CNMS Operations: Quarterly Report

NSRC Name, DOE Laboratory(s): Center for Nanophase Materials Sciences (CNMS), Oak Ridge National Laboratory (ORNL)
NSRC Director: Karren More
Date: October 6, 2022
Period covered: July 1 to September 30, 2022

1. User Program:

- **User Updates**
  - **User Project Extensions**: 62 proposals from the 2021B cycle were extended. The new expiration date is July 31, 2023.
  - **Proposal Call:** The CNMS Fall (2023A) proposal call is open and set to close on October 19, 2022.
- **User Meeting Planning:** The CNMS User Meeting was held August 8-11, 2022. Plenary speakers included David Muller, Sossina Haile, Jie Shan, and Maria Chan. The meeting included sessions on Quantum Materials, Emerging Research in Nanoscience (Early Career), Diversity in Nanoscience Research, 2D Materials, and Atomic-Scale Characterization, Advanced Analytics for Microscopy and Materials Science, and Nano- and Micro-Additive Manufacturing. The meeting will also feature three CNMS led workshops focused on Laboratory ES&H Best Practices and Hazard Analysis, Multi-Ion and Multi-Photon Direct-Write Nanofabrication, and Nano4Neuro. A total of 289 people registered to attend the User Meeting.
- **Active User Proposals as of Sept 27, 2022**: 444 (includes 38 rapid access proposals, 10 proposals for internal ORNL PIs/projects)
  - Industry-led: 13
  - University-led: 296
  - ORNL Staff-led: 107
  - Other Government Laboratory-led: 19
  - MSI and HBCU-led: 19
  - Unique Institutions: 174
  - MSIs and HBCUs: 19
- Average User Time: 3.5 days
- Science Categories: User distribution - 60% Materials Science; 12% Engineering; 8% Chemistry; 5% Instrumentation; 4% Polymers; 4% Biological/Life Sciences; 3% Physics; 3% Optics; 2% Earth/Environmental Sciences; 1% Medical

2. PIs and Technical Staff Supported by FWP:

- **Numbers:**
  - Number (Headcount) of Staff in CNMS Division (not including post-docs): 71
    - Director: 1
    - R&D Staff: 52
    - Tech Professional: 6
    - Technician: 6
    - User Office: 0 (new hire starts in November)
    - Administrative: 6 (5 are supported at the directorate level)
    - Operations (ES&H): 1 (supported at lab level)
  - Total # FTEs supported on CNMS FWP (not including post-docs): 40.6
  - Other funding sources/programs: 3 LDRD projects, 3 BES-MSED-FWPs, 1 BES-SUFD-ECA, QSC, 3 EFRCs, multiple EERE offices (BETO, HFTO, VTO)
- **New Staff Hires:**
  - Matthew Boebinger – CNMS post-doc converted to research staff in Materials MicroÅnalysis (MMÅ) Group
  - Kevin Harman – named CNMS Operations Manager, replacing Scott Hollenbeck

- Andy Lupini – named Group Leader, Scanning Transmission Electron Microscopy (STEM) Group
- Abinash Kumar – post-doc in Scanning Transmission Electron Microscopy (STEM) Group; from Massachusettes Institute of Technology
- Elizaveta Tiukalova – post-doc in STEM Group; from Nanyang Technological University, Singapore
- Michael Jacobs - post-doc in Nanomaterials Theory Institute (NTI) Group; from University of North Carolina Chapel Hill

- **Staff Departures:**
  - Victor Fung – Distinguished Staff Fellow in Nanomaterials Theory Institute (NTI) departed to take a faculty position at Georgia Institute of Technology as an Assistant Professor in the School of Computational Science and Engineering
  - Wonhee Ko – R&D Staff in Scanning Tunneling Microscopy (STM) Group departed to take a faculty position at the University of Tennessee as an Assistant Professor in the Department of Physics and Astronomy
  - Scott Hollenbeck – CNMS Operations Manager, retired on August 31, 2022
  - Vasudevan Iyer – post-doc in Functional Hybrid Nanomaterials (FHN) Group left for a research position at KLA Corp.
  - Xiangru Kong – post-doc in the Nanomaterials Theory Institute (NTI) left for a faculty position at the University of Science & Technology, Beijing, China
  - Sudhajit Misra – post-doc in the Materials MicroÅnalysis (MMA) Group left for a position as an R&D Engineer at Intel
- **Honors:**
  - Bobby Sumpter – 2022 Fellow of the Institute of Physics (IoP)
  - Bobby Sumpter – 2022 Fellow of the International Association of Advanced Materials (IAAM)
  - Jonathan Poplawsky – member of team awarded R&D 100 2022 Award for "DuAlumin-3D: An Additively Manufactured Dual-Strengthened Aluminum Alloy Designed for Extreme Creep and Fatigue Resistance"
  - Biva Talukdar – 3rd Place Poster Award, Microscopy and Microanalysis 2022, Portland, OR, August 1-4, 2022.
  - Bernadeta Srijanto – Outstanding CNMS Staff Member Award, CNMS User Meeting, August 8-16, 2022
- **Significant Invited Talks:**
  - Neus Domingo, "BE PFM and BE CRF for Functional Studies of Free-standing Ferroelectric Membranes and Thin Films," Joint ISAF-ECAPD-PFM Meeting 2022 (IEEE Society), Tours, France, June 27 – July 1, 2022.
  - Dave Geohegan, "In situ Diagnostics of Pulsed Laser Deposition Plasmas and Laser Processing for the Controlled Synthesis of Atomically Thin 2D Materials," 7th International School on Lasers in Materials Science, Venice, Italy, July 3-9, 2022.
  - R. K. Vasudevan, "Machine Learning and Automated Experiment for Manipulation and Characterization of Nanoscale Materials," Artificial Intelligence for Materials Science (AIMS), NIST, Gaithersburg, MD, July 12-14, 2022.
  - Kai Xiao, "Isotope Effect on the Thermal and Optical Properties of Atomically Thin 2D Materials" 20th International Symposium on the Physics of Semiconductors and Applications, Jeju, Korea, July 17-21, 2022, Virtual.
  - Jan-Michael Carrillo, "Multiscale Molecular Dynamics Simulations of Soft Matter and Polymers Systems," Webinar on Functional Materials, 85th Anniversary of the Chemical Society of the Philippines, Manila, Philippines, July 21, 2022, Virtual.
  - Yongtao Liu, "Machine Learning-Driven Automated SPM --Applications in Ferroelectrics," Virtual Royal Microscopical Society (RMS) AFM & SPM Meeting 2022, July 4-6, 2022, Virtual.

- Michael Zachman, "Methods for Cryo-(S)TEM Characterization of Reactive/Sensitive Materials and Samples Containing Liquids," Cryo-STEM and EELS for Material Sciences, Sunday Short Course, Microscopy and Microanalysis 2022, Portland, OR, July 31, 2022.
- David Cullen, "Improved Throughput, Statistics, and Instrument Utilization with Automated Analytical Electron Microscopy," Microscopy and Microanalysis, Portland, OR, August 1-4, 2022.
- Vasudevan Iyer, "Probing Material Dynamics with an SEM at Nanometer Length and Picosecond Time-scales," Microscopy and Microanalysis 2022, Portland, OR, August 1-4, 2022.
- Karren More, "My Interesting, Rather Turbulent, Science Journey," Women in Microscopy Breakfast, Microscopy and Microanalysis 2022, Portland, OR, August 3, 2022.
- Jordan Hachtel, "High Spatial Resolution Vibrational Signatures in Biological Whole Cells," Nion Open House 2022, Kirkland, Washington, USA. August 5, 2022.
- Steven Randolph, "Selective Photothermal Functionalization of 3D Nanostructures," NNCI Nano and Advanced Manufacturing Summit, Louisville, KY, August 9-10, 2022.
- Karren More, "CNMS User Program Update" and "CNMS Theme Science," 2022 CNMS User Meeting, August 8-16, 2022, Virtual.
- Rama Vasudevan, "From Machine Learning to Automated and Autonomous Experiments for Accelerating Scientific Discoveries," 2022 CNMS User Meeting, August 8-16, 2022, Virtual.
- Charles Pat Collier, "Soft Materials for Memristors and Memcapacitors, Synapses, and Neurons," Nano4Neurons Workshop, 2022 CNMS User Meeting, August 8-16, 2022, Virtual.
- Jordan Hachtel, "New Problems and Solutions in the Ultralow Loss Regime," 2022 CNMS User Meeting, August 8-16, 2022, Virtual.
- Kyle Kelley, "Dynamic Manipulation of Ferroelectric Structures via Automated Piezoresponse Force Microscopy," International Materials Research Conference (IMRC), Cancun, Mexico, August 14-19, 2022.
- Rama Vasudevan, "Reinforcement Learning and Automated Experiments for Scanning Probe Microscopy of Ferroelectric Materials," International Materials Research Congress (IMRC), Cancun, Mexico, August 14-19, 2022, Virtual.
- Charles Pat Collier, "Soft Materials for Memristors and Memcapacitors, Synapses, and Neurons," BTSD Crosscut Forum on "Bio-inspired Engineering Solutions – Learning from Nature on Sustainable Energy Conversion Processes," NTRC, ORNL, August 19, 2022.
- Yongtao Liu, "Machine Learning Driven Automated Microscopy for Materials Discovery—Applications in Scanning Probe Microscopy and Ferroelectric Materials," 2022 SPIE Optics & Photonics Meeting, San Diego, CA, August 21-25, 2022.
- Jan-Michael Carrillo, "Assembly of Charged Star Block Copolymers at the Oil-Aqueous Interface," ACS Fall Meeting 2022, Chicago, IL August 21-25, 2022, Virtual.
- Michael Zachman, "Probing Intact Solid-Liquid Interfaces and Reactive Materials by Cryo-FIB and Cryo-STEM," International Cryo-EM (ICE) Workshop for Advanced Materials, Albuquerque, NM, August 23-25, 2022.
- Karren More, "Opportunities at the Center for Nanophase Materials Sciences," International Cryo-EM (ICE) Workshop for Advanced Materials, Sandia National Laboratories, Albuquerque, NM, August 22-25, 2022.
- Peter Maksymovych, "The Multiwells: Switching Portraits of the Thiophosphate Family," 15th International Symposium on Ferroic Domains & Micro- to Nano-scopic Structures (ISFD-15), Kofu, Japan, August 28-31, 2022.
- An-Ping Li, "Understanding Heterogeneities in Quantum Materials," Neutrons and Complementary Techniques for Quantum Materials Workshop, Oak Ridge, TN, September 6-8, 2022, Virtual.

3. Postdocs Supported by FWP

- Total number of post-docs in CNMS Division: 23 FTE
- Number of post-docs supported by CNMS FWP: 6 FTE

4. Progress and budget summary on BES NSRC-supported QIS FWP:

FWP ERKCZ62 "Precision Atomic Assembly for Quantum Information Science," PI - Art Baddorf

- **Progress:**
  - Capital Equipment: All capital equipment budgeted for FY22 has been ordered.
    - Scanning Tunneling Microscopy (STM) Control System: Received. Key to operation and interface to AI/ML algorithms. A Nanonis system from SPECS has been received and costed.
    - Closed-cycle Low Temperature STM: Ordered. This microscope is central to the project. After competitive evaluation the Infinity STM from Scienta Omicron has been ordered. Expected delivery in November 2022 is on schedule.
  - Artificial Intelligence/Machine Learning (AI/ML): A computer enhancement for AI/ML control of experiments has been purchased and interfaced to the Nanonis control system.
  - A laboratory for the STM has been selected and is being prepared, which includes installation of cabinets, lab bench, desks, cylinder storage, and utilities. A second isolated utility room has been prepared for the compressor to enable closed-cycle cryogen cooling of the microscope.
  - The CNMS hired a new research staff scientist to contribute to STM experimentation on this project who will start work in November.
- **Budget summary:**
  - FTE funded staff/post-docs = 0.59 FTE (no post-docs on this project)
  - (a) First year of Award - Funds received in late FY21 for project start in FY22. Received $310,000 OPE and $1,190,000 EQU.
  - (b) Funds received to date (OPE and EQU) – $3,350,000
  - (c) Funds spent to date (OPE and EQU) - $788,021.31
  - (d) Committed equipment funds - $608,306.06

5. Progress and budget summary on BES NSRC-supported ECRP FWP:

FWP ERKCZ55 "Probing Electrons in Electrides and Beyond," PI - Miaofang Chi

- **Progress** Work has focused on understanding charge redistribution in electrides and other material systems by advancing and utilizing new scanning transmission electron microscopy (STEM) techniques, such as 4D-STEM-based phase-contrast imaging and cryogenic atomic resolution imaging. In this quarter, we discovered a thickness dependence in crystallographic transitions in layered binary transition *metal trihalides* at low temperatures. The number of layers defines the freedom of layer shifting, resulting in various stacking orders. Since the low-temperature magnetic behavior and spin states, e.g., Kitaev spin liquid transitions in $RuCl_3$, is strongly correlated to the interlayer interactions, our finding may reconcile the discrepancies in previous observations of magnetic properties. The other project we focused on was the development of a new algorithm to realize atomic resolution cryogenic 4D-STEM imaging. A primary goal of this project is to study quantum materials' correlated charge, lattice, and magnetic behavior using STEM. Our previous studies proved it could be achieved by 4D-STEM imaging; however, the limited stability of current cryogenic stages has limited 4D-STEM applications at cryogenic temperatures when quantum phases emerge. The developed algorithm can efficiently correct the complex distortions in atomic resolution cryogenic 4D-STEM datasets. This method allows a minimum loss of information in both reciprocal and real spaces, thereby reconstructing sample information from 4D-STEM datasets. This method is computationally cheap, fast, and applicable and will enable on-the-fly data analysis in the future for in situ cryogenic 4D-STEM experiments. A manuscript was recently submitted. Forthcoming activities will be to study the lattice and electronic structures of electrides and 2D van der Waals materials as a function of temperature and specimen thickness using cryogenic monochromated electron energy loss spectroscopy and 4D-STEM imaging.
- **Publications:**
  - K. Venkatraman, J.A. Hachtel, and M. Chi, “Visualizing Magnetic Fields at the Atomic Scale,” *Matter*, 5(8) 2414-2416 (2022). DOI: 10.1016/j.matt.2022.05.010
- **Invited Presentations:**

- Miaofang Chi - "Atomic Scale Cryogenic Scanning Transmission Electron Microscopy for Quantum and Energy Materials," International Cryo-EM (ICE) Workshop for Advanced Materials, Sandia National Laboratories, NM, August 22-25, 2022.
- Miaofang Chi - "Advanced Scanning Transmission Electron Microscopy for Energy Storage Research," International Conference on Energy Conversion & Storage (ORCAS), Friday Harbor Labs, WA, September 8-10, 2022.
- Miaofang Chi - "Atomic-scale Cryogenic STEM for Quantum and Energy Materials," Princeton-Nature Conference, 2nd Frontiers in Electron Microscopy for Physical and Life Sciences, Princeton, NJ, September 28-30, 2022.

- **Budget Summary:**
  - FTE funded staff/post-docs = 1.02/2.51 (since awarded in FY19)
  - First year of Award - FY19
  - Funds received to date (OPE and EQU) – $ 2,099,910.33
  - Funds spent to date (OPE and EQU) - $1,137,569.90

6. Progress on BES SUF-supported Data FWP(s)

CNMS staff are collaborators on three externally led BES-SUFD Data Projects:

- FWP ERKCZ59 "4D Camera Distillery: From Massive Electron Microscopy Scattering Data" led by LBNL. Continued to work with Jim Ciston at LBNL to incorporate ORNL-generated AI code into low-loss electron energy-loss spectroscopy workflows. We generated a template iPython notebook with needed libraries and functions that can easily be adjusted for new datasets and provided a test dataset to demonstrate iPython notebook functionality. New post-doc, Elizaveta Tiukalova, started her position in late September after significant delays – work will pick up in FY23.
- FWP ERKCZ60 "A Collaborative Machine Learning Platform for Scientific Discovery" led by LBNL. Building a Bayesian optimization framework to optimize spectral data acquisition with "user voting" (human-in-the-loop). Extending/customizing the VAE loss functions for our in-house built pyroVED software package to incorporate physical objectives for physics-constrained representation learning to improve robustness and reliability for applications in experiments and simulations.
- FWP ERKCZ61 "A Digital Twin for Spatiotemporally Resolved Experiments" led by ANL. We successfully incorporated variational autoencoders to enable enhanced determination of effective interactions from the coherent scattering of strongly correlated soft matter (paper being submitted).

7. Operations Equipment (OPE or EQU) Investments:

- **Ordered:** SQUID Magnetometer - EQU $650k - delivery expected in early FY23
- **Ordered:** Electron Energy Loss Spectrometer (EELS) for Nion UltraSTEM U100 - EQU $620k - ordered in FY21 but delivery not expected until early FY23
- **Ordered**: Holder heating/cleaning unit for JEOL NeoARM - OPE $60k - delivery expected in early FY23
- **Ordered:** Low temperature manipulator for STM to add new functionality – OPE $130k – delivery expected in early FY23
- **Ordered:** Gatan double-tilt, side-entry, cryo-holder and controller for Nion MAC-STEM – OPE $75k – delivery expected in early FY23
- **Delivered**: Dedicated CNMS Computational Cluster (CPUs) - OPE $400k – delivered to CADES and should come online in October
- **Delivered**: Dedicated Cloud Infrastructure - OPE $125k – delivered to CADES and should come online in October
- **Delivered:** Spin-polarized mK-STM (initial QIS Infrastructure project led by Chris Rouleau) was delivered on September 15 and will be transported to CNMS lab on main campus on October 18 to start installation
- **Repaired:** The field emitter for UHV Gemini SEM in the 4-probe SEM has been replaced and the SEM is working again. The control computer and the secondary electron detector have both failed on the UHV

JEOL SEM on the Unisoku/RHK 4-probe STM; due to the age of the instrument (installed in 2006), the vendor no longer provides service, and the SEM has been judged non-repairable. We have a plan to repurpose this STM.

8. Covid Impact:

- All ORNL staff returned to working full-time, on-site on May 16, 2022
- CNMS staff continue to work remotely with ~30% of CNMS users

9. Safety Updates:

None to report

10. Concerns and Challenges

Current challenges are primarily related to staffing:

- Kara Clayton, (long-time CNMS finance officer) retired in December 2021 and since then we have had three finance officers, which has posed many challenges. Samantha Moore (started in June) is currently an ORNL finance "floater" (meaning she takes care of any ORNL division finance issues when needed), but we are hoping she is named permanent CNMS finance officer soon. Samantha currently works 4 days at CNMS each week.
- Sandy Lowe, (long-time CNMS User Program Coordinator) retired in June 2022. Lisa Goins will assume this role in November 2022.
- Scott Hollenbeck (long-time CNMS Operations Manager) retired in August 2022. Kevin Harman is the new CNMS Operations Manager - we are currently searching for Kevin's replacement.
- Staff retention is an ongoing problem, although CNMS attrition rate is somewhat lower than ORNL's overall.
- On-boarding of new staff hires is taking longer than expected – numerous delays have been encountered, primarily due to visa issues

11. Other News:

- Neus Domingo, served as Tutorials Chair for the Joint ISAF-ECAPD-PFM Meeting 2022 (IEEE), Tours, France, June 27, 2022.
- Miaofang Chi and Michael Zachman Co-organized workshop "Cryo-STEM and EELS for Materials Sciences," Sunday Short Course, Microscopy and Microanalysis 2022, Portland, OR, July 31, 2022.
- Jonathan Poplawsky, organized symposium "Expanding the Limits of Atom Probe Tomography," Microscopy and Microanalysis 2022, Portland, OR, July 31-August 4, 2022.
- Kinga Unocic, organized symposium "Pre-Congress Meeting X62 — Real-World Data Analytics & Quantitative Liquid and Gas Environmental Electron Microscopy," Microscopy and Microanalysis 2022, Portland, OR, July 31, 2022.
- Steven Randolph, organized symposium "Ultrafast Laser Materials Processing," Microscopy and Microanalysis 2022, Portland, OR, July 31-August 3, 2022.
- Karren More, participated in the EVMS Surveillance Review for the NSRC-Recapitalization Project, August 2, 2022.
- Petro Maksymovych, Sabine Neumayer, Jon Poplawsky, and Liam Collins, co-organized Nano4Neuro – a workshop on connecting nanoscale science and neuromorphic materials research that took place on Aug 15-16, as satellite of the CNMS user meeting. The workshop comprised 22 presentations including leading research groups in neuromorphic materials and showcase of signature capabilities of CNMS in the study of materials away from equilibrium.
- Karren More and Ilia Ivanov, participated in the protocol visit of Dr. Asmeret Asefaw Berhe, Director of the Office of Science, August 17, 2022.
- Charles Patrick Collier, co-chair of organizing committee for ACS Fall 2022 Meeting, Colloids and Surface Chemistry Symposia, Chicago, IL, August 21-25, 2022.

- Rama Vasudevan, organized symposium "G1 - AI-Enabled Advances in Materials Imaging, Automation, and Analysis," International Materials Research Congress (IMRC) 2022, Cancun, Mexico (hybrid), August 14-19, 2022.
- Miaofang Chi and Michael Zachman, co-organized the International Cryo-EM (ICE) Workshop for Advanced Materials, Sandia National Laboratories, Albuquerque, NM, August 22-25, 2022.
- Rajeev Kumar and Michael Zachman, participated in the 1st SUFD Science Slam!, August 30, 2022.
- Karren More, served on an NSF Panel Review for Convergence Accelerator Program, September 15-16, 2022.

12. Science Supported by FWP

Three highlights will be submitted in Q4:

1. Z. Shen, J.-M.Y. Carrillo, B.G. Sumpter, and Y. Wang, "Fingerprinting Brownian Motions of Polymers under Flow," Physical Review Letters **129**, 057801 (2022). DOI: 10.1103/PhysRevLett.129.057801

   CNMS-only publication: There has been sustained interest in understanding molecular motions in nonequilibrium steady state. Despite the herculean experimental and computational efforts over the past several decades to probe the nonequilibrium dynamics of soft matter by inelastic scattering techniques and computer simulations, characterizing and interpreting anisotropic space-time correlation functions under flow remain a challenging task. This work introduces a new methodology for quantitative studies of polymer self-dynamics under flow, by employing a set of complementary reference frames and extending the spherical harmonic expansion technique to dynamic density correlations. Application of this approach to nonequilibrium molecular dynamics simulations of coarse-grained polymer melts reveals a number of universal features. The quantitative approach outlined here is not limited to entangled polymer dynamics, but applicable to a wide range of problems in nonequilibrium dynamics of soft matter.

2. M. Checa, X. Jin, R. Millan-Solsona, S.M. Neumayer, M.A. Susner, M.A. McGuire, A. O'Hara, G. Gomila, P. Maksymovych, S.T. Pantelides, and L.M. Collins, "Revealing Fast Cu-ion Transport and Enhanced Conductivity at the $CuInP_2S_6$-$In_{4/3}P_2S_6$ Heterointerface," *ACS Nano* **16**(9), 15347-15357 (2022). DOI: 10.1021/acsnano.2c06992

   CNMS-led publication: Van der Waals layered ferroelectrics, such as CuInP2S6 (CIPS), offer a versatile platform for miniaturization of ferroelectric device technologies. Control of the targeted composition and kinetics of CIPS synthesis enables the formation of stable self-assembled heterostructures of ferroelectric CIPS and nonferroelectric In4/3P2S6 (IPS). Here, we use quantitative scanning probe microscopy methods combined with density functional theory (DFT) to explore in detail the nanoscale variability in dynamic functional properties of the CIPS-IPS heterostructure. We report evidence of fast ionic transport, which mediates an appreciable out-of-plane electromechanical response of the CIPS surface in the paraelectric phase. Further, we map the nanoscale dielectric and ionic conductivity properties as we thermally stimulate the ferroelectric-paraelectric phase transition, recovering the local dielectric behavior during this phase transition. Finally, aided by DFT, we reveal a substantial and tunable conductivity enhancement at the CIPS/IPS interface, indicating the possibility of engineering its interfacial properties for next generation device applications.

3. G. Minnik, B.T. Safa, J. Rosenbohm, N.V. Lavrik, J. Brooks, A.M. Esfahami, A. Samaniego, F. Meng, B. Richter, W. Gao, and R. Yang, "Two-Photon Polymerized Shape Memory Microfibers: A New Mechanical Characterization Method in Liquid," *Advanced Functional Materials* (2022). DOI: 10.1002/adfm.202206739

   CNMS/user co-led publication: Two-photon polymerization (TPP) is widely used to create 3D micro- and nanoscale scaffolds for biological and mechanobiological studies, which often require the mechanical characterization of the TPP fabricated structures. To satisfy physiological requirements, most of the mechanical characterizations need to be conducted in liquid. However, previous characterizations of TPP fabricated structures are all conducted in air due to the limitation of conventional micro- and nanoscale mechanical testing methods. In this study, a new experimental method is reported for testing the mechanical properties of TPP-printed microfibers in liquid. The experiments show that the mechanical behaviors of the microfibers tested in liquid are significantly different from those tested in air. By controlling the TPP writing parameters, the mechanical properties of the microfibers can be tailored over a wide range to meet a variety of mechanobiology applications. In addition, it is found that, in water, the plasticly deformed microfibers can return to their pre-deformed shape after tensile strain is released. The shape recovery time is dependent on the size of microfibers. The experimental method represents a significant advancement in mechanical testing of TPP fabricated

structures and may help release the full potential of TPP fabricated 3D tissue scaffolds for mechanobiological studies.

- **Publications:** All Q4 CNMS publications for categories (publication types) (a) *Sole NSRC-led*, (b) *NSRC-led*, (c) *NSRC/User Co-led*, (d) *User-led Collaborative*, and (e) *User-only* are documented in the Excel spreadsheet (separate document "CNMS Q4 Publications"). Note that we added another drop-down selection in column A to include (e) *User-only* publications.
- **Invention disclosures, patents, other products:**
  - **Patent**: C.L. Cramer, R.A. Lowden, K. Unocic, J.W. McMurray, and A.M. Elliott, "Indirect Additive Manufacturing Process for Producing SiC-B_4C-Si Composites," Patent #11364654, June 21, 2022.
  - **Patent**: J.R. Cahill, V. Kertesz, and S.R. Retterer, "Porous Membrane Enabled Mass Spectrometry Characterization of Microfluidic Devices," Patent #11417508, August 16, 2022.

13. FWP Budget details

- Budget information included in Excel spreadsheet (separate document " FY22 ORNL CNMS Budget - September").