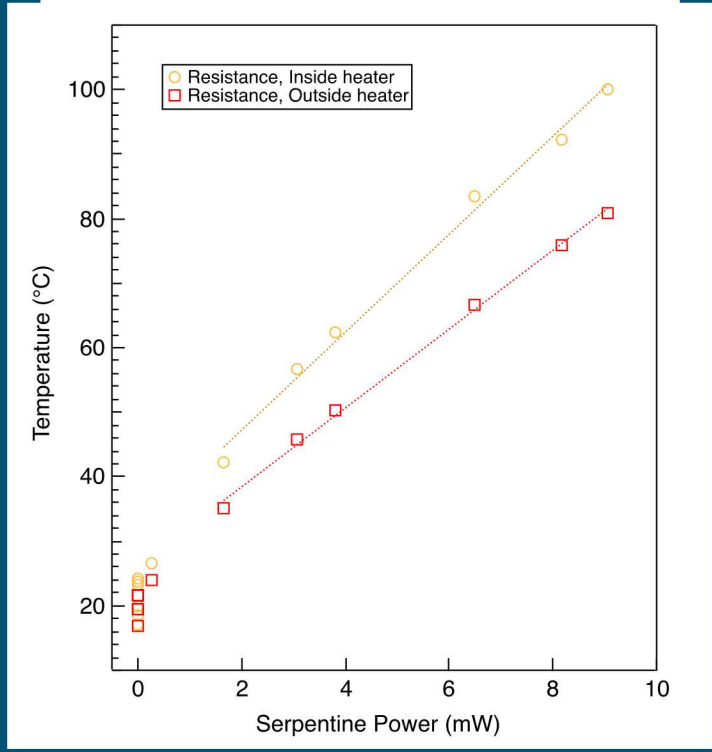
Electrochemical Closed-Cell Scanning/Transmission Electron Microscopy



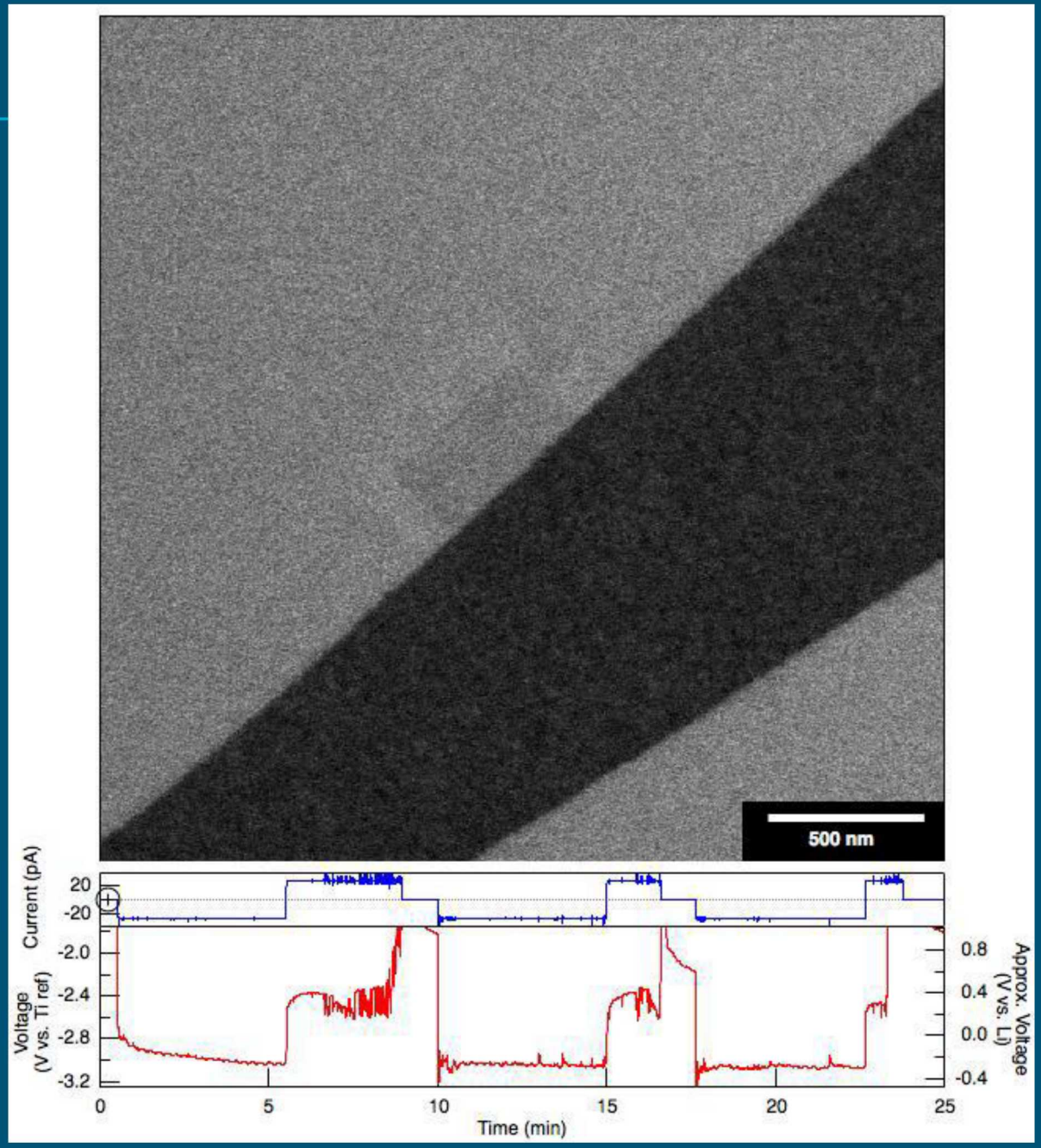


Temperature Control in Electrochemical Liquid Cell



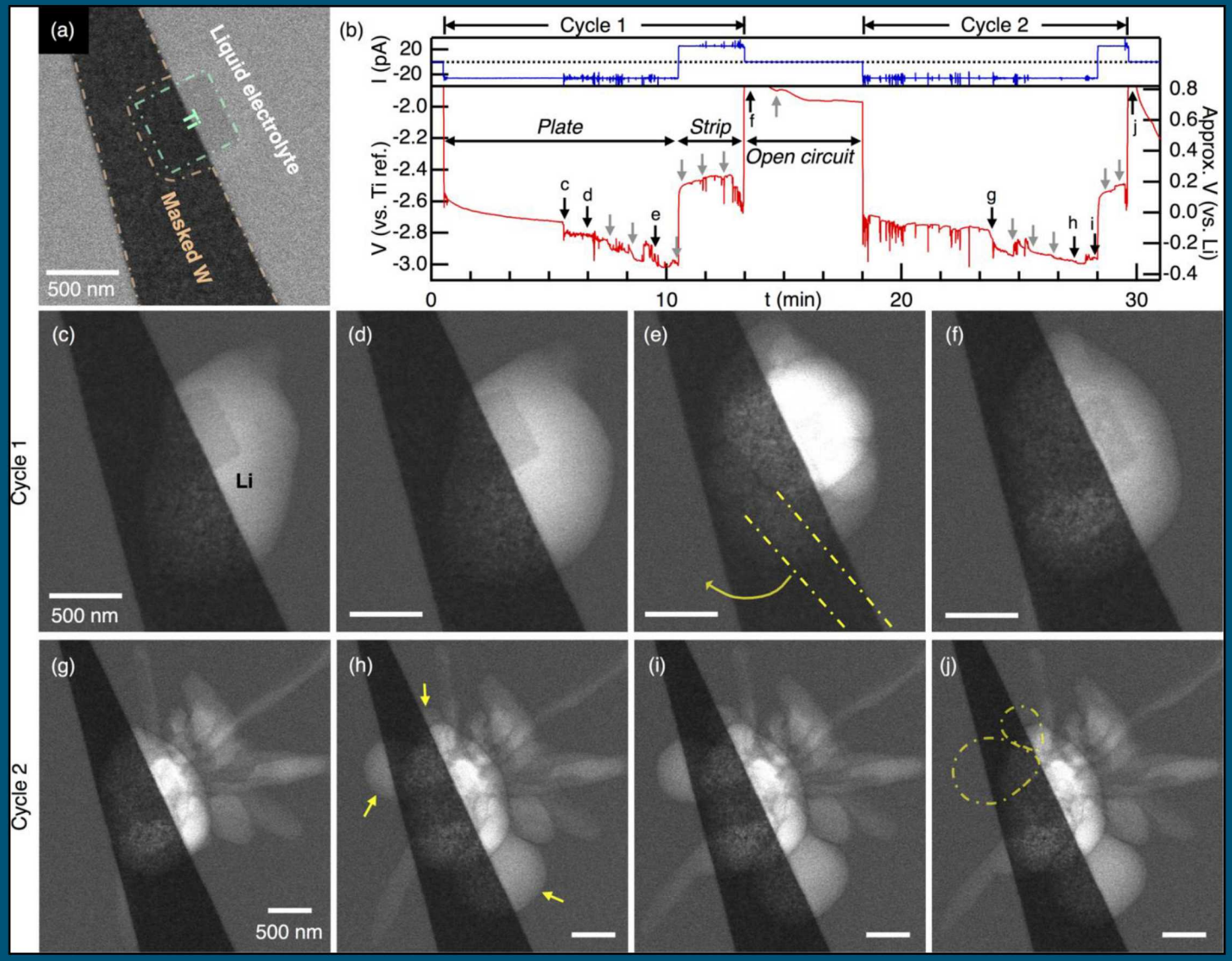


Li Metal Plating/Stripping



Leenheer et al. *ACS Nano* **9**, 4379 (2015)

Low-Dose Imaging Required for Li Plating/Stripping

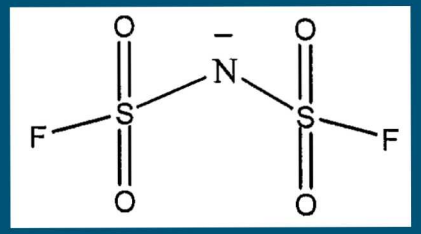


Leenheer et al. *ACS Nano* **9**, 4379 (2015)

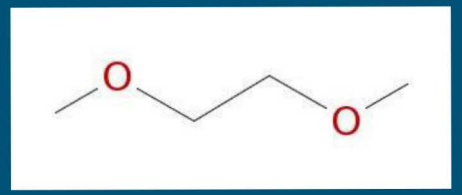
Contact Pressure Impacts Li Metal Plating Morphology

Salt

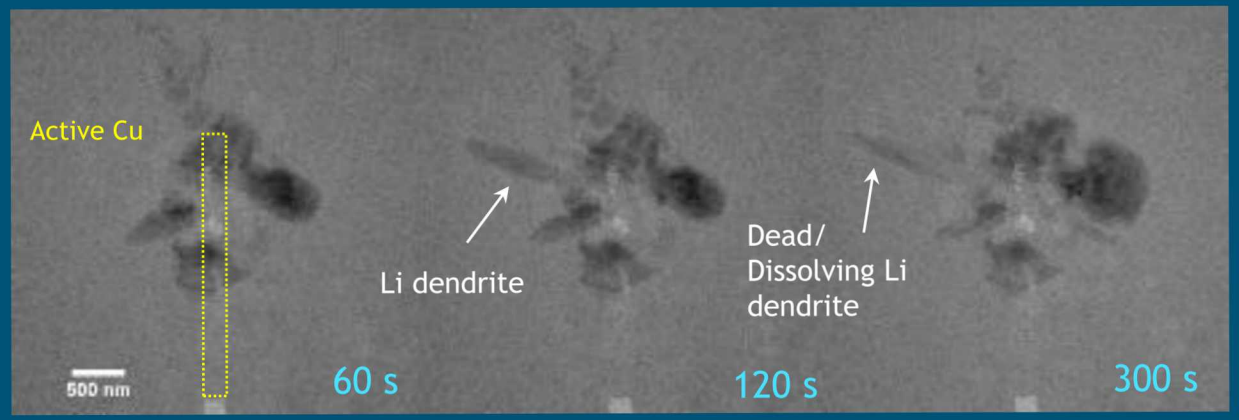
Li^+

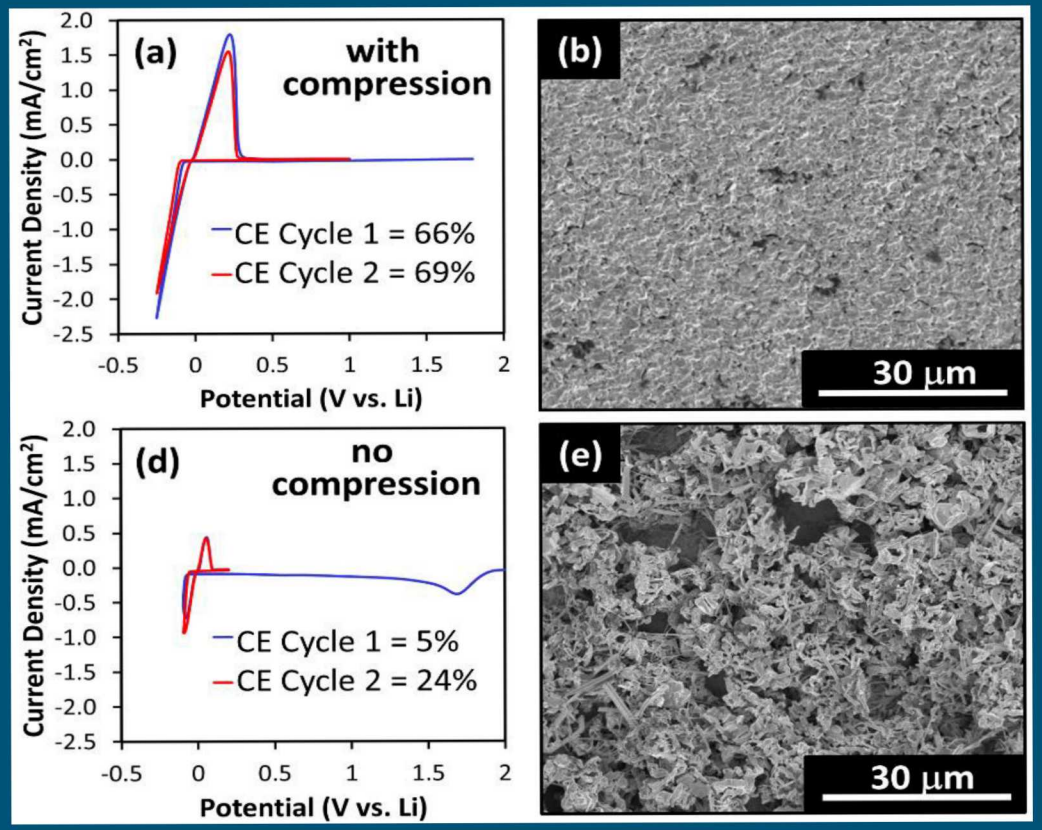


Solvent



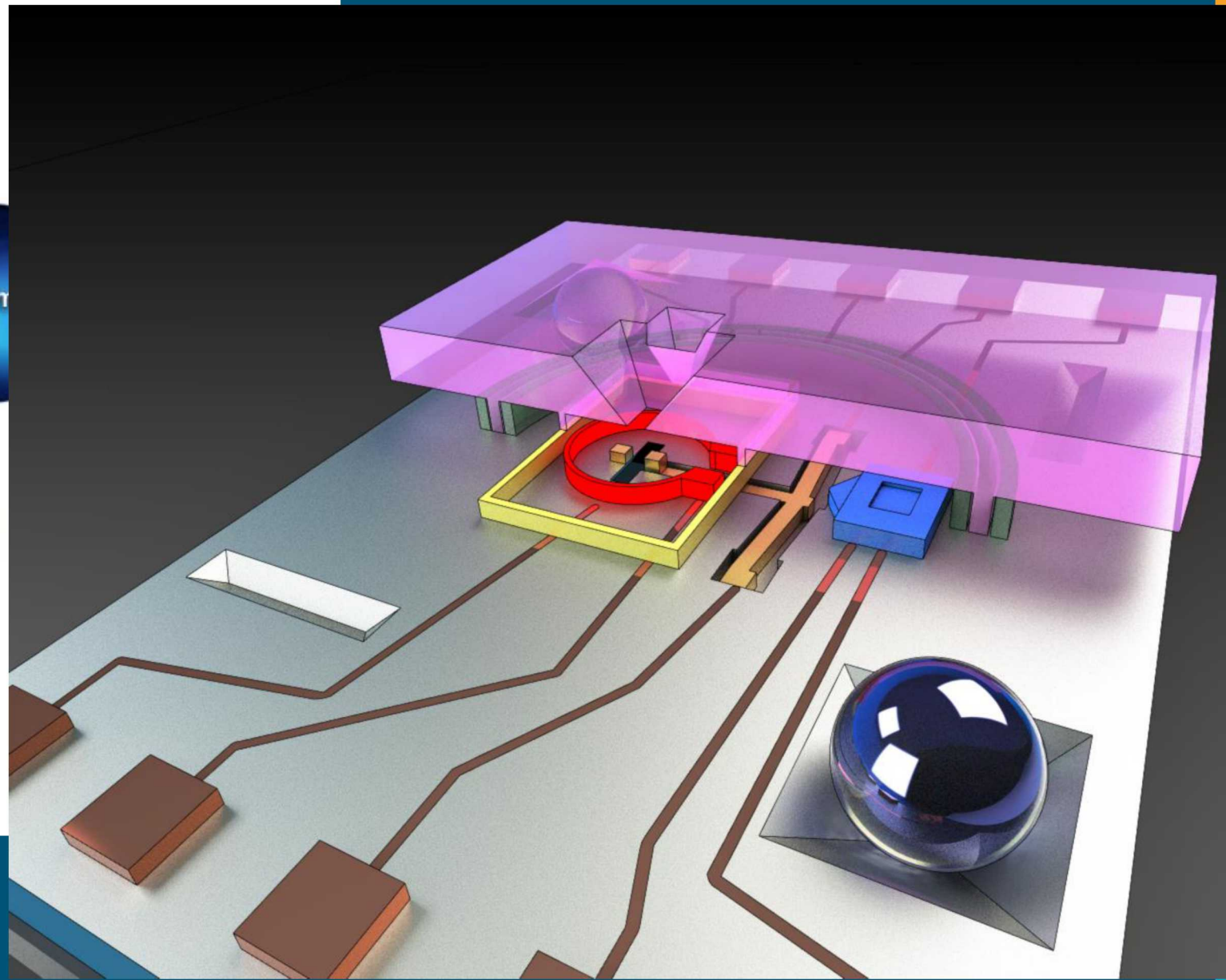
-2.25 mA/cm^2 on 0.44 µm^2 Cu: ***Cycle 10***



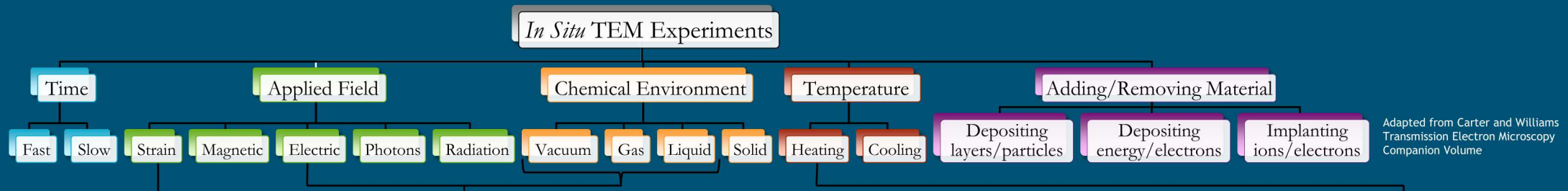


Harrison et al. *ACS Nano* **11**, 11194 (2017)

Mechanical-Environmental-Thermal TEM Discovery Platform



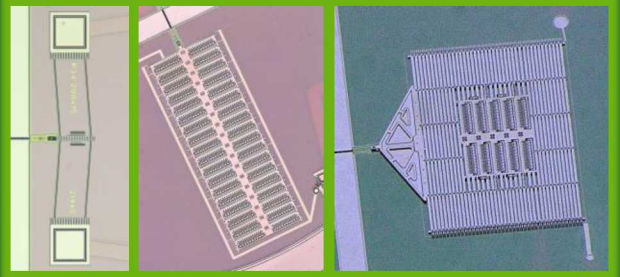
Mechanical Strain Observed in Sodiation of Antimony Nanofilms

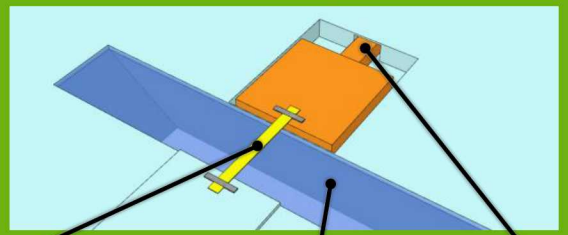


Adapted from Carter and Williams Transmission Electron Microscopy Companion Volume

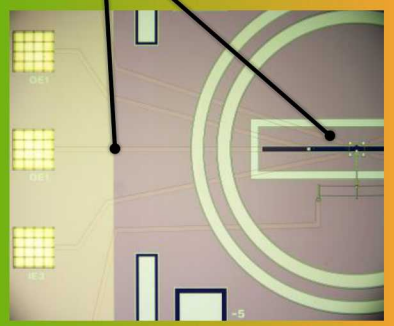
MEMS Actuator/Sensor Options

-Thermal -Electrostatic comb drive (ECD) -ECD+capacitive sensor

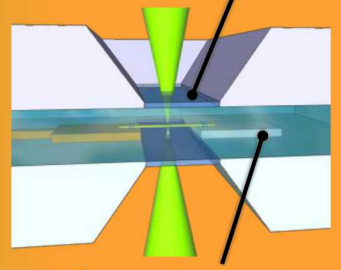




Sample affixed over Si_3N_4 window to the buried piston of the actuator

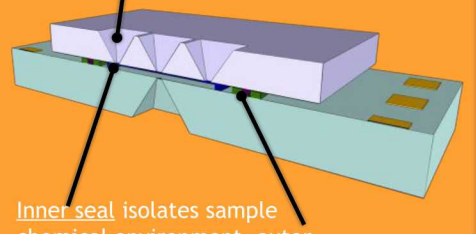


Buried electrical traces and electrical leads to the sample area



40 nm thick electron transparent Si_3N_4 windows

400 nm environmental chamber height

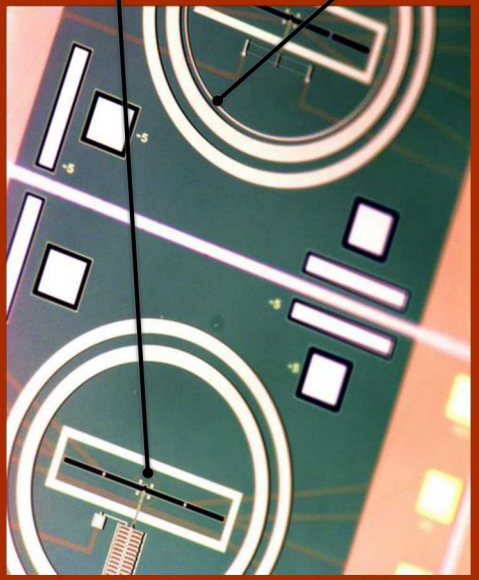


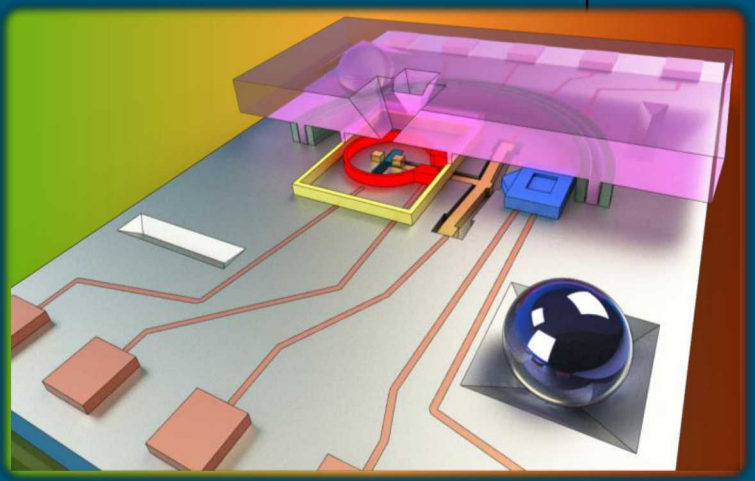
Fill ports in lid for chemical environment options: vacuum, gases, and liquids

Inner seal isolates sample chemical environment, outer hermetic seal isolates actuators operating in air

Heater Options

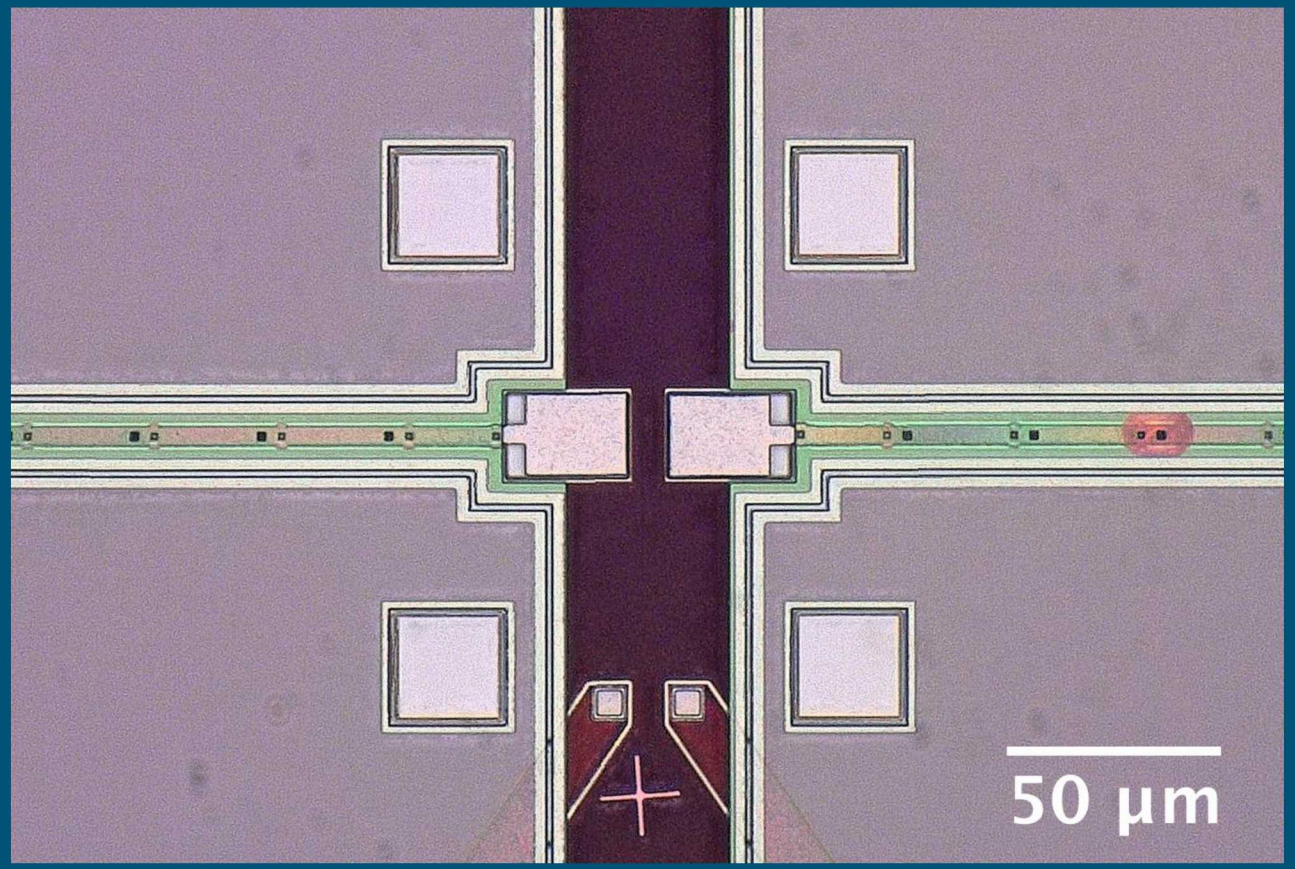
Buried inner heater Exposed outer heater

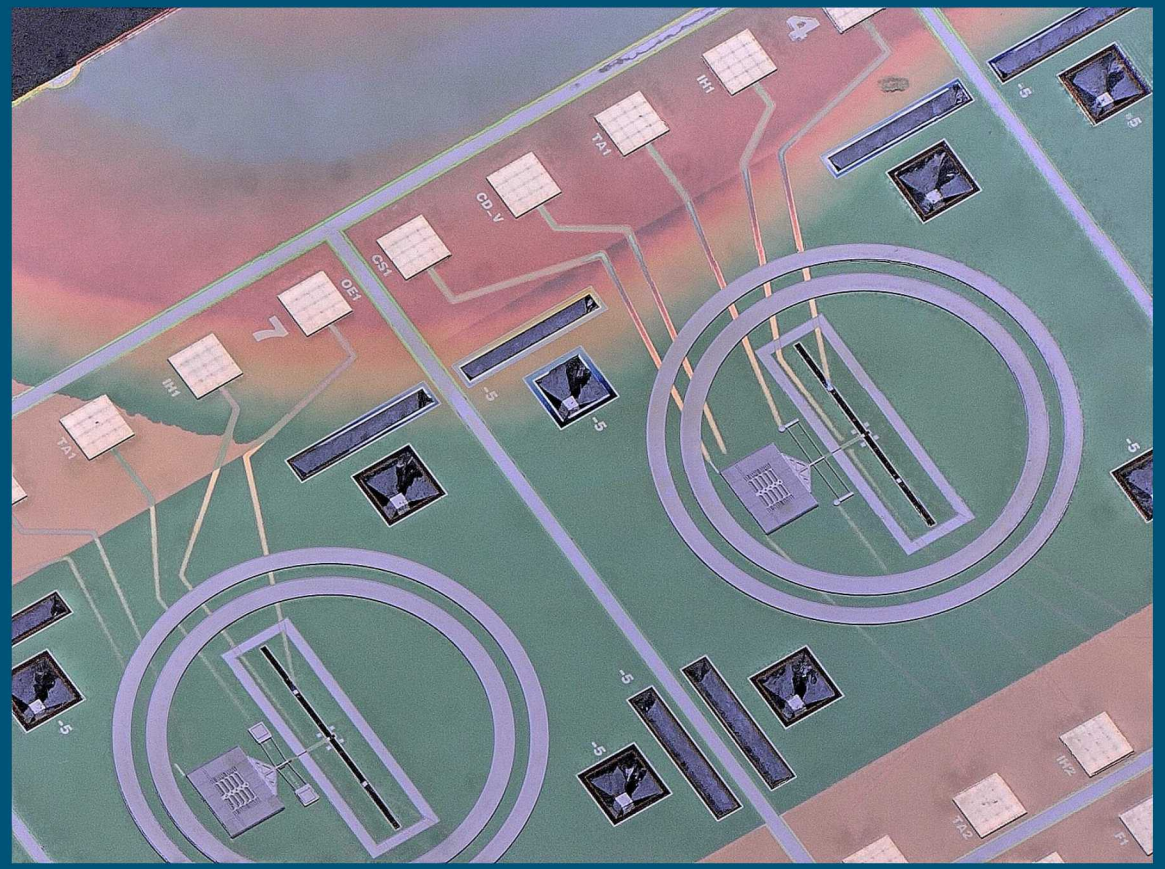




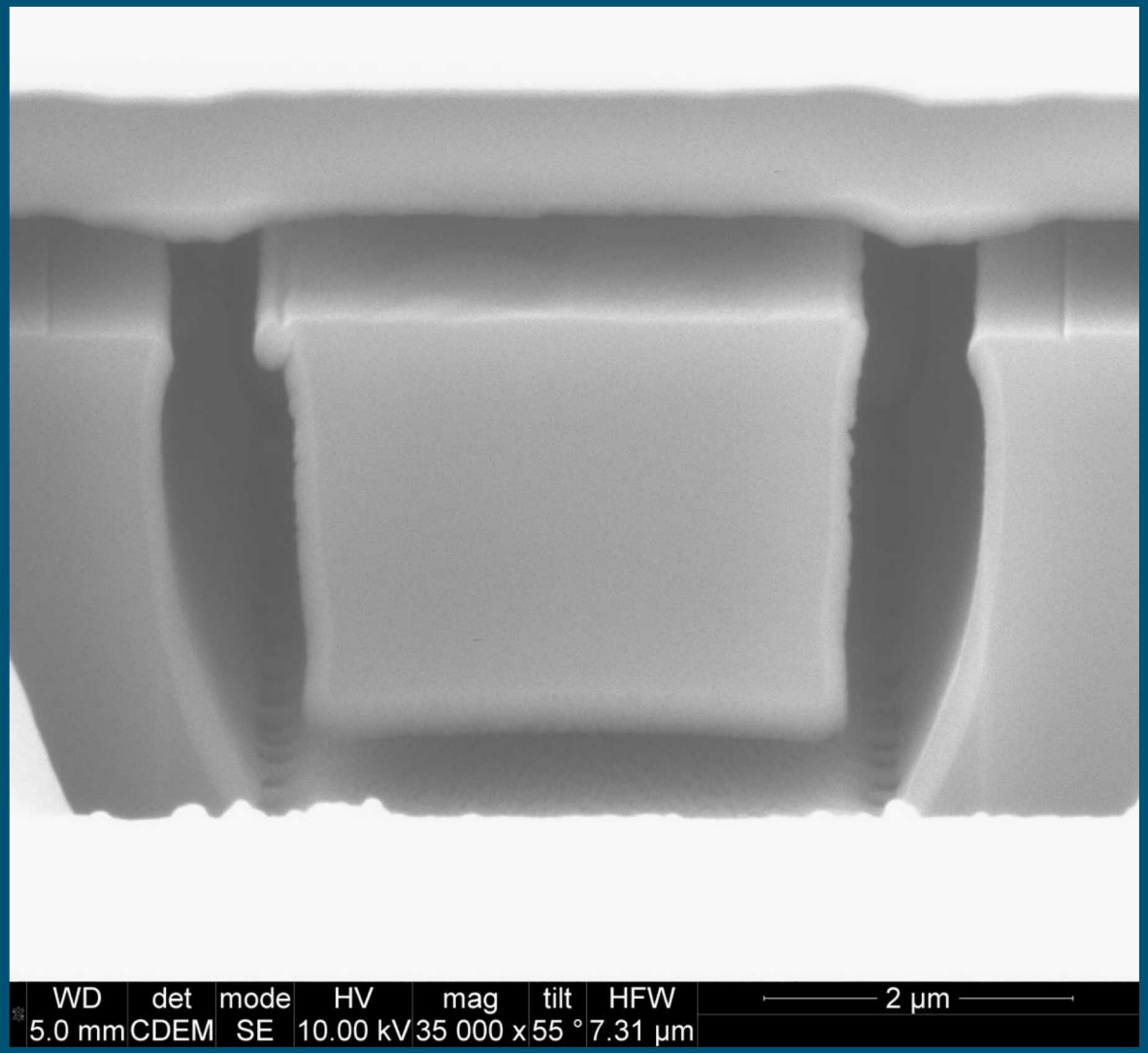
- A sample can be strained *in situ* while in a controlled operating environment (chemical, electrical, temperature).
- The sample is affixed, over a 40 nm thick electron transparent Si_3N_4 window, to a buried piston attached to a MEMS actuator (thermal or electrostatic).
- A lid, with a matching Si_3N_4 window, is aligned to the base using sapphire beads and/or optical fibers, then glued in place.
- An inner seal is created that isolates the sample chemical environment, an outer hermetic seal isolates the actuators operating in air.
- Fill ports in the lid allow for the addition of many chemistry options, including: vacuum, gases, and liquids.
- Buried electrical traces are used to control the actuator displacement, run the buried inner heater or exposed outer heater, and for patterning customized electrical contacts in the environmental chamber.

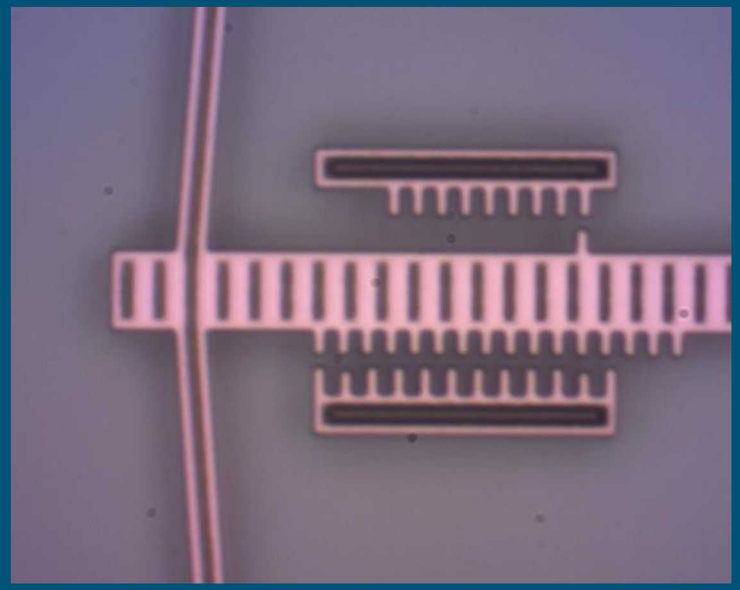
Mechanical-Environmental-Thermal TEM Platform

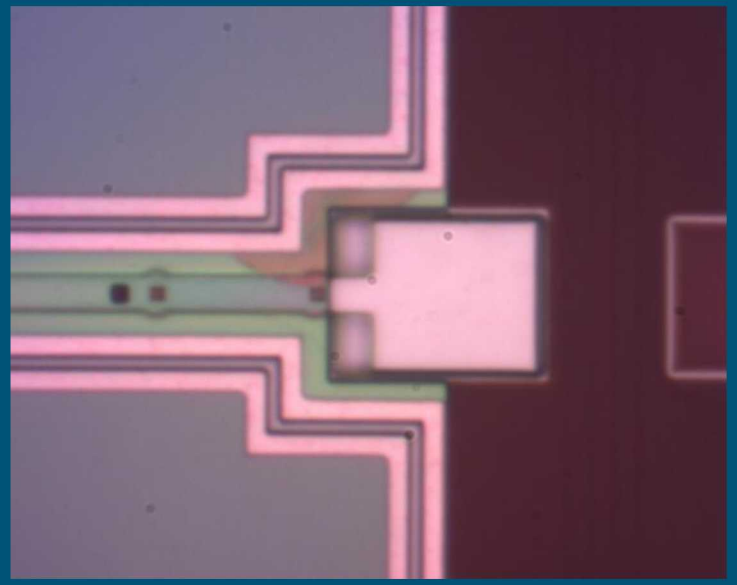




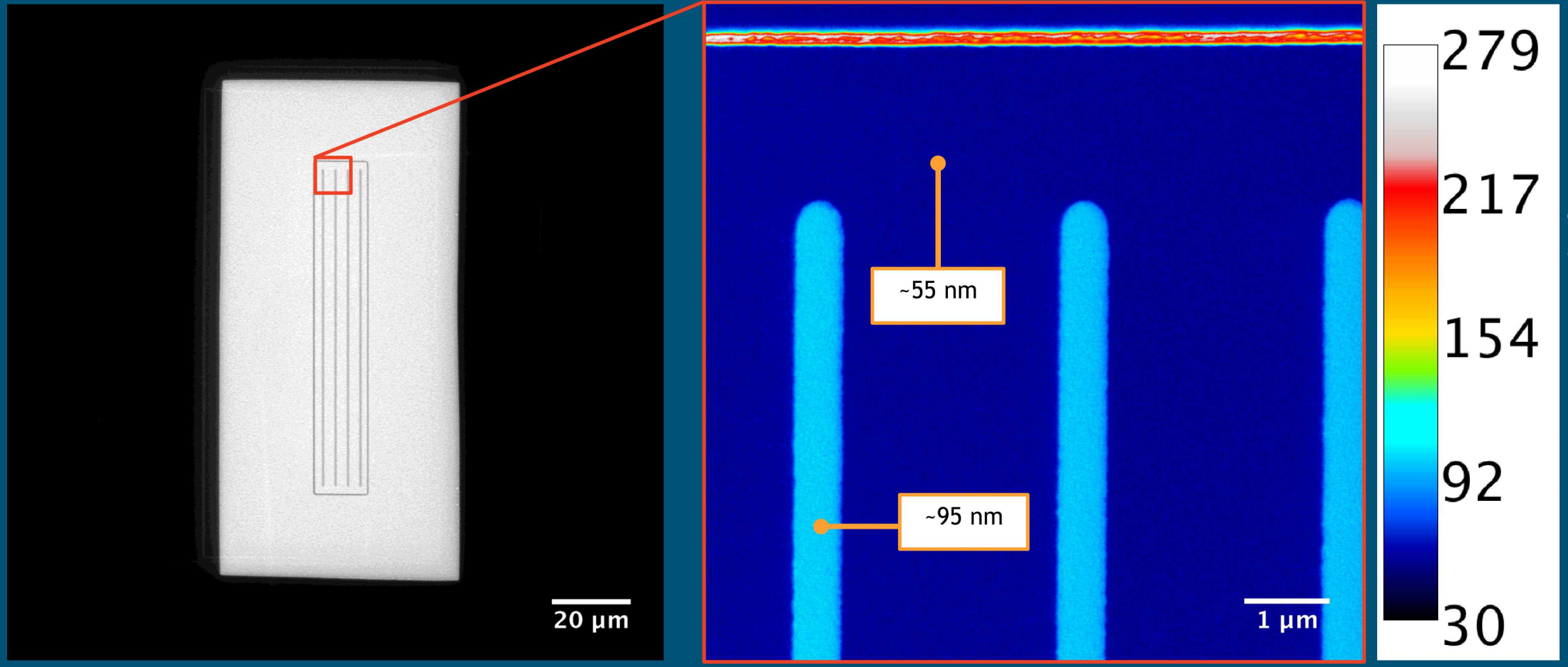
Mechanical-Environmental-Thermal TEM Platform



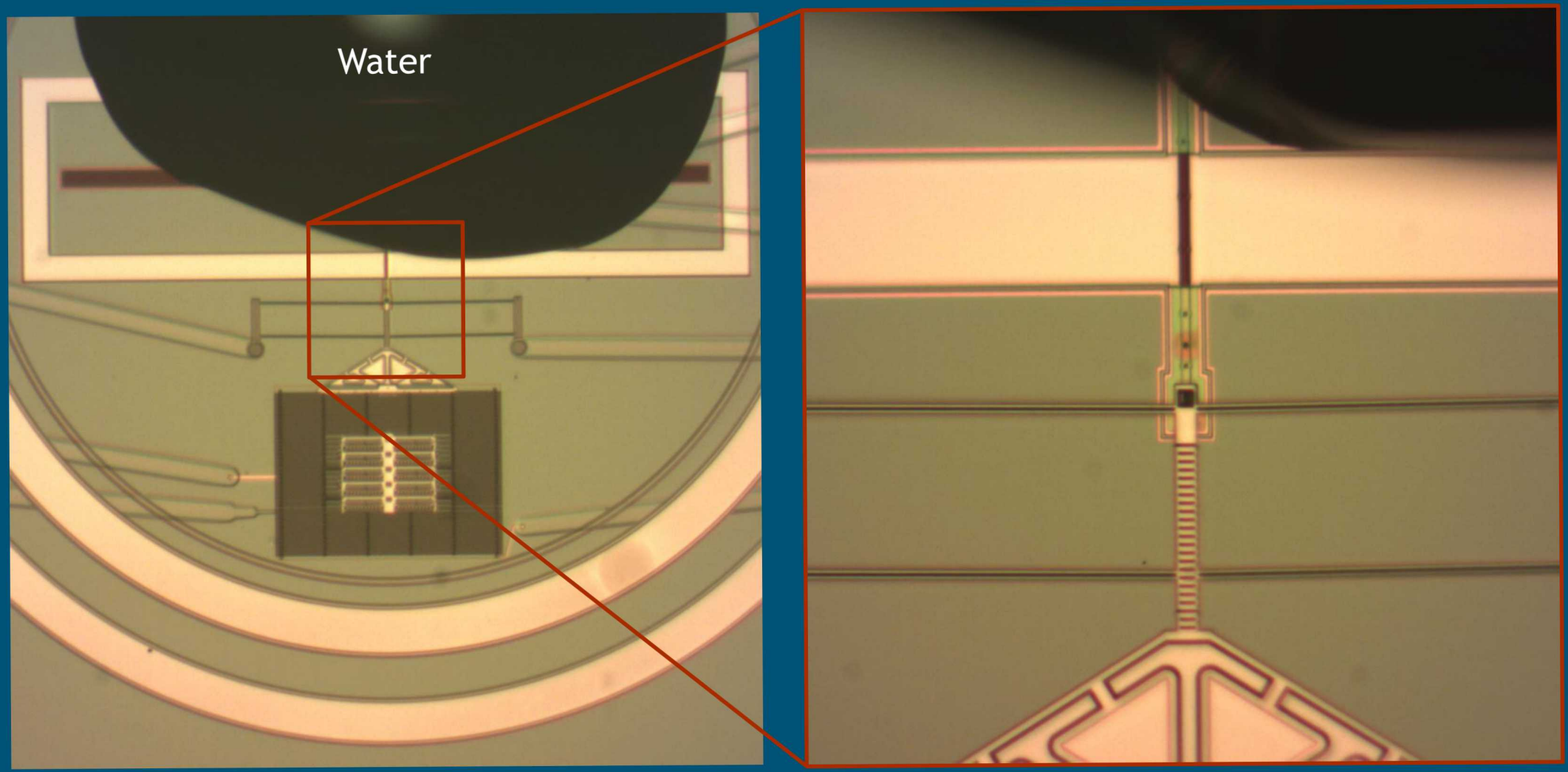




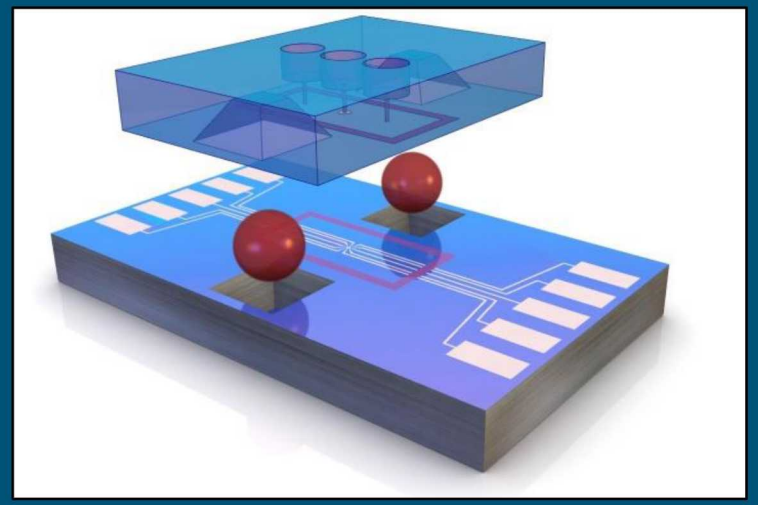
Ribbed SiN Membrane: Prevent Sticktion

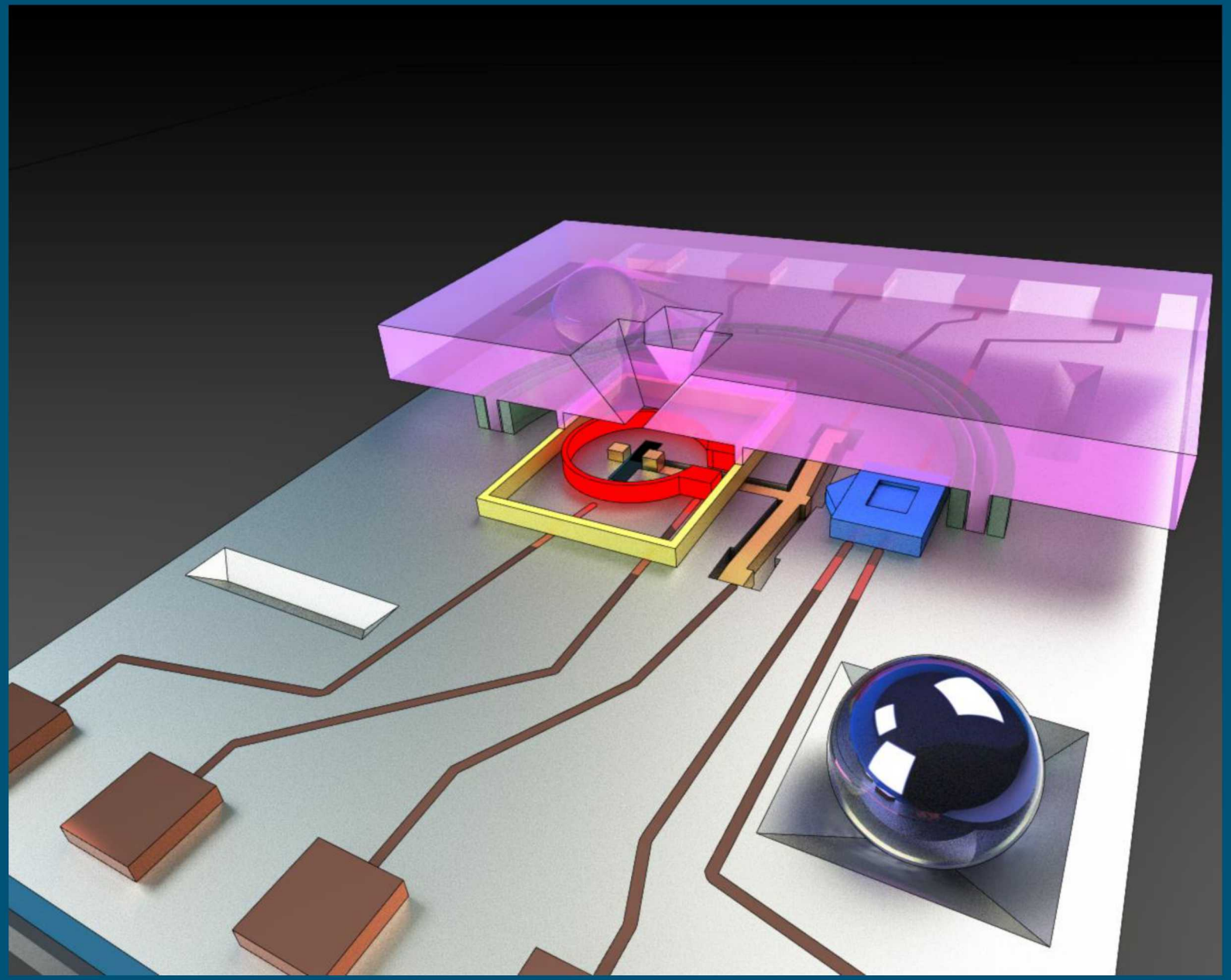


Ribbed SiN Membrane: Prevent Sticktion



CINT's Ion Transport Simulation and Characterization Capabilities





Center for Integrated Nanotechnologies

CINT Core Facility: Albuquerque, NM

CINT Gateway Facility: Los Alamos, NM

Department of Energy, Basic Energy Sciences national user facility to provide expertise and instrumentation free of charge to support accepted peer-reviewed nanoscience research

CINT's Integrated Approaches to Ion Transport

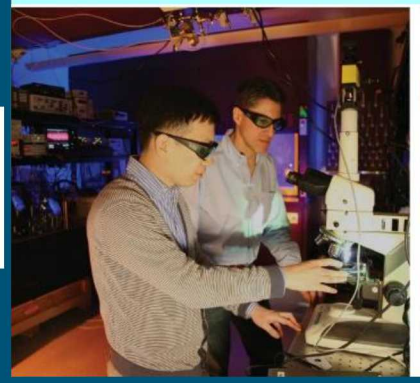
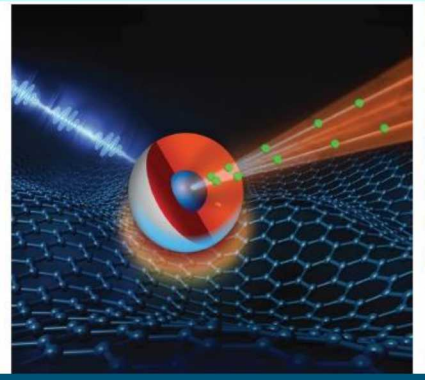
About CINT:

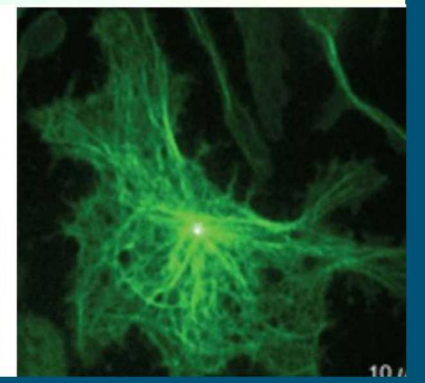
- Free access to staff expertise and equipment (if intent is to publish)
- Two proposal calls per year; proposals for short-term projects are accepted continuously
- Simple 2-page proposal
- Proprietary proposals for industry users

http://cint.lanl.gov
https://cint.sandia.gov
cint@lanl.gov

Research areas:

- **In-Situ Characterization and Nanomechanics –** Developing and implementing world-leading capabilities to study the dynamic response of materials and nanosystems to mechanical, electrical, or other stimuli.
- **Nanophotonics & Optical Nanomaterials –** Synthesis, excitation, and energy transformations of optically active nanomaterials and collective or emergent electromagnetic phenomena (plasmonics, metamaterials, photonic lattices).
- **Soft, Biological & Composite Nanomaterials –** Synthesis, assembly, and characterization of soft, biomolecular, and composite nanomaterials that display emergent functionality.
- **Quantum Materials Systems –** Understanding and controlling quantum effects of nanoscale materials and their integration into systems spanning multiple length scales.



Acknowledgements

This work was performed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525. This paper describes objective technical results and analysis. Any subjective views or opinions that might be expressed in the paper do not necessarily represent the views of the U.S. Department of Energy or the United States Government.