MONT Montana Nanotechnology Facility

An NSF NNCI Node in the Northern Rocky Mountain Region



Year 3 Snapshot



David Dickensheets NNCI Y3 Annual Conference, September 13/14, 2018

nano.montana.edu





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Overview and Core Facilities

Coordinated access to and training on shared equipment housed in 5 campus facilities:

- Montana Microfabrication Facility
- Imaging and Chemical Analysis Lab
- Center for Biofilm Engineering
- Center for Bio-Inspired Nanomaterials
- Metabolomics, Proteomics and Mass Spectroscopy facility







Core Fabrication





Processes set up for 4-inch wafers and pieces

- Wet benches
- Spinners pos, neg photoresists, ebeam resists, lots of SU-8 work
- Patterning contact litho; FE-SEM with NPGS, Dip-Pen nanowriting
- Thermal processing oxidation and anneal; solid source diffusion
- PVD Angstrom Engineering sputter, e-beam evap, thermal evap
- Dry etching cryo ICP; RIE; O2 ash;
- Critical point dryer







CBE CBIN Unique Capabilities for Bio + Nano

Center for Biofilm Engineering

- Incubation capabilities for biofilms, mammalian cells and thermophiles
- Chromatography and spectroscopy tools
- Genetic sequencing
- Advanced confocal and fluorescence imaging – live cell imaging









Core Characterization

- Thin films: Ambios profilometer, Nanospec, resistivity probes, XRD/GID
- **Device characterization**: probe stations, SPA, wire bonders
- Imaging: Metrology and stereo microscopes, fluorescence microscopes, SEMs, AFMs, TEM
- **Spectroscopy**: nanoAuger, TOF-SIMS, XPS, EBSD, EDX, and Mass Spec capabilities spanning small to large molecules









Focus Areas





- Program Emphases
 - Optical MEMS and Nanosystems with local industrial collaborations
 - **Biology and Nanotechnology** Biofilms and Microfluidics, bio-corrosion
 - Novel optical and high temperature materials
 - Nanoscale characterization SEM (cryo), nanoAuger, XPS, XRD, ToF-SIMS
 - Education and Outreach emphasizing undergraduate research, K-12 students/teachers, web-based education, local Tribal colleges







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Yearly User Data Comparison

	Year 1	Year 2	Year 3 (6 months)
Total Users	125	160	113
Internal Users	96	124	81
External Users	29 (23%)	36 (23%)	32 (28%)
External Academic	6	9	5
External Industry	22	24	26
External Government	1	3	1
External Foreign	0	0	0
Total Hours	3599	4713	2538
Internal Hours	2852	3901	2004
External Hours	747 (21%)	812 (17%)	534 (21%)
Average Monthly Users	47	51	60
Average Ext. Monthly Users	8 (17%)	8 (16%)	10 (17%)
New Users Trained	35	58	39
New External Users Trained	1 (3%)	9 (16%)	2 (5%)

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MONT User Data







Facility Enhancement

- Y3 New Equipment: Electroplater for permalloy, confocal Raman microscopes (2), AML Wafer Bonder, cryo stage for FESEM
- ion-assist e-beam evaporator on order for October 2018 delivery
- Renovating space to expand ISO 7 (class 10000) footprint to support soft lithography/microfluidics users
- Funding secured for Talos Arctica 200 kV cryo TEM with Gatan K3 DED camera (NSF MRI/Murdock Trust/MSU); to be housed in MONT

We continue to leverage our NNCI award as match to secure State and Private investment in our facility.



our NNCI capital expenditures.





Research Highlights

- Agile Focus Designs, of Bozeman, MT is commercializing fast focus and zoom devices to enable real-time imaging in microscopes, cameras, and surgical instruments.
- Agile Focus Designs uses MONT to fabricate voltage controlled micro-electro-mechanical systems mirrors capable of rapid focusing.
- Agile Focus Designs has ongoing R&D efforts, including recent NSF SBIR awards 1548737 and 1819493 and projects through the Montana Board of Research and Commercialization.
- The company broadens participation and diversity in engineering by exceeding the national company average (13%) of practicing female engineers with a 50% ratio of women to men.



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Dr. Sarah Lukes, founder and CEO, in the MONT facility inspecting a micro-electro-mechanical systems (MEMS) mirror capable of electronically actuated focus control.



AGILE FOCUS

Agile Focus Designs, LLC, Bozeman, MT Sarah Lukes, CEO Work performed at Montana State University, MONT facilities MMF and ICAL





Research Highlights

Advanced Neurofluidic Devices for Neurodegenerative Disease Studies

- Studying neuronal cell growth "on chip" to elucidate complex guidance cues.
- A neurofluidic device comprises microfluidic channels specifically designed to compartmentalize different cell body parts of neurons.
- Microchannels in PDMS are fabricated from a molding master. We use standard silicon wafers and a two-step photolithography with KMPR 1005 and 1050 to achieve channel depths of 7 µm and 70 µm, respectively.
- This project tests growth and transport behavior of degenerative signals in primary cortical neurons.







Figure 1: Two-step photolithography to fabricate microchannels in poly-dimethylsiloxane (PDMS) for axon/dendrite compartmentalization and directed growth in brain cell cultures.

Figure 2: Phase contrast microscope image shows the microfabricated channels after casting into PDMS. Shallow channels reflect upright light more then deeper channels.



Kendra Hergett, Anja Kunze, Montana State University Work performed at Montana State University, MONT facility: **MMF**





Research Highlights

Advanced Neurofluidic Devices for Neurodegenerative Disease Studies



Figure 1 (left): Livecell, false-color fluorescent microscope image of axonal growth through narrow microchannels generated from cortical rat neurons (E18). After 8 days in culture axons are reaching the larger perfusion microfluidic channel. Axons were stained with Fluo4, a live-cell, viability, calcium marker.

Figure 2: False-color TIRF microscope image shows axonal growth from several hundred neurons through a microfluidic channel to study mitochondria and calcium dynamics. (accepted at BMES,

Kendra Hergett, Prof. Anja Kunze, Montana State University Work performed at Montana State University, MONT facility: MMF

Publications in 2017

80 Publications total

- 41 Journal Articles
- 37 Conference papers
- I Book chapters
- Ipatent

Up 31% over 2016







Marketing and Recruitment of New Users

- Workshops and Meetings
 - Inviting local industry association members to Annual User's Meeting poster sessions
 - Workshop for industrial affiliates of Center for Biofilm Engineering
- On-Campus Department Visits
- User Grants for Project Initiation
 - ~\$2500 credit for facility use
 - Y3 awards
 - Marketa Hulkova, visiting Fulbright Scholar, Masaryk Univerzity, Czech Republic
 - Biomodum LLC, genetic analysis platform
 - Stephan Warnat, Assistant Professor, Mechanical& Industrial Engineering
 - NSF proposal pending





Y3 Education and Outreach

In cooperation with Carleton College Science Education Resource Center (SERC):

CO-convened nano/geo workshops at the Geoscience Society's Goldschmidt Conference (2017 and 2018) and 2018 NanoEarth

- Catalytic events to a) aggregate resources from expert leaders; b) develop new resources and teaching activities; c) disseminate to workshop participants and larger Earth Science community.
- Workshop and webinar support

Teaching Nanoscience Across the Undergraduate Earth and Environmental Science (E&ES) Curriculum Website

- "What", "Why" and "How" to teach Nanoscience; leverages NSF investments in Starting Point, On the Cutting Edge, and InTeGrate
- Need and opportunity to teach Nano in E&ES
- Tutorials on Analytical Methods, Instructional Resources
- Nano topics ready to be integrated into E&ES curriculum
 - Over 450 resources vetted by experts, organized by topics, advice and resources for faculty to integrate into Intro and courses for majors

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Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS)

David W. Mogk, Montana State University

What is Time-of-Flight Secondary Ion Mass Spectrometry (ToF-S

Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) is a surface-sensitive analytical method that uses a pulsed io to remove molecules from the very outermost surface of the sample. The particles are removed from atomic monolayers of These particles are then accelerated into a "flight tube" and their mass is determined by measuring the exact time at which time-of-flight). Three operational modes are available using ToF-SIMS: surface spectroscopy, surface imaging and depth p of ToF-SIMS include:



- Mass resolution of 0.00x amu. Particles particles with the same nominal mass (e.g. Si and C₂H₄, both with amu = 28) are easily distinguished from one another because as Mr. Einstein predicted there is a slight mass shift as atoms enter a bound state.
- Mass range of 0-10,000 amu; ions (positive or negative), isotopes, and molecular compounds (including polymers, organic compounds, and up to ~amino acids) can be detected.
- Trace element detection limits in the ppm range.
- Sub-micron imaging to map any mass number of interest.
- Depth profiling capabilities; sequential sputtering of surfaces allow analysis of the chemical stratigraphy on material surfaces (typical sputtering rates are ~100 A/minute).
- Retrospective analysis. Every pixel of a ToF-SIMS map represents a full mass spectrum. This allows an analyst to retrospectively produce
 maps for any mass of interest, and to interrogate regions of interest (ROI) for their chemical composition via computer processing after
 the dataset has been instrumentally acquired.

Fundamental Principles of Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS)

ToF-SIMS uses a focused, pulsed particle beam (typically Cs or Ga) to dislodge chemical species on a materials surface. Particles produced closer to the site of impact tend to be dissociated ions (positive or negative). Secondary particles generated farther from the impact site tend to be molecular compounds, typically fragments of much larger organic macromolecules. The particles are then accelerated into a flight path on their way towards a detector. Because it is possible to measure the "time-of-flight" of the particles from the time of impact to detector on a scale of nano-seconds, it is possible to produce a mass resolution as fine as 0.00X atomic mass units (i.e. one part in a thousand of the mass of a proton). Under typical operating conditions, the results of ToF-SIMS analysis include:



- 1. a mass spectrum that surveys all atomic masses over a range of 0-10,000 amu,
- 2. the rastered beam produces maps of any mass of interest on a sub-micron scale, and
- 3. depth profiles are produced by removal of surface layers by sputtering under the ion beam.

ToF-SIMS is also referred to as "static" SIMS because a low primary ion current is used to "tickle" the sample surface to liberate ions, molecules and molecular clusters for analysis. In contrast, "dynamic" SIMS is the method of choice for quantitative analysis because a higher primary ion current results in a faster sputtering rate and produces a much higher ion yield. Thus, dynamic SIMS creates better counting statistics for trace elements. Organic compounds are effectively destroyed by "dynamic" SIMS, and no diagnostic information is obtained.

Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) Instrumentation – How Does It Work?

ToF-SIMS instruments typically include the following components

An ultrahigh vacuum system, which is needed to increase the mean free path of ions liberated in the



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EDUCATION AND OUTREACH

Curriculum

Y3 Education and Outreach



Teaching Nanotechnology Across the Undergradu



Iontana Nanotechnology

MONT Activities MONT User Meeting Goldschmidt Workshop 2017 Mont Webinar March 2017

Mont Webinar Dec

2016

Nanotechnology is an exciting emerging field of research with applications in all STEM disciplines including the physical, chemical, life, Earth and environmental sciences and allied disciplines in materials science and engineering Nanotechnology provides unprecedented opportunities for frontier research at the interfaces between these STEM disciplines by studying the properties of materials on the nanoscale (~1 billionth of a meter!). The development of engineered nanomaterials has applications in such diverse fields as energy capture and storage devices, delivery of pharmaceuticals, environmental health and safety (related to both natural and engineered

What is Nanotechnology Why Teach Nanotechnology How to Teach

STEM

anotechnology in STEM > Methods

Nanotechnology in STEM What is Nanotechnology Why Teach Nanotechnology

How to Teach Nanotechnology

Ethics

Nanoscience in the Earth and Environmental Sciences

Methods

Nanoscience Topics in Earth Science

Instructional Resources

Goldschmidt Workshop 2017

Tutorials of Methods Used in Nanotechnology

Browse Geochemical Analytical Instruments and Techniques

Each of these pages contains information about each instrument or technique including what it is, fundamental principles, how it works, applications, strengths and limitations, sample preparation, data collection, results, and preparation, and if available, literature and teaching activities/resources.

X-ray Crystallography

- Single-crystal X-ray Diffraction--Christine M. Clark, Eastern Michigan University and Barbara L. Dutrow, Louisiana State University
- X-ray Powder Diffraction (XRD)--Barbara L. Dutrow, Louisiana State University and Christine M. Clark, Eastern Michigan University

Electron Microbeam

• Electron Probe Micro-analyzer (EPMA)--John Goodge, University of Minnesota-Duluth

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research).

Nanoscience in the Earth and Environmental Sciences--Needs and Opportunities

Nanoscience is one of the great, relatively unexplored frontiers in the Earth and Environmental Sciences. However there are very few curricula, courses, or even chapters in textbooks (Mineralogy, Petrology, Geochemistry, Hydrology....) that focus on nanoscience. (See the Virginia Tech Nanoscience program as one example). This is unfortunate because nanoscience addresses entirely new lines of fundamental research about the Earth system, and it also addresses important applications to the grand challenges facing humanity living



Nanomaterials and Crystallization: New Understanding of Processes and on Earth. The purpose Pathways approaches and appli

Classic theory of nucleation and growth of crystals assumes that crystals grow by ordering provide resources to e atoms (monomers) one at at a time in prescribed positions in the crystal structure. However, modern studies of growth mechanisms of crystals shows that crystals more Environmental Science typically grow by aggregation of nanoparticles. Crystallization pathways may involve formation of multi-ion complexes from dissociated ions, to organization of these interested in introduci complexes into isolated nanoparticles with very short range order, to oriented aggregate of nanoparticles, and ultimate formation of macroscopic crystals. Examples of this type of crystallization pathway of crystals forming from aggregates of nanoparticles can be found in calcium carbonates, calcium phosphates, ferric hydroxides and hydroxyl-sulfates, and aluminosilicate nanoparticles. For a more detailed description of crystallization by particle attachment (CPA), see DeYoreo et al., 2015, Crystallization by particle attachment in synthetic, biogenic, and geologic environments, Science, vol 349. issue 6247, aaa6760-1

owth of crystals from appreciated nanopart

 See the Powerpoint presentation by Dr. Manual Caraballo, University of Chile. Crossroads in analytical chemistry and non-classical crystal nucleation: deciphering the role of inorganic polymers in poorly crystalline nanominerals nucleation presented at the 2017 Goldschmidt Nanoscience Workshop.

 Caraballo, M. A., Michel, F. M., and Hochella Ir. M. F., 2015. The rapid expansion of environmental mineralogy in unconventional ways: Beyond the accepted definition of a mineral, the latest technology, and using nature as our guide: American Mineralogist, v. 100, no. 1, p. 14-25. Environmental mineralogy is rapidly expanding in technological directions that allow for the detection, characterization, and understanding of noncrystalline and poorly crystalline phases, crystalline-amorphous mixed phases, and nanosized naturally occurring materials. Specifically, this article provides a perspective view of the broad range of structural complexity/heterogeneity observed in environmental minerals a



Impact of Education & Outreach Activities

Green boxes show activities which were evaluated.

MONT 2017-2018 Education & Outreach Events				
MSU location of MONT	# Participants			
K-12 Students	370			
MAP Student Engagement	14			
REU and Convocation	3			
Teacher Education (MSSE)	7			
Science Olympiad	50			
Technical Events	108			
Public Events	240			
Total	792			

Fifth graders explore NanoLand



Summary Solar Cells for Teachers

N = 7	Not at all	Somewhat	Agree	Strongly
knew more about how solar cells operate and how they are made after the course	0	1	0	6
knew more about how to use engineering examples and applications in their classrooms	0	1	0	6
felt more comfortable using engineering applications and talking about engineering in their classrooms	0	0	0	7





NNCI Cooperative Network Activities

Network-Wide

- Participation in subcommittees (New Equipment and Research, Entrepreneurship, Assessment and Evaluation) and working groups (Microscopy, SEI and Education), resulting in shared reports and best practices
- Attendance at REU convocation and NNCI annual conference
- Participation in Nano Day activities

Multi-Site

- Proposals pending leveraging access to aberration-corrected TEM at nano@stanford
- User support and staff technical interactions with **SDNI**, **MINIC**, **nano@stanford and CNS**
- Working with **SENIC**, leveraging their expertise and investment in facility management software



Workshop organization with NanoEarth (2018 Goldschmidt Conference, NanoEarth 2018 workshop)

On Behalf of the Network

Helped with NNCI exhibit booth at TechConnect 2018







Y3 Societal and Ethical Implications



https://serc.carleton.edu/msu_nanotech/index.html



- Equipment Roadmap
 - Equipment Committee local stakeholders
 - Opportunistic acquisitions (startup agreements)
 - Shiny new toys vs. tired workhorse tools
- In-house vs. network access; how specialized can we get?
- Is leasing an option?
- Space, equipment decommissioning, user relations



