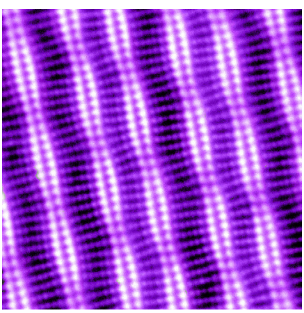
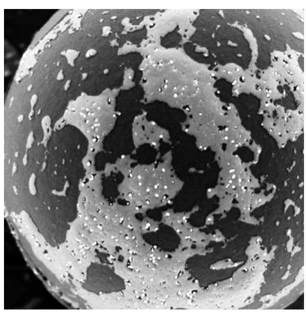
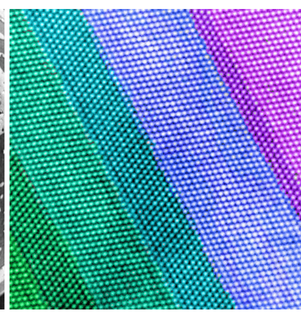
NC STATE UNIVERSITY

# AIF
# analytical instrumentation facility

www.aif.ncsu.edu

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# **ABOUT THE AIF:** THE ANALYTICAL INSTRUMENTATION FACILITY AT NC STATE

Monteith Engineering Research Center on Centennial Campus

The AIF, founded in 1923, is within the College of Engineering and is NC State's primary shared research facility for materials characterization. The mission of AIF is to enable state-of-the-art research through acquisition, development, maintenance, training, and access to major analytical and materials characterization instrumentation. Professional technical staff engage in technique and applications development and are available to provide support to users and clients. Students, faculty, and scientists at NC State and from across the region can be trained to operate instruments independently and interpret and quantify data through individualized training or participation in workshops.

By continuing to enhance our capabilities and engaging faculty and students from across NC State, the AIF seeks to become the preeminent university-based analytical characterization facility in the US.

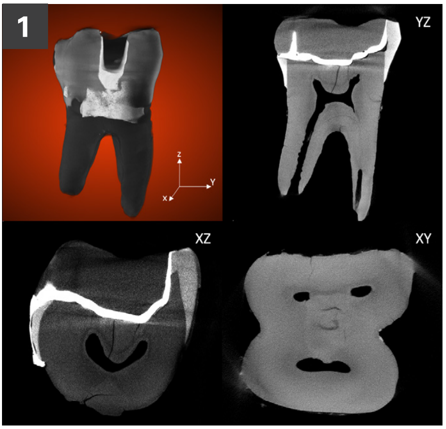
**NC STATE** Engineering

# X-RAY TOMOGRAPHY AND MICROSCOPY

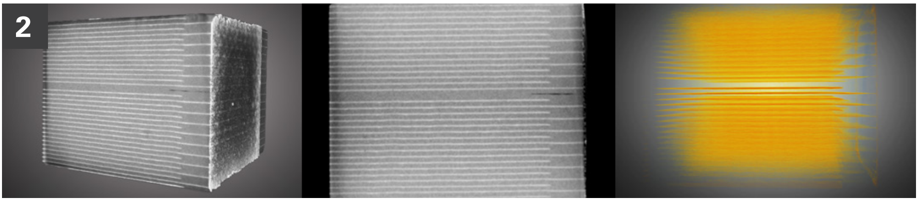
ZEISS Xradia 510 Versa X-ray tomography and microscopy (XRM) is a high-resolution 3D X-ray imaging system for non-destructive analysis. Xradia 510 allows submicron imaging of samples from mm to inches with weight up to 15 kg and sample size up to 300 mm. Specifically, the resolution of the system achieves < 0.7 μm true spatial resolution with minimum achievable voxel size < 70 nm of samples. The high resolution and contrast with flexible working distances provides excellent non-destructive imaging performance.

## INSTRUMENT SPECS

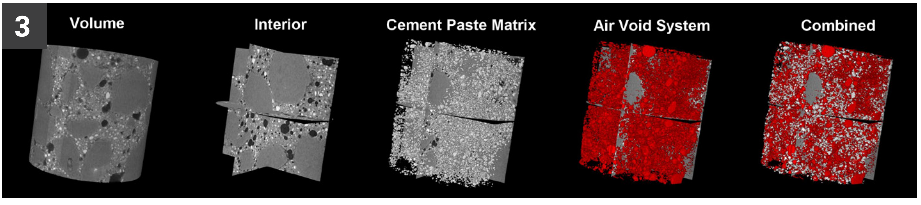
- 30-160 kV, 10 W maximum power and 12 filters for energy selection
- < 0.7 μm true spatial resolution and below 70 nm voxel size
- Dual-stage detector system with 2K x 2K pixel. The detector turret of multiple objectives (0.4X, 4X, 20X) at different magnifications with optimized scintillators for highest contrast
- Flat panel detector allows imaging for large samples up to 300 mm in size




1. Examples of the 3D visualization of human molar tooth on nano-CT datasets. This figure shows a lateral view (top-left image) and cross-section views (rest of the images) of the molar tooth.




2. Detection of cracks in multilayer ceramic capacitors (MLCC) by X-ray imaging. This figure shows an 3D reconstruction image, 2D cross-section view, and electrode layers of a MLCC (left to right).



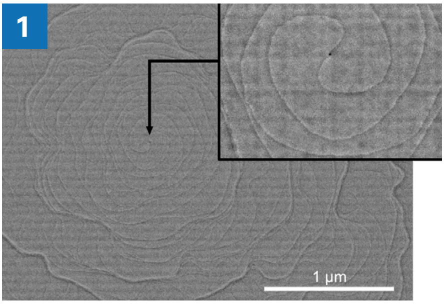

3. X-ray CT images showing 3D lateral view, 2D cross-section view, unhydrated cement, air voids and combined view in a mortar sample. *Courtesy of Laura Dalton, College of Engineering.*

# FIELD-EMISSION SCANNING ELECTRON MICROSCOPY (FESEM)

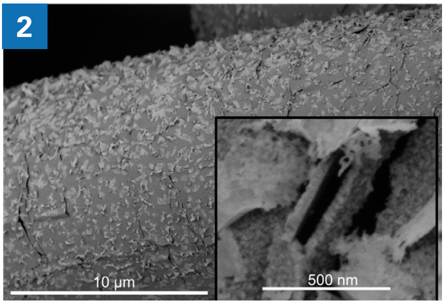
FESEM enables the imaging of samples with high electron brightness and small spot size. The FEI Verios was acquired in January 2014. It is an extreme high-resolution SEM, providing sub-nanometer resolution from 1 to 30 kV and enhanced contrast needed for precise measurements on insulating materials including advanced semiconductor manufacturing and soft materials. The Verios FESEM is equipped with five electron detectors and an energy dispersive X-ray spectroscopy (EDS) detector.

## INSTRUMENT SPECS

- Resolution: 0.6 nm from 2 kV to 30 kV, 0.7 nm at 1 kV, and 1 nm at 500 V. The ability to operate at low voltage while maintaining high resolution enables extreme surface sensitivity and minimizes beam damage on soft samples
- Detectors: Everhart-Thornley secondary electron detector (SED), retractable solid state concentric backscatter electron detector (BSED), in-column solid state BSED, in-column SED, through lens SED, and retractable concentric STEM detector Oxford AztecEnergy EDS detector, 127 eV energy resolution (at Mn Kα line), and Oxford CMOS-based EBSD detector
- Ability to bias the stage from -50V to -4000V to improve imaging on insulating samples without a conductive coating and to allow high resolution imaging at voltages down to 100V
- Five axis, high-resolution piezo drive stage with 100 mm x 100 mm x 20 mm XYZ range, -10° to +60° tilt, and 360° rotation




1. Screw dislocation in homoepitaxial GaN. *Courtesy of Zlatko Sitar, MSE.*




2. Polyamide-6 fibers with ZnO coating and Ag nanoflakes deposited via atomic layer deposition. *Courtesy of Jesse Jur, TECS.*

# ABERRATION CORRECTED S/TEM

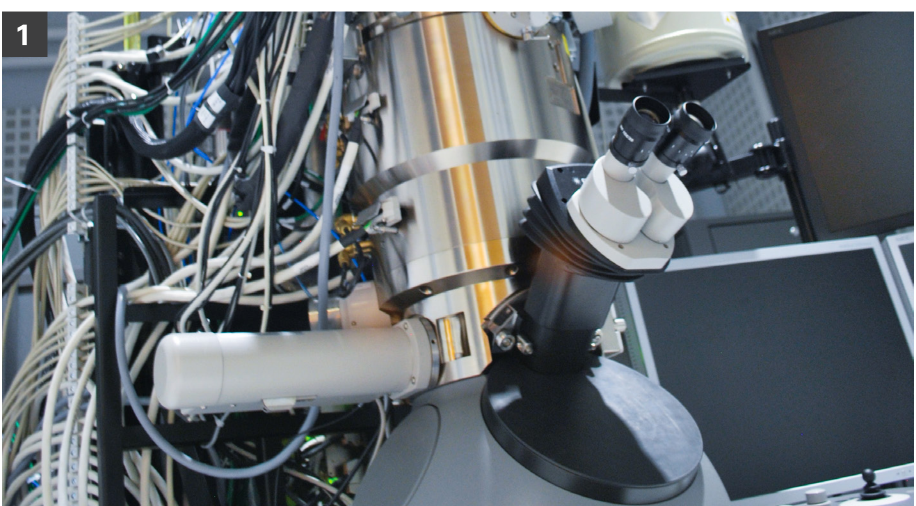
The FEI Titan is an aberration-corrected Scanning Transmission Electron Microscope (S/TEM) with state-of-the-art spectroscopy capabilities. The combination of features on this microscope enable atomic resolution imaging and chemical analysis.

## INSTRUMENT SPECS

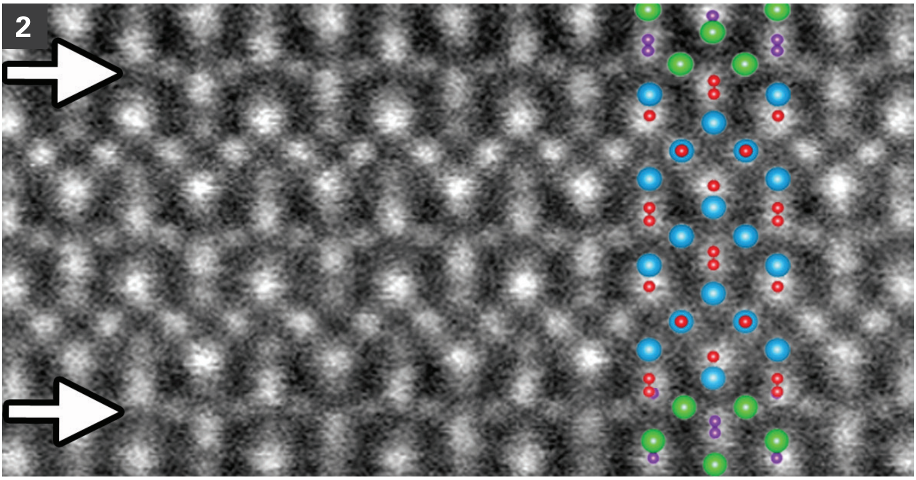
- Accelerating voltages: 80, 200, and 300 kV
- TEM resolution: point 0.20 nm, information limit 0.10 nm
- STEM resolution: 0.07 nm (sub-Angstrom)
- Energy resolution: 0.8 eV (non-monochromated), 0.15 eV (monochromated)
- Super X EDS system: 4 silicon-drift detectors (SDD)
- 3-condensor lens system: parallel beam in Angstrom size
- Electron energy loss spectroscopy, Gatan Enfinium with DualEELS
- Holders: single-tilt and double-tilt analytical holder, tomography, in-situ heating and biasing holder, and in-situ atmosphere holder

## APPLICATIONS

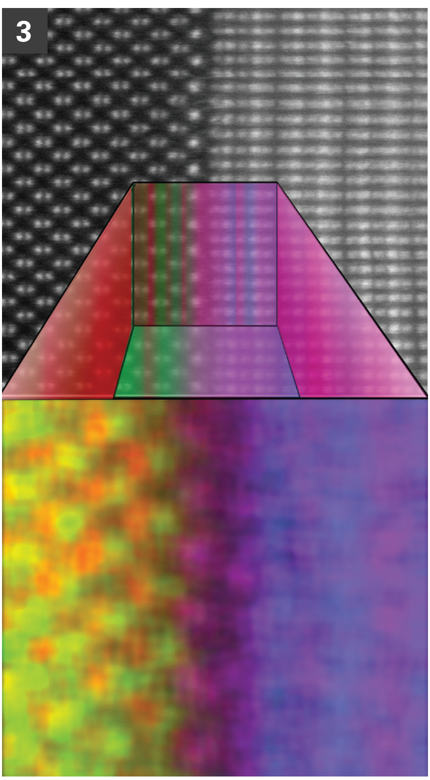
- Sub-Å resolution STEM Imaging
- Atomic resolution TEM imaging
- Sub-eV resolution EELS
- Atomic resolution EELS/EDS
- In situ heating/biasing and gas experiments



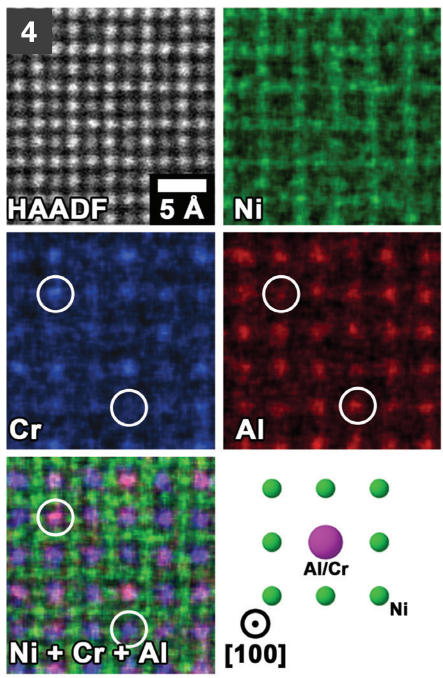

1. FEI Titan G2 60-300 Aberration Corrected S/TEM.




2. Planar defect observed in a complex $Ti_6Sn_5$ intermetallic compound.




3. Interface between GaAs (left, a semiconductor) and $Bi_2Te_3$ (right, a thermoelectric) explore with atomic resolution HAADF STEM and EDS imaging.




4. Distribution of Cr alloy elements imaged directly with atomic resolution EDS.

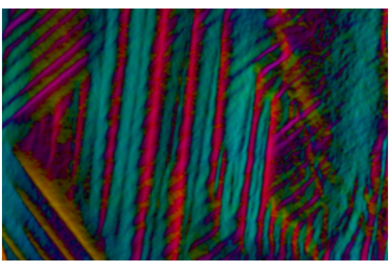
# FIELD EMISSION S/TEM

The Talos F200X G2 is a field emission analytical Scanning Transmission Electron Microscope (S/TEM). It is designed for fast, precise, and quantitative characterization of functional materials, and it is ideal for in-situ and operando experiments.

## INSTRUMENT SPECS

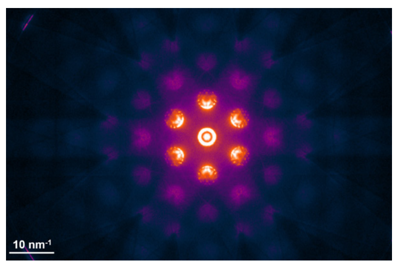
- Accelerating voltage: 80 kV to 200 kV
- TEM information limit 0.12 nm, STEM HAADF resolution 0.16 nm
- Holders: Single-tilt and high-visibility and low-background double-tilt holder, multi-specimen holder, Gatan 70 degree field of view cryo-transfer holder, double-tilt LN2 cooled holder, Tomography, Protochips Aduro double tilt heating holder, Poseidon electrochemistry liquid holder and in situ atmosphere gas holder
- SuperX EDS system: 4 silicon-drift detectors (SSD)
- Segmented STEM detectors including bright field, HAADF and two annular dark field detectors, Gatan energy filtered TEM, and Ceta 16M camera
- Gatan 655 dry pumping station for Cryo holder and Vitrobot sample preparation for Cryo-electron microscopy

**1**



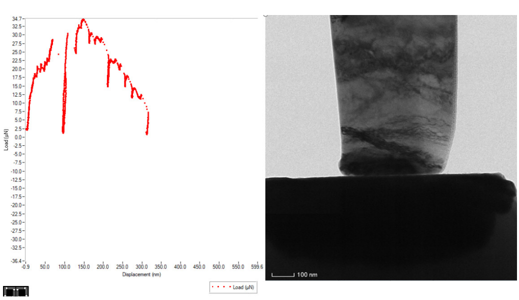
1. A differential phase contrast image reveals arrays of ferroelectric domains in Barium Titanate (BaTiO3). *Courtesy of Dr. Elizabeth Dickey group, MSE.*

**2**



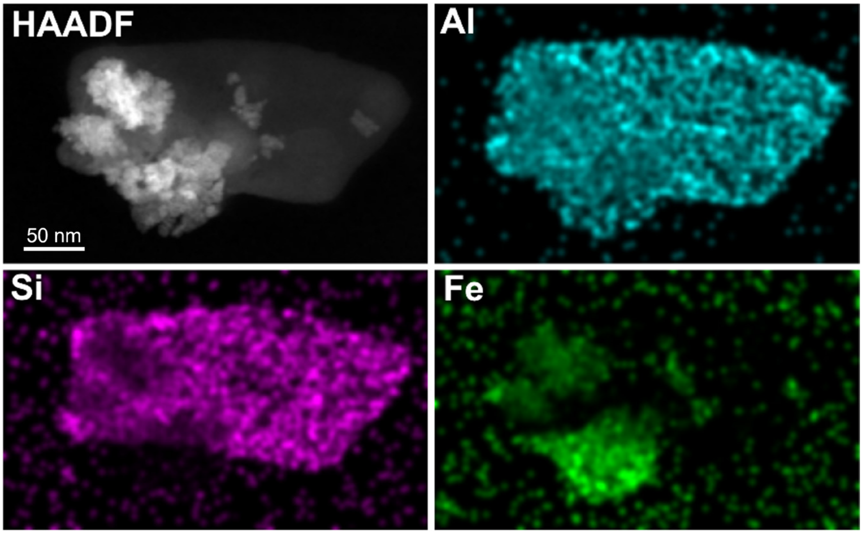

2. A false color convergent beam electron diffraction pattern of single crystal silicon. *Courtesy of Alex Hsain, Young Huwan Lee and Adam Rigby, MSE.*

**3**




3. A martensitic steel pillar is crushed inside the Talos, and the load vs. displacement is mapped in real time. *Courtesy of Djamel Kaoumi, Nuclear Engineering.*

4

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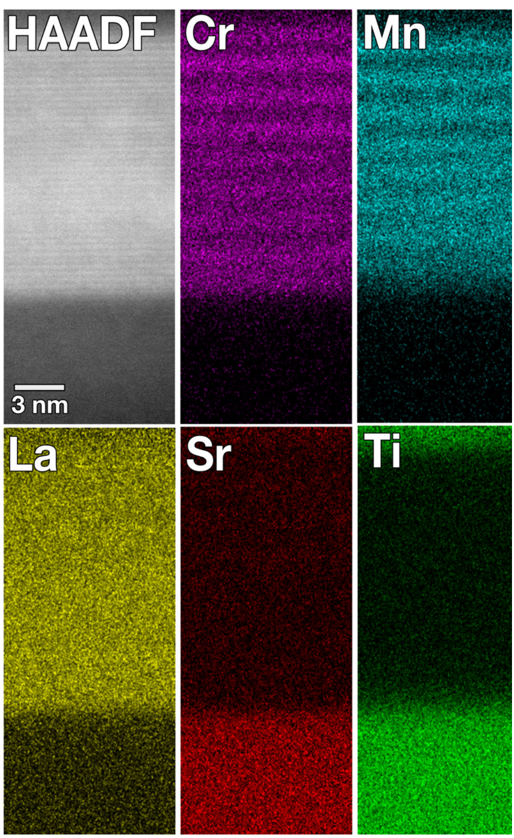



4. STEM-EDS mapping identifies the location of Iron Oxides forming on Aluminosilicates in soil samples. *Courtesy of Dean Hesterberg group, Soil Science.*

5.STEM-EDS mapping of LSCO and LSMO thin films with alternating layering. *Courtesy of Dr. Divine Kumah's group, Department of Physics.*

# THREE STATE-OF-THE-ART X-RAY DIFFRACTOMETERS

X-ray Diffraction (XRD) enables the determination of microstructural and crystallographic information about materials using diffracted X-rays. Some representative information that can be determined from XRD includes phase identification, atomic structure including lattice parameters, preferred orientation, and crystallite size and strain effects.

## PANALYTICAL EMPYREAN

- 4 different non-ambient stages
- Temperature ranges between -193 °C and 2300 °C
- Atmosphere including air, inert gas, vacuum (<$10^{-4}$ Torr), and reactive gas; 1 mbar to 10 bar operating pressure
- Application of electric fields in situ up to ±10 kV for polycrystalline and thin film samples
- Spinning stage for Rietveld-quality powder diffraction measurements in reflection and transmission geometries
- PIXEL1D detectors allows rapid measurements
- Grazing incidence for thin films

## BRUKER

- Bruker D-5000 with HighStar area detector
- Four-circle Eulerian cradle can accommodate polycrystalline and single crystalline samples
- Collimators down to 50μm in diameter
- HighStar detector features excellent signal to noise ratio capabilities that enable one to detect extremely fine grains, extremely thin films, and extremely weak scatters.
- HighStar detector allows for efficient measurement of texture and characterization of coarse-grained materials.

## RIGAKU SMARTLAB

- Operating modes include Bragg-Brentano, grazing incidence, rocking curve, phi scan, and X-ray reflectivity
- Ge (220)x2 monochromator allows high resolution XRD measurements
- Cross-Beam optics allow rapid switching between parallel beam and Bragg-Brentano modes
- An Eulerian cradle with $\phi$ and $\chi$ rotation for texture, reflectivity, and residual stress measurements

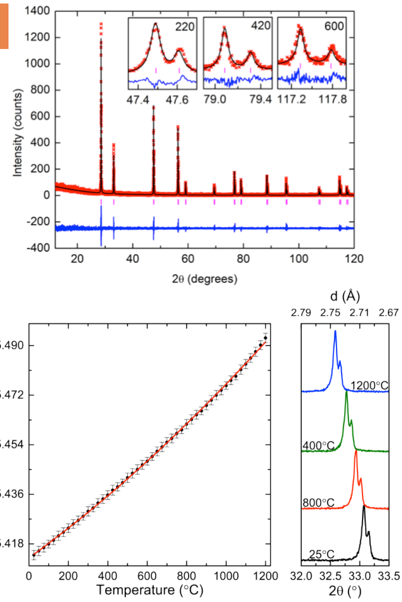
## ANALYTICAL SUITE

- HighScore plus analysis suite with Rietveld capability
- ICDD database for pattern matching and phase identification

1

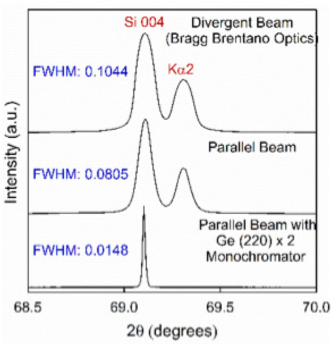

1. PANalytical Empyrean X-ray Diffractometer.

2



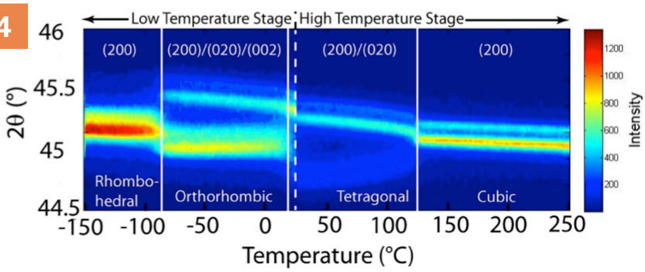

2. (Top) Room temperature diffraction pattern of $CeO_2$ and profile fitting using the Rietveld method. (Bottom) High temperature diffraction patterns and analysis of $CeO_2$ demonstrates nonlinear thermal expansion up to 1200°C.

3




3. A Ge (220)x2 monochromator provides a highly conditioned beam (low divergence and $k\alpha_2$ can be removed) to the sample.

4



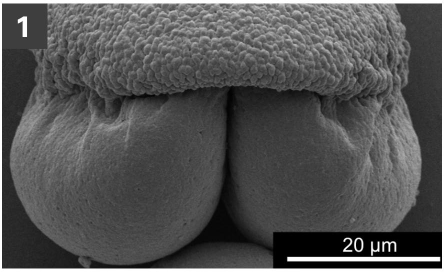

4. 4 non-ambient stages enable measurement and characterization of phase transitions. In this example, the (200) reflection of cubic $BaTiO_3$ splits into multiple reflections in the lower temperature phases.

# VARIABLE PRESSURE SCANNING ELECTRON MICROSCOPY (VPSEM)

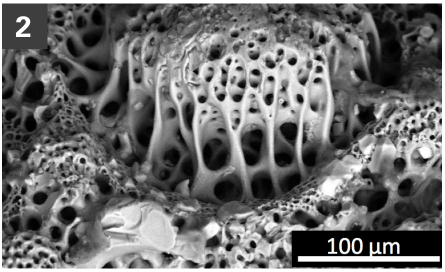
The Hitachi S-3200N VPSEM is capable of imaging from conductors and insulators without coating with energy dispersive X-ray spectroscopy (EDS).

## INSTRUMENT SPECS

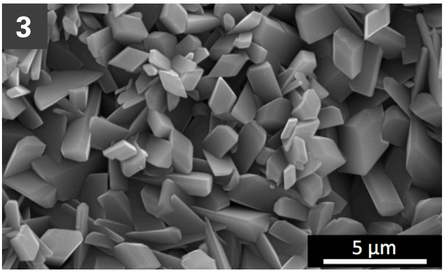
- Resolution: 3.5 nm at 30 kV in SE mode
- Detectors: Everhart-Thornley secondary electron detector, Robinson backscatter detector, and specimen current meter
- Oxford/AAT EDS detector, 133 eV energy resolution (at Mn K$\alpha$ line)
- Digital scan generator with user-specified pixel resolution in both X and Y up to 16384 x 16384 pixels
- Five axis stage with 50 mm x 40 mm x 30 mm XYZ range, -10° to +90° tilt, and 360° rotation
- In VPSEM mode, the chamber can be back filled with nitrogen, helium, or any other non-corrosive, non-reactive gas. This allows for imaging insulating samples with no conductive coating.



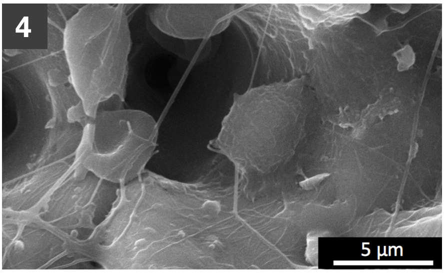

1. Pine pollen. *Courtesy of Chuck Mooney, AIF.*




2. Surface of a sand dollar from the North Carolina coast. *Courtesy of Jonathan Pierce, MSE.*




3. Surface of a commercial steel coated with manganese phosphate. *Courtesy of Chuck Mooney, AIF.*



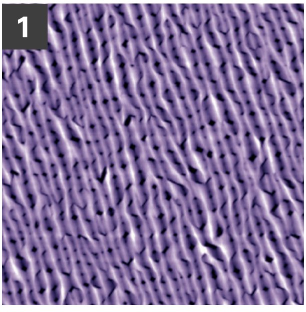

4. Experimental microfluidic device containing red blood cells, activated platelets, and protein filaments. *Courtesy of Roger Narayan, BME.*

# ATOMIC FORCE MICROSCOPY (AFM)

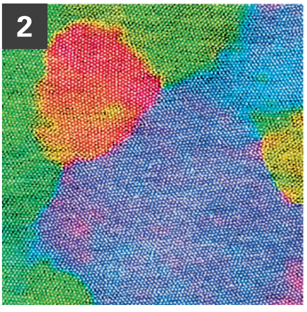
Atomic force microscopy involves using a cantilever with a nanometer-scale tip to scan the specimen surface for local structure and properties. The Asylum MFP-3D classic AFM uses a single closed-loop scanner to span the range from atomic resolution scans up to much larger micron-scale scans. This instrument is capable of imaging surface topography, and other surface interactions for applications in materials science, life science, polymers, nanolithography, and nanoindenting.

## INSTRUMENT SPECS

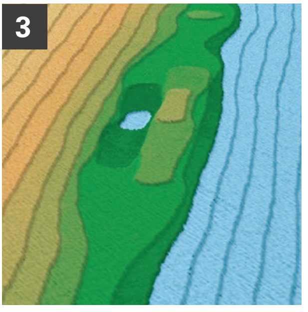
- XY resolution: 90 μm scan range with <0.5 nm sensor noise
- Z resolution: Sensored Z scanner with >15 μm range with <0.25 nm sensor noise. Low-coherence 860 nm SLD with <0.02 nm DC deflection noise
- Modes of operation: Contact/LFM, AC mode/tapping mode with phase and Q-control, electric force microscopy, surface potential microscopy, magnetic force microscopy, piezoresponse force microscopy, Dual AC and Dual AC resonance tracking (DART), loss tangent imaging, force spectroscopy and force mapping, nanolithography, nanomanipulation
- Accessories: closed fluid cell, dual gain conductive AFM cantilever holder kit, AM-FM viscoelastic mapping mode cantilever holder kit




1. Strain-induced corkscrew pattern on MBE grown AlGaAs, 12 μm scan. *Image courtesy of S. MacLaren, UIUC.*




2. Block copolymer self-organized into a close-packed lattice of spherical microdomains, 16 μm scan. *Image courtesy of Asylum research.*




3. Sapphire crystal following annealing at 1400°C leaving a clean surface with atomic steps (~3Å tall) and occasional defects, 12 μm scan. *Image courtesy of S. MacLaren, UIUC.*

# AIF CORPORATE AFFILIATES PROGRAM

The Analytical Instrumentation Facility (AIF) is the leading university materials characterization facility in the Southeastern United States. We enable access and training on state-of-the-art, high-valued instrumentation for students and researchers from industry, academia, government, and nonprofits. AIF reaches widely across the university. Every day, we are training students from disciplines as diverse as materials, animal, plant, and soil science to chemical and mechanical engineering. The tools and capabilities of the AIF bring these researchers together in a dynamic, interdisciplinary environment that seeks to discover, innovate and translate technologies into society. The AIF partners with other networks like the National Nanotechnology Coordinated Infrastructure (NNCI) and the Research Triangle Nanotechnology Network (RTNN) to run novel programs like Kickstarter, which seeks to rapidly enable initial results for new startups, small companies, and researchers from diverse disciplines and backgrounds. We also welcome countless K-12 student groups and tours of our laboratories and workshops to inspire the next generation of scientists and engineers. These unrestricted cash donations will allow us to continue corporate engagement, improve instrumentation for research and education, and enhance our interactions with the public.

**$2,500 annually (July-June) – Silver Level**

- Company name and logo listed on the AIF website and in the AIF building hallway
- Company listed as corporate sponsor on all AIF-wide emails
- Access to AIF staff
- Rapid response to service requests
- Invitation to attend the annual Carolina Science Symposium, sponsored by AIF and RTNN, and present a company poster
- Invitation to attend AIF-wide seminars and events
- Introductory company product literature made available in AIF office spaces
- Access to non-exclusive visitor office space in AIF office suites (in Monteith Research Center, Centennial Campus), upon pre-approved request and availability

## Program Levels

**$5,000 annually – Gold Level**

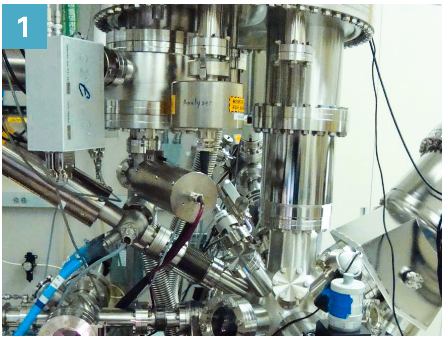
- All of the benefits of Silver, plus:
- Invitation to present at the beginning of one AIF-wide seminar or event
- Company featured with short description in an email/newsletter sometime during the year
- Invitations to the Student-Company mixer at the Carolina Science Symposium
- Limited number of complimentary seats for company employees in AIF workshops
- Invitations to attend technique-specific "AIF user group meetings"
- Ability to post job openings on AIF electronic job boards

# X-RAY PHOTOELECTRON SPECTROSCOPY (XPS)

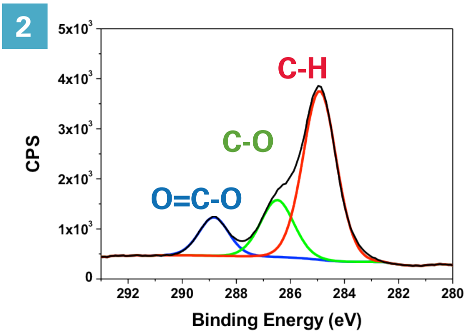
XPS is a surface-sensitive (< 10 nm) spectroscopy technique that measures the elemental chemistry and electronic states of materials. The AIF XPS has five excitation sources that include a monochromator and a high performance ultraviolet source. The 150 mm hemispherical design analyzer permits selection of a detected area with multiple apertures. A spin detector provides a unique capability. A number of accessories, such as sputter gun and charge neutralization, make this a versatile instrument.

## INSTRUMENT SPECS

- Five sources: Mg/Al X-Ray, Al/Ag monochromator X-Ray, Ultraviolet
- Unique detectors: Array of 6 channeltrons, spin detector
- Ar sputter gun to remove surface contamination, electron flood gun for charge neutralization
- Sample stage has X, Y, Z, and tilt capability
- Sample temperature range from -140°C to 600°C
- Base pressure in the analyzer chamber in low $10^{-10}$ mbar range
- Up to six samples can be loaded at same time




1. SPECS XPS/UPS/Spin Resolved Photoelectron System (FlexMod).




2. Binding energy difference between different Carbon bonds is readily measured in polyethylene terephthalate. *Courtesy of Jesse Jur, Textiles.*

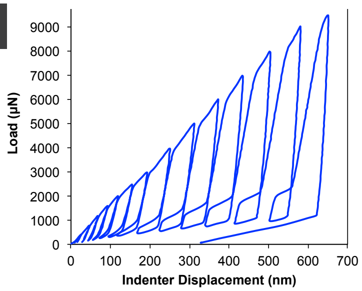
# NANOINDENTATION

The Bruker Hysitron TI980 Triboindenter is a quasistatic indentation system for nanomechanical testing of mechanical properties, including Young's modulus, hardness, fracture toughness, creep, and stress relaxation. It is ideal for measuring mechanical properties of coatings and films, as well as the spatial dependence of hardness and elastic modulus. It's three-plate capacitive transducer design allows for a high displacement sensitivity and a low thermal drift. It is also capable of tribological testing including nanoscratch testing and wear testing.

## INSTRUMENT SPECS

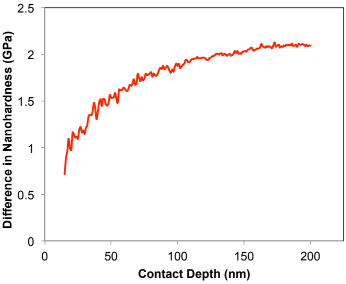
- Three-plate capacitive transducer design provides:
  - high displacement sensitivity (0.0064 nm resolution, 0.1 nm noise floor)
  - high load sensitivity (1 nN load resolution, 20 nN noise floor)
  - low thermal drift (< 0.05 nm / sec)
- In situ scanning probe microscopy (SPM) for imaging sample surfaces before and after indentation, scratch, or wear testing
- Berckovich, NorthStar cube corner, cono-spherical, and fluid cell indentation tips available to users

1



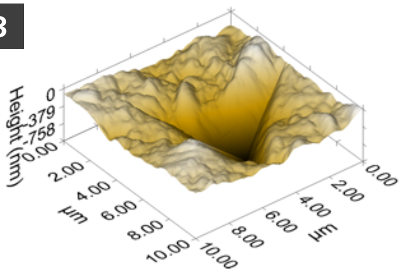

1. Load vs. displacement curve from progressive indentation into $He_2$+/- irradiated ferritic steel.

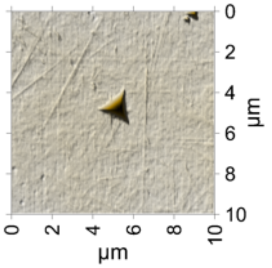
2




2. Plot of the difference in hardness between irradiated and non-irradiated sample as a function of indentation depth, the shape of which is associated with the He penetration depth. *Courtesy of Lulu Li & Prof. Ronald Scattergood, MSE.*

3



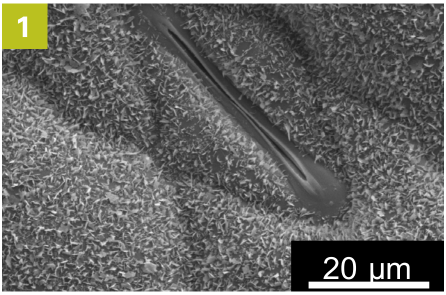

3. SPM images of indentations into the surfaces of bovine dentin (top) and fused quartz (bottom).

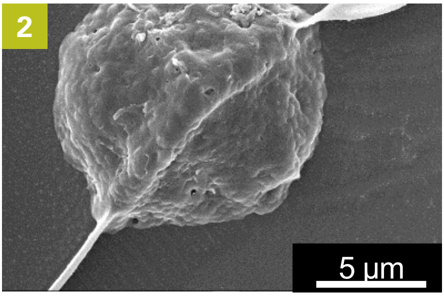
# CRYOGENIC SCANNING ELECTRON MICROSCOPY
(CRYO-SEM)

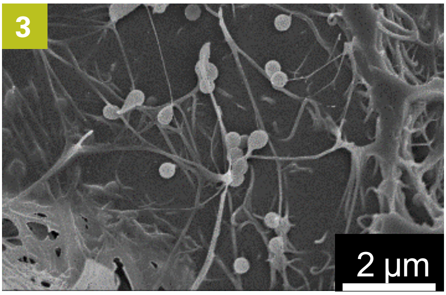
The JEOL JSM-7600F Scanning Electron Microscope outfitted with a cryogenic transfer system and stage can be used to evaluate hydrated samples without the need for drying, making it an ideal imaging tool for volatile or beam-sensitive samples in the areas of life, food, biology and polymer sciences. The Horiba H-CLUE, acquired in July 2017, offers a complete Cathodoluminescence (CL) solution to analyze CL signals in keeping optimum performance over a wide spectral range from 200 nm – 2200 nm.

## INSTRUMENT SPECS

- Gatan Alto Cryo-transfer system, including a variable temperature cold stage, anti-contaminator, in-situ cold knife fracture device, and cold magnetron sputter coater
- Horiba H-CLUE CL detector for CL imaging and a wide spectral range from 200 nm - 2200 nm. Motorized X and Y positioning under vacuum
- Sample biasing to decrease sample damage and enhance surface detail
- High resolution imaging using a field emission (FE) electron gun (1.0 nm at 15 kV and 2.3 nm at 1 kV in SEM mode and 1.5 nm at 1 kV with biasing)
- Low-angle backscattered and transmitted electron detectors



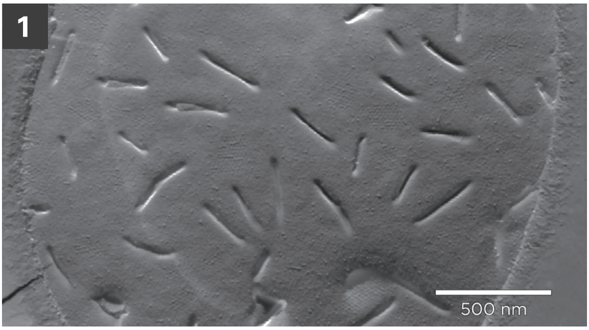




CryoSEM images of plunge frozen:

1. Maze leaf surface showing the stomata
2. Mammalian cell surface
3. Fibrin clot hydrogel network polymerized in the presence of pNIPAm microgels

# BIOLOGICAL AND SOFT MATERIAL SAMPLE PREPARATION

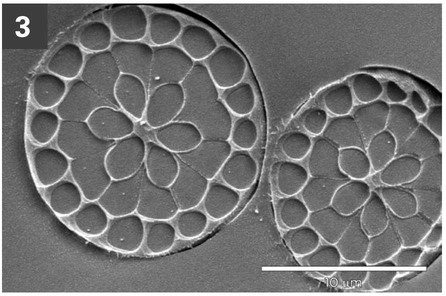
**Freeze Fracture:** Freeze fracturing is useful for observing internal cell structures, particularly membrane proteins since fracturing of the sample frequently occurs between lipid bilayers of the cell membrane. Freeze fracture can also be used on small samples of various soft materials such as polymer solutions and gels, wood pulp, oil-water-surfactant emulsions, lyotropic and thermotropic liquid crystals, colloidal dispersions, detergents, and even ice cream! The Leica ACE900 Freeze Fracture combines all of these functions in a single semi-automated instrument. The delivery of carbon/metal mix coatings by e-beam combined with a rotating cryo stage and flexible shadowing options provides about 1.5 nm resolution imaging capability for any TEM and SEM analysis.




1. Bright field STEM image of yeast replica prepared with freeze fracture.

**Cryomicrotoming:** The Leica UC7 Cryo Ultramicrotome can be used to prepare thin sections of 90-500nm from soft materials for TEM, SEM, AFM, OM or SIMS analysis. The UC7 also has an attached cryo unit that allows for sectioning at low temperatures.  AIF has Epon embedding media and a temperature controlled oven for repeatable and consistent curing of the epoxy.


2. Microtomed sections floating in boat prepared by cryomicrotoming.




3. SEM image of Island in the Sea fibers prepared by cryomicrotoming.

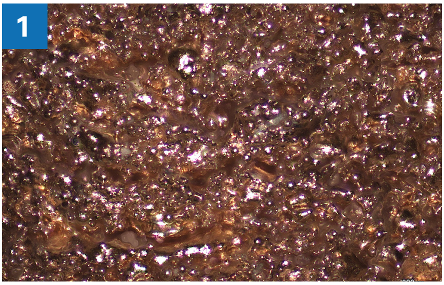
# CONFOCAL LASER SCANNING MICROSCOPE

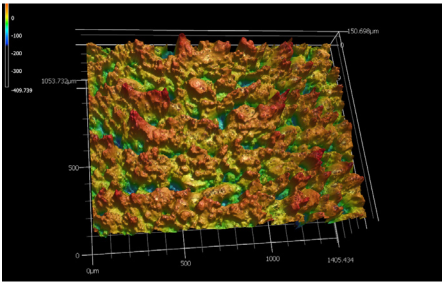
Confocal Laser Scanning Microscope (CLSM) combines optical microscopy with laser profilometry, making it possible to obtain high resolution optical images and subsequently measure profile and surface roughness. It can be used for almost any type of material and is especially suited to measure surface features and roughness of samples which traditional stylus profilometry would not be feasible.

## INSTRUMENT SPECS

- **Source:** 404 nm violet laser source and white light source
- **Detector:** High-sensitivity 16-Bit photomultiplier and high-definition color CMOS
- **Resolution:** 5 nm lateral solution and 0.5 nm Z-axis movement of objective lenses
- **Stage:** Motorized XY stage with 100 mm x 100 mm area; Micrometric motorized Z translation; Automated image stitching
- **Optical microscope:** Pinhole confocal optical system with 5 objectives and USB camera

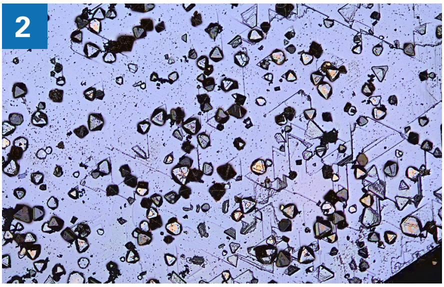
1

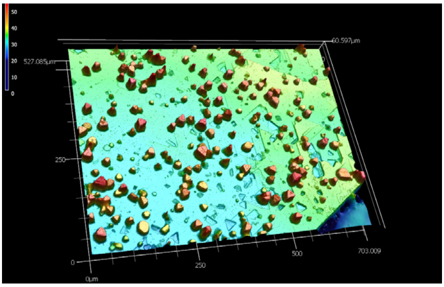





1. Optical image (left) and surface roughness (right) of 3-dimensional printed copper. *Courtesy of Tim Horn and Chris Ledford, CAMAL.*

2





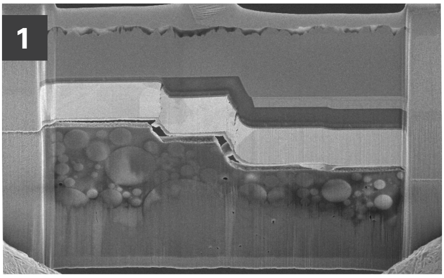

2. Laser plus optical image (left) and surface roughness (right) of Cs2AgBiBr6 perovskite materials *Courtesy of Dr. Ge Yang's group, Nuclear Engineering.*

# **FOCUSED ION BEAM** (FIB) **WITH FIELD-EMISSION SCANNING ELECTRON MICROSCOPY** (FESEM)

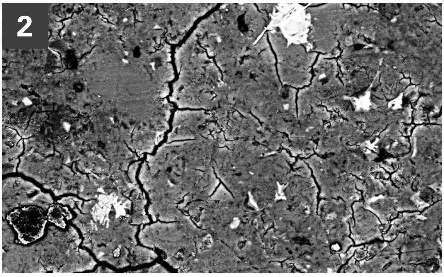
FIB uses a focused beam of ions to enable site-specific deposition, ablation, and analysis of materials. Coupled with an FESEM in the same instrument, the tool is a superior solution for fast preparation of large samples over a wide range of materials and is used for 3D characterization and nanoanalysis, TEM sample preparation, and structural modification of sample surfaces at the nanometer scale. The FEI Quanta 3D Dual Beam FIB-FESEM is ideal for high-resolution imaging and nanofabrication.

## INSTRUMENT SPECS

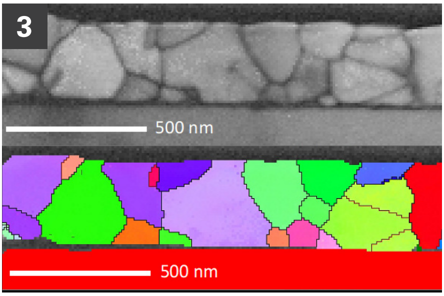
- SEM Resolution: 1 nm at 30 kV in high vacuum
- FIB Resolution: 7 nm at 30 kV
- Stage: Eucentric tilt -20° to +70°, 360° rotation, x = 80 mm, y = 40 mm, z = 26 mm
- Oxford EDS and Electron Backscatter Diffraction (EBSD) detectors, retractable directional back scattered (DBS) detector
- Transmitted detector capable of bright field, dark field, and HAADF images
- Protochips Aduro in situ heating and electrical biasing stage
- Omniprobe micromanipulator for in situ mechanical manipulation of nanofabricated samples
- Gas injection system (GIS) for in situ deposition of Pt metal




1. Cut out of a light emitting diode showing different layers.




2. Cement viewed with Directional Back-Scattered (DBS) detector.




3. TKD band contrast map (top, related to crystalline quality) and crystallographic orientation map (bottom) of a cross-sectioned thin film of BaTiO3 deposited on sapphire. The orientation map is color-coded according to the inverse pole figure color map displayed in the bottom right corner; grain boundaries are identified by the solid lines. *Courtesy of Matt Burch, Jon-Paul Maria and Beth Dickey, MSE.*

# HARD MATERIAL SAMPLE PREPARATION LABORATORIES

AIF offers a comprehensive and evolving suite of sample preparation facilities and techniques for subsequent imaging and characterization. These facilities include established sample preparation techniques such as mechanical polishing and ion milling. New approaches are also used to enhance the capabilities of certain analyses. For TEM specimen preparation, multiple ion milling instruments are available as well as an electropolisher. The Allied High Tech instrument offers very precise mechanical polishing.

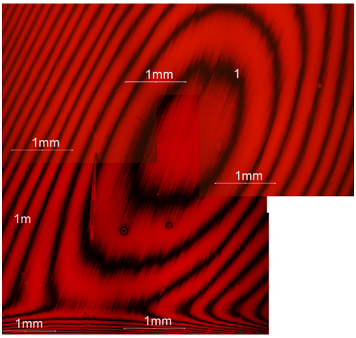
## SAMPLE PREP EQUIPMENT

- Allied High Tech Multiprep
- Low speed diamond saw
- Stuers Tenupol 2 Electropolisher
- Fischione 1050 Ion Mill
- Fischione 1060 SEM Ion Mill

## SAMPLE PREP TECHNIQUES

- Metallography
- TEM Wedge Polishing
- Backside Polishing for SIMS Analysis. With this approach, the sample is thinned by polishing the sample from the back until about 200nm of the substrate remains. The analysis, usually SIMS, is then conducted on the thinned sample. AIF has been a leader in this area (e.g., see reference below) and provides thinned samples as a service and a training course for the technique.

  "Back side SIMS analysis", F. A. Stevie, R. Garcia, C. Richardson and C. Zhou, *Surface and Interface Analysis*, vol. 46, page 241 (2014).



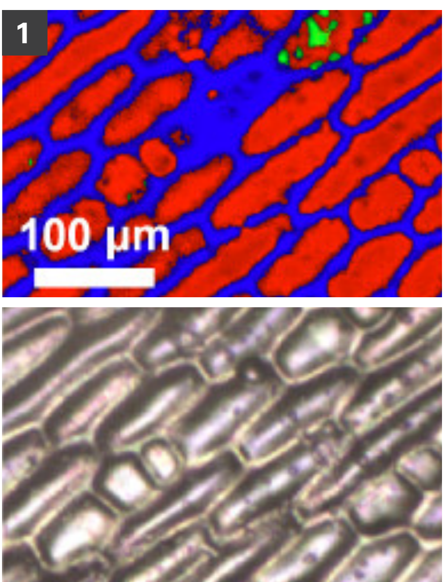

A silicon sample that was thinned using backside polishing is illuminated with red light. The circular region in the center of the image will be used for analysis and is greater than 1 mm in diameter (large enough for multiple SIMS analyses).

# CONFOCAL RAMAN MICROSCOPE

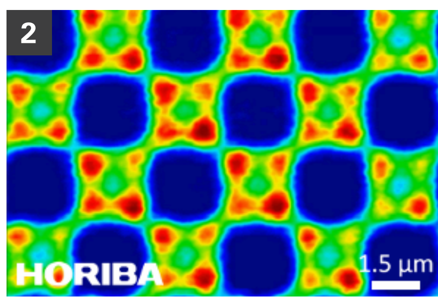
Confocal Raman Microscope combines Raman spectrum and optical microscope, providing sample identification and chemical imaging on a microscopic scale. The interaction of laser light with a sample results in a detailed chemical fingerprint. The Horiba XploRA PLUS offers fast confocal imaging, and automated laser wavelength switching. It is an ideal technique for non-destructive, non-contact, water/aqueous phase sampling in pharmaceuticals, semiconductor, geology, polymers, and forensic applications.

## INSTRUMENT SPECS

- Research grade optical microscope – 2 position motorized white light illuminator, 2 objectives and USB camera
- Integrated imaging spectrometer with 4 gratings: 600, 1200, 1800, 2400 gr/mm
- Horiba Scientific CCD detector: 1024x256 pixels
- Motorized computer controlled 6 position NC filter wheel, confocal pinhole, entrance slit and coupling optics, laser, and filter selection
- Excitation: 532 nm laser kit and 785 nm laser kit
- Motorized XY stage and micrometric motorized Z translation
- 0.5 µm resolution spatial resolution in XY direction




1. SWIFT™ ultra-fast Raman imaging of onion cells, illustrating general cell structure (red/blue) and isolated zones of carotenoid species.



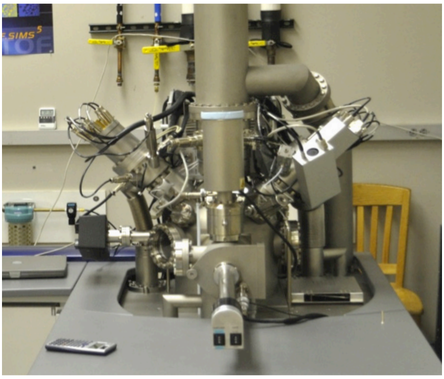

2. Raman image acquisition on a structured semiconductor device, with 40,200 spectra acquired in less than 50 seconds. *Courtesy of Horiba Scientific.*

# TIME-OF-FLIGHT SECONDARY ION MASS SPECTROMETRY (TOF SIMS)

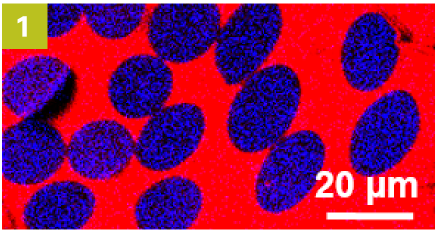
ToF SIMS is a highly sensitive surface analytical technique using a pulsed and focused ion beam and time-of-flight analyzer to produce positive and negative ion mass spectra and mass spectral images from the outer 1 to 2 nm of materials, which provides detailed elemental and molecular analysis of surfaces, thin layers, and interfaces with sub-micron lateral resolution.

## INSTRUMENT SPECS

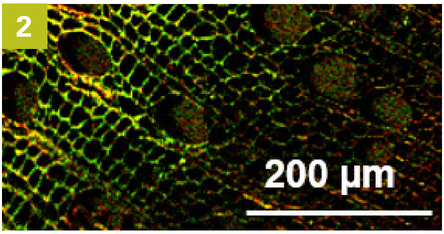
- Mass spectrum of detailed information about the molecular structure of surfaces
- Mass spectral image of different species to yield surface reactivity maps
- Depth profile of elements and/or molecular ions as a function of depth
- Mass range: 1-10,000 amu
- Mass resolution: >10000
- Spatial resolution: 300 nm



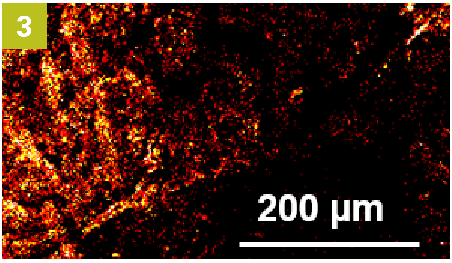
ION TOF: TOF SIMS5

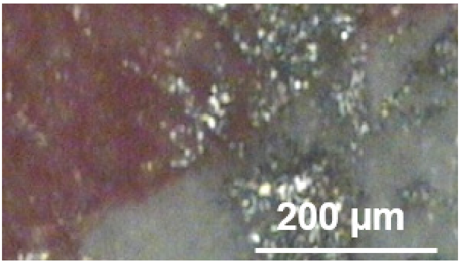



1. Dye identification on fiber cross section.




2. Distribution of Syringyl (in green) and Guaiacyl (in red) in a wood cell wall.




3. TOF SIMS Image of ion specific to ink.

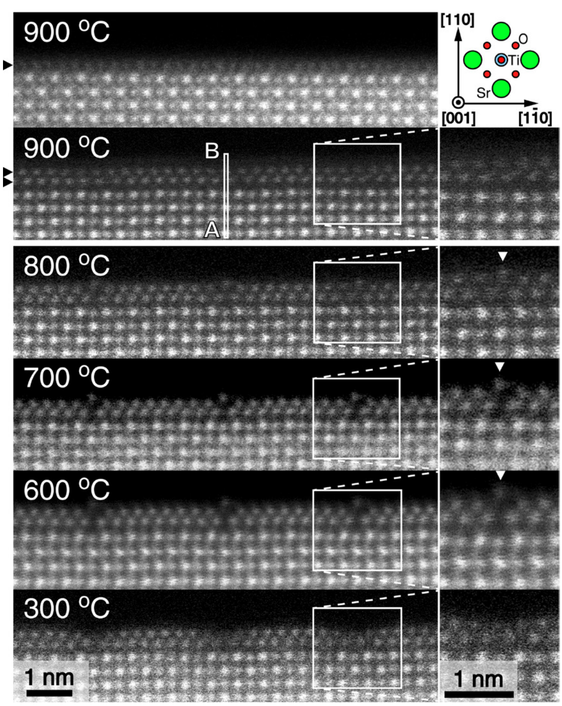
# IN SITU ELECTRON MICROSCOPY AT AIF

## LIQUID ENVIRONMENTS INSIDE TEM

- Protochips Poseidon holder: flow, mixing, or static operation modes
- Space between two SiN window membranes: 50 nm, 150 nm, 500 nm and 5 µm
- Catalysts size, distribution and aggregation in liquid environments
- Growth of nanostructures (nanoparticles, nanowires, etc.) in liquid
- Chemical reactions in liquid with adjustable flow rates
- Surface functionalization on nanostructures
- Direct observations of protein, emulsion, gels, oils, creams, inks, paints, etc.

## GAS ENVIRONMENTS INSIDE TEM

- Protochips Atmosphere TEM Environmental Gas Cell
- Samples heated up to 1000˚C
- Pressure control: 1 Torr to 1 atm (760 Torr)
- Automated control of three gas sources, for example: inert, oxidizing and reducing gases
- Space between two SiN window membranes: 5 µm
- Structural and chemical evolution of catalysts in different environments
- Reaction of materials with gases under elevated temperatures




Left, HAADF-STEM images of reconstructed $SrTiO_3$ (110) surfaces at the indicated temperatures decreasing from 900°C to 300°C. Atoms protrude from the surface, as highlighted by arrows in the corresponding inset left. These protruding atoms are lost from the surface at 300°C.

Latex spheres with DNA-tethered
Au nanorods, imaged using the FEI
Verios extreme high-resolution SEM
*(courtesy of Thom LaBean, MSE)*


2 μm

AIF is a member of the Research Triangle Nanotechnology Network (RTNN) and the National Nanotechnology Coordinated Infrastructure (NNCI).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