

MATERIALS RESEARCH FACILITY (MRF)

COLLABORATIVE, STATE-OF-THE-ART LAB SPACE PROMOTES INNOVATIVE MATERIALS WORK THAT LEADS TO BREAKTHROUGHS







FROM OUR DIRECTOR

Telcome to the University of North Texas' Materials Research Facility (MRF), a unique state-of-the-art integrated facility for processing, fabrication, characterization and analysis of a wide range of materials. The seeds of MRF were planted in 2004 with the creation of the Center for Advanced Research and Technology (CART) at UNT, made possible by an Army Research Laboratory grant. CART comprised more than 10 pieces of state-of-the-art equipment for advanced microscopy and spectroscopy, including a high-resolution analytical transmission electron microscope (TEM), a dual-beam focused ion beam scanning electron microscope (FIB-SEM), a 3D atom probe microscope, X-ray photoelectron spectroscopy (XPS) and Auger electron spectroscopy (AES) systems.



From the beginning, one of the distinctive attributes of CART was the complementary nature of the characterization and analysis tools present in the facility and the coupling of dedicated microscopy and dedicated spectroscopy tools. This level of coordination is not often observed in other advanced microscopy facilities, nationally or internationally. Subsequently, in 2010, UNT won a highly competitive National Science Foundation grant under the Academic Research Infrastructure (ARI) program for the purpose of designing a new facility to consolidate this equipment under one roof, with complementary equipment located in adjacent rooms. Additionally, a nanofabrication cleanroom facility completed in 2012 was combined with CART's microscopy and spectroscopy equipment under one centralized MRF in 2016. The success of the MRF is made possible by significant continuing support from UNT's Division of Research and Innovation.

MRF is closely partnering with UNT's latest initiative on additive manufacturing funded by the State of Texas, the Center for Agile Adaptive and Additive Manufacturing (CAAAM). Additive manufacturing is a rapidly developing technology that builds 3D objects through the successive layering of metals and ceramics using lasers and other high-energy technologies. Because of its unprecedented efficiency, limitless applications and a transformational shift in design, manufacturing and supply approaches, additive manufacturing and related technologies will be in high demand and use for the aerospace, biomedical, and oil and gas industries.

Such a coupling of processing and fabrication from the macro (CAAAM) to the nano scale (cleanroom) with advanced microscopy and spectroscopy techniques, all located in adjacent areas of the same building, makes the MRF one of a kind in the nation. Researchers from departments across multiple colleges of the university are using these facilities for both fundamental and applied research. A number of local and regional industries also use MRF facilities, largely for solving specific technological problems. We also have had active collaborators from other U.S. and international academic and research institutions. Looking into the future, we are enhancing the MRF to become a center for 3D materials characterization from atomic- and nanometer-length scales to millimeters.

I personally invite all of you to discover our facilities, use them and develop a collaborative relationship with us. We look forward to working with you.

Best regards,

Rajarshi Banerjee Director of UNT's Materials Research Facility Presidential Professor and University Regents Professor rajarshi.banerjee@unt.edu

UNT's Cutting-edge Research Spaces Drive Innovation and Creation Across Disciplines

WHO WE ARE

SINCE 1890, THE UNIVERSITY OF NORTH TEXAS HAS BEEN A CATALYST FOR DISCOVERY AND INNOVATION.

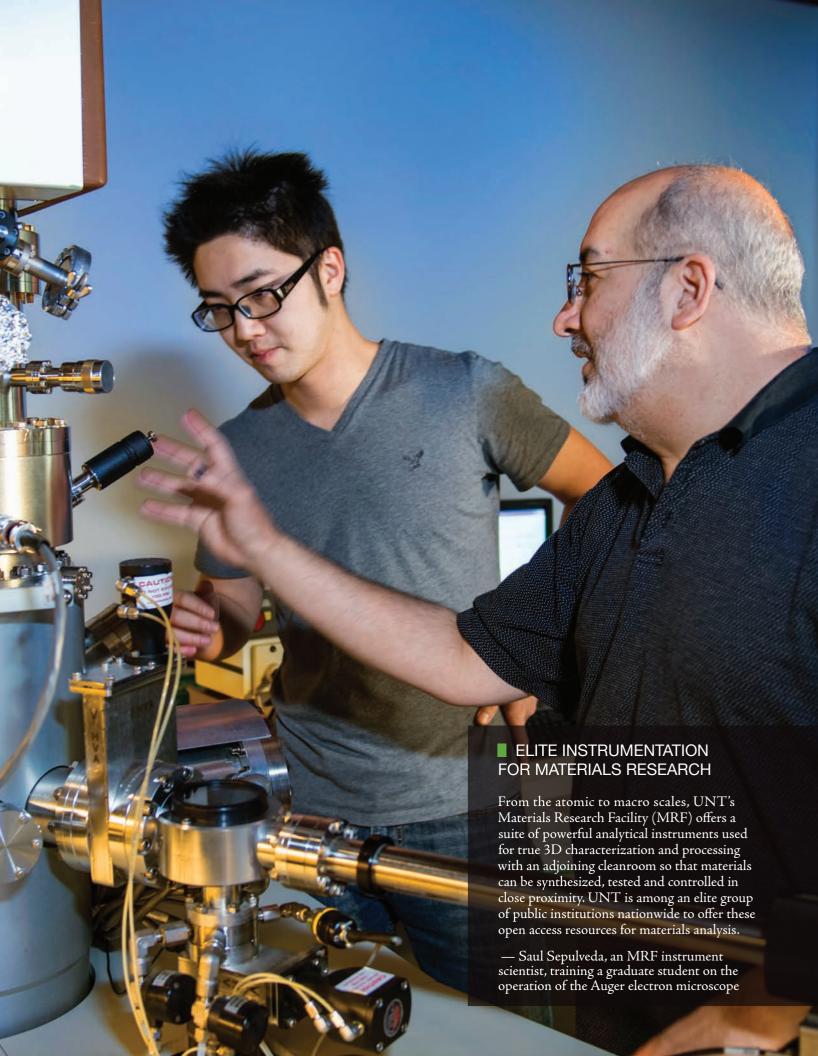
Today, UNT is ranked a Tier One research university by the Carnegie Classification — a recognition that speaks to its quality and impact — and high-tech research facilities are part of the UNT culture that makes this possible. The university's Materials Research Facility (MRF) is one such example.

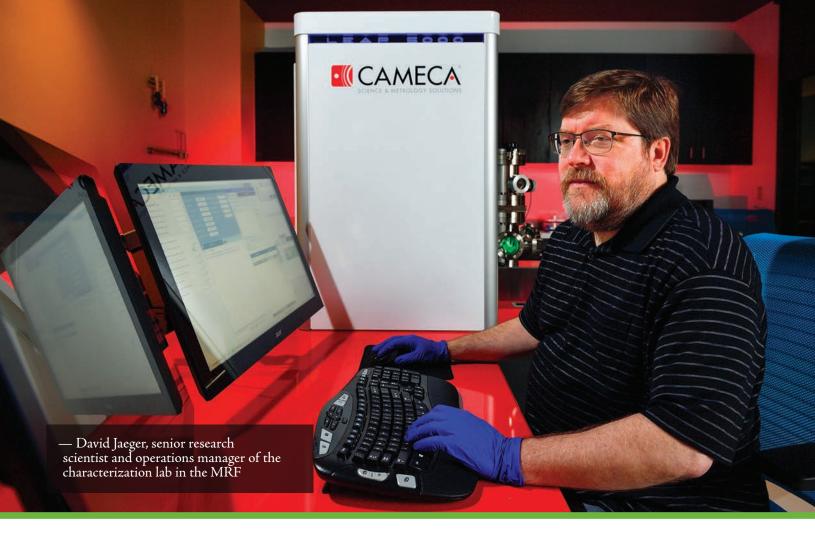
Researchers from across the UNT campus and beyond use the more than two dozen instruments at the university's MRF laboratories to multidimensionally fabricate, characterize and analyze a wide range of materials. Numerous projects bring in millions of dollars in research funding and span numerous areas of expertise in disciplines such as engineering, materials science, physics, chemistry and biology. The research collaborations in these laboratories are no doubt at the cutting edge of cross-disciplinary synthesis, characterization and analysis.

From the atomic- and macro-length scales, the MRF is one of the most advanced university research facilities in the nation for materials analysis. The facility offers a suite of powerful analytical instruments used for true 3D characterization and processing with an adjoining cleanroom so that materials can be synthesized, tested and controlled in close proximity. UNT is among an elite group of public institutions nationwide to offer these open access resources.

UNT's Discovery Park — the university's 300-acre research campus and the largest in the North Texas region — houses the MRF, as well as numerous other laboratories and state-of-the-art instrumentation that enable faculty researchers to push the boundaries of science, technology and creativity while providing students hands-on experience to ready them for their future careers.

In many of UNT's facilities like the MRF, faculty and student researchers already are partnering with each other and collaborators from research institutions around the world and industry. UNT labs can be "put to work" to investigate, beta test, and produce solutions and real-world applications. Faculty and students work alongside industry leaders to apply their knowledge in solving problems and creating new products and technologies.





MULTI-DIMENSIONAL CHARACTERIZATION LABORATORY

UNT'S MULTI-DIMENSIONAL CHARACTERIZATION LABORATORY HOUSES ADVANCED INSTRUMENTATION FOR PROBING THE STRUCTURE, CHEMISTRY AND COMPOSITION OF MATERIALS DOWN TO THE NANOMETER-AND ATOMIC-LENGTH SCALES.

Such a combination of instruments and their layout makes this lab a unique facility both locally and among major research universities in the U.S. The instruments in this lab are complementary to each other, and combining the experimental information from these different instruments gives a robust and realistic picture regarding the structure and chemistry of complex materials. A good example of this complementary set of instruments is the trio comprising the dual-beam focused ion beam microscope (FIB-SEM), the high-resolution analytical transmission electron microscope and the 3D atom probe microscope. The FIB-SEM allows one to carry out high-resolution scanning electron microscopy on various materials and components from industry and site-specific or location-specific sample preparation, and these samples can immediately be analyzed using the TEM and 3D atom probe.

Not only are these three powerful characterization instruments highly complementary in nature, but they also are located in close proximity within the Materials Research Facility (MRF) — literally in adjacent rooms. Additionally, the lab has a range of other microscopy, spectroscopy and X-ray diffraction instruments, all located in close proximity in a central location.

The facilities in this lab are used by researchers from industries such as aerospace, defense, and oil and natural gas, as well as academic investigators from all around the world.

SURFACE AND THIN FILM ANALYSIS

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XPS IMAGING SPECTROMETER

The PHI Versaprobe II performs surface-sensitive chemical and molecular analysis. The instrument's technology provides a monochromatic, micro-focused scanning X-ray source, which provides excellent large area and superior micro-area spectroscopy performance. It can measure sample composition, as well as chemistry within the sample. Applications of this instrument include the analysis of inorganic compounds such as atomically thick films of graphene for next-generation transistors, the analysis of wear-resistant coatings used in advanced aircraft manufacturing and the determination of contaminants that lead to failures in electronic circuits.

SCANNING AUGER MICROSCOPE

The PHI 670XI is a unique high-performance instrument that uses advanced detectors to examine the elemental and chemical distribution of the elements in a material's sample. In doing so, users are able to see how a material's structure affects it properties at the nanoscale. This high-performance Auger is used to analyze contamination of microelectronic samples and the corrosion of metals used in oil and gas processing.

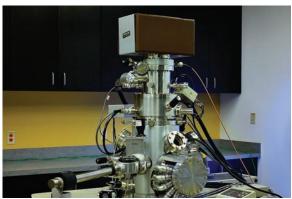
SPECTROSCOPIC ELLIPSOMETER

The J.A. Woollam VASE Ellipsometer is a noncontact, nondestructive instrument that measures surfaces and very thin films on surfaces. It measures how polarized light interacts with samples to measure optical properties, roughness, thickness, electrical conductivity and many other properties for materials such as semiconductors, dielectrics, polymers and metallic multilayered thin films.

SCANNING PROBE MICROSCOPE

The Veeco Nanoscope IIIa Multimode is a versatile highperformance SPM that measures the surface topography and physical properties of samples down to the atomic scale using a variety of interchangeable scanners and probes. It can nondestructively analyze a wide variety of materials, such as metals, semiconductors, ceramics, biological materials, polymers and nanoparticles.



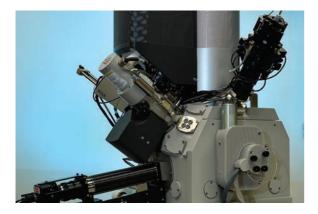


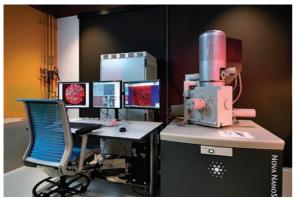




ELECTRON AND ION MICROSCOPY







3D ATOM PROBE MICROSCOPE

The Cameca LEAP 5000 XS is a cutting-edge atom probe microscope that uses voltage and laser pulses to strip single atoms from sample materials. The identity of each stripped atom is subsequently analyzed using a time-of-flight mass spectrometer. This allows for a true 3D mapping of the atomic distribution within samples. This instrument has been used to analyze the distribution of atoms within advanced metallic alloys used in aerospace applications, complex interfaces within microelectronic circuits and solar cell devices.

HIGH-RESOLUTION ANALYTICAL TEM

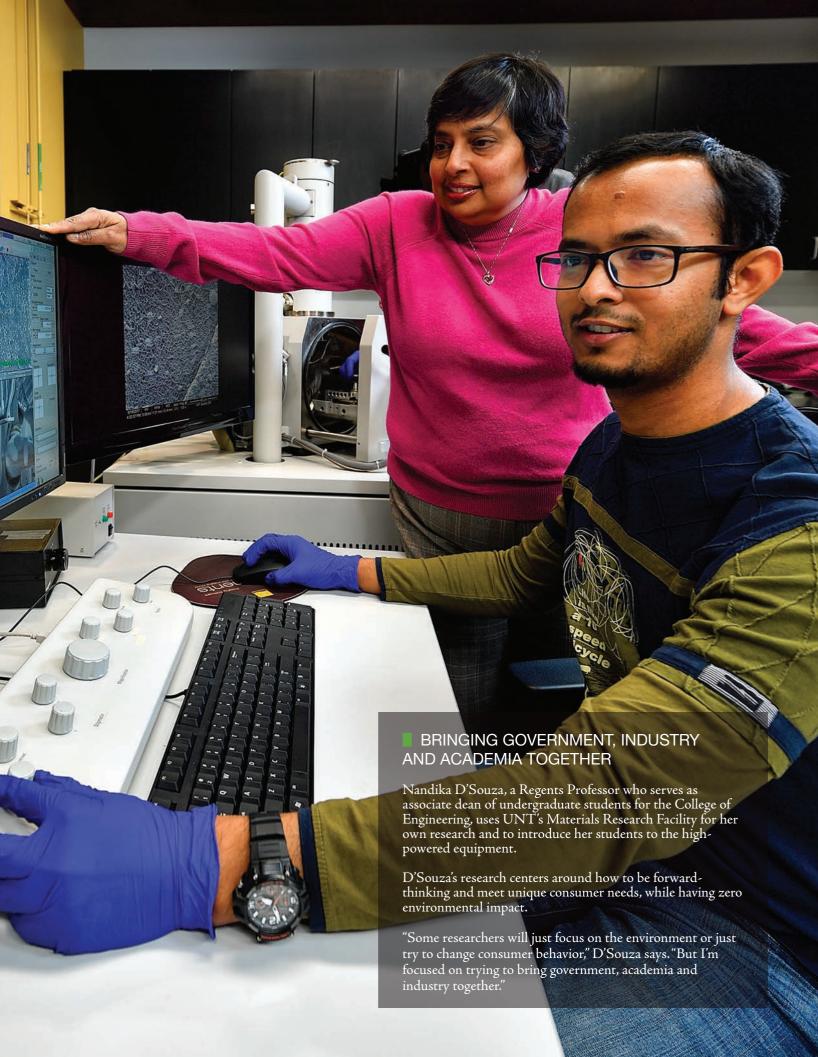
The FEI Tecnai G2 F20 ST scanning transmission electron microscope provides information on the structure and chemistry (or composition) of samples prepared from a wide range of materials. The system is equipped with an S-TWIN lens and high-brightness field emission electron gun and has the ability to cross correlate analytical techniques, allowing researchers to accurately determine the structure and chemistry of materials from the nanoto micro-scale.

DUAL BEAM FIB/SEM

The FEI Nova NanoLab 200 is a focused ion beam and scanning electron microscope designed to be a complete nanotechnology laboratory in one instrument. This instrument is used to examine materials of a few millimeters to a hundred nanometers and has the ability to do micro-machining using a focused ion beam and create 3D reconstructions of the material structure and crystallography. This instrument is widely used across multiple disciplines for diverse applications such as analyzing failed transistors in integrated circuits (ICs) for microelectronics and determining why aircraft components cracked in service.

ANALYTICAL FEG-SEM

The FEI Nova NanoSEM 230 is an ultra high-resolution scanning electron microscope equipped with a field-emission gun and specifically configured with an array of advanced detectors to get the most structural and analytical information out of the widest selection of samples such as complex metallic, ceramic and semiconducting materials. Equipped with a state-of-the-art electron backscatter detector (EBSD), this SEM analyzes the phase and crystallography of the material. This system has been used to investigate a host of advanced engineering metallic alloys used in aerospace, transportation and medicine, as well as microelectronic materials and magnetic materials used in the memory storage industry.





ENVIRONMENTAL SEM (ESEM)

The FEI Quanta 200 is an easy-to-use, unique and versatile highperformance variable pressure scanning electron microscope, capable of imaging dry and wet samples and performing dynamic in-situ experiments. The instrument allows for the analysis and imaging of the widest range of materials from microelectronic circuit boards and metal gas pipeline components to tree leaves and insects.

VIBRATIONAL SPECTROSCOPY



FOURIER TRANSFORM INFRARED SPECTROMETER (FTIR)

The Thermo Electron Nicolet 6700 spectrometer can handle nondestructively both exploratory research and everyday routine analysis. In measuring the infrared spectrum obtained after a monochromatic light beam interacts with a sample, researchers from the biomedical, microelectronics and pharmaceutical industries are able to rapidly determine the chemical and molecular structure of a wide range of both solid and liquid samples.

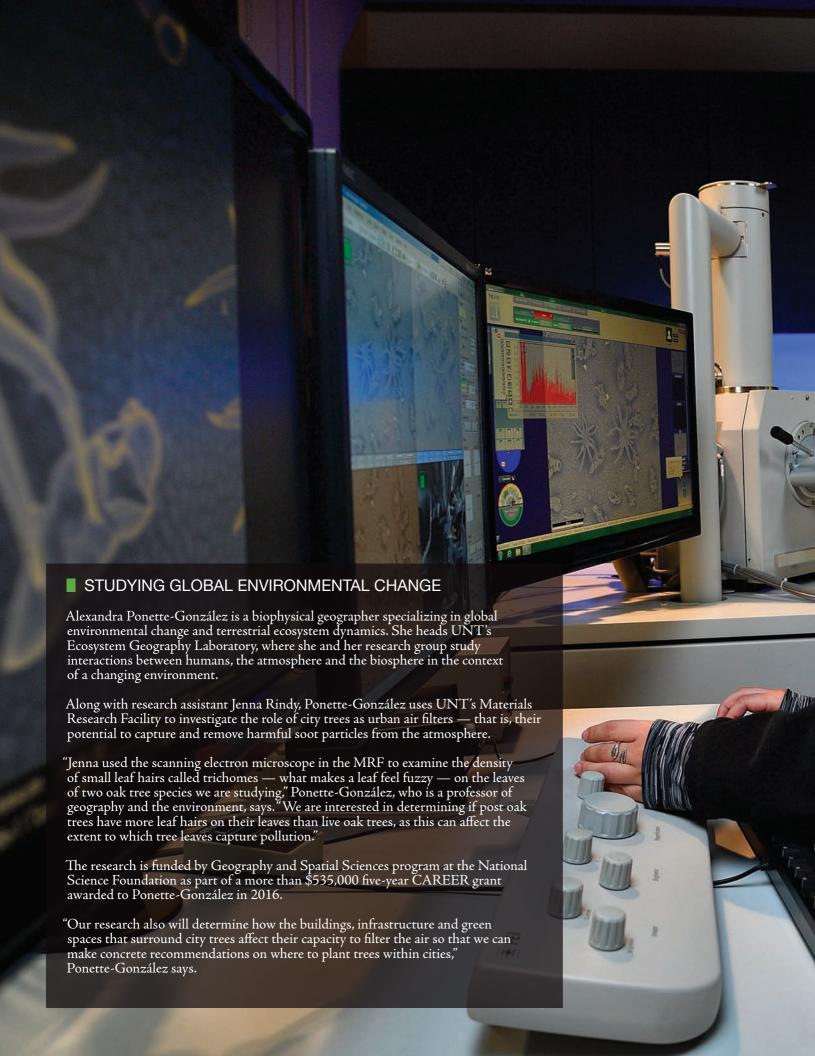
X-RAY DIFFRACTION



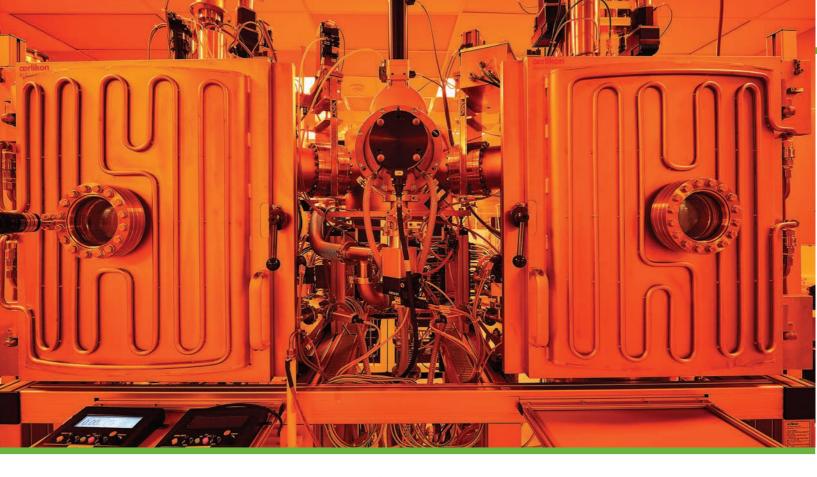
HIGH RESOLUTION X-RAY DIFFRACTOMETER (XRD)

The Rigaku Ultima III is an advanced multi-purpose X-ray diffraction system that performs in-plane and normal geometry phase identification, quantitative analysis, lattice parameter refinement, depth-controlled phase identification and many different measurements, including crystallite size, structure refinement, density, roughness and multilayer thicknesses. This allows researchers to create a pattern that can be analyzed to determine the structure of metals, ceramics, gemstones and pharmaceuticals.









NANOFABRICATION CLEANROOM

UNT'S NANOFABRICATION CLEANROOM IS AN ADVANCED RESEARCH FACILITY EQUIPPED WITH AN EXTENSIVE SET OF INSTRUMENTS FOR NANO- AND MICRO-DEVICE FABRICATION AND CHARACTERIZATION TO SUPPORT THE RESEARCH NEEDS OF FACULTY AND STUDENTS IN A BROAD RANGE OF ENGINEERING AND SCIENCE DISCIPLINES.

The cleanroom features about 3,000 square feet of clean space and includes a class 100 lithography area and a class 10,000 metallization wet and dry processing and characterization area. Its capabilities emphasize nano and micro-device development, biomedical, advanced materials, photomask fabrication, OLED device fabrication and thin film techniques. This open-access facility is used in a wide variety of engineering disciplines, including materials, mechanical, electrical and biomedical, along with chemistry and physics.

LITHOGRAPHY





The Laurell WS-650Mz spin coater is used for applying photoresist coating through high-speed spinning on substrates for photolithography process.

JEOL JSM-7001F SCANNING ELECTRON MICROSCOPE AND XENOS XPG 2 ELECTRON BEAM LITHOGRAPHY PATTERN WRITER

The JEOL JSM-7001F and XENOS XPG 2 together are the ideal platform for demanding micro- and nano-patterning applications as well as those requiring high-resolution imaging and ease-of-use. The microscope is used for obtaining images of patterns created on chemically coated small samples, and then the patterns are created through exposure to electron beam patterning of transistor gate structures for advanced materials, electrical property testing and graphene ribbon fabrication for next-generation integrated circuits and systems.



HEIDELBERG DWL 66 MASKLESS LITHOGRAPHY LASER WRITER

The Heidelberg Instruments DWL 66 Maskless Lithography Laser Writer is a high-precision, direct-write system for creating patterns on samples through exposure to light. The system can be used for mask making or direct exposure to basically any flat material coated with photoresist. Applications include photomask fabrication for advanced circuitry design and patterning biomedical delivery channel device.



DEPOSITION

OERLIKON SPUTTERING AND ION ASSIST EBEAM SYSTEM

The Oerlikon Sputtering and Ion Assist Electron Beam System uses both a focused electron beam and plasma in separate chambers to create nanometer-thin films for electrical contacts commonly used in all the electronic devices. Conductive thin films for adhesion layers such as titanium and chromium also can be deposited to enhance the adhesion property of device layers.





NANOMASTER NEE-4000 DUAL EBEAM SYSTEM

The Nanomaster NEE-4000 Dual Electron Beam Evaporation System uses a focused electron beam to create nanometer-thin films for electrical contacts. Applications of this system include various metals such as gold, silver and copper as electrical contact and non-conductive thin films as insulator layers for transistor structure.



TROVATO 300C ORGANIC DEPOSITION SYSTEM

The Trovato 300C Organic Deposition System uses intense heat to create nanometer-thin films. The films are used for research and development of organic light-emitting diodes (OLEDs), solar cells and microelectronics devices. Applications of this system include OLED device and photovoltaic solar cell device fabrication.



SCS PDS 2010 PARYLENE DEPOSITION SYSTEM

The SCS PDS 2010 Parylene deposition system is a vacuum system used for the vapor deposition of the Parylene polymer type C and N onto a variety of substrates. Parylene deposition is used for coating of devices or in some cases even using the material as an insulating layer in the device structure.

PACKAGING

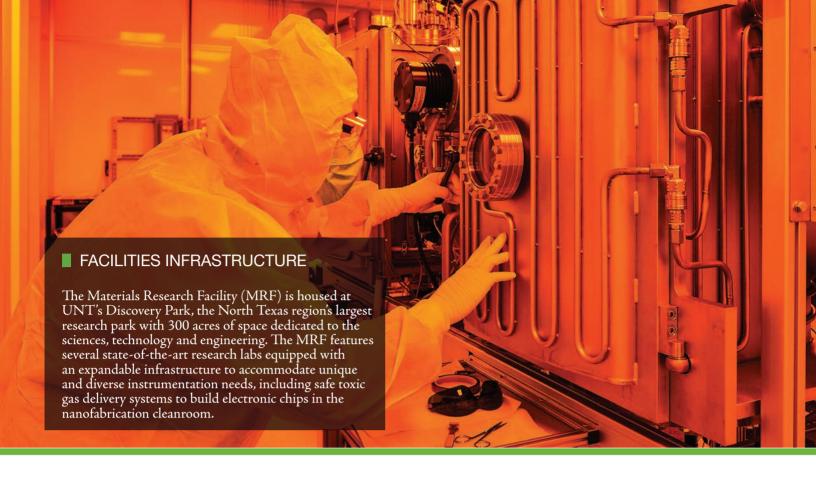




WIRE BONDER WEST BOND INC. 7476D

The West Bond 7476D wire bonder is used for connecting the semiconductor chip to external contact leads by applying ultrasonic energy to attach the wire.





ETCHING

AGS MPS-150 REACTIVE ION ETCHER

The AGS MPS-150 RIE system is used for dielectric and metal thin film etching processes for removing thin films from a surface. This is used for microchip fabrication. Applications of this system include photoresist ashing, graphene film etching and patterned silicon etching for biomedical devices.



LAURELL WS-1000MH-CP7-D CHEMICAL PROCESSING STATION

The Laurell chemical processing stations are designed for all the steps of lithography wet processing with automated programming capacity. It is used for cleaning, development, lift-off and etching wet processes with ease and automation. Its application includes producing photomasking and assisting the fabrication process of patterning devices.





NANOSCIENCE UNDER PRESSURE

Hao Yan, assistant professor of chemistry, uses facilities at the MRF to study nanoscale materials under mechanical pressure. High pressure is traditionally the realm of geologists, where people search for new phases of minerals in extreme environments such as the center of planets. These works have largely focused on bulk materials. On the other hand, it is well known that new properties emerge in materials at the nanometer scale; however, how these properties are modulated by external pressure is largely unknown.

Hao's research bridges these two fields — high pressure science and nanoscience — to create and manipulate functional nanomaterials for applications ranging from catalysis to quantum information technology. MRF provides a wide variety of the tools — from atomic-resolution microscopy to surface spectroscopy — that are essential for the highly interdisciplinary research in his group.



METROLOGY



J.A. WOOLAM M-2000V AUTOMATED ANGLE ELLIPSOMETER

The J.A. Woolam M-2000V Ellipsometer is used for measuring thin film thickness and optical constants through non-contact optical analysis.



JEOL JSM-7001F SEM

The JEOL JSM-7001 SEM is a high-resolution field emission SEM that is capable of imaging the fine structure of materials with nanometer resolution. In addition to conventional SEM imaging, it is capable of high-resolution, low-voltage imaging of nonconductive samples and imaging magnetic materials. Substrates ranging from 200 mm diameter wafers down to small pieces can be imaged with the system.



NIKON OPTIPHOT 66 OPTICAL MICROSCOPE

The Nikon Optiphot 66 optical microscope provides reflection and transmission mode imaging for photo-mask and backside inspection using objective lenses with 2.5x to 100x magnification and coupled with a digital camera for image capture and storage.



KLA-TENCOR D300 PROFILER

The KLA-Tencor D300 Profiler offers high-resolution 2D profiling in an easy-to-use platform. It uses a stylus to scan across a sample area to measure thin film thickness, etching depth and surface roughness.





PROTECTING THE ENVIRONMENT

Dornith Doherty, a Distinguished Research Professor of studio art, uses UNT's SEM in the Materials Research Facility to capture images of the microscopic details of migratory bird feathers, focusing on the minute details of airborne dust and damaged feathers that would be otherwise invisible to the unaided eye. After capturing the scans, Doherty creates artworks by adjusting and stitching together five or more individual scans to make works that are approximately 6 feet tall and still maintain the clarity and accuracy of the original scanning electron microscope imagery.

"In conversation with my scientist collaborators, I edit the images with artistic intent," Doherty says. "The resulting research-based artworks in my ongoing project, *Atlas of the Invisible*, use metaphor and beauty to pose open-ended questions to engage viewers. My art counters the common misapprehension of the atmosphere as a void and reveals our entanglement with, and dependence on, the atmospheric domain."

A 2012 Guggenheim Foundation Fellow, Doherty's work was featured in a solo exhibition at the National Academy of Sciences Art Gallery in Washington D.C. in 2019 and is currently on display at the Smithsonian National Museum of Natural History's *Unsettled Nature: Artists Reflect on the Age of Humans* in Washington D.C., and the Museum of Contemporary Art in Toronto.









"The UNT Materials Research Facility is, by any measure, a world-class resource staffed by extraordinary scientists, who, together with our research laboratories, are helping to discover scientific insights every day."

> — Mark McLellan, Vice President for Research and Innovation

Materials Research Facility (MRF) 3940 North Elm Street, E-178G Denton, Texas 76207-7102 940-369-8106 MRF.Research@unt.edu

Learn more about UNT research by visiting *mrf.research.unt.edu*.