FY 2015 - 2016
Annual Report

MichiganTech
Multi-Scale Technologies Institute

July 2016
Multi-scale technologies are those that bring together functional elements to form systems where the relative size of components within the system spans from the nano through the micro and into the macro domain. The systems-focus of MuSTI emphasizes the challenges associated with integrating technologies that have relative feature sizes that are orders of magnitude apart, and operating characteristics that are size dependent. MuSTI became operational near the end of 2005 and is currently authorized until December 2020. The number of proposals submitted by MuSTI affiliates was down by 8 (17%) from the prior reporting year, and are shown in the Appendix. This can be attributed to the fact that several faculty who were prolific proposal submitters departed Michigan Tech, and that several others became understandably affiliated with the new Henes Institute of Quantum Phenomena. The number of agencies to which proposals were submitted remained the same. The new and incremental awards showed a sharp decrease due to the reason above, as well as the last reporting period included a MRI award of nearly $2 million for the Scanning TEM. The educational outreach activities of MuSTI remain strong as highlighted.

During 2016-17, MuSTI will make a significant contribution to help facilitize the Scanning TEM. The current commitment is $115,000 and may increase depending on the status of other campus-wide initiatives including upgrades and expansion of the Superior computing cluster which MuSTI has supported in the past. This was anticipated and is the primary reason that MuSTI expenditures during 2015-2016 were lower than in past years.

During the 2015-2016 reporting period:

- Proposals submitted during the period 39
- Number of different PIs and Co-PIs 19
- Number of different departments/units of PI/Co-PIs 5
- Number of different agencies submitted to 23
- Approximate total request $10,222,332
- Number of PhD funding years requested 74
- Number of new and incremental awards during the period 10
- Total funding of new and incremental awards $1,073,417

MuSTI had a fiscal year beginning IRAD balance of $181,838 and an ending balance of $253,277 with IRAD revenue of $110,094 and expenditures of $38,655. Expenditures supported:

- $ 1,146 graduate student support
- $ 13,262 travel to potential sponsors and conferences
- $ 19,764 cost share and non-mandatory transfers, including nanotechnology course support
- $  4,403 supplies and use fees

Craig Friedrich, Director
Paul Bergstrom, Associate Director for Fabrication and Facilities
John Jaszczyk, Associate Director for Education and Outreach
Computing Infrastructure Support

In 2013, MuSTI financially supported the acquisition of 64 additional processors via four compute nodes for Superior. This queue (musti.q) is used to provide highest submission priority to MuSTI affiliates and when not fully utilized is open for other users, and brings the overall compute capacity to nearly 31 TFLOPS.

During the current reporting period, musti.q was used for 3,277 simulations using 430,178 CPU-hours. At a nominal $0.10 per CPU core per hour, this usage amounts to $43,017 for this reporting period.

Since its introduction, 5,272 simulations have been run through musti.q with a total utilization of 858,122 CPU-hours with worth equivalent to $85,812. This can be treated as the level of funds that MuSTI researchers would have had to spend on external computing services (e.g., Amazon/Google Cloud Computing services) if these nodes were not available for use. The one-time cost of acquiring these nodes was $24,524. This measurable 3.5:1 benefit-cost ratio is an example of the ongoing reinvestment of MuSTI resources in campus-wide initiatives to grow the Michigan Tech research enterprise.

Educational Initiatives

Outreach and Education: Nanotechnology Workshop to High School Underrepresented Students

- Shiva Bhandari, PhD Candidate, Department of Physics
- Bishnu Tiwari, PhD Candidate, Department of Physics, Engineering Physics
- Mingxiao Ye, PhD Candidate, Department of Physics
- Yoke Khin Yap, Professor, Department of Physics

A workshop on nanotechnology was conducted on April 11, 2016. This year, we engaged the L’Anse, Michigan High School, which is about 30 miles from Michigan Tech. The L’Anse High School is located near the L’Anse Indian Reservation, a federally recognized Keweenaw Bay Indian Community of the historic Lake Superior Chippewa. The high school has an underrepresented student enrollment of 24%. In view of this, we invited all 9th and 10th grade students to attend the workshop. The workshop was conducted in collaboration with the GEAR-UP (Gaining Early Awareness and Readiness for Undergraduate Programs) program of the Center for Pre-College Outreach at Michigan Tech.
As shown in **Figure 1**, the number of student attendees was 91, who were accompanied by four teachers. In addition to the instructor (Professor Yoke Khin Yap), the workshop was assisted by graduate students Bishnu Tiwari, Shiva Bhandari, and Mingxiao Ye, and Liz Fujita from the GEAR-UP program. As shown in **Figure 2**, the workshop started with a 40-minute introduction module, followed by a 40-minute hands-on session. Students were first introduced to the concept of nanometer, nanotechnology, atomic bonding in diamonds, and graphite. They were engaged to make ball and stick models of diamonds and graphite by using Play-Doh and wood sticks. Students then returned to a 40-minute seminar session on carbon nanotubes (CNTs). They learned about the structures of a CNT by folding a graphene sheet (honeycomb network printed on a transparent plastic sheet), their properties, and potential uses (with animations and video clips). Finally, these students were inspired with a series of futuristic applications of nanomaterials before we ended the workshop for lunch.

During the workshop, eleven questions were posed to these students to keep track on their learning progress. Some of the interesting outcomes are summarized as follows,

- Among the students, 15 were Native American, 31 female, and 43 male.
- Before the workshop, all 91 students stated they love/like new technology (internet, smart phones) but only 80 of them love/like science.
- After the lectures, almost 100% of the students knew what is “nano”, “1 nm”, and the definition of “nanotechnology”.
- After the workshop, 87 students said that they love/like science.
- Based on these outcomes, we believe that the workshop has promoted interest of high school students in science, in addition to giving them direct exposure to scientists and inquiry.
Fundamentals of Nanoscale Science & Engineering

- John Jaszczak, Professor, Department of Physics

Michigan Tech offers an interdisciplinary undergraduate minor in **Nanoscale Science and Technology**. Administered through **MuSTI**, the minor focuses on multi-disciplinary nanotechnology studies in emerging fields of science, engineering, and technology. The minor:

  - introduces students to the basic issues and overall scope of the field,
  - encourages them to pursue interdisciplinary work outside their major and beyond as careers and research interests develop,
  - emphasizes fundamental sciences, current and potential applications, modern instrumentation, and impact on society.

In Spring 2016, the offering of UN 2600 **Fundamentals of Nanoscale Science & Engineering**, a required core course of the Minor, was enhanced with the introduction of popular hands-on experiences using...
Atomic Force Microscopy and Scanning Tunneling Microscopy labs for the first time (below). Enrolled students were 2nd through 4th year students from Biomedical Engineering, Materials Science and Engineering, Mathematics, Mechanical Engineering, and Physics. Students examined different memory chip designs for EPROMs (AFM) and also measured atomic bonding distances in graphite (STM). The course was a team-taught introduction to the fundamentals of nanotechnology, emphasizing the interdisciplinary nature of this field. Modern instrumentation, key scientific foundations, and current and potential applications were discussed. Real and potential societal implications of nanotechnology were explored.

Research Highlights

During the reporting period there were 39 new proposals submitted with 10 new or incremental awards. The research of MuSTI affiliates continues to be diverse spanning many dimensional scales. In this report, two examples are detailed.

The first, Ferrofluid Interface Deformation and Spray Onset under Electric and Magnetic Stresses, investigates an innovative technology for integrating electric and magnetic spacecraft propulsion systems that spans from the macro-scale down to the atomic (ionic) scale. This technology does not require bulky chemical-based thrusters for small satellites or deep-space propulsion systems.

The second, Generating Heterogeneous Pericellular Matrix of Chondrocyte Sub-Populations In-Vitro by Microencapsulation, is working to develop a novel approach to the manufacture of engineered tissue cell sources targeting osteoarthritis that affects more than 27 million people in the US costing more than $180 billion annually.
**Ferrofluid Interface Deformation and Spray Onset under Electric and Magnetic Stresses**

- Brandon A. Jackson, PhD Candidate, Mechanical Engineering – Engineering Mechanics

The deformation of a fluid meniscus in an electric field has many practical applications including mass spectrometry, pharmaceutical production, nano-fabrication, and spacecraft propulsion. Traditionally, this phenomenon of emission is achieved only in the presence of an electric field with a magnetically neutral fluid and is termed "electrospray." However, it has been demonstrated that emission can be assisted through the use of a magnetic field and a superparamagnetic fluid, changing the dynamics of the fluid meniscus and emission. The aim of this research was to develop a modeling tool to predict the deformation of a fluid interface under simultaneous electric and magnetic stress and to analyze the dynamics leading up to spray emission.

When a uniform electric field is applied to a droplet that has a closed bounding interface with another fluid (or vacuum) the droplet elongates in the direction of the applied field. Previous investigations into the deformation of sessile droplets under the influence of either electric or magnetic fields has been studied numerically, but little attention has been given to the meniscus behavior of a sessile droplet under the combined action of electric and magnetic fields.

For strong electric fields, the fluid meniscus will deform into a sharp point until a threshold is reached at which time the pointed meniscus begins to emit a jet, or even individual molecular ions. Work reported here was motivated by a desire to understand the onset conditions for a new type of electrospray, where both electric and magnetic fields are applied to a superparamagnetic fluid to achieve spray. In this technique the fluid is neither coated on the external surfaces of a solid needle nor fed through a hollow capillary and thus there is no imposed geometric length scale. Instead liquid peaks are formed in the free surface of a pool by exciting a normal-field instability via applied magnetic field; an applied electric field then acts in concert with the magnetic field to achieve spray.

The goal of this research was to develop a predictive modeling tool to analyze the flowfield and interface deformation of liquids subject to simultaneous electric and magnetic stresses. Images of deformed fluid volumes were analyzed using electro/magnetostatic simulation to determine the coefficient of surface tension for the fluid under study. Using this value of surface tension a dynamic Navier-Stokes and Maxwell stress model was employed to predict deformation shapes of droplets under the action of combined fields. The resulting simulated droplet geometries were verified against laboratory images.

**Meniscus Imaging Study Under Magnetic Stress**

An imaging apparatus was utilized to image a sessile ferrofluid droplet under controllable electric and magnetic fields. This setup, shown in *Figure 1*, backlights the ferrofluid droplet, enabling silhouette
images to be taken. This method of imaging creates well defined silhouettes, allowing for precise edge detection.

Figure 1: Diagram of imaging setup. (1) Blue LED collimated backlight source, (2) Ferrofluid droplet on slide within Helmholtz coil (not shown), (3) Variable zoom imaging lens, (4) Monochromatic CMOS camera.

A Helmholtz coil, shown in Figure 2, generated a variable uniform magnetic field. The coil consisted of a pair of solenoids, each containing 100 windings. The coil and power supply are capable of generating a maximum field of 310 Gauss. Slots in the coil core allowed for light to pass through to backlight the droplet. Coil current was measured with the use of a shunt resistor, and a calibration relationship was obtained with the use of an Alpha Labs GM-2 Gauss Meter with a High Stability Universal Probe. The fluid droplet was biased by applying a voltage to the electrode (2) in Figure 2 using a Stanford Research Systems PS350 high-voltage power supply with a range of 5kV.

Figure 2: (Left) Cross section of Helmholtz coil apparatus. The coil inner diameter is 3.8 cm. (1) Coil with 100 wraps per solenoid, (2) Biased electrode supporting droplet, (3) Ferrofluid droplet, (4) Grounded electrode. Spacing between the two electrodes is 4 mm. (Right) Image of Helmholtz Apparatus.

The ferrofluid used for all work reported here was commercially available EFH1 from Ferrotec. Aluminum, brass, stainless steel, and ITO glass were investigated as electrode material to qualitatively determine the wettability with the ferrofluid. It was found that the fluid had near perfect wettability to all surfaces tested. Ultimately, polished brass was selected for the supporting slide. It was found that applying the droplet to the slide in the presence of a magnetic field prevented the fluid from spreading - ensuring that only a single tip formed. Images were then taken, decreasing the field for each subsequent image.
Simulation Results and Comparison with Data

Two sets of laboratory images will be discussed in this section. Elements of these sets are presented below in Figure 3. In the upper set, the droplet was exposed to only a magnetic field. In the lower set, taken with a different droplet on a different day, the droplet was placed in a constant magnetic field and images were taken under varying electric fields. The mean value for the surface tension in the magnetic-only case was found to be 17.6 mN/m. This value differs from the 13.8 mN/m for the droplet imaged under both electric and magnetic fields. No correlation between surface tension and variables such as drop volume, magnetic field, electric field, or temperature has been identified. To eliminate surface contamination along the fluid-solid contact plane, the brass electrode was cleaned thoroughly with isopropyl alcohol between tests and allowed to dry. The authors therefore believe that the most likely form of this variation between droplets is a result of free surfactant in the ferrofluid which was not consistent between droplets.

![Figure 3: (Top) Droplet meniscus under magnetic field only. Droplet volume of 0.350 mm³. (Bottom) Droplet meniscus under magnetic and electric field held under a constant magnetic field of 216 Gauss. Droplet volume of 0.43208 mm³.](image)

The dynamic model was used to study a ferrofluid meniscus in the presence of only a magnetic field. To perform this study, the fluid droplet presented in Figure 3 for the magnetic-field-only case was selected. This droplet was modeled using a constant surface tension of 17.6 mN/m obtained from the static analysis. In Figure 4, the experimentally observed peak height is compared against the peak height that was obtained as the steady-state solution of the transient simulation for a magnetic field range of approximately 40 Gauss. As can be observed, the dynamic simulation was capable of accurately matching both apex height and droplet profile for the range of magnetic fields generated by the Helmholtz coil.
Next a droplet under combined magnetic and electric fields was modeled as shown in Figure 5. The calculated surface tension of 13.8 mN/m was used in the simulation. The permittivity of the fluid was not known experimentally and was determined by fitting a droplet contour at 2000 V while varying $\varepsilon_r$ of the ferrofluid. Ultimately $\varepsilon_r = 23$ yielded an acceptable fit. This permittivity value was then used to simulate the droplet shape in a range between 0 and 3100 V.

The dynamic simulation incorporating electric and magnetic fields was capable of accurately matching the meniscus profile for a droplet within a strong electric field up to approximately 2300 V. Above 2300V,
the laboratory droplet formed a sharp peak, analogous to a Taylor cone instability that was not captured in the dynamic simulation. The onset of this peak corresponded with measured current emission from the sharpened meniscus. The dynamic model captured the run-up to this instability as evidenced by an increasing growth rate, however an abrupt transition was not observed.

To conclude, we have dynamically simulated a single-peak normal-field instability for a ferrofluid in the presence of a magnetic field and combined magnetic and electric fields. The fluid surface tension was determined through a static model of the droplet interface. For the dynamic model in the presence of only a magnetic field, the tip was accurately modeled over the magnetic field range imaged. For the combined field case, the simulation yielded a high degree of accuracy for most of the range of electric fields imaged. However, for voltages near the experimentally observed onset of emission, the model was less accurate at predicting the apex geometry. It is likely that the true instability arises from a perturbation to the axisymmetric geometry that grows exponentially – a situation that is suppressed in the numerical simulation.

**Generating Heterogeneous Pericellular Matrix of Chondrocyte Sub-Populations In-Vitro by Microencapsulation**

- Shuo Wang, PhD Candidate, Mechanical Engineering – Engineering Mechanics
- Dr. Chang Kyoung Choi, Mechanical Engineering – Engineering Mechanics

More than 27 million Americans suffer from osteoarthritis, resulting in aggregate annual medical care expenses of $185.5 billion. Articular cartilage (AC) tissue engineering (TE) is a promising strategy to treat this disease. One key factor that determines the success of an engineered AC tissue is to employ a favorable cell source. Tissue engineering (TE) is a promising method to treat diseases that cannot be cured by traditional approaches. One key factor to determine the success of TE is to prepare favorable cell sources. A favorable cell source should be the cells whose viability, phenotype, and functions can be effectively and efficiently characterized and/or maintained in vitro. For articular cartilage (AC) TE, the main cell sources are primary and/or stem cell-induced chondrocytes. As engineering heterogeneous zonal AC tissues has gained popularity recently, cell sources used for ACTE are required to maintain not only their chondrogenic phenotype, but also the zonal phenotypes of chondrocyte subpopulations (in superficial, middle and deep zones of AC). It has been proven that the pericellular matrix (PCM) surrounding the chondrocytes could protect the inner cells, enhance their chondrogenic phenotype and induce the growth of AC tissues. These findings indicate that “chondrons” (a structural unit composed of chondrocytes with surrounding PCM) can be a better cell source. However, the availability of autologous primary chondrons is too limited to be utilized in practice, so investigators have attempted to culture chondrocytes in vitro to regenerate PCM. In-vitro generated PCM (neoPCM) is inferior to natural PCM in terms of morphology, structure, biocomposition and biomechanical properties. This neoPCM is still not a functional cell source for ACTE to engineer heterogeneous AC tissues.

The contribution of the outcomes of this study will be significant because: 1) The photopolymerization method will highly improve current alginate microencapsulation in terms of system robustness, production
accuracy and efficiency; 2) Knowledge gaps in how chondrocytes regenerate PCM in vitro given specific microenvironments will be filled; 3) An alternative cell source for ACTE, chondrocyte subpopulations with neoPCM, will be created. The proposed research is innovative because: 1) The concept of using microencapsulation to culture chondrocytes to regenerate PCM is originally proposed; 2) Photopolymerization will for the first time be employed for alginate microfluidic encapsulation; 3) Artificial chondrons (chondrocytes with neoPCM) will be created for ACTE.

**Approach**

**Isolation of primary chondrocytes and chondrons:** We successfully isolated bovine chondrocytes and chondrons based on enzymatical protocols. The primary chondrons will be the positive control groups for comparing with neoPCM. Here, AC samples were digested overnight in Ham's medium composed of 0.15% type I collagenase for chondrocyte isolation; and 5 hours in DMEM medium composed of 0.2% type II collagenase and 3.3 unit/ml Dispase for chondron isolation. Cell morphology and viability were justified under phase contrast microscopy (Fig. 1). To accurately isolate zonal chondrocyte or chondron subpopulations in the proposed experiments we will employ a microtome to harvest zonal AC tissues.

**Photocrosslinking microfluidic encapsulation:** By using our photo-crosslinking microfluidic encapsulation system, we have successfully photo-crosslinked 8% (w/v) oxidized methacrylated alginate (OMA) microbeads with 1% (w/v) VA-086 photoinitiator under ~10 mW/cm² UV (Fig. 2). This result proved the feasibility of our proposed OMA encapsulation approach. In the project, we will modify the microchannel dimensions to produce non-spherical shaped microbeads (disk and rod) to match the heterogeneous morphology of zonal PCM.
**Atomic force microscopy (AFM) characterization of cells:** We have simultaneously tested the topography and Young’s modulus distribution of cell subjects using an AFM (Fig. 3). We will employ AFM to test the mechanical properties of engineered AC cartilage at the microscale. We will also study the relationships between bio-structure, engineering parameters, and biomechanical properties of engineered AC with this AFM.

**Manufacturing and characterization of OMA microbeads:** OMA microbeads with various encapsulation factors will be generated in polydimethylsiloxane (PDMS) microchannel chips by controlling several engineering parameters in two successive procedures: droplet generation and curing. First, by controlling the flow rates of disperse phase (OMA solution with photoinitiator) and continuous phase (mineral oil with 4% w/w lecithin) OMA and the microchannel dimensions, microdroplets will be generated with different sizes and shapes, which are aligned with the morphology of natural chondron subpopulations. Second, UV will be applied to the wavy section of the microchannel to photocrosslink OMA droplets into micro gel beads. The UV exposure duration will be adjusted by controlling the flow rates and by covering the channel with aluminum foil. Gelled OMA microbeads will be washed with hexadecane, centrifuged, and collected, to verify the success of photocrosslinking. The droplet production rate will be calculated by analyzing the videos using a high-speed camera.

**Anticipated Results:** We expect that OMA microencapsulation will facilitate generation of significantly more biomimetic PCM compared to the previous bulk alginate culturing method. The neoPCM is expected to help maintain the zonal phenotypes and improve the chondrocytic metabolism for AC tissue engineering. We will also understand the kinematics of generation of PCM in controlled microenvironments *in-vitro*. In addition, the advantage of tunable degradation of OMA microbeads will be justified by comparing to conventional alginate beads.

**Future studies**

Several aspects of encapsulating chondrocyte to generate PCM can be further investigated. First, natural chondron subpopulations (MZ and DZ) have multiple chondrocytes per chondron. Further study of generating PCM will examine how to encapsulate multiple chondrocytes to generate the biomimetic columnar chondron. Second, we plan to employ human chondrocytes and/or human stem cell-induced chondrocytes in this method to fabricate human chondrocytic PCM. There are limitations in the use of autologous human chondrocytes, such as limited availability of human chondrocytes. Stem cell (e.g. mesenchymal stem cells) differentiated chondrocyte subpopulations can therefore be a promising cell resource. The knowledge of optimal microenvironments defined in this study can provide insights on stem cell chondrogenic differentiation in the future. Third, we plan to apply neochondrons into *in-vitro* and *in-vivo* AC tissue engineering, e.g. 3D printing, in order to identify the advantages of using this proposed new cell source to engineer heterogeneous AC tissues.
Appendix

Titles of Proposals Submitted by and/or Awarded to MuSTI Affiliates

Because proposals and ongoing research may involve companies or sub-contractors with which non-disclosure agreements are in place, proposal and awards details such as sponsors or amounts are not included here. Proposals and awards are listed in the approximate chronological order of submission or award. There may have been multiple awards for the same titled proposal in cases of multiple collaborators outside of Michigan Tech. The titles are indicative of the breadth of research that is conducted at the forefront of multi-scale technologies.

1. In-Situ Transient Analysis of Two-Phase Flow Pressure Drop in PEMEC Flow Channels
2. Foundation of Next-Generation Electrospray Propulsion
4. In-Situ Liquid Microscopy of Fiber-Fluid Interactions
5. Giant Enhancement of Nonreciprocal Response in Nanoscale Magneto-Optic Films
6. High Brightness Fluorophores
7. Toward Intuitive Ankle-foot Prosthesis Control: Gait Anticipation with Artificial vision
8. In-Situ Liquid microscopy of Biological Materials
9. Collaborative Research: Molten Polymer Microfluidics: Multilayer Film Manufacturing on a Benchtop
10. Collaborative Research: Multi-Scale Atomistic modeling of Fracture and Compressive Failure in Nanocarbon Reinforced Polymers
11. Electrospray from Magneto-Electrostatic Instabilities
12. A New Hybrid Plasma Flow Simulation Scheme with Directly Solving the Vlasov Equation for Electrons
13. Collaborative Research: Interfacial Stability and Evolution of convective Structure in Evaporating Films under Non-Stationary Conditions
14. Investigation of Liquid Film Instabilities on Evolution of Churn flow in microchannels and Derivation of a Capillary-Scale Two-Phase Flow Formulation
15. Study of Shock Waves Intensity Amplification Due to Cavitation
16. Comprehensive Studies on Gaseous Micro-Flows
17. Engineering Articular Cartilage Scaffolds with Microscale Zonal Heterogeneities by Applying Controlled Environments and Fluid Flow Shear Stimulation
18. Exploring the Compressibility Effect in Capillary Scale Gas-Liquid Two Phase Flows
19. A Particle-Gas Kinetic Method for Plasma Flow Simulations by Solving the MHD Equations for Electrons
20. Investigation of Thermal Interface Materials
21. I/UCRC: Novel High Voltage/Temperature Materials and Structures
22. SNM: SSIR, FIOM and Network Transition Behavior: Scalable Nanomanufacturing of Neat CNTs Pseudo-Crystalline Assemblages
23. Effect of Drop Deposition Dynamics on Nano and Microscale 3D Porous Structure for Advanced Manufacturing Electrodes for Electrochemical Technologies
24. Magnetically Enhanced Electrospray from Ferrofluids
25. NRI: From Biomechanics of Agile Gait to Control of 2-DOF Powered Ankle-Foot Prostheses
26. Mass Measurements of an electrospray Beam from a Single Emitter Ionic Liquid Ferrofluid Electrospray Source
27. Novel Ionomers and Electrode Structures for Improved PEMEC Electrode Performance at Low PGM Loadings
30. Development of a Swept Sine Vibration Measurement System