nano.purdue.edu

CHARACTERIZATION

MEASURE IT AT BIRCK

PURDUE UNIVERSITY

Pushing the boundaries of *POSSIBLE*

Research at the Birck Nanotechnology Center leads to fundamental science discoveries and development of new technologies that can revolutionize industries. Our center is home to a talented team of researchers, engineers and scientists who are committed to pushing the boundaries of what's possible in nanotechnology.

We welcome users from academia and industry to our leading-edge characterization facility. In-depth training by research scientists is available for users who wish to use the equipment; our team of experts also offers measurement and analysis services.

All laboratories are designed for low acoustic noise, less than 1 milligauss EMI, and ± 1 °C temperature stability. Additionally, the first-floor laboratories achieve NIST A vibration rating. The TEM laboratory has yet tighter temperature controls, specialized airflow patterns, and special acoustic materials on the walls and floors.

Front cover: Hakan Salihoglu, Research Scientist in near-field optics lab at Birck (Purdue University/ Charles Jischke)

ELECTRON MICROSCOPY

RESEARCHER IN CHARGE

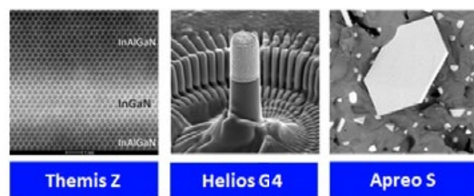
Dr. Christopher Gilpin
Director, Purdue Electron Microscopy Center
gilpin@purdue.edu
(765) 494-7750

As part of the 10 electron microscopes in the campus-wide Purdue Electron Microscopy Center, Birck houses scanning and transmission electron microscopes and focused ion beam (FIB), including these instruments:

- *Themis Z Double Aberration Corrected S/TEM*
- *Helios G4 UX Dual-Beam SEM/FIB*
- *Apreo Field emission gun-SEM (in cleanroom)*

The EM Center staff of 6 also provide these services:

- One-on-one sessions and user support through assisted use
- In depth training to allow for self-use
- Imaging coupled to chemical analysis using EDX and EELS (SEM and TEM)
- Ion beam patterning and milling
- Slice-and-View for bulk 3D reconstruction
- Auto-TEM for unattended lamellar sample preparation for TEM analysis
- Tomography and high-resolution 3D reconstruction from images and chemical composition
- Integrated differential phase contrast for imaging of low Z atoms in a matrix of high Z material



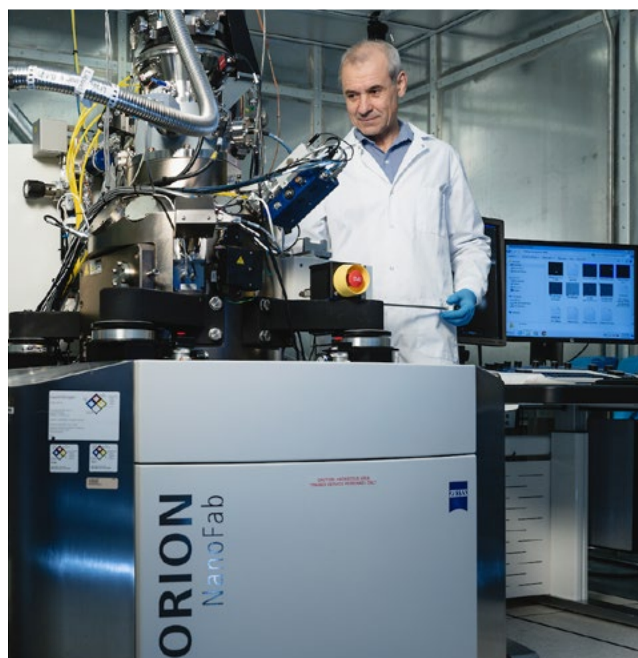
	Themis Z	Helios G4	Apreo S
Type	TEM/STEM	SEM/FIB	SEM
Resolution	0.65 Å @ 300kV 1.8 Å @ 80kV	0.6nm @ 30kV	0.8nm @ 30kV 1nm @ 1kV
Detectors	EDX, EELS, ADF, BF, HAADF, 4-Quadrant DF	EDX, STEM TLD (SE/BSE) ETD (SE), ICE SI/SE	IC T1 (BSE) IC T2 (SE) ETD, IC T3 (SE/BSE)
Accelerating Voltage	60 – 300kV	e- : 350V – 30kV Ion: 500V – 30kV	0.8 – 30kV

HELIUM ION MICROSCOPY/ NANOFABRICATION

RESEARCHER IN CHARGE

Dr. Dmitry Zemlyanov
dimazemlyanov@purdue.edu
(765) 496-2457

Zeiss ORION NanoFab is a revolutionary helium ion microscope (HIM) that takes nanoscale imaging and nanofabrication to new heights. This tool offers innovative capabilities for imaging and nanofabrication using high-brightness, highly focused ion beams (He^+ or Ne^+) generated from a single atomic source.



SCANNING PROBE MICROSCOPY AND PROFILOMETRY

ENGINEERING MANAGER

Ron Reger
rreger@purdue.edu
(765) 494-6667

Atomic Force Microscopy (AFM):

A variety of scanning probe microscopes are available. Samples can be imaged in air or liquid environments. Measurement modalities include: Contact mode and tapping mode; Force displacement; Contact resonance and bimodal (AM-FM); Magnetic force microscopy (MFM); Electrostatic force microscopy (EFM); Piezoelectric force microscopy (PFM); Kelvin probe force microscopy (KPFM).

Available AFMs include *Park NX20, Asylum Cypher S AFM, Asylum MFP-3D-Bio AFM*, and *Bruker Catalyst.*

For vacuum AFM/STM, see **Surface analysis** section.

Cleanroom Profilometry:

Several stylus-based profilometers from *KLA-Tencor (P7, P17, Alpha-Step IQ)* for studying up to 8" wafers, also a *Bruker GT-K* optical profilometer.

Park NX20 AFM (credit: parksystems.com)

SURFACE ANALYSIS

RESEARCHER IN CHARGE

Dr. Dmitry Zemlyanov
dimazemlyanov@purdue.edu
(765) 496-2457

X-ray Photoelectron Spectroscopy (XPS) is used to examine the chemical composition of a solid surface.

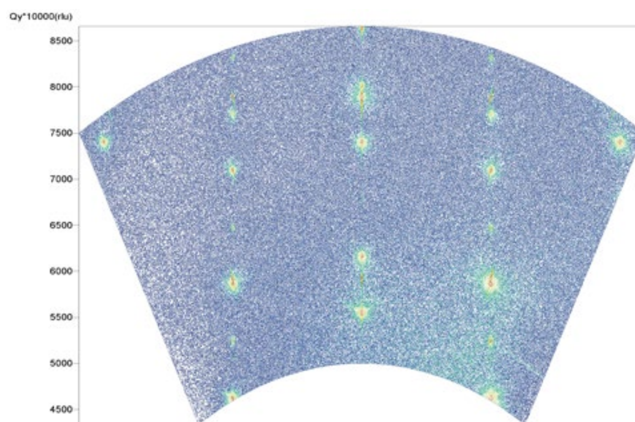
Kratos Axis Ultra DLD Imaging XPS is equipped with a monochromatic X-ray source and a charge neutralizer for studying non-conducting samples, features real-time imaging XPS for x-y chemical mapping, a reaction cell (CatCell) that allows sample treatment under various gas mixtures at pressures up to 6 bar and temperatures up to 1000 °C, a sputtering gun capable of using either Ar^+ ions or coronene (C_{24}) for non-destructive depth profiling, a UV lamp for performing Ultra-Violet Photoemission Spectroscopy (UPS), an Ar-filled glove box attached directly to the entry lock of the XPS instrument for analyzing air- and moisture-sensitive samples.

Omicron Surface Analysis Cluster is a unique, multi-tool instrument that integrates XPS, low-energy electron diffraction (LEED), ultra-high vacuum (UHV) scanning tunneling microscopy and atomic force microscopy (STM/AFM), and high-resolution electron energy loss spectroscopy (HREELS). The system includes a UHV treatment chamber where samples can be heated up to 1000 K or cooled with liquid nitrogen down to 120 K, and exposed to various gases, including metal-organic precursors. The treatment chamber is further equipped with a mass spectrometer, an e-beam evaporator, an atomic hydrogen source, a metal sputtering source, and several thermal evaporators.

RESEARCHER IN CHARGE

Dr. Hakan Salihoglu
hsaliho@purdue.edu
(765) 494-3106

X-ray diffractometry (XRD)
Panalytical MRD X'Pert Pro can determine the atomic crystalline structure, orientation, and quality as well as density and film thickness; samples can be films, crystals and polycrystalline materials up to 4" wafer; measurements up to 900°C; Spatial mapping (100 x 100 mm motion) of sample surfaces; High speed wide angle x-ray diffraction and fast mode reciprocal space mapping; Pole figure; Low angle measurement down to 0.1 °.

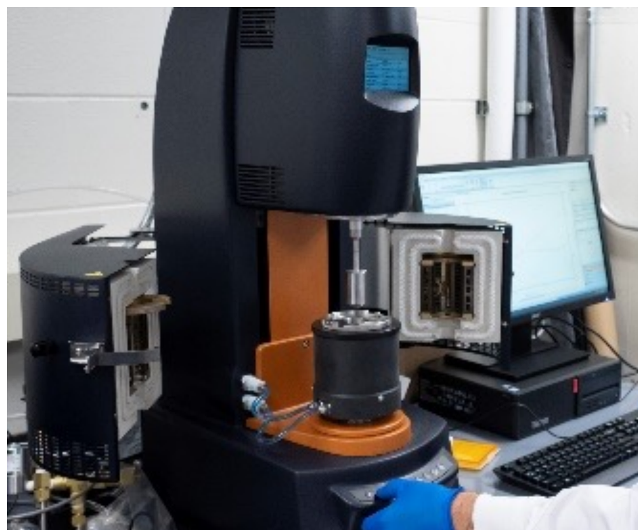


MATERIALS AND ROLL-TO-ROLL CHARACTERIZATION

RESEARCHER IN CHARGE

Dr. Nicholas Glassmaker
nglassma@purdue.edu
(765) 494-4312

Rheometry *(TA Instruments DHR-3 Rheometer):* Accessories are available for measurement of viscometry, viscoelastic properties, dynamic mechanical analysis, and dielectric properties of solids, liquids, and viscoelastic materials. Many tests have temperature control over a wide range, and some can also be carried out under humidity control. UV accessory is available to measure property changes during UV curing processes.



Thermogravimetric Analysis, Dynamic Scanning Calorimetry *(TGA Netzsch TG 209 Tarsus, DSC Netzsch DSC 214 Polyma):* Automated temperature ramps, cycling, and hold tests are available, or advanced programming is possible to simulate process. N2 atmosphere.

Instrumented Drier *(custom apparatus "JOEY")* Measure weight, thickness, temperature, and optical birefringence to determine time to dry polymer solutions, as well as dynamic development of anisotropy.

Mechanical Load Frame *(custom apparatus)* Tensile testing with temperature control and in-situ optical measurements for thermomechanical and opto-mechanical property measurements on solid films.

ELECTRICAL MEASUREMENTS

RESEARCHER IN CHARGE

Dr. Angshuman Deka
dekaa@purdue.edu
(765) 496-9156

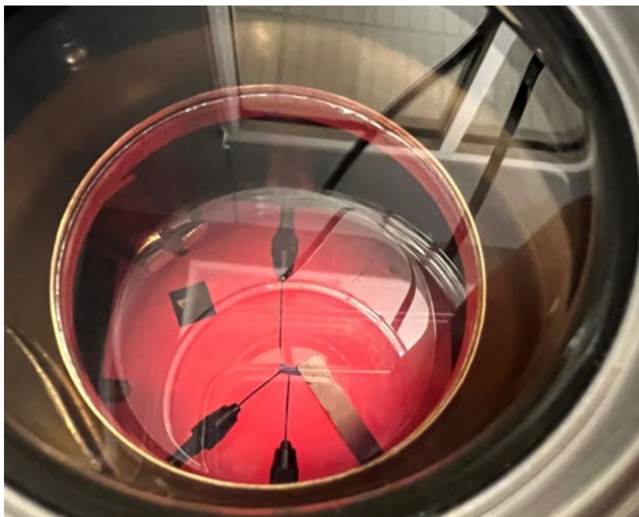
Electrical properties such as carrier mobility and concentration, resistivity, dielectric constant and breakdown can be measured on films, bulk samples, or devices using a suite of probe stations and cryostats in conjunction with impedance analyzers, semiconductor parameter analyzers and lock-in amplifiers.

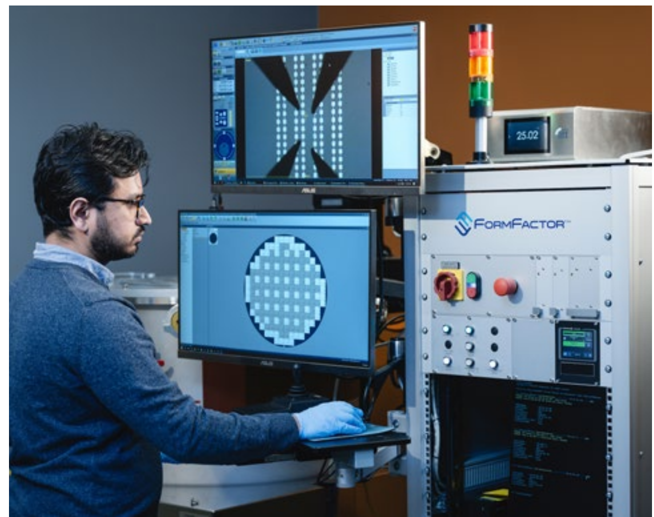
High bandwidth *Copper Mountain* vector network analyzers allow microwave characterization up to 44 GHz. In addition, a *Keysight* 13 GHz, 80 GSa/s, 4-port oscilloscope and a fast-switching arbitrary waveform generator with 4 ports, 65 GSa/s sampling and 16 GSa memory allow users to measure high frequency properties in cryogenic to elevated temperatures.

Advanced low frequency noise analyzer suite from *Keysight* enables noise measurements in 30 mHz – 100 MHz range. High power DC or pulsed reliability testing of electronics from is possible for up to ± 3000 V and ± 50 A using Keithley 2657A and 2651A.

Probe stations include semi-automated probe stations from Formfactor and Micromanipulator, in addition to probers from *MicroXact, Lake Shore,* and *Jandel.* Compatible with DC and RF (40 GHz) measurements and providing temperatures from 4 – 1173 K (900 C) and out-of-plane magnetic fields up to 2.5 tesla. Measurements can be performed on coupons or wafers up to 8” (200 mm), with automated probing for large device arrays. Environments include air, vacuum, and controlled atmosphere.

In addition to the Lake Shore cryogenic probe stations, other cryogen-free systems for electrical measurements include a *Quantum Design DynaCool PPMS* (temperatures from 1.8 K – 400 K and magnetic fields up to 9 tesla) and closed-cycle *Bluefors* dilution refrigerator (10 mK, 12 tesla) providing a platform for optical spectroscopy and electrical transport measurements from DC to RF.



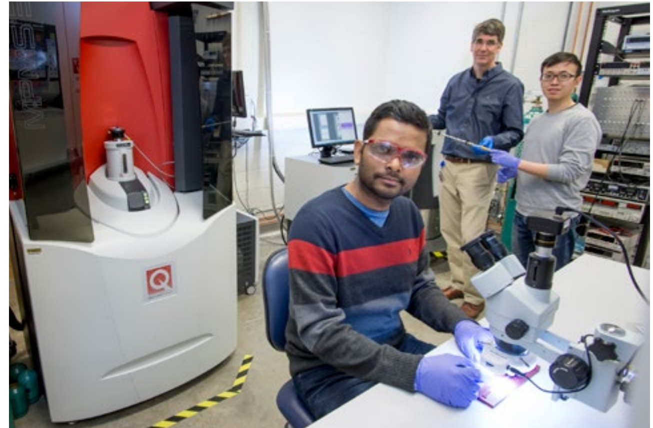


MAGNETIC AND THERMAL PROPERTIES

RESEARCHER IN CHARGE

Dr. Neil Dilley
ndilley@purdue.edu
(765) 496-6080

Measure such properties as magnetization, permeability, magnetic anisotropy, phase transitions and GHz spin dynamics. Samples can be thin films, powder, bulk or single crystals. Magneto-optic Kerr effect (MOKE) of micron-scale materials is also available (see **Other Advanced Measurements** section at the end of this brochure).



SQUID magnetometry *(Quantum Design MPMS-3 EverCool):* high sensitivity 10^{-8} emu (10^{-11} A·m ^{2}); SQUID-AC susceptibility from 0.1 to 1000 Hz; temperatures 1.8 – 1000 K; magnetic fields up to 7 tesla.

Broadband ferromagnetic resonance (FMR) spectroscopy *(Quantum Design DynaCool PPMS)* to 18 GHz; vibrating sample magnetometry (VSM); temperatures 1.8 – 400 K, magnetic fields up to 9 tesla.

Thermal properties of heat capacity, thermal conductivity, and Seebeck effect can be measured on bulk materials. The cryostat for bulk thermal measurements is the Quantum Design DynaCool PPMS (temperatures from 1.8 K – 400 K and magnetic fields up to 9 tesla).

OPTICAL CHARACTERIZATION

RESEARCHER IN CHARGE

Dr. Alexei Lagoutchev
lagutch@purdue.edu
(765) 496-3539

Thermo Scientific DXR3xi Raman Imaging Microscope (credit: thermofisher.com)

Raman spectroscopy is a method of measuring frequencies and strengths of molecular vibrations. These frequencies typically belong in the infrared area of electromagnetic spectrum, but Raman spectroscopy allows measuring them using visible or near-IR light. Vibrational spectra of molecules are used to identify different species or crystalline structures of solids.

Thermo Scientific DXR3xi Raman Imaging Microscope:

- Excitation: 455 nm and 532 nm laser lines
- Raman mapping: fast 3D positioning stage with 100-nm step sizes in the X-Y direction and 200-nm in the Z direction
- Light detection: EMCCD camera
- Spectral resolution: 2 to 5 cm^{-1}, depending on the spectral window

Ellipsometry is used to infer information about thickness, permittivity and refractive index of thin films (down to ~nm).

J.A. Woollam RC2 Spectroscopic Ellipsometer: measurements of refractive indices, permittivities and Mueller matrices of bulk materials and thin films; composition, roughness and thickness of thin films, scatterometry and transmission, 210–2500 nm.

J.A. Woollam V-Vase Spectroscopic Ellipsometer: 200–3000 nm range, in air, temperature range of 80–1273 K

Grating spectrophotometry *(PerkinElmer Lambda 950):* capable of measuring transmittance, absorbance, reflectivity and scattered light spectra over a 190–3000 nm range, and is equipped with an integrating sphere for reflectivity and scattered light spectra measurements in various geometries.

For access to the clean room optical microscopes listed below, please contact: Ron Reger, rreger@purdue.edu, (765) 494-6667.

Digital 3D microscopes

Keyence VHX-950F: 20x-2000x; compound light with multiple lighting modes, image contour enhancement, glare removal, and vibration compensation. The depth composition feature produces sharp (focus-stacked) images and enables a full 3D reconstruction of the sample.

Keyence VK-X3000: roughness/flatness measurement confocal system. Measures roughness down to 0.5 nm on wafers up to 8″ diameter.

Keyence VK-7000 Digital microscope: 20x-6,000x with Elemental Analysis which can detect 75% on the table of elements in an acquisition time of 2 seconds.

Optical microscopes *(several, including Olympus BX-60* and *Nikon Eclipse L150)* offering 5x-150x lenses, bright/dark field and Nomarski, and CCDs with SPOT software for image capture.

Keyence VHX-950F Digital Microscope (credit: keyence.com)

NANOSCALE OPTICAL MICROSCOPY AND SPECTROSCOPY

RESEARCHER IN CHARGE

Dr. Hakan Salihoglu
hsaliho@purdue.edu
(765) 496-3106

Nano-Raman spectroscopy is a method of measuring frequencies and strengths of molecular vibrations with nanoscale resolution. Excitation wavelength is 532 nm, and the Andor spectrometer detects photoluminescent and Raman emissions up to 950 nm.

Nano-FTIR spectroscopy is a technique to obtain spectral optical response of material with nanometer resolution. Accompanied with a pulsed laser covering a wavelength range from ~4 μm to 15 μm, this instrument collects local absorption and reflection spectra.

Near-field scanning optical microscopy enables optical imaging at nanoscale using a background-free interferometry technique. Coupled to an Optical Parametric Oscillator (OPO) laser, the instrument images structures over 100 x 100 μm^2 area with a resolution down to 20 nm over a wavelength range of 1.5 μm to 18 μm. One can also perform imaging at 532 nm.

Ultrafast spectroscopy is a pump-probe technique enabling femtosecond pump excitation and mid-infrared probing over a broad IR spectrum. This technique is coupled to the nano-FTIR system and delivers femtosecond resolved spectroscopy.

Kelvin-probe force microscopy (KPFM) enables high-resolution surface potential and topography mapping of nanostructures, surfaces and devices.

Piezo force microscopy (PFM) enables nondestructive visualization and control of ferroelectric nanodomains as well as direct measurements of the local physical characteristics of ferroelectrics, such as nucleation bias, disorder potential, energy dissipation, and domain wall dynamics.

Conductive AFM maps local variations in a sample's electrical conductivity with an applied voltage between sample and a conductive tip and generates a current image. It also enables local I-V curves at a desired position.

OTHER ADVANCED MEASUREMENTS AVAILABLE IN RESEARCH LABS

Several research groups at Birck are making select instruments available for collaborations:

WiTec Alpha300R Raman/ Photoluminescence(PL)/ Photocurrent Imaging Microscope
wavelengths of 405 nm, 488 nm, 532 nm, 785 nm; temperature range of 80–600 K
Prof. Thomas Beechem lab
tbeechem@purdue.edu

Horiba Xplora Raman/PL microscope
red and green laser wavelengths (638 nm and 532 nm); temperatures down to 10 K
Prof. Yong Chen lab
chen276@purdue.edu

Horiba LabRAM800 Raman spectrometer
Prof. Xianfan Xu lab
xxu@ecn.purdue.edu

JA Woollam IR-VASE Mark II Spectroscopic Ellipsometer
temperature range of 80–600K
Prof. Thomas Beechem lab
tbeechem@purdue.edu

Magneto-optic Kerr effect (MOKE)
is an optical measurement of magnetic properties of films. We have a MOKE optical cryostat (temperatures down to 6 K) equipped with a 5 tesla superconducting magnet.
Prof. Yong Chen lab
chen276@purdue.edu

Ultrafast pump-probe thermoreflectance (Coherent Mira)
for thermal conductivity and interface resistance
Prof. Xianfan Xu lab
xxu@ecn.purdue.edu

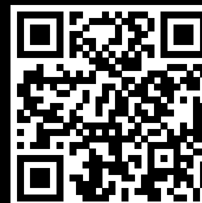
Ultrafast VIS-IR pump-probe spectroscopy system (Coherent Legend) to measure ultrafast carrier dynamics
Prof. Xianfan Xu lab
xxu@ecn.purdue.edu

FTIR and Continuum IR microscopy
(Thermo Electron Nicolet IS50R)
Prof. Xianfan Xu lab
xxu@ecn.purdue.edu

CONTACT US

BIRCK NANOTECHNOLOGY CENTER
1205 Mitch Daniels Blvd.
West Lafayette, IN 47907-2057
nano.purdue.edu

For general information about our facilities, please contact Birck's engineering manager:
Ron Reger
rreger@purdue.edu
(765) 494-6667



For more information on Birck's facility resources including characterization and nanofabrication.

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