ND*nano* Undergraduate Research Fellowship (NURF)
2012 Project Summary

1) Student name: Adam Talbot
2) Faculty mentor name: Dr. David Go
3) Project title: Plasma Jets for Nanomaterials Synthesis

4) Briefly describe any new skills you acquired during your summer research:

- Learned how to create an electrochemical cell using a micro-hollow plasma cathode
- Learned how to operate the Magellan Scanning Electron Microscope and use it to both take images and measure data
- Learned how to use X-ray Photoelectron Spectroscopy to identify the surface chemistry of a sample
- Learned how to find relevant scientific articles and use them in my research
- Learned how to prepare scientific posters and presentations

5) Please briefly share a practical application/end use of your research:

Silver nanoparticles have shown promising applications in enhanced field emission. Silver nanoparticles also have applications in medicine as antibacterial coatings and in agricultures as pesticides.

Project summary:

I. Background

Plasma, or the conduction of current across a gas, has often been called the fourth state of matter. When at least one of its dimensions is sub-millimeter in length, the plasma is considered a microplasma. The unique electrochemistry attributed to microplasmas makes them favorable for the synthesis of nano-scale materials. Plasmas contain and emit large quantities of energized electrons. When used as a cathode in an electrochemical cell, a microplasma directly reduces metal ions in solution to form nano-scale materials. Microplasma electrochemistry has led to the successful production of nanoparticles, nanostructures, nanotubes, nanostructured films, and nanocrystals. In particular, this project uses microplasma jets for the rapid synthesis of colloidal silver nanoparticles.

II. Activities and Results

This project aimed to create silver nanoparticles from microplasma interactions with a silver nitrate ($AgNO_3$) solution. The set-up included a glass electrochemical cell and a power supply. The anode for the cell was a relatively unreactive piece of platinum foil with dimensions 2.5 cm x 0.5 cm. The cathode was a stainless-steel capillary with an opening of

about 100 microns in diameter, through which flowed an argon gas neutral. The tip of this cathode was positioned 1-2 cm above the solution of $AgNO_3$.The microplasma consistently ignited at about 1.2 mA of applied direct current.

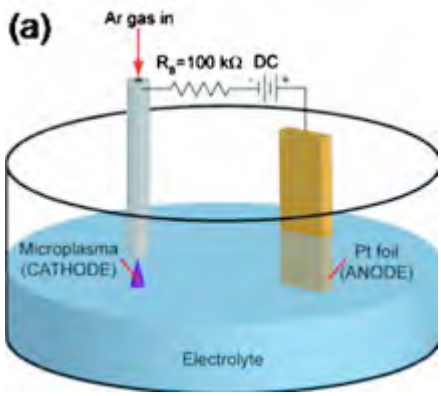

Fig. 1. Liquid-plasma electrochemical cell.

Just like in an ordinary electrochemical cell, oxidation occurs at the anode and reduction occurs at the cathode. However, when a microplasma replaces a metal cathode, positive Ag^+ ions are reduced directly and remain in solution, rather than adhering to the surface of the cathode itself. The associated oxidation-reduction reactions within this cell are:

Anode (oxidation): $2\ H_2O(l) \rightarrow O_2\ (g) + 4\ H^+(aq) + 4e^-$

Cathode (reduction): $Ag^+(aq) + e^- \rightarrow Ag(s)$

Overall: $4\ AgNO_3(aq) + 2\ H_2O(l) \rightarrow 4\ Ag(s) + 4\ HNO_3(aq) + O_2(g)$

Silver nitrate concentration, microplasma current, and operation time were varied in a series of trials. Trials were run at AgNO3 concentrations of either 0.01M or 0.001M, at currents of either 1.2mA or 1.6mA, and with operation times of 5, 10, 15, or 20 minutes. Parameters were mixed and matched for a total of 16 trials.

A fructose surfactant was consistently added to the $AgNO_3$ electrolyte solution at a concentration of 0.01M. The fructose reduces the surface tension of the solution so that particles do not agglomerate. However, purification to remove the fructose surfactant from samples containing silver nanoparticles was necessary, since organic molecules have adverse effects on the Scanning Electron Microscope (SEM). This proved to be difficult. Samples were centrifuged and washed with de-ionized water. Other samples baked on a titanium-coated silicon wafer substrate underwent dip purification in a number of solvents, including de-ionized water, methanol, and ethanol. The best method of purification involved baking the samples on the titanium substrate, followed by dip purification in de-ionized water then in ethanol.

X-ray Photoelectron Spectroscopy (XPS) confirmed the presence of pure silver nanoparticles on samples. It also showed significant purification of the fructose surfactant. SEM imaging revealed the spherical shape of the nanoparticles and allowed for the collection of data measurements. Silver nanoparticle size over all trials ranged from less than 5 nm to about 50 nm in diameter. The average silver nanoparticle size was about 15 nm in diameter. Trials 8 and 16 were run at 1.6mA for 20 minutes each and differed only in $AgNO_3$

concentration. Trial 8 was set at 0.001M and Trial 16 at 0.01M. Trial 8 had a mean diameter of 32.61 nm and Trial 16 had a mean diameter of 14.45 nm.Early data suggests that higher concentrations of $AgNO_3$ reduce nanoparticle size. However, more data must be collected to confirm this correlation.

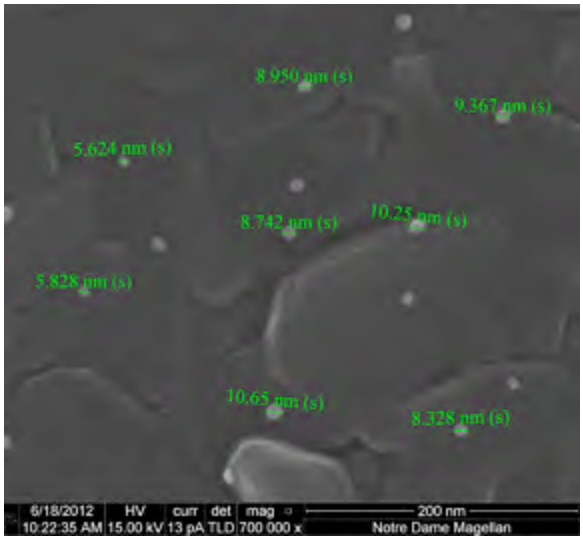

Fig. 2. Silver nanoparticles on titanium-coated silicon wafer substrate.

Publications (papers/posters/presentations):

1) Adam V. Talbot, Paul Rumbach, R. Mohan Sankaran, and David B. Go, "Rapid Synthesis of Colloidal Silver Nanoparticles via plasma-liquid electrochemistry," 2012 Undergraduate Research Summer Symposium, Notre Dame, IN, 2012.