Northwestern University Atomic and Nanoscale Characterization Experimental Center

Northwestern

EXPLORING INNER SPACE

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Front cover, clockwise from top left: Staff with user on the ARM300F; Image of butterfly wing on the FEI Quanta 650 (Image credit: Elizabeth King); NUFAB characterization bay; Users on the Hitachi HD2300; Image of moss on the Hitachi S3400 (Image credit: Elizabeth King)



This page:

Image of beads with proteins on the Hitachi S4800 (Image credit: Elizabeth King)

Welcome to the NUANCE Center



Professor Vinayak P. Dravid Founding Director, NUANCE Center

Founding Director Professor Vinayak Dravid welcomes you to the NUANCE Center!

Northwestern University's Atomic and Nanoscale Characterization Experimental (NUANCE) Center was established in 2001 to integrate complementary characterization, fabrication, and analytical capabilities toward the development of soft, hard and hybrid materials, structures, and devices.

NUANCE provides and continually updates state-ofthe-art nanoscale resources, with 24/7 open access, to all research endeavors at Northwestern, the greater Midwest, and beyond. Our resources include world class instrumentation, expert technical assistance, advanced hands-on training, novel technique development, interdisciplinary research collaboration, inspired education, and engaged outreach.

We look forward to exploring inner space with you!

Education & Outreach:

- Magnifying Minds middle school program
- Research Experience for Teachers (RET): Bringing nanoscience to the classroom
- Research Experience for Undergraduates (REU)
- Community college partnerships
- Women in Microscopy Annual Conference
- "Art of Science" Annual Image Contest
- 10+ academic courses taught in labs

Diversity by Field

Materials Chemistry Life Sciences Education Physics Electronics MEMS/Mechanical Engineering **Biomedical**

By the Numbers

Team

26 Total Technical & Administrative Staff



Capabilities 80+ Instruments, Tools. and Services

Users



200+ Faculty Groups 1,000+ Total Users



Space

24,000+ sq. ft. **Research Space**



Research

\$50+ million in research, education, infrastructure & facilities

Publications 350+ publication acknowledgments each year

Regional Impacts:

- Workshops
- Seminars
- Demonstrations
- Tech Talks
- Tours
- International Conferences
- Open House
- Collaborations

EPIC: TEM

Industries:

Chemistry / Materials / Physics / Engineering / Biomedical / And many more...

Instruments

- JEOL 2100F
- JEOL ARM300F
- JEOLARM200CF

Specimen Holders (In Situ)

Besides conventional room temperature single- and double-tilt holders, we have various in-situ holders present in our facility:

- Protochips Atmosphere Gas System & Holder
- Hummingbird Liquid-Flow Holder & Tomography Holder
- Nanofactory Instruments Biasing Holder
- Gatan Heating Holder
- DENS Solutions Wildfire Holder

Advanced Specimen Preparation

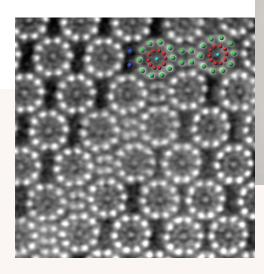
- Fischione 1050 TEM Mill, 1040 NanoMill, Electropolisher
- FEI Helios Nanolab 600 FIB-SEM

Services

- 1. Conventional and high-resolution S/TEM imaging
- 2. Electron transparent sample preparation (both conventional and focused ion beam)
- 3. (Scanning) electron diffraction and structural defect analysis
- **4.** Localized composition and electronic structure analysis enabled by Energy Dispersive X-ray Spectroscopy (EDS) and Energy Loss Spectroscopy (EELS)
- **5.** Microstructural variations under various external stimuli (heat, bias, gas, liquid)

- 1. Simultaneous HAADF/LAADF and BF/ABF imaging
- 2. Simultaneous dual-EELS and EDS analysis
- **3.** Atomic-scale spectrum imaging analysis
- 4. 4D STEM using direct electron detectors
- 5. Various kinds of in-situ TEM applications







Top: Staff with user on the ARM300 Above: Protochips Atmosphere gas holder for in-situ (S)TEM experiments Left and opposite: Deformed array of NWs

Deformed array of NWs (Image credit: Hee Joon Jung)

Testimonial

We are grateful to the NUANCE Center where we have been utilizing the ARM200CF electron microscope for our ongoing research on high entropy alloys and battery materials. The EPIC teams' assistance in training my students how to utilize the S/TEM and its in-situ electron microscopy holders has been a tremendous help for moving our projects forward. We look forward to continuing our collaboration with the NUANCE Center for many years to come.

Professor Reza Shahbazian-Yassar

University of Illinois at Chicago, Department of Mechanical and Industrial Engineering

EPIC: SEM

Industries:

Biological Sciences / Geology / Chemistry / Medicine / Physics & Astronomy / Environmental / Materials Science / Art Conservation / Engineering / And many more...

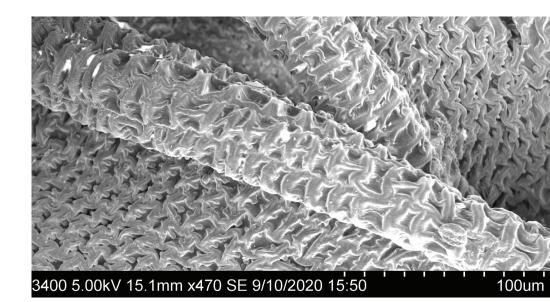
Instruments

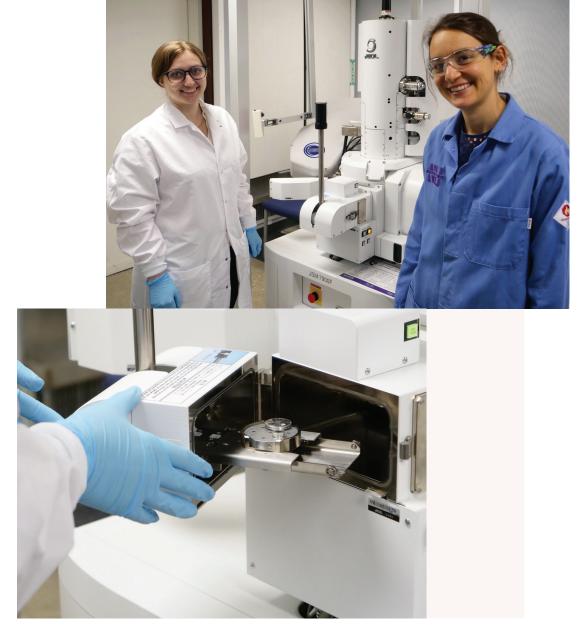
- Hitachi S3400N-II SEM
- Hitachi S4800-II cFEG SEM
- Hitachi SU8030 cFEG SEM
- FEI Quanta 650F ESEM
- JEOL JSM-7900 FLV-SEM

Services

- 1. Ultra-high-resolution Scanning Electron Microscopy (SEM)
- 2. X-ray Microanalysis (EDS, WDS)
- **3.** Electron Backscatter Diffraction (EBSD) and Transmission Kikuchi Diffraction (TKD)
- 4. Environmental SEM (ESEM)
- **5.** Electron Beam Lithography (eBL)
- 6. In-situ heating and cooling stages

- 1. Surface morphology characteristics
- 2. Defect and contaminate analysis
- **3.** Site-specific elemental analysis
- 4. Microstructural and crystallographic characterization
- 5. Fabrication and characterization of nano-devices





Top: Staff with user in front of the JEOL 7900 Above: JEOL 7900 exchange chamber **Opposite:** Image of moss on the Hitachi S3400 (Image credit: Elizabeth King)

Testimonial

Throughout my research, EPIC has played an essential role in collecting the crucial data needed for my master's thesis. During Covid when other facilities were closed, the staff at EPIC were flexible and creative; and with their help, I was able to safely acquire high-quality SEM images of my paleobotanic specimens. The friendly and knowledgeable staff, along with the state-of-the-art instruments available, make EPIC a great place to conduct research.

Maya Bickner

Graduate Research Assistant at the Chicago Botanic Garden, Program in Plant Biology and Conservation, Northwestern University

EPIC: FIB

Industries:

Biological Sciences / Geology / Chemistry / Medicine / Physics & Astronomy / Environmental / Materials Science / Art Conservation / Engineering / And many more...

Instruments

• FEI Helios Nanolab 600 FIB-SEM

Specimen Holders (In Situ)

• see EPIC: TEM

Advanced Specimen Preparation

- Denton Desk-IV Sputter Coater
- Lesker Nano38 Deposition System
- SPI Osmium Coater

Services

- **1.** Two- or three-dimensional (2D/3D) structural characterization from micro- to nanoscale
- 2. Surface morphology and cross-sectional analysis
- 3. Site-specific micro- to nanoscale sample preparation
- 4. 2D/3D defect analysis
- 5. In-situ nanomanipulation and probing

- 1. Site-specific ion beam and electron beam assisted material deposition or etching
- 2. Site-specific nanoscale high precision ion beam milling
- **3.** (In-Situ) Transmission Electron Microscopy (TEM) and Atom Probe Tomography (APT) sample preparation using a combination of deposition and milling techniques
- **4.** Scanning Transmission Electron Microscopy (STEM) detector for STEM-in-SEM analysis
- Focused Ion Beam (FIB) Tomography for three-dimensional (3D) structure visualization





Testimonial

The Helios Nanolab FIB-SEM at NUANCE provides my students with the opportunity to prepare cross-sectional TEM samples, which is essential for many projects in my group at the University of Illinois at Chicago. The services provided by the NUANCE staff enable a faster sample preparation time and offer valuable experiences for both my undergraduate and graduate students.

Professor Robert F. Klie

University of Illinois at Chicago, Department of Physics

Clockwise from top left:

User in front of the Helios Nanolab FIB-SEM, Staff with user on the Helios Nanolab FIB-SEM, Helios Nanolab FIB-SEM

Opposite, left to right:

MEMS-based E-chip (left) containing a FIB-prepared thin section between gold (Au) contacts (right) for performing in situ (S)TEM electrochemistry experiments (Image credits: Paul Smeets)

BioCryo

Industries:

Life Sciences / Materials Science / Biotechnology and Pharmaceuticals / Food and Agriculture / And many more...

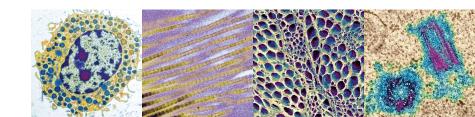
Instruments

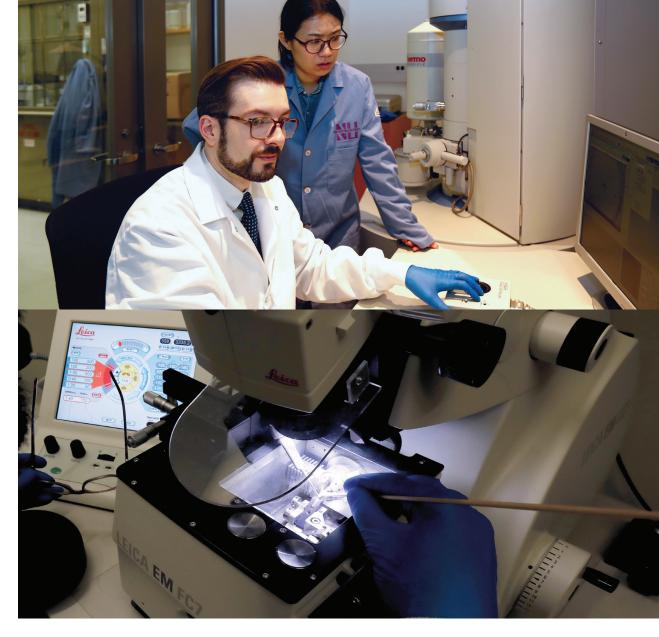
- JEOL 3200FS TEM, 300kV with in-column Energy Filter and K2 Direct Detection camera
- JEOL 1400 TEM, 120kV with Gatan OneView camera
- Hitachi HT-7700 S/TEM, 120kV for biological imaging
- Hitachi HD2300 STEM, Schottky FEG, dedicated STEM with Dual-EDS
- Hitachi S-4800-II, FE cryo-SEM
- FEI Vitrobot IV and III Plunge Freezer
- Leica HPM100 High-Pressure Freezer
- Leica AFS2 Automated Freeze Substitution
- Leica VT1200S Vibrating Blade Microtome
- Leica UC7/FC7 Cryo-Ultramicrotome
- Tousimis Samdri-795 Critical Point Dryer

Services

- 1. Electron Microscopy and Microanalysis
- 2. Cryo S/TEM and cryo SEM
- **3.** Advanced cryogenic and conventional sample prep of biological, soft matter, and hybrid samples
- 4. Training and assistance, consulting
- **5.** Customized experimental design

- Cryo-TEM, cryo-STEM, cryo-SEM, and conventional TEM, STEM-EDS
- **2.** Plunge freezing, high-pressure freezing, and freeze substitution
- 3. (Automated) resin embedment, flat embedment of monolayers
- Cryo-Ultramicrotomy of ultrathin sections for S/TEM and thicker sections for LM and other applications
- **5.** Freeze fracture, cryo etching and coating





Top: Staff with user on the Hitachi HD2300 Above: User on the Cryo-Ultramicrotome Opposite, left to right: Mast cell, collagen, decellularized kidney, and centriole (Image credits: Reiner Bleher and Eric Roth)

Testimonial

It's all about the specimens!

We have relied on the sample preparation expertise of the BioCryo facility at NUANCE for many years to create ultrathin sections for TEM. Their extensive experience in soft matter sample preparation and sectioning transferred beautifully to our catalyst specimens. We have explored these in a broad spectrum of catalyst studies on a wide range of different catalyst types from supported metal systems to zeolites. Ultramicrotomy avoids redeposition problems common in FIB sample preparation and allowed us to achieve excellent nanoscale quantitation.

Matthew Kulzick, INEOS, A Global Chemical Company

Keck-II

Industries:

Art Conservation / Failure Analysis / Semiconductor / Materials Synthesis / Elemental Detection / Corrosion Chemistry Pharmaceutical / Materials Modification / Battery Materials / And many more...

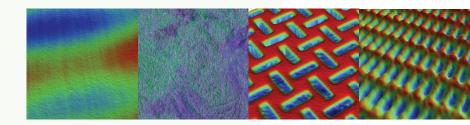
Instruments

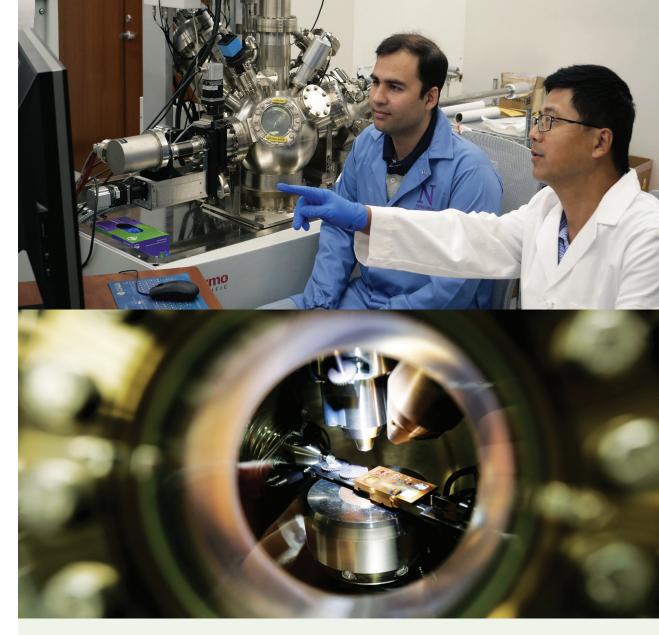
- Thermo Scientific™ Nexsa G2 X-Ray Photoelectron Spectrometer (XPS) System
- Thermo Scientific™ ESCALAB™ Xi+ XPS Microprobe
- IONTOF M6 Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS)
- Bruker ContourGT Optical Profiler
- Bruker Dektak 150 Surface Profiler
- Thermo Nicolet iS50 Fourier Transform Infrared Spectroscopy (FT-IR)
- Bruker LUMOS FT-IR Microscope
- J.A. Woollam M2000U Spectroscopic Ellipsometer
- Malvern Zetasizer Ultra

Services

- 1. Composition analysis on top surface
- 2. Surface contamination analysis
- 3. Chemical state identification at elemental level
- 4. Isotope and impurity analysis
- 5. 3-dimensional surface profiling and mapping

- 1. X-Ray Photoelectron Spectroscopy (XPS)
- 2. Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS)
- 3. Fourier Transform Infrared Spectroscopy (FT-IR)
- 4. High Resolution Surface Profiling
- 5. Dynamic Light Scattering (DLS) Particle Size Characterization





Top: Staff with user on the XPS Above: Keck-XPS 3 Opposite, left to right: Diamond-indent-50x, 50um SIMS image of alloy, AFM standard-4-2, etched SiO2 pattern (Image credits: Xinqi Chen)

Testimonial

Our materials science lab is at UChicago, and we have been using the Keck-II facility since 2013. Dr. Xinqi Chen has been extremely helpful and supportive in pushing the quality of our work. He has co-authored a paper in Science and a paper in Science Advances with us. Our lab has been very fortunate to use the Keck-II facility, and we look forward to more opportunities to do so in the future.

Professor Bozhi Tian

University of Chicago, Department of Chemistry

SPID

Industries:

Biological Sciences / Chemistry / Applied Physics / Materials Sciences / Engineering / Metallurgy / Cultural Materials / Biomaterials / Pharmaceutical / And many more...

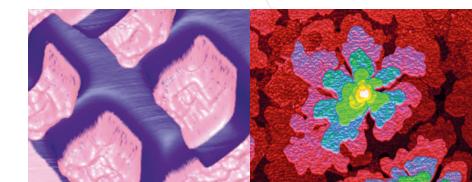
Instruments

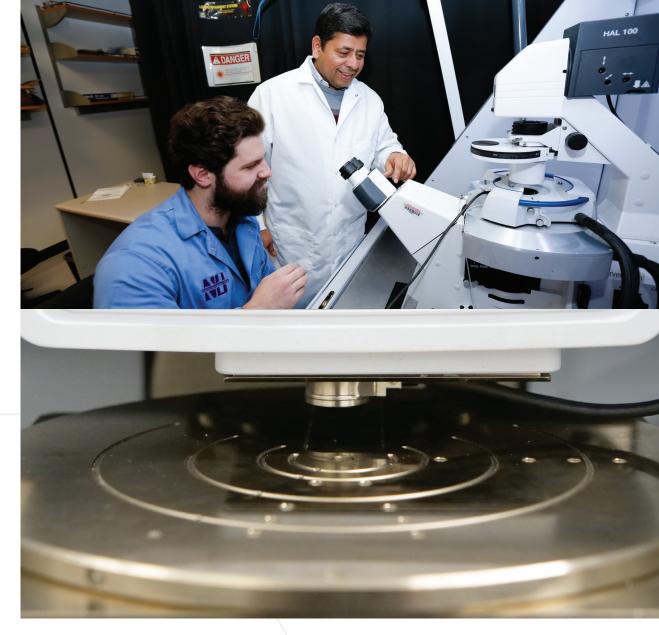
- Bruker Dimension ICON AFM & Fastscan
- Bruker Bioscope Resolve Life Science Imaging System
- Bruker Photoconductive and Functional AFM
- Bruker Triboindenter System with XPM (High speed modulus mapping)
- Horiba Confocal Raman System
- AFM-Raman Integrated System (Co-localized measurements and TERS)
- High Resolution Thermal Imaging System

Services

- 1. High Speed Imaging and nanomechanical analysis
- 2. Advanced Functional AFM Imaging
- **3.** Confocal Raman Mapping, Photoluminescence and TERS
- 4. Micro and nanoscale indentation and high speed mapping
- **5.** Life science imaging system integrated with confocal/ fluorescent microscopy

- 1. High Resolution molecular and atomic imaging
- 2. Quantitative nano-mechanical, chemical, electrical analysis
- 3. Chemical mapping and spectroscopy
- 4. Nanopatterning of soft nanostructures and biological materials
- 5. Absolute temperature and thermal conductivity measurements





Top:

Staff with user on the Bruker Bioscope Resolve Above: Bruker FastScan AFM

Opposite, left to right: 2D dopant profiling on the SRAM using Dark Lift Mode, Pentacene layers (Image credits: SPID)

Testimonial

I worked with Dr. Gajendra Shekhawat of SPID on solid and liquid sample analysis using SPM techniques for our nano-mechanical analysis. I am very happy with the excellent technical service provided by Dr. Shekhawat and his team. A number of direct measurements on our solid and colloidal particles were performed using SPID's advanced tools, which lead to a better understanding of our materials. SPID is really an amazing facility with advanced functional imaging capabilities.

Sajo Naik, PhD, Sr. Research Analytical Scientist at CMC Materials

NUFAB

Industries:

Semiconductor / Microelectronics / MEMs / Biotechnology / Materials / Nanotechnology / Optoelectronics

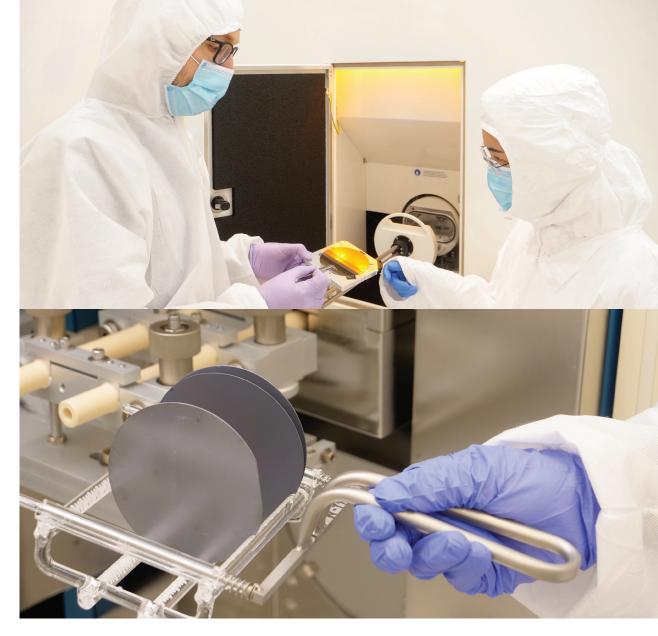
Services

- 1. Consultation
- 2. Training
- 3. Staff-assisted processing
- 4. Full-service processing
- 5. Self-service

- 1. Deposition (PVD, CVD, polymer)
- 2. Etching (RIE, DRIE, XeF2)
- **3.** Lithography (photo, e-beam, direct-write with front and backside alignment)
- **4.** Characterization/testing (electrical, optical, topography, thickness)
- 5. Dicing/packaging (saw, laser, wire bonding)







Top:

Staff loading a sample into the Raith Voyager 100 E-Beam Lithography System

Opposite, left to right: Staff in NUFAB's Deposition Bay, User on iBond 5000 wire bonder

Above:

User loading wafers in Tystar furnace

Testimonial

NUFAB staff were helpful to me as I worked on developing new technologies such as RIE, PECVD, and LPCVD recipes. The facility assisted me with running the tools and sample transferring across Northwestern's departments. As such, I recommend NUFAB because of their prompt and responsible staff.

Aleksandra Gunko, Knowles Corporation

NUFAB cont.

Characterization

- 3D Optical Profiler: Zygo Nexview
- Atomic Force Microscope: Bruker Edge
- Contact Angle Measurement: VCA Optima XE
- Electrical Test Station
- Microscope: Nikon LV150
- Reflectometer: Filmetrics F20
- Spectroscopic Ellipsometer: J.A. Woollam Alpha-SE
- Stylus Profilometer: Veeco Dektak-8
- Hall Effect Measurement System: LakeShore HMS8404

Deposition

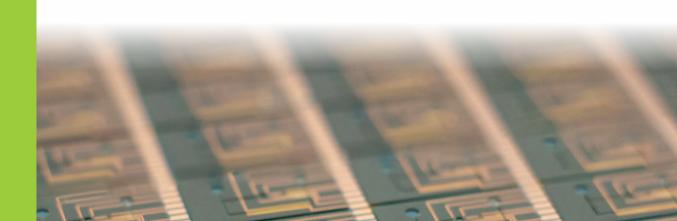
- Atomic Layer Deposition
 Arradiance GEMStar XT-P
 Cambridge NanoTech Savannah S100
- E-Beam Evaporator: AJA & Edwards
- Parylene Coater: SCS Labcoter2 Parylene Deposition System (2 systems)
- Plasma Enhanced Chemical Vapor Deposition (PECVD): STS LpX CVD
- Sputter I: AJA Orion; Sputter II: AJA Orion
- Thermal Evaporator: Denton Vacuum Explorer 14

Dry Etching: Ashing

- Deep Reactive Ion Etcher (DRIE): STS LpX Pegasus
- Plasma Cleaner: Samco PC-300
- Reactive Ion Etcher (RIE): Samco RIE-10NR
- Xenon Difluoride Etcher: Xactix

Furnaces

- Doping/Oxidation–Tystar TYTAN Mini Four Stack Horizontal Tube Furnace (Bank 1: Atmospheric)
- LPCVD–Tystar TYTAN Mini Four Stack Horizontal Tube Furnace (Bank 2: Low Pressure)
- Rapid Thermal Processor: AW-610
- Vacuum Rapid Thermal Processor: Annealsys AS-Micro



Dicing/Packaging

- Laser Cutter: LPKF ProtoLaser R
- Wire Bonder: Ball-iBond5000; Wedge-West Bond 747677E
- Wafer Dicing System: Advanced Dicing Technologies ADT 7122

Photolithography

- Automatic Develop System: Osiris UNIXX S 20 D
- Convection Ovens: Blue M DCC-146-C-ST350
- Develop Bench
- Mask Aligner: Suss MABA6; Suss MJB4
- Maskless Aligner: Heidelberg MLA150, Heidelberg uPG501
- Microscope: Nikon LV150
- Polyimide/SU8 Bench
- Spinner/Bake Bench
- Ultraviolet Flood Exposure System: Inpro Technologies F300S
- Vacuum Oven

E-Beam Lithography

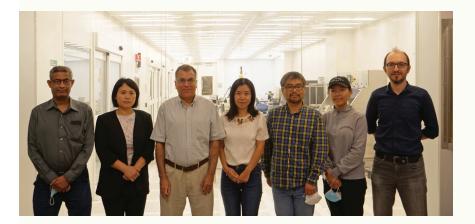
- High-Performance E-Beam Writer: Raith Voyager 100
- Coat/Bake/Develop Station

Wet Processing

- Acid Hoods
- Automatic Dry In/Dry Out Acid Etch System: Osiris CHEMIXX MASK 30 E
- Critical Point Dryer: Tousimis Automegasamdri–915B, Series C
- Ultrasonic & Megasonic Cleaners

Software

• Semiconductor Process & Device Simulation-Silvaco TCAD



Above:

NUFAB team in front of cleanroom, left to right: Anil Dhote, Shaoning Lu, Nasir Basit (director), Ying Jia, Sung Oh Woo, Jiaxin Duan, Serkan Butun

Opposite:

TEM liquid cell sample holders fabricated in NUFAB (Image credit: Serkan Butun)





Connect with Us

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- Founding Director, NUANCE Center
- Abraham Harris Chaired Professor of Materials Science and Engineering, McCormick School of Engineering
- Director of Global Initiatives, International Institute for Nanotechnology

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