NC STATE UNIVERSITY

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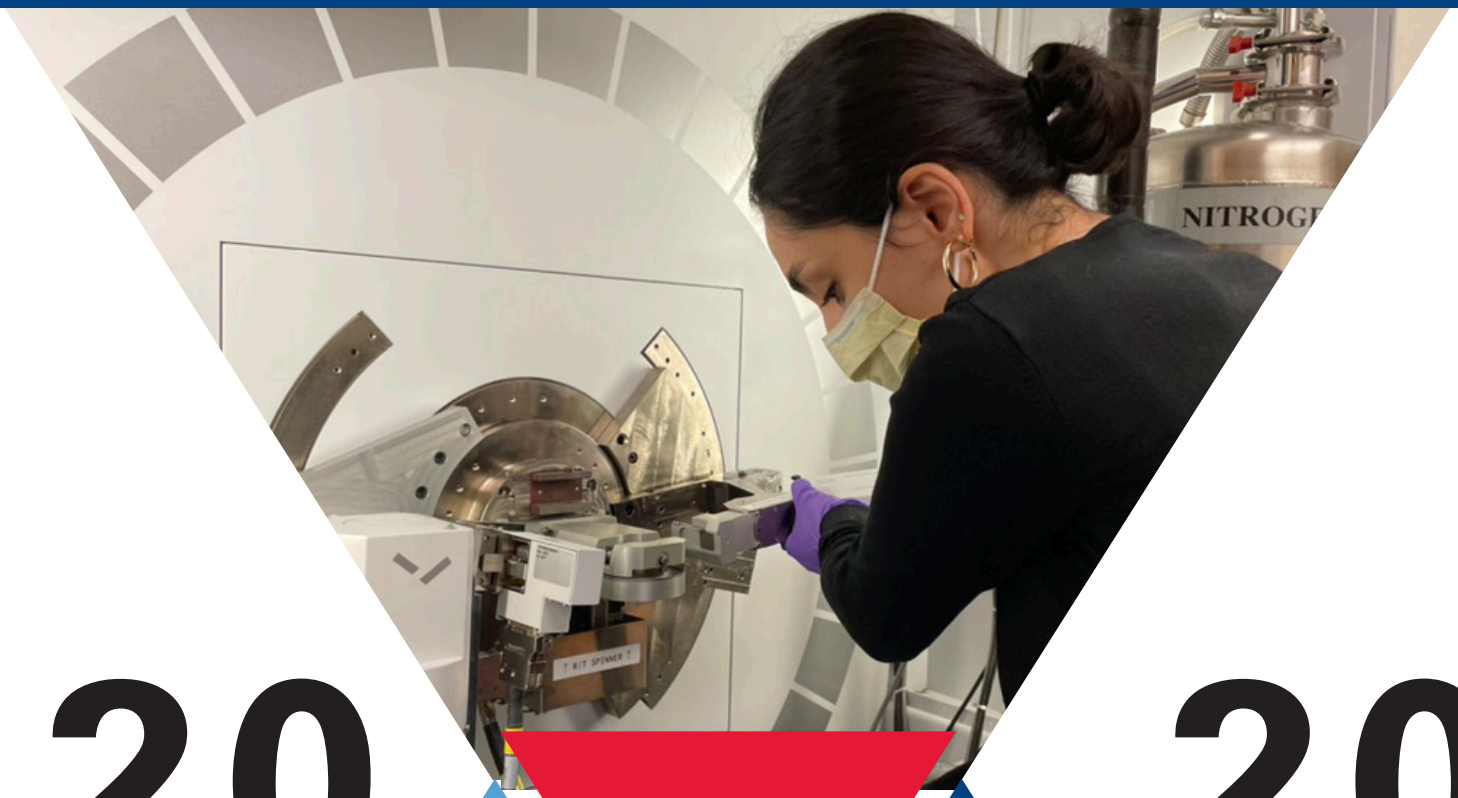
NSF

National Nanotechnology Coordinated Infrastructure



2020

RTNN

ANNUAL REPORT

RESEARCH TRIANGLE NANOTECHNOLOGY NETWORK

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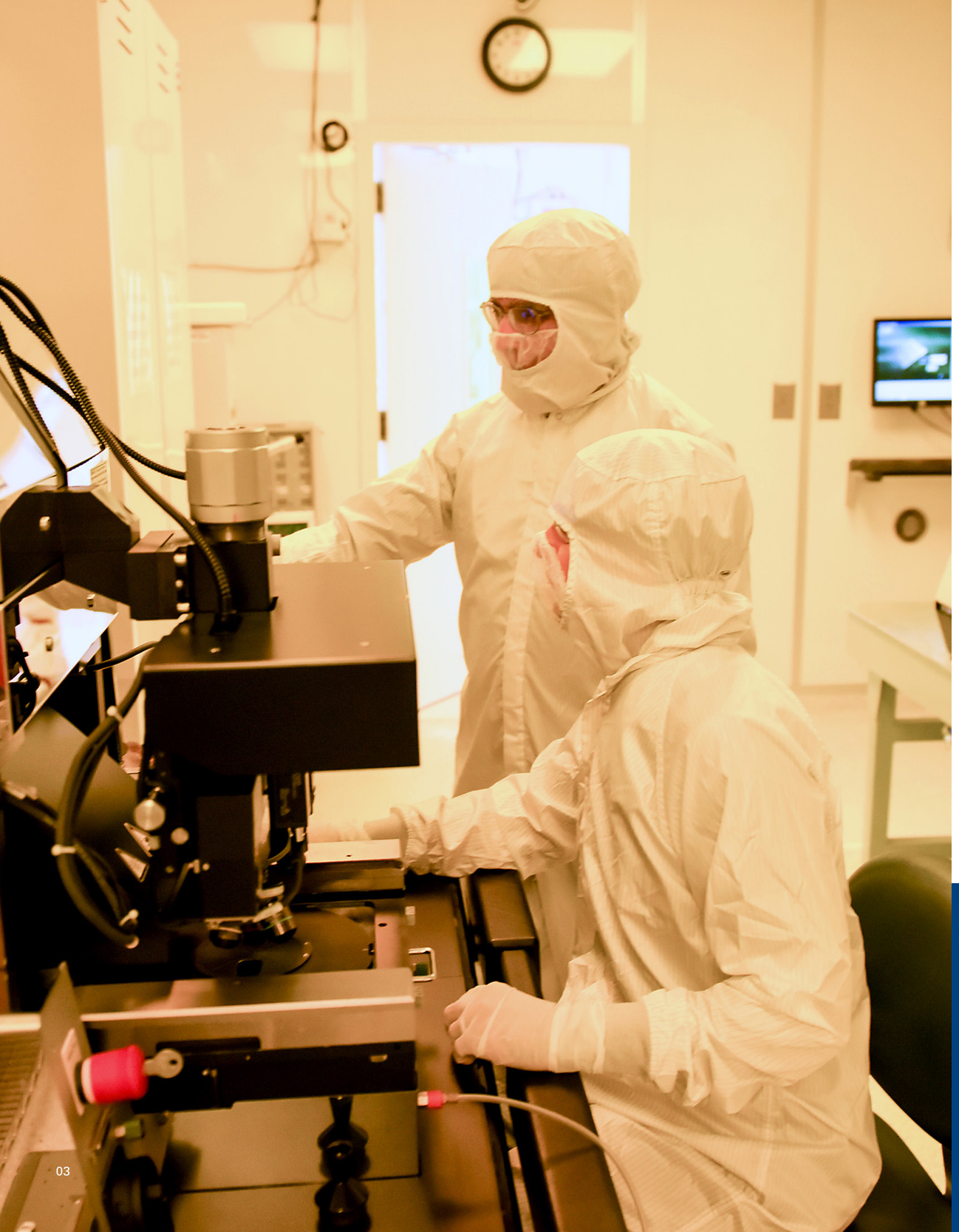


ABOUT RTNN

Nanotechnologies are currently in development for use in diverse fields. These technologies take advantage of unique properties achievable at the nanometer (10^{-9} m) scale to tackle complex problems (e.g., make more efficient solar panels or deliver cancer therapeutics). To support and expand the growth of nanotechnology, the National Science Foundation (NSF) established the National Nanotechnology Coordinated Infrastructure (NNCI) in 2015. The NNCI is made up of 16 sites across the U.S. whose work is focused on the development and analysis of unique nanotechnologies. The Research Triangle Nanotechnology Network (RTNN) is one of such sites.

The RTNN is a collaborative partnership between North Carolina State University (NC State), the University of North Carolina at Chapel Hill (UNC) and Duke University (Duke). Collectively, these institutions house four open-access nano-fabrication and characterization facilities and over 100 principal faculty members whose research encompasses broad aspects of nanotechnology. The overarching goal of the RTNN is to dramatically enhance access to university nanotechnology resources, such as fabrication and characterization facilities and techniques as well as expert research personnel, by lowering barriers to access such as distance, cost and awareness. Through its activities, the RTNN is able to advance basic research at the nanoscale as well as support development and commercialization of innovative nanotechnologies.

2020 Program Highlights

Our activities are designed to raise awareness of our nanotechnology facilities and their capabilities as well as how they can be accessed. These activities are innovative, comprehensive and effective with continual assessment and revision. The COVID-19 pandemic spurred a shift to virtual programming, and RTNN launched several new initiatives to reach the community. Highlights from the past year include:

- Despite the pandemic, **over 1,000 unique annual users** accessed facilities for **over 43,000 hours** of experimental time
- ***Nanotechnology: A Maker's Course***, a Coursera online course, saw a significant increase in enrollment with **more than 23,000 learners in >170 countries**
- The **Kickstarter program** provided ~115 hours on nanotech tools to 10 non-traditional and new users
- *Take-out Science* **virtual SEM sessions** attracted more than **4,000 views** on YouTube
- RTNN visited rural communities in Asheville, NC and Hickory, NC to provide hands-on experiences to K-gray participants, like operating a **portable scanning electron microscope (SEM)**
- **11 in-person and 20 virtual technical workshops and short courses** exposed over 200 participants to new nanotechnology tools and ideas and brought them together to spark new collaborative work

VISION

The aspirational vision of the RTNN is to identify and respond to emerging nanotechnology infrastructure needs that will enable researchers to address societal grand challenges of the next decade, using our facilities as a focal point for discussion and convergence of academic disciplines, industries, the public and private sector, and individuals.

DIVERSITY, EQUITY AND INCLUSION

The RTNN is committed to ensuring equitable access to our facilities by eliminating barriers to access such as distance, cost and awareness, and our programs are intentionally designed to engage participants from underrepresented groups. The RTNN strives to create a culture of inclusion within its facilities by fostering open dialogue and continual discussions with our users and staff and enforcing strict, no tolerance policies in regards to harassment and discrimination. To enact and promote change across the nation, Jacob Jones leads the NNCI subcommittee on diversity. The subcommittee includes members from several NNCI sites who are passionate and committed to addressing racism and discrimination and creating an inclusive environment at their respective sites and institutions.

In response to the violent deaths of George Floyd, Breonna Taylor, Ahmaud Arbery, Rayshard Brooks and numerous other African Americans, the subcommittee held an Anti-Racism Town Hall. This event brought together over 200 users, staff members and faculty members from across the NNCI to discuss complex issues of racism and discrimination. Attendees openly shared their own experiences and perspectives as well as provided innovative ideas to combat racism at our sites and institutions.

NNCI STATEMENT OF DIVERSITY

The NNCI embraces diversity and welcomes, recruits, educates, employs, serves and engages a diverse group of users, students, faculty and staff with a wide variety of backgrounds, perspectives, interests and talents, creating a community of teachers, learners and researchers that exemplifies the best in all of us — in our intellectual pursuits, our diversity of thought, our personal integrity and our commitment to excellence.

We believe that diversity includes the individual differences among people, including

- Gender
- Social, racial or ethnic backgrounds
- Disabilities or handicaps
- Socioeconomic class
- Gender identity or expression
- Sexual orientation
- Appearance or personal characteristics
- Political affiliation and opinion
- Language
- Religion or beliefs
- Economic circumstances
- Philosophical outlooks
- Veteran status
- Life experiences

All of these characteristics, both singularly and in combination, contribute to the richness of the NNCI community.

RENEWAL AND FUTURE PLANS

After undergoing the NSF renewal process, the RTNN was successfully renewed on September 1. In future years, the RTNN will enhance existing programs and implement several new initiatives in response to participant and user feedback.

Kickstarter Program: The RTNN will continue to grow our successful Kickstarter program. We will lower barriers to travel by offering travel support. Participants who are unable to travel will connect to site technical staff remotely during fabrication and/or analysis of samples. This new approach provides more **interpersonal interactions and networking** with users.

"Coursera Nano 2.0": The RTNN will add unique stand-alone modules focused on **Making Devices** to our Coursera course. For each module, videos will provide background on the general area of research and describe how RTNN facilities are used to make a device. RTNN will begin with modules on the fabrication of solar cells as well as biomedical microfluidic devices.

Lunch-and-Learn Events: To promote **awareness**, particularly to non-traditional user communities, the RTNN will host lunch-and-learn events at local economic development organizations and technology incubators to disseminate RTNN capabilities.

STEM Internship Program: To facilitate the successful matriculation of transfer students from community colleges to RTNN institutions, we will create two STEM internship positions per year at RTNN facilities.

Social Media: RTNN will maintain and extend its presence by posting on Russian and Chinese social media sites. RTNN will also champion a social media "Takeover" exercise wherein posting opportunities are turned over to nanotechnology organizations or student groups at universities in India, China or Russia.

Rural K-gray Outreach: The RTNN will reach outside of the Triangle by taking hands-on activities and portable SEMs to rural schools, libraries, museums and other events such as Science Olympiad Regional competitions where there is limited access to R1 institutions.

RTNN Affiliates Network: The RTNN will establish an RTNN Affiliates Network with **local** university and government facilities in the Triangle. The goals of the Affiliates Network are to: 1) **allow regional facility managers/directors to more readily connect researchers** to complementary facilities, 2) enable facility leadership and staff from across the Triangle to **communicate timely information efficiently and effectively**, and 3) **develop strategic partnerships** on emerging opportunities and technical needs.

Research Community: The NNCI Coordinating Office has organized Research Communities as a new mechanism to support and advance compelling research topics of national priority. The RTNN leads the Research Community for Convergence Research and will host a one-day workshop in 2021 to bring together leading researchers from diverse disciplines, facilitate their collaboration using established team science approaches, and work toward a common vision for open-access facilities to address the research needs in **Food and Nutrition Security**.

IMPACT OF COVID-19

The RTNN, similarly to other academic endeavors, was impacted significantly by the COVID-19 pandemic. All RTNN facilities were closed to users for two months during North Carolina's stay-at-home order with the exception of researchers conducting work directly related to the pandemic.

As facilities began to reopen, modified procedures were put into place to ensure the safety of users and staff. Appropriate PPE was purchased, and disinfection protocols were developed and implemented. Reservation procedures were modified to control occupancy in lab spaces and limit face to face interactions. Both staff and users adapted successfully to operating with these new conditions in place. And, despite the closures, 1,000 unique users accessed RTNN facilities.

COVID-19 Related Research

Protochips uses the NC State Nanofabrication Facility and Analytical Instrumentation Facility to manufacture and analyze in situ TEM holders and sample supports. One of Protochips's customers, the McLellan Lab at the University of Texas, determined the 3D structure of the SARS-CoV-2 spike protein, a critical first step towards developing a vaccine. This reconstruction is widely used, including on the homepage of the CDC.

Scientist Priyamvada Acharya from Duke's Human Vaccine Institute is using the Krios cryo-TEM to image the coronavirus spike protein to learn how it functions and translate this knowledge for vaccine design. *Publication: Henderson, R. et al. Controlling the SARS-CoV-2 spike glycoprotein conformation. Nat. Struct. Mol. Biol. 27 (2020).*

The liquid array printer in the Shared Materials Instrumentation Facility (SMIF) cleanroom bio bay was used to produce test kits for the COVID-19 virus.

Researchers at UNC are using the TEM to analyze nanoparticles being developed for drug / vaccine delivery.

Serving the Community

- A carload of SMIF gloves and face masks was delivered to Duke University Hospital to be used for PPE during the COVID-19 crisis.
- SMIF's 3D printer was used to create filter adapters for breathing equipment.
- Two new virtual programs were designed to reach K-gray participants in their homes, *Take-out Science* and *Sciencing with Abby*. In *Take-out Science*, RTNN live-streamed a scanning electron microscopy session and looked at a different type of sample weekly. During *Sciencing with Abby*, participants conducted nano-focused experiments with supplies in their homes, guided by staff member Abby Carbone.

CORE FACILITIES

The RTNN provides a broad foundation of core technical capabilities in nanotechnology fabrication and characterization. The RTNN also contributes unique expertise and facilities in the areas of "soft, wet" materials (e.g., textiles, plants and biological nanomaterials), heterogeneous integration and in situ characterization.

Analytical Instrumentation Facility (AIF) provides nano-characterization of both hard and soft materials and has over nine in situ stages (liquid cells, heating, mechanical loading, electrical biasing) for microscopy and diffraction.

Chapel Hill Analytical and Nanofabrication Laboratory (CHANL) offers standard and specialized nanofabrication and characterization capabilities including rapid prototyping of nano- and microstructures in a variety of substrate materials.

NC State Nanofabrication Facility (NNF) operates in a class 100/1000 cleanroom, which houses a comprehensive toolset for deposition, etching and patterning of nano- and micro-devices and structures with additional space dedicated to characterization.

Shared Materials Instrumentation Facility (SMIF) offers a comprehensive fabrication and characterization facility with unique cleanroom fabrication and characterization capabilities for research in bio / soft matter nanoscience, environmental nanotechnology, heterogeneous integration and metamaterials / plasmonics.

BY THE NUMBERS

- **3** major research universities
- **4** core shared user facilities
- More than **40,000** sq. ft. of laboratory space
- More than **230** major fabrication and characterization tools
- More than **28** technical staff members
- More than **40,000** annual hours of collective use

RTNN AFFILIATES NETWORK

Public Communication of Science and Technology (PCOST) improves public communication in science and technology, including emerging and converging technologies such as nanotechnology, bionanotechnology, neurotechnology, geoengineering and synthetic biology.

Molecular Education, Technology and Research Innovation Center (METRIC) provides researchers with world-class state-of-the-art measurement science facilities encompassing three key molecular characterization technologies including mass spectrometry, magnetic resonance and X-ray.

The NC State Nuclear Reactor Program (NRP) provides non-destructive testing and characterization of materials using neutron imaging, neutron powder diffraction, the intense positron beam and neutron activation elemental analysis.

The Duke Magnetic Resonance Spectroscopy Center (DMRSC) offers ultra-high-field NMR instruments with cryogenically cooled probes as well as conventional instruments.

The Zeis Textile Extension Education for Economic Development Center (ZTE) provides professional education and services such as hands-on training for all aspects of textile processing. The Analytical Services Laboratory offers comprehensive chemical analysis of nanofibers, dyes and other associated chemistries.

Chemical Research Instrumentation Teaching and Core Laboratories (CRITCL) supports research at all levels in the acquisition and analysis of data from state-of-the-art instrumentation. Core laboratories include Nuclear Magnetic Resonance, Mass Spectrometry, Small Molecule X-ray Diffraction, Electronics and Glass Shop.

The Nanomedicine Characterization Core Facility provides assistance in in-depth characterization of nanomaterials, including studies on: polymer chemical composition and structure, nanoparticle characterization, formulation characterization and nanoparticle preparation and isolation.

Capabilities of the RTI Analytical and Material Laboratories include energy-dispersive X-ray fluorescence, inductively coupled plasma mass spectrometry, inductively coupled plasma optical emission spectrometry, advanced surface area and porosity analysis, organic elemental analysis thermogravimetric analysis, chemisorption analysis and environmental chambers.

Specialized Equipment and Expertise

In addition to providing a strong foundation of fabrication and characterization facilities and capabilities, the RTNN offers highly specialized nanotechnology fabrication and characterization equipment and expertise. Representative examples of these unique capabilities include:

- Hot embosser
- Electrospinning of nanofibers
- High temperature furnaces for SiC
- Positron annihilation spectroscopy
- Small-angle X-ray scattering (SAXS)
- Extreme-resolution scanning EM (SEM)
- Vibrational microscopy
- In situ (heating, gas and liquid stages) transmission EM (TEM)
- Chemically-sensitive, atomic-resolution scanning TEM (S/TEM)
- Cryo-TEMs for biological and soft materials imaging and single particle analysis
- High-resolution NMR spectroscopy
- X-ray microscopy

NEW TOOLS AND CAPABILITIES

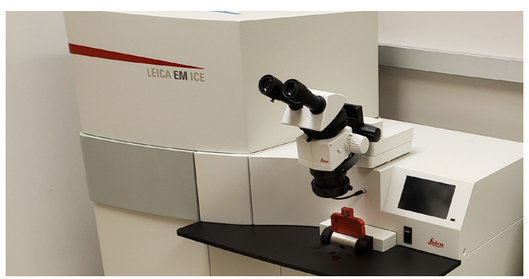
Through the support of our universities and by leveraging external funding proposals, the RTNN has acquired and / or upgraded >52 tools valued at >$15.4 million. The equipment was selected through discussions with users, principal investigators and long-term strategy of the facilities. In 2020, several new pieces of equipment were acquired:

- **The Hitachi TM4000 Tabletop SEM** provides a solution for SEM users to easily obtain high-quality data and quickly generates reports enabling a very efficient workflow.

Hitachi TM4000 Tabletop SEM

It accommodates sample sizes of up to 80 mm in diameter and 50 mm in thickness. This instrument was acquired to build on our successful outreach program.

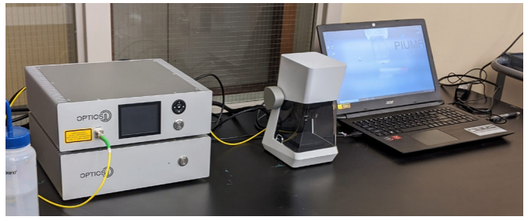
- **The Leica EM ICE** high-pressure freezing system enables users to preserve samples in a near native state and capture and resolve highly dynamic processes at the nanometer scale with millisecond precision. The system is equipped with light stimulation to enable photosensitive experiments. Once preserved, high-pressure frozen samples can be analyzed with cryo-EM. The Leica EM AFS2 freeze substitution system is able to progressively lower temperature for low temperature embedding and polymerization of resins. This is typically used in conjunction with high-pressure freezing.



Leica EM ICE High Pressure Freezing System

- **The PIPS II ion mill** features adjustable ion sources with voltages ranging from 100eV to 8keV, a liquid nitrogen cooled stage and a vacuum/inert atmosphere transfer capability for milling air-sensitive samples.

- **The Optics 11 Piuma system** is a nanoindenter designed specifically to make force measurements on biological samples in fluid to extract elastic modulus information. The system can operate in three different modes (displacement, load or indent) to provide different types of feedback during the experiment, and can also do dynamic experiments to determine viscoelastic properties of materials.



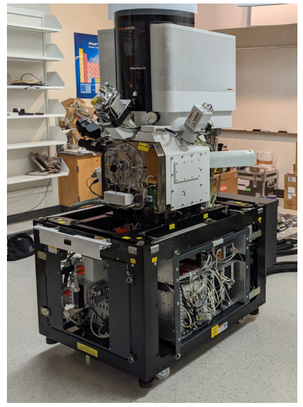
Optics 11 Piuma Nanoindenter

- **The ThermoScientific Talos F200X** scanning / transmission electron microscope (S/TEM) combines outstanding high-resolution S/TEM and TEM imaging with industry-leading energy dispersive X-ray spectroscopy (EDS) signal detection and 3D chemical characterization with compositional mapping. The Talos F200X S/TEM allows for the fastest and most precise EDS analysis in all dimensions (1D-4D), along with the best HRTEM imaging with fast navigation for dynamic microscopy. The Talos F200X S/TEM does all this while also providing the highest stability and longest uptime.

RTNN staff member Amar Kumbhar loads a sample in the Talos S/TEM.

- **The FEI EasyLift nanomanipulator system** allows precise cutting and removal for TEM sample preparation.

- **The Thermo Scientific Helios 5 Hydra DualBeam**, plasma focused ion beam scanning electron microscope, PFIB-SEM, (NSF MRI Award, PI Elizabeth Dickey, professor emerita in the NC State Department of Materials Science and Engineering) is equipped with multiple ion species.



Helios 5 Hydra DualBeam

The instrument enables and supports a diverse range of research projects and programs, within academia and industry, that involve three-dimensional, multi-modal characterization and patterning of microstructures. Such three-dimensional analysis is particularly important for developing materials processing strategies for additive manufacturing and for understanding mechanisms leading to material degradation and failure. In addition, the nano-patterning capabilities of the instrument provide precision engineering of novel nanometer-scale optical and mechanical devices.

The maximum current on the ion beam is 2.5μA providing greater removal rates for an increased throughput. This instrument also allows for polishing at 500V to greatly reduce damage in TEM samples. The Hydra FIB equips Multichem deposition of Pt, W or C and with the Thermo Easy Lift NanoManipulator for extraction and placement of TEM samples. The instrument features several detectors: In-lens SE/BSE detector (TLD-SE, TLD-BSE), in-column SE/BSE detector (ICD), Everhart-Thornlry SE detector (ETD), Ion Detector (ICE) for secondary ions (SI) and electrons and a retractable solid state backscatter detector (DBS). The instrument also has EBSD and EDS detectors.

- **The Hitachi HT7800 RuliTEM** is a 120 kV transmission electron microscope (TEM) that facilitates analysis in a wide range of fields, from biomedical research to soft materials science. The HT7800 has multiple lens configurations including a standard lens for unsurpassed high contrast and a class-leading HR lens for high resolution. The dual-mode objective lens supports easy observation under low magnification, wide-field high contrast, high resolution and more. Normal room light operation and automated functions allow both novice and experienced operators to use the system effectively. Advanced stage-navigation function enables whole-grid searching and efficient image acquisition.

Hitachi HT7800 RuliTEM

- A **Jackfish Spectrochemical cell fixture** has been acquired for one of our FTIR systems. This cell enables fundamental studies of the electrified metal-solution interface with applications in self-assembly, interfacial sensing and next generation energy solutions.

Jackfish Spectrochemical Cell Fixture

- **Axon software** integrates with the Titan and Talos TEMs to dramatically improve sample stability during in situ experiments. Axon tracks sample location and minimizes sample motion during sample heating, cooling, or when gasses or liquids are introduced in sample chamber.

- An **integrated RGA (residual gas analyzer)** is now attached to our Protochips Atmosphere system. The RGA enables quantitative control and examination of nanoscale reactions within the gas cell holder, and allows for improved control over gas chemistries introduced to the sample environment.

WHO USES RTNN?

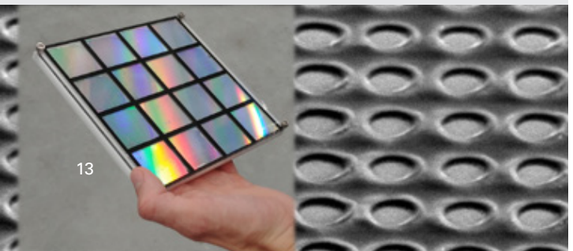
In 2020, more than 1,000 unique users accessed the facilities for over 40,000 hours. RTNN currently draws the majority of its users from the host institutions (76 percent), and greater than 85 percent of the use is on site. These users come from a broad range of disciplines including non-traditional disciplines such as the life sciences and medicine. Our impact on small businesses is tremendous. 58 percent of industry users are from companies with less than 50 employees.

Smart Material Solutions

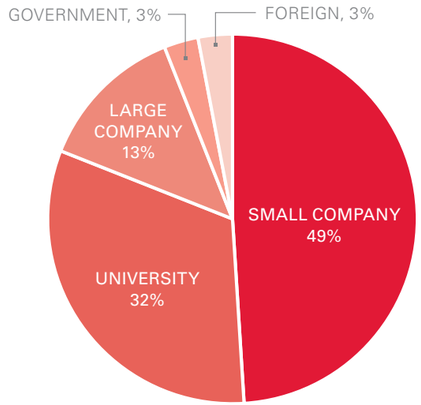
START-UP HIGHLIGHT

Initial Cohort of RTNN Kickstarter Projects in 2016:

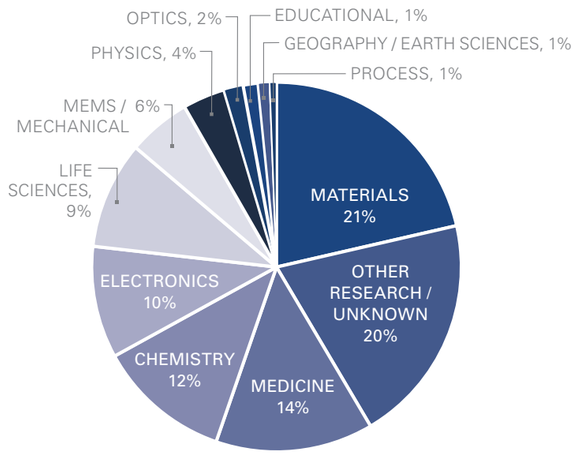
- Developed “nanocoining” process to create nanostructured surfaces
- Nanopatterns can manipulate light or change surface interactions, useful in a range of applications
- Received subsequent NSF SBIR (2017) and U.S. Army STTR (2019), SBIR (2020) funding
- First product launched in 2020: flexible micropatterned mold for nanoimprint lithography



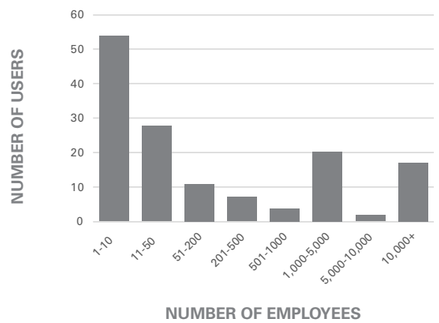
EXTERNAL USERS BY AFFILIATION



ALL USERS BY DISCIPLINE



2020 RTNN INDUSTRY USERS AS A FUNCTION OF COMPANY SIZE



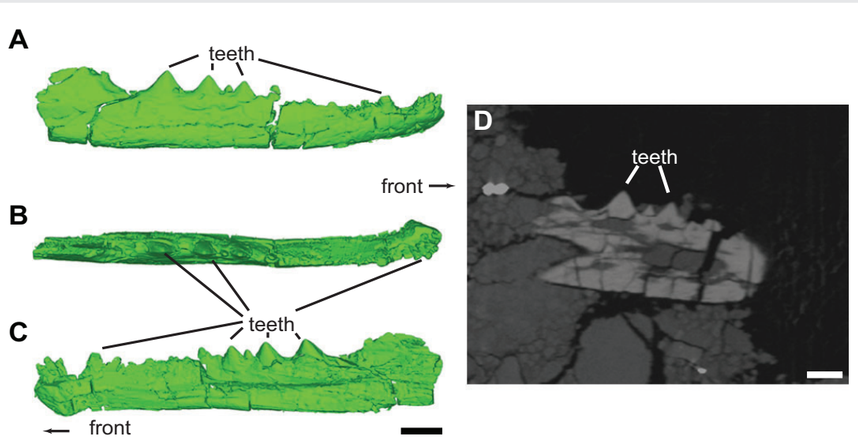
EXTERNAL USER HIGHLIGHT: VIRGINIA TECH

The rise of terrestrial vertebrates during the Triassic Period

After the end-Permian extinction, the largest extinction of all time, the planet fundamentally changed. Starting in the Triassic Period (252-200 million years ago [Ma]), terrestrial vertebrates transformed from the ‘alien’ (e.g., dicynodonts) to the more familiar (amphibians, mammal relatives and reptiles). However, chronicling this great transformation has been challenging because we lack robust anatomical information from these forms which inform their relationships, rates of evolution and their ecologies. In this long-term project, we target the members of Triassic faunas that have living members (e.g., mammals, birds, turtles, lissamphibians and squamates) as well as the Triassic forms that evolved and went extinct within the Triassic (e.g., phytosaurs, early dinosaurs). Computed tomography has revolutionized our ability to study tiny terrestrial vertebrates and delicate internal anatomy without removing fragile specimens from their encasing rock and to quantify morphology (e.g., tooth shape). We have already identified some of the world’s oldest amphibians, and we hope to understand the evolution of ecological shifts in reptiles

Publication: Wynd, B. M., S. J. Nesbitt, M. R. Stocker, and A. B. Heckert. A detailed description of Rugarhynchos sixmilensis gen. et comb. nov. (Archosauriformes, Proterochampsia) and cranial convergence in snout elongation across stem and crown archosaurs. J. Vertebr. Paleontol., 39 (2020).

PIs: Michelle Stocker and Sterling Nesbitt
Students: Chris Griffin, Candice Stefanic, Ben Kligman, Brenen Wynd, Kiersten Formoso
Virginia Tech Department of Geosciences



3D reconstructions from high quality CT data. A delicate and rare reptile jaw from the Triassic (in **(A),** lateral, **(B)**, occlusal, and **(C)** medial views) from CT data **(D)**. Scale bars = 1 mm

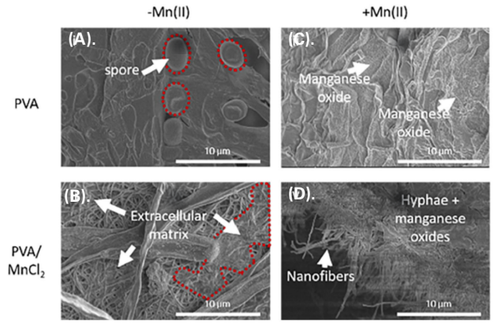
RESEARCH AND DEVELOPMENT HIGHLIGHTS

Biohybrid Nanofibers for Heavy Metal Removal from Water

Adsorption is an inexpensive and effective means to remove heavy metals from groundwater. In this work, polyvinyl alcohol (PVA) was electrospun into nanofibers containing $MnCl_2$. Magnanese-oxidizing fungi *(Coprinellus sp. and Coniothyrium sp.)* from a Superfund site water treatment system were immobilized onto the nanofiber surfaces. Upon attachment to manganese chloride–seeded nanofibers, fungi catalyzed the deposition of manganese oxide that served to adsorb heavy metal ions. These fungi–nanofiber hybrids removed heavy metals from groundwater.

Publication: Park, Y. et al. Biohybrid nanofibers containing manganese oxide-forming fungi for heavy metal removal from water. J. Eng. Fibers Fabr., 13 (2020)

Graduate Student:
Yaewon Park
PI: ***Ericka Ford***
Department of Textile Engineering, Chemistry and Science, NC State University



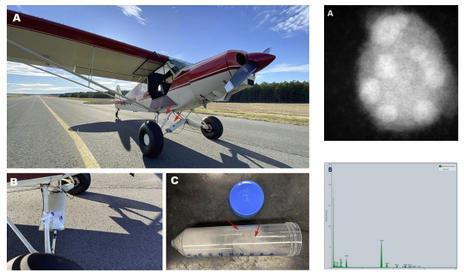
SEM images confirmed attachment of fungi to nanofibers **(A).** Presence of spores indicated that nanofibers did not inhibit fungal reproduction **(C).** *Coniothyrium sp.* catalyzed the growth of sheet-like manganese oxide particles in Mn(II)-containing media **(B).**, **(D).** Extracellular matrix appeared at the interface of nanofibers and hyphae (fungal cells)

Electron Microscopic Characterization of Exhaust Particles Containing Lead Dibromide Beads Expelled from Aircraft Burning Leaded Gasoline

Piston powered aircraft burning leaded gasoline contribute ~70 percent of the lead in the atmosphere in the U.S. The physical size, composition and structure of aircraft exhaust particles containing lead dibromide are poorly understood and heretofore have not been examined directly by electron microscopy (EM), in particular when captured from an aircraft in flight. To accomplish this, exhaust samples were trapped on EM supports within 10–15 ms of exiting the aircraft exhaust pipe. High angle annular detector dark field scanning EM revealed irregular particles with a mean diameter of 13 nm consisting of a 4 nm microcrystal of lead dibromide surrounded by a halo of hydrocarbons. In contrast, exhaust particles from an automobile burning leaded fuel averaged 35 nm in diameter and contained 5–10, 4 nm lead beads. Of significant concern, the smaller aircraft particles could penetrate mucosal barriers in the lung and be readily taken up by epithelial cells.

Publication: Griffith, J. Electron microscopic characterization of exhaust particles containing lead dibromide beads expelled from aircraft burning leaded gasoline. Atmos. Pollut. Res., 11 (2020).

PI: ***Jack D. Griffith***
Lineberger Comprehensive Cancer Center, Departments of Microbiology and Immunology, and Biochemistry and Biophysics, University of North Carolina at Chapel Hill



Left: Aircraft used in the study, arrows point to the location of the collecting tube **(B-C)** 50 ml tube with glass wool and EM supports (arrows).

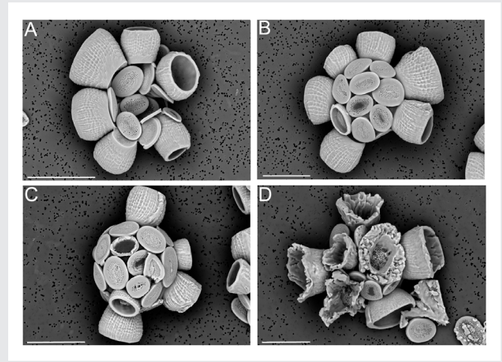
Right: (A) HAADF image of a single large exhaust particle from the motor vehicle exhaust collection is seen to be composed of many small lead dibromide microcrystal beads. **(B)** EDX spectra obtained from the exhaust particles detects presence of lead (Pb) and bromine (Br).

Using Analytical SEM to Investigate Trace Element Incorporation During Biomineralization of the Alga *S. apsteinii*

Coccolithophores are bloom-forming, unicellular marine phytoplankton that are the most significant producers of biogenic calcite in the world's oceans through the production of small CaCO3 plates known as coccoliths. The goal of this study was to investigate Sr incorporation in Scyphosphaera apsteinii coccoliths. This species exhibits an order of magnitude higher coccolith Sr/Ca ratios and partitioning coefficients (DSr) when compared to any other coccolithophore species. The RTNN users hypothesized that this unusually elevated Sr/Ca ratio indicates a functional role of Sr in biomineralization for this species. They grew cells in a range of Sr concentrations and analyzed the effects on coccolith morphology (SEM) and calcite DSr (SEM-EDS) (Figure 37) using AIF. Coccolithophores grown in deplete Sr/Ca did not exhibit disrupted calcification, but those grown in higher than ambient Sr/Ca conditions displayed a significantly higher frequency of malformed or aberrant coccolith morphologies. This implies that Sr is not necessary for normal coccolith morphology in S. apsteinii, but Sr can disrupt mineralization. The investigators propose that differential Sr- and Ca-binding capacities of associated organics and a less selective Ca2+ transport pathway may account for the unusually high Sr incorporation into the calcite coccoliths of this species. Understanding how the biomineralization process affects trace metal incorporation among species is important because the trace element composition of microfossil coccoliths sampled from marine sediments is used to develop paleoproxies for reconstructing past physiochemical properties of the surface oceans.

Publication: Meyer, E.M. et al. Sr in coccoliths of Scyphosphaera apsteinii: Partitioning behavior and role in coccolith morphogenesis. Geochimica et Cosmochimica Acta. 285 (2020).

Graduate student:
Erin Meyer
PI: ***Alison Taylor***
Department of Biology and Marine Biology, University of North Carolina at Wilmington



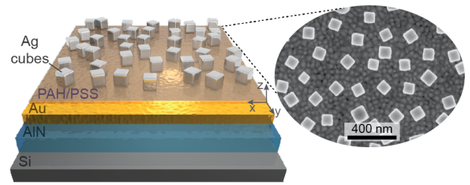
SEM images acquired with an electron backscatter detector showing the effects of **(A)** deplete, **(B)** ambient, and **(C, D)** high (36 mmol/mol Sr/Ca and 72 mmol/mol Sr/Ca, respectively) Sr on *S. apsteinii* coccolith morphology. Scale bars represent 20 µm.

Ultrafast Pyroelectric Photodetection with On-Chip Spectral Filters

Thermal detectors are uniquely capable of sensing incident radiation for any electromagnetic frequency though response times are typically on the millisecond scale. By combining a pyroelectric thermal detector with wavelength-selective nanoparticle absorbers, researchers achieve ultrafast response of a pyroelectric sensor with near-infrared responsivity. On-chip spectral filters are realized through control of nanoparticle size.

Publication: Stewart, J. et al. Ultrafast Pyroelectric Photodetection with On-Chip Spectral Filters. Nat. Mater., 19 (2020).

Graduate student:
Jon Stewart
PI: ***Maiken H. Mikkelsen***
Department of Electrical and Computer Engineering, Duke University

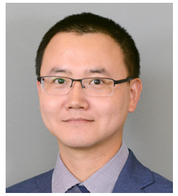


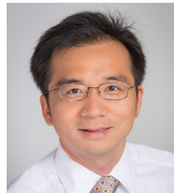
Left: Schematic of the vertical detector structure with a plasmonic metasurface deposited on a polycrystalline AlN pyroelectric layer. **Right:** An SEM image of the fabricated metasurface with 100 nm Ag cubes.

Acoustofluidic Synthesis of Particulate Nanomaterials

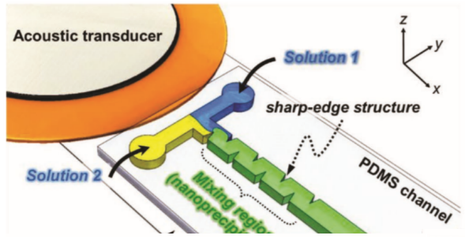
In this work, an acoustofluidic platform was used to synthesize nanoparticles and nanomaterials in a controllable, reproducible manner through acoustic-streaming-based active mixing of reagents. This process allows for the dynamic control of reaction conditions by adjusting the strength of the acoustic streaming. The work demonstrated the synthesis of diverse nanoparticles / nanomaterials: polymeric nanoparticles, chitosan nanoparticles, organic–inorganic hybrid nanomaterials, metal–organic framework biocomposites and lipid-DNA complexes. Platform flexibility in establishing various reaction conditions enables the synthesis of versatile nanoparticles and nanomaterials with prescribed properties.

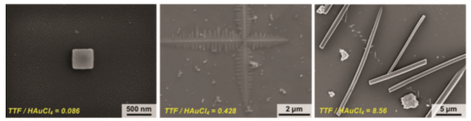
Publication: Huang, P. et al. Acoustofluidic Synthesis of Particulate Nanomaterials. Adv. Sci., 6, 365 (2019).





Post-doc:
Nanjing Hao
*PI: **Tony Jun Huang***
Department of Mechanical Engineering and Materials Science, Duke University





Top: Schematic of acoustofluidic synthesis device composed of a microfluidic channel with multiple pairs of sharp-edge structures. When flowing through the channel, Solutions 1 and 2 are rapidly mixed in the presence of the acoustic streaming effect. **Bottom:** SEM images of nanohybrids synthesized by mixing tetrathiafulvalene (TTF) and $HAuCl_4$ solutions at different flow rate ratios.

Ratcheting Quasi-ballistic Electrons

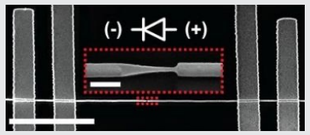
Ratcheting effects play an important role in systems ranging from mechanical socket wrenches to biological motor proteins. In this work, a semiconductor nanowire with precisely engineered asymmetry was capable of ratcheting electrons at room temperature.

Modulation of the nanowire diameter creates a cylindrical sawtooth geometry on a nanometer-length scale. This structure funnels electrons preferentially in one direction through specular reflection of quasi-ballistic electrons at the nanowire surface. The ratcheting effect causes charge rectification at frequencies exceeding 40 gigahertz, demonstrating the potential for applications such as high-speed data processing and long-wavelength energy harvesting.

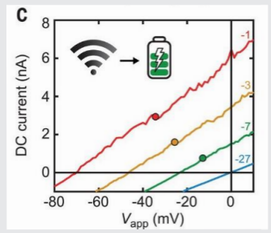
Publication: Custer, J. et al. Ratcheting quasi-ballistic electrons in silicon geometric diodes at room temperature. Science, 368 (2020).

Graduate student:
James Custer
*PI: **Jim Cahoon***
Department of Chemistry, University of North Carolina at Chapel Hill

SEM image of a nanowire with three geometric diodes in series (Scale bar = 200 nm).



SEM image of a single nanowire device with electrical contacts (scale bar = 5 µm). Inset shows geometric diode and circuit diagram (scale bar = 250 nm).

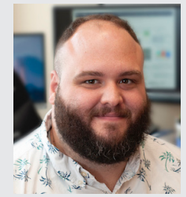


I-V response of a geometric diode with 5.2-GHz ac applied at varying powers.

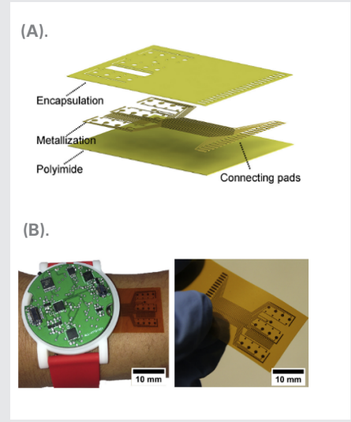
Wearable Multiplexed Biosensor System Toward Continuous Monitoring of Metabolites

Comprehensive metabolic panels are the most reliable and common methods for monitoring general physiology in clinical healthcare. Translation of this clinical practice to personal health and wellness tracking requires reliable, non-invasive, miniaturized, ambulatory and inexpensive systems for continuous measurement of biochemical analytes. In this work, researchers designed and characterized a wearable system with a flexible sensor array for non-invasive and continuous monitoring of human biochemistry. The system includes signal conditioning, processing and transmission parts for continuous measurement of glucose, lactate, pH and temperature. The operation of the system is demonstrated in vitro by simultaneous measurement of glucose and lactate, pH and skin temperature. This miniaturized wearable system enables future evaluation of temporal changes of the sweat biomarkers.

Publication: Yokus, MA. et al. Wearable Multiplexed Biosensor System Toward Continuous Monitoring of Metabolites. Biosens. Bioelectron., 153 (2020).



Graduate student:
Murat Yokus
*PI: **Michael Daniele***
Joint UNC / NC State Department of Biomedical Engineering



(A) Exploded view of the flexible sensor array showing the polyimide (bottom), metallization (intermediate), and encapsulation (top) layers. **(B)** Left: Picture of wrist-worn, wearable multiplexing system in a 3D printed watch enclosure for demonstration purposes only. Right: Picture of the flexible sensor array.

Collaborative Research Award

The 2020 recipients of the Collaborative Research Award were **Carrie Donley** and **Fred Stevie** for their work in developing educational resources for X-Ray Photoelectron Spectroscopy (XPS). Recently, a few publications have illustrated that a large percentage of research papers that include XPS data have seriously flawed interpretation of the data. As a response to this crisis in the field, the American Vacuum Society (AVS) sponsored a collection of publications to improve the overall understanding of XPS. Donley and Stevie prepared two papers for the AVS collection. These papers will not only help RTNN researchers but the larger XPS community as well.

Publications: Stevie, F. and C. Donley. Introduction to X-Ray Photoelectron Spectroscopy (XPS). J. Vac. Sci. Technol., A38 (2020). Stevie, F. et al. Sample handling, preparation and mounting for XPS and other surface analytical techniques. J. Vac. Sci. Technol., A38 (2020).

Carrie Donley

Fred Stevie

BUILDING THE USER BASE

The overarching goal of the RTNN is to build the user base. The RTNN has identified three barriers to engaging new users:

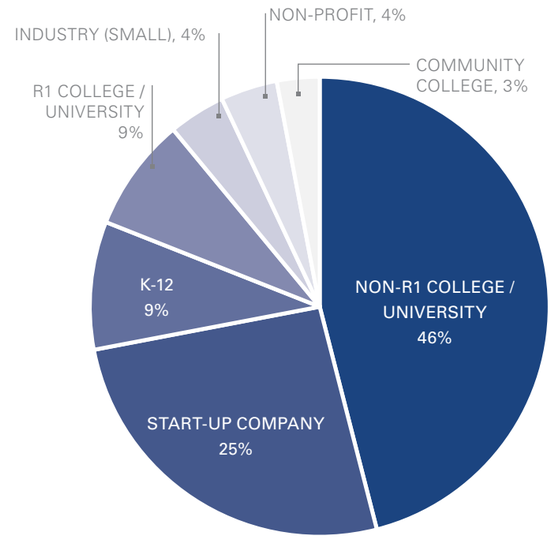
- **Knowledge** of the existence of the facilities and how to access them;
- **Distance** to travel to the facilities;
- **Cost** of accessing the facilities.

To address these barriers, the RTNN has implemented targeted, innovative new programs and activities and strengthened existing ones to attract and retain new and current users.

Kickstarter Program

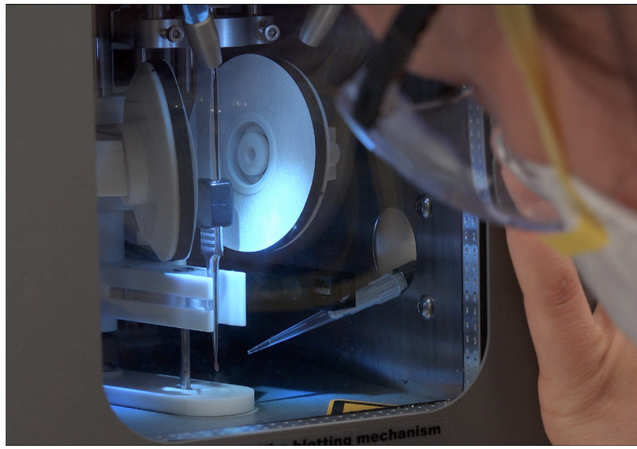
This program supports initial use of RTNN nanotechnology facilities by new, non-traditional users by providing **free access to facilities** for work valued at up to $1,000. Proposed projects must be focused on research, preliminary development activity or educational programming in an area of nano-scale science, engineering and/or technology. Seventy-five projects have been selected for the Kickstarter program, receiving over 1,300 hours in facility use. The majority of participants hail from non-R1 colleges / universities and start-ups. **Thirty-six percent of participants in this program have continued to use facilities with their own financial support leading to an additional $200,000 in revenue to the facilities**. In addition, several participants have indicated that they are using the data from their projects to support research proposals and include RTNN facility usage in the proposed work.

KICKSTARTER PARTICIPANTS



Nanotechnology: A Maker's Course

RTNN developed a free online course to give an overview of nanotechnology tools and techniques and demonstrate equipment within RTNN facilities. The goal of the course is to introduce nanotechnology concepts to students and give them a better sense of the various tools' capabilities. The course includes eight modules, each focused on a different fabrication or characterization concept. Students first learn the science behind a specific technique or instrument. The lectures make the information accessible to a large audience, using simple language and relatable analogies to everyday things. In-lab demonstrations of the equipment follow each lecture with an explanation of each step in the process. **Since the course launched in September 2017, >184,000 people have visited the course site and >39,000 have enrolled in the course**. The course has attracted learners from more than 170 countries across the world garnering scores of positive reviews. During the pandemic, there was a significant bump in enrollment, as the demand for virtual educational resources increased. ***coursera.org/learn/nanotechnology***



Demonstration of sample preparation for cryo-TEM in *Nanotechnology: A Maker's Course*

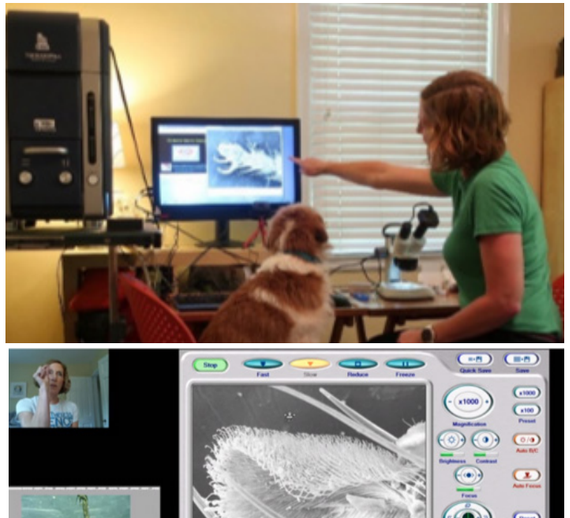
K-GRAY ENGAGEMENT

» **> 900 people reached in person**

» **> 19,700 people reached remotely**

Take-out Science

During the COVID-19 pandemic, the RTNN designed a program to connect our facilities to people at home and bring them *Take-out Science*. Staff member Holly Leddy set up one of our portable scanning electron microscopes (SEMs) in her guest bedroom. During the spring of 2020, the RTNN live-streamed a new show weekly focused on a different theme. Leddy guided participants through observation of samples related to that theme with both a light microscope and an SEM. She also described how the two instruments worked and compared their capabilities. To engage participants, in each episode, the RTNN teased the following week's theme and invited people to guess it on RTNN's website and social media pages. Correct guesses were highlighted during the live show. With colleagues at Hitachi, RTNN began to add Spanish subtitles to each episode. To date, the show has **generated >4,800 views on YouTube over 14 episodes**.



Top: RTNN staff member, Holly Leddy, is assisted by June "The Science Dog" during *Take-out Science* from her guest bedroom. Below: YouTube live screen showing Leddy, the light microscope view, and the SEM view.

Sciencing with Abby

In this video series, also launched during the pandemic, kids (and adults) are invited to conduct science experiments with the RTNN using supplies they can find at home. Each experiment has two levels: beginner videos are aimed at K-5 audiences and advanced at grades 6-12. Videos are hosted by Abby Carbone, a recent NC State graduate and former AIF staff member. Carbone's videos have **received over 1,100 views on YouTube**.

Girls STEM Day

Annually, Girl Scouts and their families traveled to Duke University in May 2020 to learn from and work with women in STEM careers across the Triangle. In 2020, the event shifted to a virtual setting in November, Girl Scouts STEM Power Hour, with **124 girls participating**. STEM kits were mailed to girls to enable them to participate in hands-on activities like extracting DNA and observing a variety of insects with the SEM.

Kids follow Abby Carbone's instructions to paint with food coloring in milk during an episode of *Sciencing with Abby.*

Visits to RTNN Facilities

Before the pandemic, over **150 K-12 students** had the opportunity to visit the facilities to learn the size of a nanometer and discover real-world nanotechnology applications, see the equipment in action and participate in fun hands-on activities.

Visits to Classrooms

RTNN staff also traveled to many K-12 classrooms and schools to introduce nanotechnology, interacting with over **150 students**. These visits were paired with hands-on activities to engage students. RTNN staff members often travel with a portable, desktop scannng electron microscope (SEM) making it possible to take facilities to the classroom. The desktop SEM is user-friendly and approachable. Students can begin using it right away without complex and lengthy training sessions.

Rural Outreach

The RTNN has expanded outreach efforts with pilot events in rural areas of North Carolina including Fayetteville (70 miles from the Triangle), Hickory (160 miles) and Asheville (230 miles). These multi-day events split time between schools / community colleges and a public space (e.g., library) to reach a broad swath of the community. To support these events, the RTNN partners with NNCI colleagues at the Joint School of Nanoscience and Nanoengineering (JSNN). The pilot events engaged over **800 K-gray participants**.

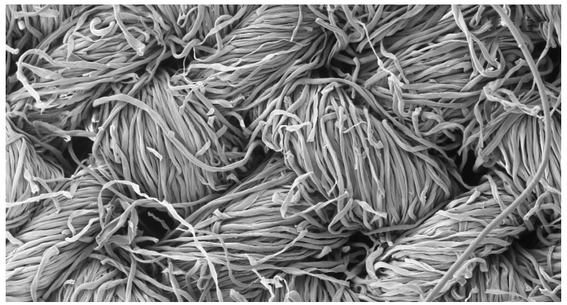
Community College Engagement

RTNN staff members also worked with Durham Technical Community College faculty members to incorporate the SEM into engineering, physics and biology coursework. This included both a lecture and hands-on component with the portable SEM.

Remote Use of Facilities

Users can access nano-facilities remotely with the assistance and expertise of RTNN students and staff. This capability was critical when many facilities and schools were closed due to the COVID-19 pandemic. Fabrication and / or characterization are performed on site and

JSNN graduate student Klint Davis demonstrates the process of photolithography to patrons at Pack Memorial Library in Asheville, NC.



SEM image of a woven fabric taken during a remote session.

RTNN Student Ambassador Abby Carbone describes use of the SEM to visitors at the Catawba Science Center in Hickory, NC.

streamed live to the remote users.

Research Experience for Teachers (RET)

Due to the COVID-19 pandemic, the *Atomic Scale Design and Engineering RET Program* had to be canceled. The 2019 participants were surveyed to determine how the program impacted their teaching. The teachers' reflections demonstrate the positive and lasting impact of the program on the teachers' professional development. The teachers appreciated the opportunity to participate in research and recognized the importance such experiences can have on their classroom instruction.

Technical Workshops and Short Courses

The RTNN hosts training and technical workshops at member institutions. These workshops are provided at low cost to internal and external users. They provide technical and / or educational training on nano-fabrication and / or – characterization equipment and techniques. This year, RTNN held **11 in-person workshops and short courses** with **over 82 participants** and **20 virtual workshops and short courses** with **>200 participants.**

Attendees at the In Situ Microscopy Congress, an RTNN workshop organized with industry sponsors Protochips and ThermoFisher Scientific.

RTNN Student Ambassadors Program

The RTNN Student Ambassadors Program continued this year to engage students in the RTNN's mission to bring nanotechnology tools and expertise to new researchers and the public. Ambassadors represent the RTNN to visitors and program participants, increase awareness of the RTNN and extend outreach activities. RTNN ambassadors traveled to local and rural schools and museums, guided tours and got people excited about nanotechnology!

Student Outreach Awards

This award recognizes students who have dedicated their time to support RTNN education, outreach and engagement activities. This award shows our appreciation to the energy that these students have devoted to bringing in a future generation of users. RTNN Student Ambassadors **Jessica Chestnut** (NC State), **Anna Fraser** (UNC) and **Beatriz Medrano** (Duke) were the 2020 recipients of this award.

Jessica Chestnut

Anna Fraser

Beatriz Medrano

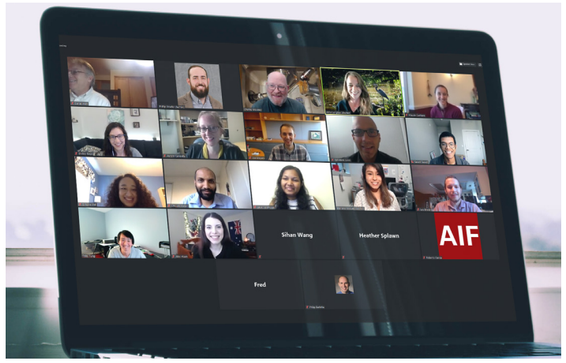
NANOTECHNOLOGY LEADERSHIP IN THE TRIANGLE

The RTNN engages with the community to promote nanotechnology in the Triangle. RTNN staff work with community college educators and other local educators to help them to incorporate nanotechnology concepts and equipment into the classroom. The RTNN has partnered with Morehead to distribute nanotechnology educational activities to K-12 schools across the state. The RTNN is engaged with North Carolina Girl Scouts, Science Olympiad and The Science House to strengthen our ability to reach remote parts of our state.

The RTNN connects researchers on emerging scientific topics at the forefront of nanotechnology. In addition, we actively seek out opportunities to strengthen and broaden our capabilities including proposals for Research Experience for Undergraduates and Major Research Instrumentation at all three institutions. The RTNN Research Experiences for Teachers (RET) Program resulted from one of these efforts.

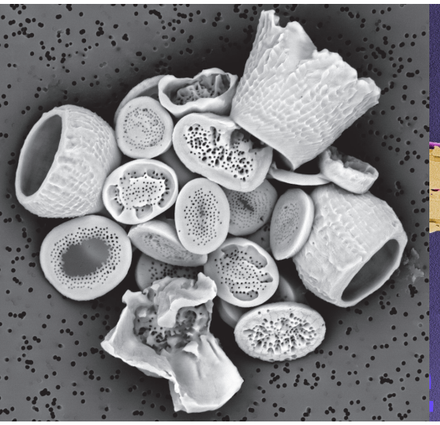
The RTNN has also organized multiple events at the Chapel Hill Public Library and the Museum of Life and Science. These events are widely publicized by the library / museum but also provide a means to reach populations who may be patronizing the venue for other reasons. At these events, the RTNN brings the portable scanning electron microscope (SEM) and other hands-on activities to attract library / museum visitors.

In honor of National Nanotechnology Day on October 9, the RTNN hosted a special *Take-out Science* session. During this event, RTNN staff gave a brief introduction to length scales and nanotechnology, took a trip to the clean room for a fashion show and observed various samples on our SEM. Leading up to this event, the RTNN hosted an image contest, *There's Plenty of Beauty at the Bottom*. The winning images represented work conducted at our facilities and are featured below.

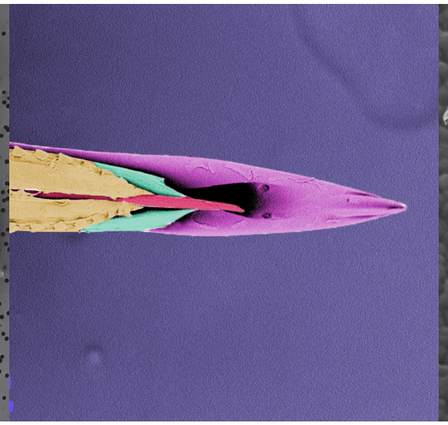


Student award winners at the virtual Carolina Science Symposium.

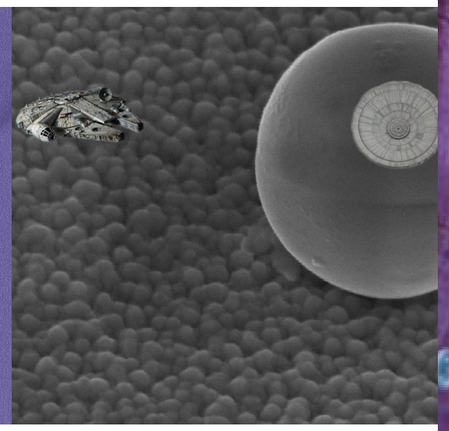
Each fall, the RTNN helps to organize the Carolina Science Symposium. This student-focused conference gives early career students their first opportunity to present their work in a professional setting. This year the symposium transitioned to a virtual meeting. There were four, two-hour sessions over two days. The new format enabled a greater number of student presentations and was well received by the more than 100 attendees.



Erin Meyer



Kun Luan



Phil Barletta

NATIONAL IMPACT AND ENGAGEMENT

The RTNN is actively involved in NNCI working groups and committees:

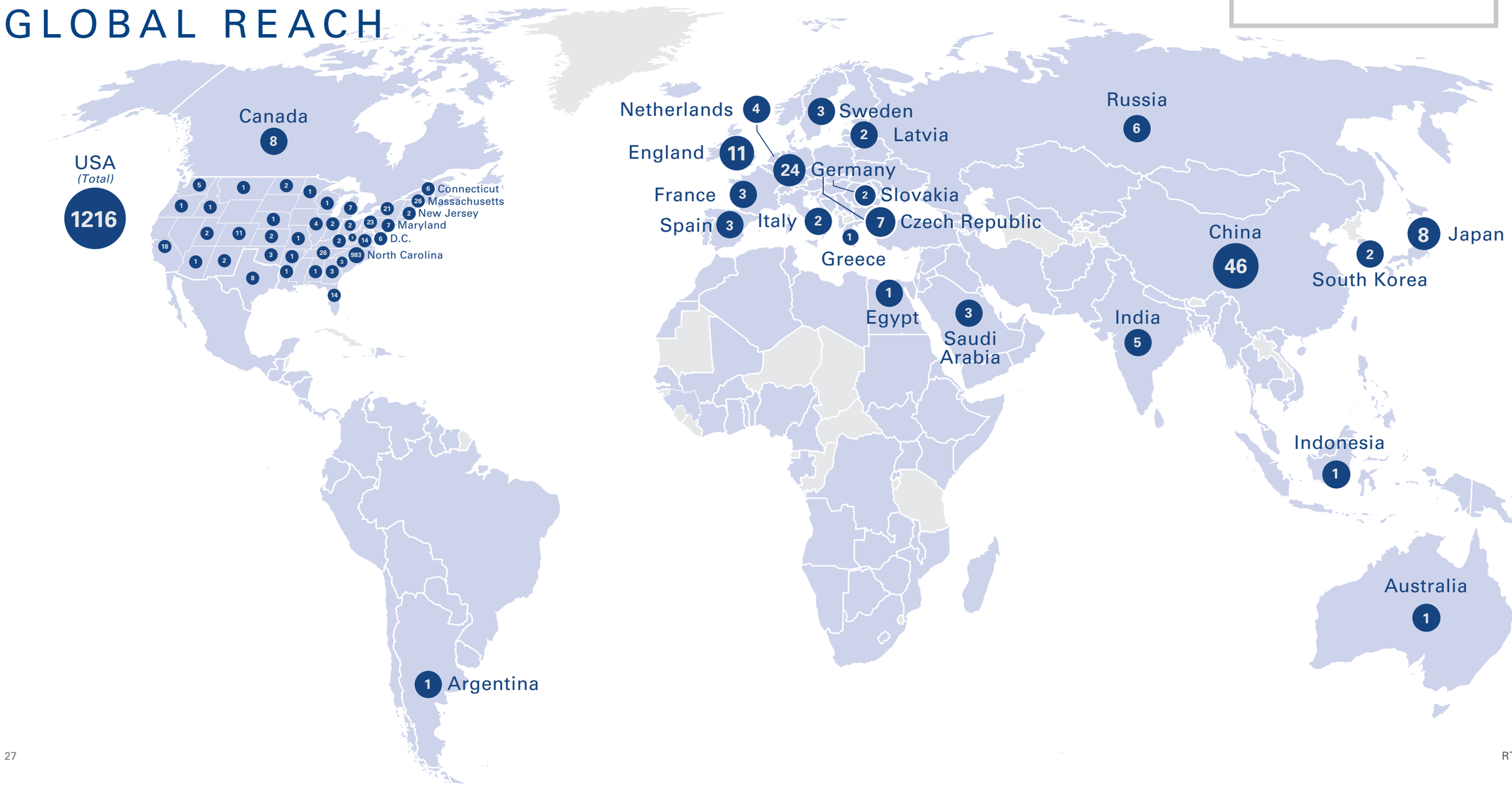
- Leadership of diversity subcommittee
- Membership in K-12 education and outreach, workforce training, online learning, environmental health and safety, EBL and XPS working groups

In 2020, RTNN had winners in all three categories of the NNCI Awards: Carrie Donley (CHANL) won in the category of User Support, Justin Gladman (SMIF) won in Technical Staff and Nicole Hedges (NNF) won in Education and Outreach. These awards acknowledge the leading role RTNN plays amongst its NNCI colleagues.

RTNN staff attend diverse national events and conferences, including education and research-focused conferences, to raise awareness of RTNN and NNCI's educational and technical programs. This year RTNN attended the MNT-eSIG Live 2020 conference and the NC Science Education Summit.

The RTNN continues to grow internationally. Nanotechnology: A Maker's Course draws learners from around the world, and some of these learners have begun to use RTNN programs and facilities. In addition, many publications acknowledging RTNN facilities have international authors (*Note: authors listed on multiple publications are counted for each publication in graphic).*

GLOBAL REACH



NANOTECHNOLOGY OUTCOMES

OUTCOME NUMBERS

$58.9M
in research activity, as defined by annual research expenditures, for projects that utilized the facilities

$338.9M
in research activity, as defined by the total contract value, for projects that accessed the facilities

154 Degrees earned

213 Publications that cite RTNN Facilities

53 Patents awarded

157 Patents filed

56 Invention disclosures

RTNN user satisfaction and programming are assessed regularly by David Berube's Social and Ethical Implications of Nanotechnology (SEIN) team within PCOST (Public Communication of Science and Technology).

Kickstarter Program Feedback

To assess the Kickstarter program, semi-structured interviews have been conducted with 24 participants. The feedback from participants in the Kickstarter program was overwhelmingly positive. Respondents were happy with the overall program and indicated they will return to the facilities. **A common theme from respondents was gratitude for RTNN staff**. Many staff members were thanked by name.

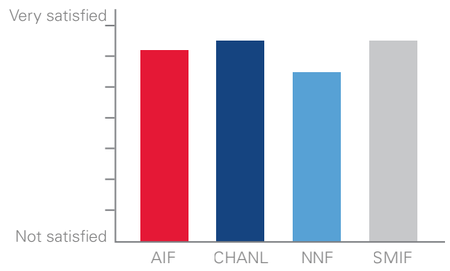
"[The staff] were very helpful. Every time I needed something, I asked them, and they were ready to help."

Nanotechnology: A Maker's Course

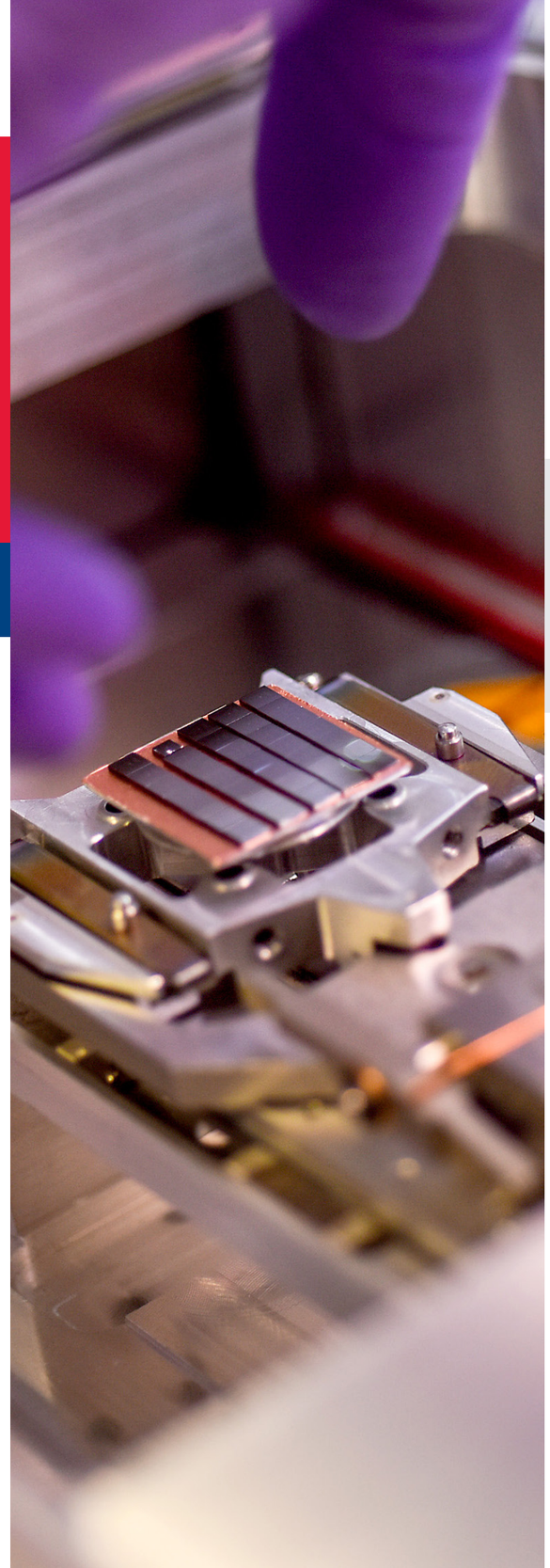
After completing the course, students receive a survey through the Coursera platform. Overall, students are very satisfied with the course. **Over 89 percent of respondents** noted that they were *likely* or *very likely* to recommend the course to others. **79 percent of respondents** noted that they had a better knowledge of the capabilities of RTNN's facilities.

User Assessment: Satisfaction

All users received an online survey to obtain demographic and satisfaction data. Facility users were *very satisfied* in the facility they used, and facility satisfaction was consistent between RTNN's four core facilities. **Greater than 97 percent of users indicated that they would return to the lab** if further work was necessary. The RTNN began to see significant differences comparing user discipline. For example, users in more non-traditional disciplines (e.g., agriculture, geosciences) are more satisfied than users from traditional disciplines like physics. Berube and colleagues recently published their findings. The Social and Ethical Implications team is investigating the source to determine why different users experience the facilities differently.



Publication: Berube, D. et al. Social science and infrastructure networks and the human-technology interface. J. Nanopart. Res. 22 (2020).



GETTING STARTED WITH RTNN

Connect with Experts For:

- Training to independently operate equipment
- Fabrication and analytical services
- Consultation, collaboration and support for process and instrumentation development
- Interactive educational opportunities for students
- Continuing education programming in nanotechnology

Contact Us

Drop us a line	**rtnanonetwork@ncsu.edu**
Give us a call	**919.515.6171**
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facebook.com/RTNNSocial

Reach Out to our Directors

Jacob Jones
RTNN Director and NC State Site Director, jacobjones@ncsu.edu
Nan Jokerst
Duke Site Director, nan.jokerst@duke.edu
Jim Cahoon
UNC-CH Site Director, jfcahoon@unc.edu
David Berube
SEIN and Assessment Coordinator, dmberube@ncsu.edu

Research Triangle Nanotechnology Network
Monteith Research Center
Campus Box 7531, Room 246 | 2410 Campus Shore Dr.
Raleigh, NC 27695-7531
919.515.6171 | rtnanonetwork@ncsu.edu

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