

FY20 Annual Report (July 1, 2019 to June 30, 2020) Analytical Instrumentation Facility (AIF) NC State University

### **Mission and Capabilities**

The AIF is NC State's primary shared facility for materials characterization with a mission to enable and lead state-of-the-art research through acquisition, development, maintenance, training, and access to major analytical and materials characterization instrumentation. Through the support of engaged faculty and experienced staff, the AIF supports state-of-the-art scanning and transmission electron microscopes, X-ray scattering and spectroscopy instruments, mass and electron spectrometry, scanning probe and Raman microscopy, laser scanning microscopy, nano-CT system, nanoindentation, freeze



fracture system, high pressure freezer system and extensive sample preparation facilities. Some of the extraordinary capabilities of these instruments include chemically-sensitive atomic-scale imaging, extremeresolution SEM of insulating and soft materials, *in situ* high temperature and electric-field-dependent Xray diffraction, cryogenic SEM of biological and soft materials, low-temperature TEM and *in situ* microscopy during heating, electrical current, immersion in fluids, and in flowing gases (to name a few). The AIF is positioned within the College of Engineering and is named an NC State Core Facility by the Office of Research and Innovation (ORI).

#### Usage

In FY20, the AIF was accessed by 157 NC State Principal Investigators (PIs) at NC State and 395 users from those groups, who are mostly students and postdocs. These individuals come from the Colleges of Agriculture and Life Sciences, Natural Resources, Engineering, Sciences, Textiles, and Veterinary Medicine. In addition, the AIF supported materials characterization services for 99 unique external government, industrial, and other academic researchers through managing 330 active contracts (451 contracts in FY19, 301 in FY18, and 256 in FY 17). Overall, the AIF provided 15,912 lab user hours in FY20, and the decrease due to prior years is attributed to the shutdown during the COVID-19 pandemic. ~20.5% of AIF users are external to the university, demonstrating our service to researchers from North Carolina and around the world.



# Distribution of the 15,9121 hours across the AIF laboratories





## **Education and Engagement**

The AIF is heavily involved in student training/education and engagement with the community. During FY20, the AIF offered 53 training workshops, short courses, and other major in-lab events for hands-on training and learning on topics including vacuum technology, SEM, TEM, XRD, surface analysis, and sample preparation. AIF staff members helped to organize the annual Carolina Science Symposium in November of 2019, an event that attracted over 104 researchers, and 22 vendors. In March 2020, the AIF, Protochips and Thermo Fisher Scientific co-hosted a two-day "In Situ Microscopy Congress." ~50 attendees were from NC State, Ohio State University, Virginia Tech, NC A&T, University of Pittsburgh, Clemson University, the University of Florida, and several local companies. Numerous talks (National Institute of Standards and Technology, Lawrence Berkeley National Lab, etc.) and demonstrations on in situ liquid cell and gas TEM/STEM were provided, including details on atomic-scale dynamic imaging with chemical analysis to probe gas-solid reactions. The demonstrations included examples of application of in situ X-ray diffraction experiment on the PANalytical instrument with an Anton Par heating stage. The AIF also contributes to numerous NC State for-credit courses through laboratory demonstrations and hands-on exercises for both undergraduate and graduate students. In FY20, the AIF gave over 56 tours for visitors, industry researchers, and classrooms. The AIF engages with and enhances the missions of other research centers on campus to provide short courses and workshops specialized for those constituencies, and proposal development to support such activities. Some examples include Vacuum Technology, XRD, SEM, and FIB short courses. Starting in late March and throughout the stay-at-home orders during the COVID-19 pandemic, each lab manager held weekly online office hours to answer general user questions (calendars of Lab Managers' office hours). AIF also launched several online introduction courses via Zoom video conferencing and posted on the a newly created AIF YouTube channel. New online videos were created in this channel including TEM, ToF-SIMS, SEM, AFM, FIB, and Nanoindentation Technique courses.







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## **New Instrumentation and Upgrades**

The AIF acquired several new instruments. In April 2020, a new Hitachi TM4000 Tabletop SEM was delivered. The TM4000 Series provides a solution for SEM users to easily obtain high-quality data and quickly generates reports enabling a very efficient workflow. It accommodates sample sizes of up to 80 mm in diameter and 50 mm in thickness. AIF also is home to a new Leica High Pressure Freezer-Automated Freeze Substitution (HPF-AFS) System, acquired through support from the Office of Research and Innovation (ORI). The system allows native ultrastructures and architectures to be preserved and maintained by freezing rapidly at very low temperature under high pressure conditions. Once preserved, high-pressure-frozen samples can be analyzed with AIF cryo-EM; The addition of the HPF-AFS system helps NC State become a leader in bio-EM in our region. In FY20, the AIF also acquired the AXON Synchronicity system from Protochips which includes the hardware and software necessary to stabilize the imaging of materials during in situ experiments on the Talos TEM instrument.



Hitachi Tabletop SEM



Leica High Pressure Freezer - Automated Freeze Substitution System

# Assessment

AIF users are annually surveyed by an external evaluator (as part of the RTNN project), allowing us to determine the level of satisfaction of AIF users with the facility. Notably, 97.7% of users would come back again for future work (N=132). 93% of users are satisfied or very satisfied with AIF (N=129) with similar satisfaction levels for staff and physical facilities. We acknowledge Prof. David Berube for the assessment.

# **Organizational Innovation**

In FY20, we enhanced our *communications with our customers and stakeholders*, including the release of a *newly created YouTube Channel* (link here to subscribe) and dissemination of our AIF Newsletter (link here to access recent newsletters). The AIF newsletter is now sent to over 3,500 individuals monthly. In FY 20, we have organized two Bio-imaging users group meetings in October 2019 (in-person) and April 2020 (via video conference). The first meeting attendants came from 10 different NC State Colleges, including the College of Agriculture and Life Sciences (CALS; 35.3%), College of Engineering (COE; 26.5%), College of Sciences (COS; 17.6%), and College of Veterinary Medicine (CVM; 14.7%). The goal for these meetings was to learn more about AIF's Bio-Imaging capabilities and to assess short and longterm instrumentation needs for this research community. Each month, AIF features a "User Spotlight," where selected users' backgrounds and projects are introduced and primary instrument(s) they are using for the research are discussed. In FY2020, Chris Ledford (PhD student in Industrial and Systems Engineering at the Center for Additive Manufacturing and Logistics (CAMAL), working under Prof. Tim Horn), Rachel Nye, Jung Sik Kim and Sarah Smith (PhD students in Chemical Engineering, working under Prof. Gregory Parsons), Prof. Larry Blanton (Professor of Plant Biology in NC State's Department of Plant & Microbial Biology), Ruocun (John) Wang (PhD student in Materials Science and Engineering, working under Prof. Veronica Augustyn), Laura Dalton (PhD student in Civil, Construction, and Environmental Engineering Department, working under Prof. Mohammad Pour-Ghaz) were featured. These individuals were from diverse disciplinary backgrounds and career trajectories and are brought together through utilizing instruments at AIF.



#### **Research Outputs and Impact**

The AIF was authorized to work on 186 unique 5account projects in FY20, representing >\$258M in research awards on campus. Users of the AIF published over >154 peer-reviewed technical publications (full list here) in the calendar year 2019 (virtually consistent with 152 in 2018, 158 in 2017, up from 120 in 2016, 93 in 2015, 68 in, and 44 in 2014). Many of these publications receive national attention through news media or recognition in scientific communities. The AIF holds a contest each year for the best papers written by students. The 7th Annual Best Paper Award Winners were Emily



Mihalko from the group of Professor <u>Ashley Brown (BME)</u> for a publication in <u>ACS Nano</u> on "<u>Targeted</u> <u>Treatment of Ischemic and Fibrotic Complications of Myocardial Infarction Using a Dual-Delivery</u> <u>Microgel Therapeutic</u>" and Zhiren Luo from the group of Professor Chih-Hao Chang (MAE) for a publication in <u>ACS Nano</u> on "<u>Magnetically Actuated Dynamic Iridescence Inspired by the Neon Tetra</u>." The AIF identifies diverse "<u>Cool Science</u>" regularly, such as <u>Solar Cell Operation</u>, the <u>Younger Dryas</u> <u>impact hypothesis</u>, a study on <u>Plant Health</u>, and a study on <u>Nanotubes and Lungs</u> by Dr. Mary Jane Thomassen's group at East Carolina University were featured in newsletters.

#### **Personnel**

A total of 13 technical and business staff members and postdocs staff the facility with two tenured faculty members serving in director-level leadership roles and undergraduate students working as research assistants. The technical staff are experienced, collaborative, energetic, and student/training focused. Dr. Anton Jansson joined AIF in 2020 as a postdoc with responsibility for the X-ray computed tomography facilities. Dr. Anton Jansson received his M.S. in Engineering Physics from Karlstad University, Sweden, in 2014. After five years as a Ph.D. student at Örebro University, Sweden, he was awarded a Ph.D. in Mechanical Engineering. Anton's main research interest is applications and method development of X-ray computed tomography. Through this interest he has developed a taste for many different research topics that can use X-ray CT to its advantage such as additive manufacturing, material development, failure analysis, etc. Dr. Wenpei Gao joined AIF in 2020 as a TEM Principal Scientist. Dr. Gao's primary appointment is an Assistant Professor of Materials Science and Engineering at NC State. Prof. Gao's research focuses on developing correlative in situ imaging techniques in state-of-the-art advanced Scanning/Transmission Electron Microscopy to study the dynamics of nanostructured catalysts, which bridge the atomic scale mechanisms with reaction and transformation in chemistry. AIF's Director, Dr. Jacob Leo Jones, was named an IEEE Fellow. He is being recognized for development of X-ray scattering methods to understand electromechanical properties of ferroelectrics. The IEEE Grade of Fellow is conferred by the IEEE Board of Directors upon a person with an outstanding record of accomplishments in any of the IEEE fields of interest. The total number selected in any one year cannot exceed one-tenth of one-percent of the total voting membership. IEEE Fellow is the highest grade of membership and is recognized by the technical community as a prestigious honor and an important career achievement. Former and current AIF Associate Directors, Dr. Jim Lebeau and Dr. Shadow Huang, respectively, both received Presidential Early Career Award for Scientists and Engineers (PECASE). The PECASE is the highest honor bestowed by the United States Government to outstanding scientists and engineers who are beginning their independent research careers and who show exceptional promise for leadership in science and technology. At the AIF, Director Jacob Jones and former Associate Director Elizabeth Dickey both received the White House PECASE earlier in their academic careers.



The AIF Associate Director, Dr. Shadow Huang, also won the 2020 NC State Outstanding Teacher Award. The award recognizes excellence in teaching at all levels and recipients of the Outstanding Teacher Award become members of the Academy of Outstanding Teachers and be eligible for the Board of Governors Award for Excellence in Teaching and the Alumni Distinguished Undergraduate Professor Award. Roberto Garcia, AIF Operations Manager and FIB Lab Manager, also won the 2020 Safety Award. The award recognizes that Roberto has led a successful effort in combining and documenting the Environmental Health and Safety and internal required training modules. Anna Lumpkin, University Program Specialist at AIF, has received the 2020 College of Engineering Award of Excellence and it is the most prestigious honor bestowed upon non-faculty employees. She is one of only two COE SHRA winners. Anna was nominated in the category of Customer Service, in an effort that she consistently goes above and beyond the call of duty in service to customers through improving and innovating new customer-centric programs and interfaces.

## **Finances and Business**

The AIF operates as a cost-recovery center, managing ~\$1.59M in annual expenditures across 27 different university accounts. The largest fraction of expenses is recovered from user fees (\$930k, or ~61%), with support also provided by the College of Engineering and ORI. Note that these numbers do not include support for equipment acquisitions. *Operational support from the university is critical* to maintain a state-of-the-art facility, enable quality training of students and researchers, and offer competitive user rates for research activities. *Service contracts for the instruments* constitute a major portion of the annual expenditures (approximately \$360k), and this number will grow as new instruments come online and the factory warranty periods of newly commissioned instruments terminate. Though large, this is a necessary expense to minimize downtime of instruments critical to the research enterprise and minimize the volatility of maintenance costs. As these expenses grow, we aim to grow facility usage concurrently (by both internal and external researchers) in order to maintain our competitive user rates.

## **Outlook**

We learned a lot through the shutdown and stay-at-home phase of the COVID-19 pandemic. Most importantly, it reinforced to us the importance of our facilities to our customers. Some of our customers rely on the AIF to perform antiviral research, research on antiviral surfaces, and to develop new textiles and fabrics for next-generation face masks. We were able to keep a few critical instruments running under university approval to support some of these research and manufacturing activities that were critical to the medical supply chain and virus research (an example of one project is highlighted <u>here</u>). Many external businesses rely on the AIF to keep their businesses running, for example by manufacturing or qualifying their products. The AIF is now starting to serve even more of these businesses during a Phase 1 Research Restart process.

During the shutdown and stay-at-home phase, we also realized the extent to which AIF can respond and adapt to a rapidly evolving situation. AIF staff members quickly moved their availability and communications online, hosting for the first time online office hours where users can just "drop by" online and say hello or ask questions. The staff hosted online user group meetings and introduction tutorials. They recorded the tutorials for archiving and viewing on AIF's new YouTube Channel (link <u>here</u>). Recent NC State graduate and AIF staff scientist Abby Carbone created a new online educational presence for children and their families called "Sciencing with Abby" (highlighted <u>here</u>).



Two new instruments were received during the shutdown phase including a new high-pressure freezer and freeze substitution system as well as a new desktop SEM for education and outreach activities. The AIF leadership communicated timely and comprehensively to our customers and stakeholders as new information was received and operational status adjusted (see updates from March 16, March 19, April 1, and May 13). These types of virtual activities and communications will continue as AIF moves forward in our different and constantly changing future. As of the date of finalizing this annual report, all of the AIF instruments are either back online or in the process of coming online and technical staff are able to report to work on a voluntary basis. Most importantly, we are currently performing work in support of internal and external user research projects, though the number of individuals allowed within each laboratory is still severely constrained.

As we move forward among many uncertainties, training and enabling student users is an important activity that will take a priority in our planning for subsequent phases of reopening. Our student users rely on hands-on work in our shared laboratories as part of their research and educational experiences. The challenge for our staff is how to undertake this type of training safely given the distancing challenges experienced in close spaces and near instruments. This problem is not unique to AIF, but is shared by research labs and core facilities across NC State and at universities around the world. Therefore, the AIF is currently participating in conversations with countless groups of other shared facilities in order to vet and adopt best practices. Moreover, the creativity and adaptability that our staff demonstrated during the shutdown and stay-at-home phase of the pandemic provides a high level of confidence that we will find solutions to the challenges that lie ahead. Ultimately, it is the ingenuity, care, and determination of our entire AIF community, including all users and stakeholders, that will help us move forward in a manner that provides quality research and education training and support in a safe and effective environment.

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