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### Electrochemical Cell Design and Experimental Setup for Passive Two-Phase Cooling

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### **Abstract**

The goal for the three credit-hour independent study in the Nanoscale Energy and Interfacial Transport laboratory was to familiarize myself with research methodology, energy storage on the micrometer scale, passive cooling for advanced electronics, and provide general aid in setting up the new laboratory space. Dr. Damena Agonafer, the principal investigator, assigned two projects to undergraduate researchers from the months of January 2017 to May 2017: novel methods for increased nanocapacitor performance, and passive two-phase cooling for microelectronics. Initial work consisted of background research into hybrid nanocapacitors, including materials, ordered quasi 2-D structures, and synthesis methods. This project was cancelled in progress in favor of the cooling project. After transitioning projects, work primarily consisted of designing and prototyping an electrolyte cell for synthesizing the required microstructures, modeling experimental setups in 3-D CAD software, and continuing to construct the laboratory. The outcomes of this independent study include a deep understanding of electrochemical energy storage and transfer and research methodology, a functional design for an electrochemical cell for synthesis of a copper inverse-opal structure, and a nearly complete physical laboratory space.

### **Introduction**

The Nanoscale Energy and Interfacial Transport Lab (NEIT Lab) is still in its primary stages, so much of the work from January to May of 2017 involved ordering equipment, writing grant proposals, and creating presentations for outside entities. A significant amount of the non-administrative work involved background research relating to unique microstructures, hybrid nanocapacitors, and micrometer scale two-phase cooling methods.

The first project assigned to me was the hybrid nanocapacitor project. Capacitors play a crucial role in high performance electronics, and because of the rising demands of these electronics, research into increasing gravimetric and volumetric energy storage and power delivery is vitally important, as well as finding an ideal balance between total energy stored versus deliverable power (energy per unit time). Batteries are able to store large amounts of energy, while traditional capacitors are able to deliver small amounts of energy very quickly; an ideal hybrid capacitor can store and quickly deliver large amounts of energy, bridging the gap between batteries and traditional capacitors.

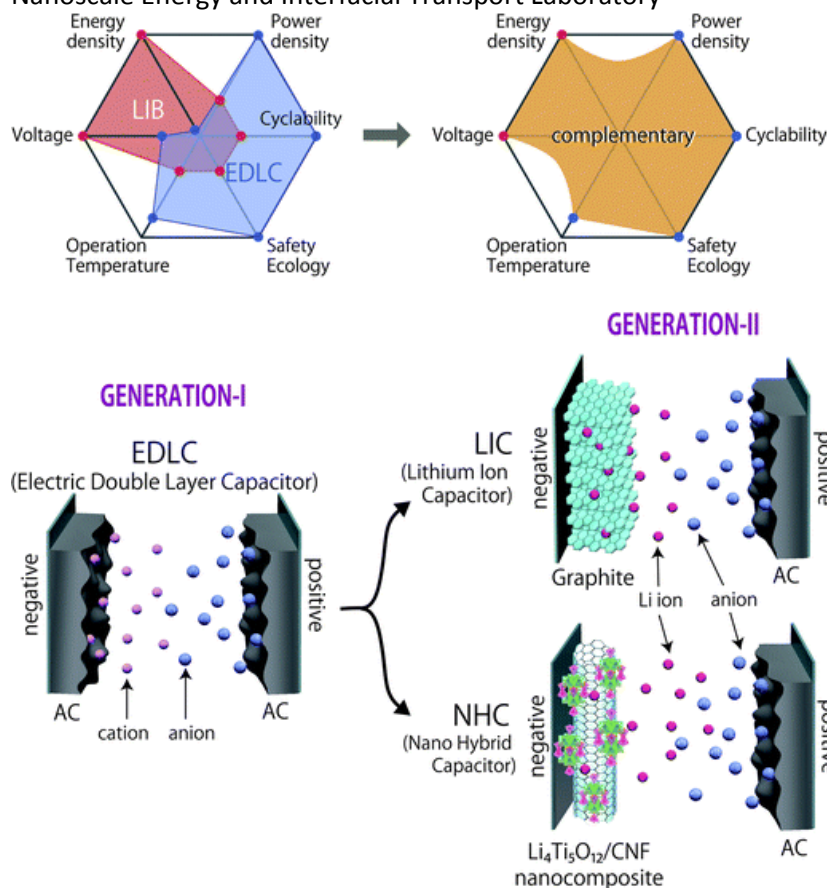


Figure 1: Differences between traditional capacitor design and doped carbon structures.

Energy Environ. Sci., 2012,5, 9363-9373

Unique solutions investigated include ordered graphene sheets interwoven with proprietary molecular chains to decrease charge propagation resistance and increase surface area available for charge storage. However, I transferred projects in order to consolidate efforts to the two-phase cooling project before actual synthesis of proposed nanocapacitors occurred.

The second project, two-phase passive cooling, is currently underway. The goal of this project is to develop and industry usable cooling solution, specifically for high density electronic applications where traditional cooling methods may not be used. Manufacturers of electronics need increased cooling capability as well as decreased size as waste heat increases from increases in performance and miniaturization.

The NEIT Lab is currently investigating a micrometer scale cooling solution integrated into 3-D electronics using dielectric liquids in place of water or other fluids, since these fluids can directly contact electronics without damage, and increase the heat transferred from the chip to fluid.

**Materials & Procedures**

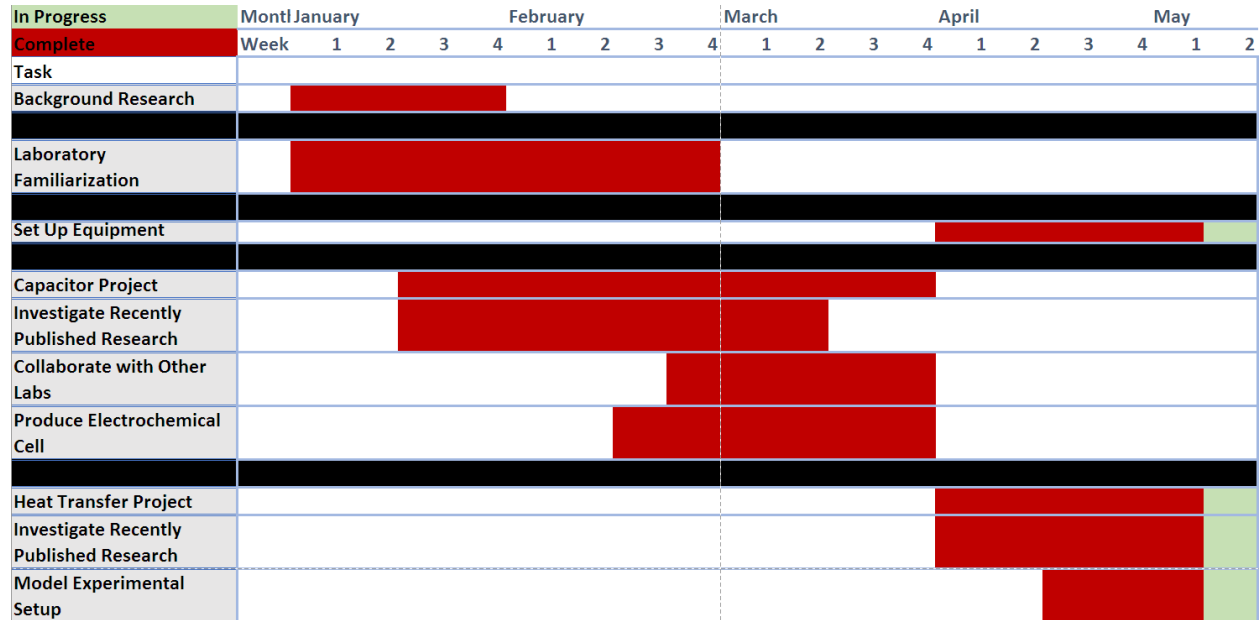
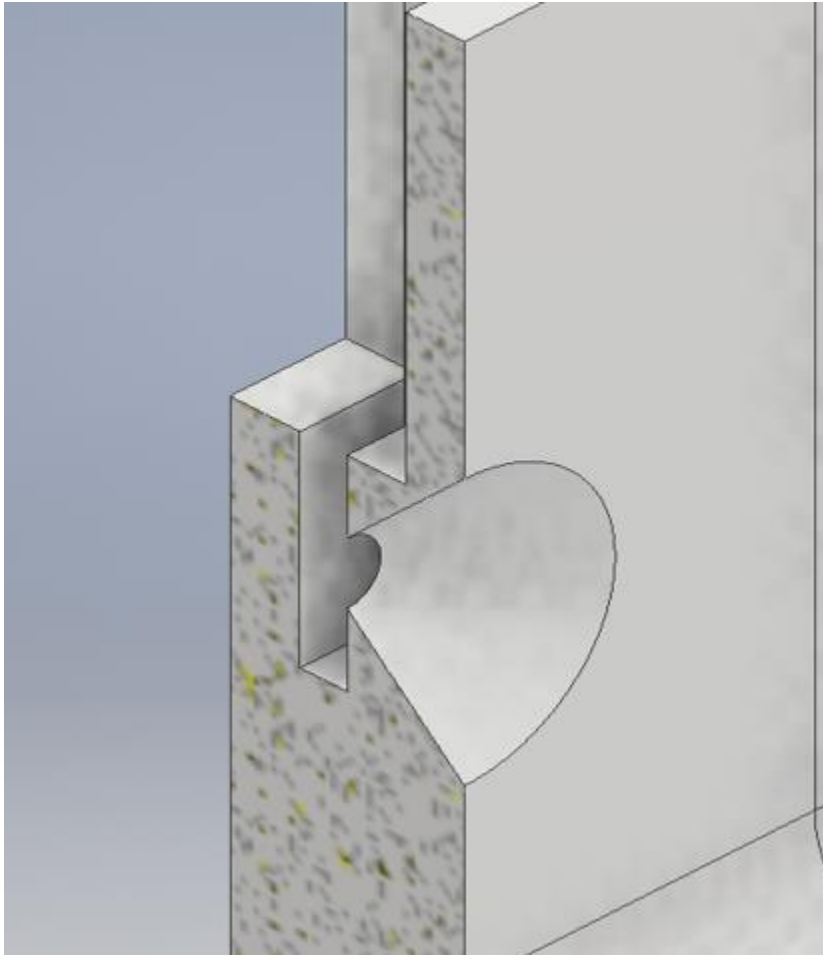


Figure 2: Gantt Chart showing student goals for the Spring 2017 semester

Initial work consisted of researching published data relating to capacitor performance, design, modeling, and synthesis. Peer-reviewed articles were found that gave insights into the theory behind nanocapacitors, modelling the physical phenomena, ideal materials and tweaking material properties, and how structures affect performance. Two main parameters were chosen for focus on improvement: volumetric and gravimetric energy storage. In order to produce a functioning prototype, a copper inverse opal (CIO) structure had to be synthesized to test the mechanical properties of the structure and establish a baseline to compare inverse opal geometries of different materials. An electrochemical cell was needed to hold the necessary fluids and supports and contain an electrical current to produce CIO structures; the cell had to minimize fluid stagnation points, especially near the substrate, and allow easy fluid mixing, as well as precise control over where the substrate and fluid came into contact. The cell design process was an iterative process. The first iteration was a simple 3-piece box; a slide holder (CIO growth substrate is on a microscope slide), the container, and a cap to hold electrodes. However, the seal between the fluid and slide holder proved to be an issue, as well as fluid stagnation points near sharp corners and edges. The second iteration attempted to solve the issue by using lip seals, but the manufacturing process required to make an effective seal would be cost prohibitive. Another issue would be having to indirectly connect the slide to the cathode, as the design did not allow for direct contact. After considerations from Dr. Agonafer were taken into account, the third iteration incorporated a two-piece design that could fulfill the requirements as well as be manufactured by machining or molding easily. The electrodes, as well as a small stir bar, could be press-fit into the cap, and the microscope slide could be easily inserted into and held by the container, while allowing for direct contact without supports to the cathode.



*Figure 3: 2nd Iteration sectioned view with a simplified geometry. The microscope slide would fit into the slot, with the substrate-fluid contact region in the small diameter hole. Small cuts & features not reflected.*

As the energy project was put on hold, the primary use of the cell did not change—the two-phase cooling project requires a CIO structure in certain regions of the proposed device.

As I transitioned into the cooling project, the supplies for testing menisci on proposed geometries were in the process of being ordered. Syringe pumps, goniometric equipment, and data capture equipment had to be ordered and set up to proceed with the testing of fluid-surface contact angles on the microfabricated geometries to be used in the cooling project. Before all supplies and experimental equipment was ordered, a model of the setup had to be produced to 1:1 scale in order to determine supply sizing and orientation. As of writing, the laboratory is still being set up and new team members are being trained in operation of the clean room,

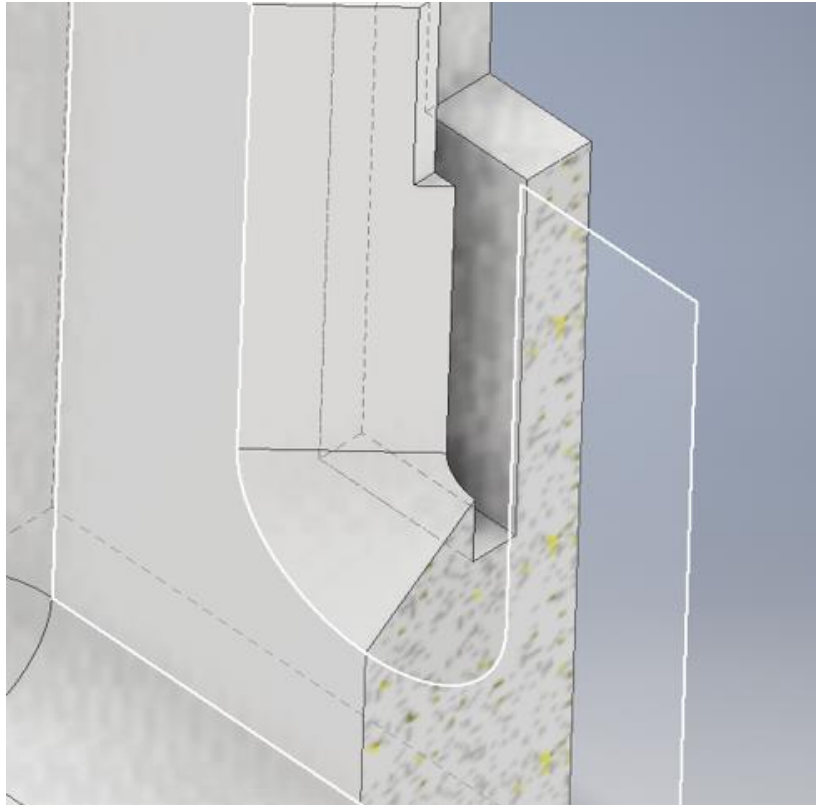
where menisci testing is to take place.

## Results

After the initial research phase for the hybrid nanocapacitor project, ideas to improve these performance metrics using previously published data were produced, with a likely candidate involving ordered graphene sheets and titanium dioxide ( $\text{TiO}_2$ ) showing promise. As investigation into the properties of materials and structures continued, the first iteration of NEIT Lab's nanocapacitor gave way to an inverse opal network structure of graphene doped with  $\text{TiO}_2$ . The final iteration before project transition was an inverse opal graphene network doped with annular polymer rods to increase both charge storage surface area and internal conductivity.

The final design of the electrochemical cell consisted of two parts: a small cap that could secure three electrodes and a stirring rod without outside parts to contaminate the electrode surfaces. The container

could hold a variable amount of fluid as needed without contacting the cathode, which is connected to a slide with a substrate for the ClO growth. Polytetrafluoroethylene (PTFE) was chosen as the cell material as it was inert with the chemicals to be used as well as non-conductive.



*Figure 4: 3rd Iteration. Simpler to manufacture, provides adequate protection from fluid for non-contact regions, as well allowing variable fluid/substrate vertical height. Small cuts & features not reflected.*

A 3-D CAD model of the meniscus experimentation setup was produced using part files sourced from the manufacturers to ensure proper spatial requirements before purchase. After the parts were ordered, the syringe pump, goniometric equipment, and data capture equipment were assembled in the laboratory clean room.

#### **Discussion & Conclusion**

Although I switched projects midway through the semester, the laboratory achieved significant progress in theory for the project. It could be possible to continue the hybrid nanocapacitor project at the prototyping and synthesis phase using work developed over the first half of the semester in conjunction with other

laboratories at Washington University. This project has major applications in high-tech industry, with companies that produce cutting edge military equipment to high performance vehicle manufacturers expressing interest, and moving forward could contribute greatly to the field of nanoscale electronics.

Passive two-phase cooling also shows potential with the same manufacturers, and continued research and advancement of the project could greatly impact the spatial and monetary costs of large-scale electronics cooling. Further research planned involves testing working fluid properties, such as the meniscus that forms from the surface of one component, as well as developing a small environmental chamber that allows close-up viewing of experiments in precisely controlled conditions.