

## Session N – Materials Science (Alphabetical)

### **A New Method in Determining Unknown Crystal Lattice Using Electron Diffraction**

Yutong Chen

*Mentors: William Goddard and Wenxi Liu*

The unit-cell is an important tool in characterizing unknown crystalline. To compensate for measurement errors, conventional method used to determining the unit-cell with transmission electron microscopy (TEM) utilizes large amount of diffraction films. Analyzing this large volume of data is time consuming and inefficient since thousands of samples with unknown crystallines are produced for a single product in the chemical, material, and pharmaceutical industries. A new efficient method in determining the unit-cell of unknown crystalline is developed. This method makes two improvements: 1) three diffraction patterns with a fixed reciprocal vector are used to correct measurement errors 2) a new set of twenty-seven cells with higher symmetry is proposed in place of the current forty-four Niggli cells. Using this method, the unit-cell of an unknown crystalline can be accurately determined within minutes.

### **Weibull Analysis of Compression and Tension Tests on Nanoscale Zr-based Metallic Glass Pillars**

Cameron Gross

*Mentors: Julia Greer and Dongchang Jang*

The mechanical properties of nanoscale pillars of metallic glass are not well understood. One can gain understanding of these properties through compressing and tensioning the pillars and observing mechanical failure. I utilized the data gathered by my co-mentor's compression and tensions tests of pillars with diameters less than  $2\mu\text{m}$  and performed standard Weibull analysis on these data. The cumulative probability of failure formula was taken from current literature. I determined a Weibull modulus of 12.3 for tension and of 12.2 and 16.1 for microscale and nanoscale compression tests, respectively. All sets of analysis showed a linear  $R^2$  value  $>0.9$ , demonstrating our data has a good linear fit. These results demonstrate our nanopillars are consistently more brittle than their larger microscale counterparts, and are less brittle than ceramics. This information can be used in the development of a mathematical model for failure in nanopillars.

### **Development of FeTi Based Alloys for Improved Hydrogen Absorption and Desorption Characteristics**

Sarah Howell

*Mentors: Brent Fultz and Channing Ahn*

Storage difficulties have limited hydrogen's use despite its great potential as an energy fuel. Metal hydrides, including FeTi, have useful qualities including the ability to absorb and release large amounts of hydrogen gas repetitively without deteriorating and their absorption and release rates that can be controlled by adjusting temperature or pressure. Modifications to these alloys can enhance stability during cycles of charging and discharging, allow them to undergo cycles at ambient pressure and temperature, increase their hydrogen storage capacity, and increase their hydrogen absorption and desorption rate.  $\text{Fe}_{1-x}\text{Ti}_x$  alloys with  $M = \text{Pd}, \text{Ni}, \text{or Cr}$  for different values of  $x$  were systematically synthesized with two methods of alloying: arc melting and mechanical alloying. The hydrogenation properties of some of these alloys were measured, the two methods of synthesis were compared, and each measured alloy's absorption and desorption isotherms at room temperature were analyzed.

### **Improving Magnetic Media Storage Capacity using Magnetic Thin Films – The Effect of Substrate on Magnetic Perpendicular Anisotropy of Co|Ni Multilayers**

Daniil Kitchaev

Mentor: Kathryn Krycka

As magnetic media shrinks and approaches the goal of Tb/in<sup>2</sup>, magnetic interactions between bits make it increasingly difficult to read and write individual bits without perturbing its neighbors. One solution is to reorient the magnetic easy-axis of thin-film structures perpendicular to the media surface by exploiting interface effects. It has been shown that very thin ( $t = 1$  to  $6 \text{ \AA}$ ) repeating layers of  $[\text{t Co} | 2\text{t Ni}]$  grown on Au produce perpendicularly magnetized structures<sup>[1]</sup>. When these layers are grown on Cu, however, the perpendicular magnetism is unexpectedly lost<sup>[2]</sup>.

It was found that the similarity of the lattice structures of Cu, Co, and Ni causes the thin Co|Ni layers to be strained – a phenomenon energetically sufficient to rotate the magnetic moments in-plane. However, to confirm this hypothesis, it was necessary to determine the degree of layering of the multilayer samples (nominally  $[\text{t Co} | 2\text{t Ni}] \times 12$  on Cu with  $t = 3 \text{ \AA}$  and  $6 \text{ \AA}$ ). For this purpose, a combination of x-ray and neutron reflectometry was used. This method is a depth-sensitive probe, with the x-rays sensitive to the

electron density, characterizing the overall sample layer thicknesses, and with the polarized neutrons sensitive to both structural and magnetic differences between the Co and Ni, It was found that in the t=6A sample, the multilayer superlattice peak unequivocally indicated significant layering, with the Co|Ni scattering length density (SLD) within 25% and magnetic scattering density (MSD) within 50% their bulk values, while in the t=3A sample, both the neutron and xray experiments suggested alloying. Thus, the combined reflectometry method proved to be effective in precisely determining the nuclear and magnetic structure of the Co|Ni samples, demonstrating that lattice strain is a significant factor in determining the magnetic anisotropy of our thicker samples.

- [1] G. H. O. Daalderop, et al, Physical Review Letters, 68, 682 (1992);
- [2] J-M. . Beaujour, et al, The European Physical Journal B, 59, 475 (2007);
- [3] T. Burkert, et al, Physical Review B, 69, 104426 (2004)

### **Mixed Conducting Electrodes for Solid Oxide Fuel Cells**

Kyung-ha Lee

*Mentors: Sossina Haile and Yoshihiro Yamazaki*

Among many different types of fuel cells, the solid oxide fuel cell (SOFC) is receiving attention because it is not limited under the Carnot cycle limit and does not require an expensive catalyst. It is reported that Y-doped barium zirconate (BYZ20) can be a good electrolyte for SOFC because of its proton conductivity. An appropriate cathode for this electrolyte necessitates both proton and electronic conductivities. Cobalt was doped to BYZ20 to create this cathode. In the experiment, various Co-doped BYZ20 powders with different compositions of cobalt were prepared through sol-gel method. To make the pellet, powder was calcined, pressed and sintered. From the dilatometer measurement for shrinkage and densification of the pellet depending on the temperature, it was found that 1300°C is the optimum sintering temperature for pellets. X-ray diffraction (XRD) and Electron Probe Micro Analysis (EPMA) were performed to confirm the sample's composition and structure. From AC impedance measurement at 450°C for a 40% Co-doped BYZ20 pellet, it was proven that the original goal of this project was achieved. A cathode with both proton and electron conductivity was made. Further experimentation will be concentrated on finding the optimum concentration of Co doping for the pellet.

### **Effects of Focused Ion Bombardment on Single-Layer Graphene**

Josue Lopez

*Mentor: Julia Greer*

We report the results of exposure of a single layer of graphene on a SiO<sub>2</sub> (300nm) / Si substrate to a focused beam of Ga<sup>+</sup> ions. In order to simultaneously monitor and control the sputtering of the material, experiments were performed in a dual-beam SEM/FIB system with a liquid metal Ga<sup>+</sup> source. Ion beam exposure was carried out at an acceleration voltage of 30kV and a beam current of 10pA. Electron-beam and Ion-beam magnifications were coupled in order to image the sample and identify damage after each subsequent single-frame exposure to the ion beam. The ion flux was determined at these conditions by intentionally etching a specified area in a thin amorphous carbon film of known thickness deposited on the same substrate via Plasma-Enhanced Chemical Vapor Deposition (PECVD). Our experiments show that a single layer of graphene is capable of withstanding greater ion beam exposure than calculated based on the ion flux obtained for the PECVD carbon film. This resilience suggests that the sputtering mechanism of this unique 2-D material is different from other carbon-based systems, leading to its potential use as a protective layer.

### **Synthesis and Characterization of Proton Conducting Solid Electrolytes:**

**Cs<sub>5</sub>H<sub>3</sub>(SO<sub>4</sub>)<sub>4</sub> and (Cs<sub>1-x</sub>Rb<sub>x</sub>)<sub>5</sub>H<sub>3</sub>(SO<sub>4</sub>)<sub>4</sub>**

Nicholas Scianmarello

*Mentors: Sossina M. Haile and Chatr Panithipongwut*

Solid acid compounds of the family M<sub>5</sub>H<sub>3</sub>(AO<sub>4</sub>)<sub>4</sub> are of interest for the room temperature stability. In addition, it is of academic interest why for rubidium Rb<sub>3</sub>H(SO<sub>4</sub>)<sub>2</sub> forms instead of Rb<sub>5</sub>H<sub>3</sub>(SO<sub>4</sub>)<sub>4</sub>, and vice-versa for cesium. Research was therefore done on the (Cs<sub>1-x</sub>Rb<sub>x</sub>)<sub>5</sub>H<sub>3</sub>(SO<sub>4</sub>)<sub>4</sub> binary phase diagram in an attempt to create a stable phase of Rb<sub>5</sub>H<sub>3</sub>(SO<sub>4</sub>)<sub>4</sub> that has an existence suggested by the high temperature decomposition of Rb<sub>3</sub>H(SO<sub>4</sub>)<sub>2</sub> into a new superprotonic phase, as well as the existence of Cs<sub>5</sub>H<sub>3</sub>(SO<sub>4</sub>)<sub>4</sub>. After a survey of four possible synthesis methods, small drop injection into acetone with a 4% metal cation deficiency, which is a quick precipitation of an ion solution of the reactants, was found to be most consistent and yielding the purest phase. This method was then used to study the desired binary phase diagram. Incorporation of rubidium is suspected to have occurred in the synthesized sample only up to 12% rubidium substitution. Observations through DSC and TGA have shown melting temperatures for Cs<sub>5</sub>H<sub>3</sub>(SO<sub>4</sub>)<sub>4</sub> in (Cs<sub>1-x</sub>Rb<sub>x</sub>)<sub>5</sub>H<sub>3</sub>(SO<sub>4</sub>)<sub>4</sub> up to x = 0.12 near the expected melting temperature of 540 K. Furthermore, the existence of a possible phase transition near 423 K was observed, as well as its

dependence on structural water. Single frequency conductivity measurements, however, did not yield any noticeable phase transition into a high or lower conductive phase.

### **Towards Synthesis of Gold Nanopillars Using the Vapor-liquid-solid Process**

Juying Shang

*Mentor: Julia Greer*

Nanoscale materials have demonstrated enhanced mechanical properties relative to their bulk counterparts. The result is a trend that for decreasing pillar diameter, the yield strength increases. However, below 200 nm in diameter, pillars are expected to change in their deformation mode. Identifying and characterizing this transition is important for the continued development of mechanical nanoscale systems. One possible way of synthesizing gold nanopillars with diameters below 200 nm is by the vapor-liquid-solid (VLS) mechanism with silicon as the catalyst and gold as the reactant for nanopillar growth. In a tube furnace, gold vapor is flowed towards a silicon substrate deposited with silicon nanoparticles. In order to determine the conditions for growth according to the gold-silicon eutectic phase diagram, the temperature profiles of the furnace are determined and the silicon nanoparticles are sonicated for better dispersion. The positions of the gold source and substrate, temperature settings, and flow rate and pressure of carrier gas are varied systematically to determine growth possibilities by looking at the samples under the SEM. Once consistent nanopillar growth is achieved, the pillars will be compressed uniaxially for evaluation of their mechanical properties.

### **Supercritical Activation of Cu-BTC and MIL-101**

Yi J. Wang

*Mentors: Brent Fultz and Channing Ahn*

Using renewable energy requires an efficient way to store excess energy for times when the energy source is not available. Hydrogen is a promising storage medium because it is clean burning, but storing hydrogen also presents a problem. Microporous materials can be used to adsorb hydrogen onto its internal surface area. Synthesized microporous materials, however, often have smaller internal surface area and hydrogen capacity than predicted. One possible explanation is pore collapse when the material is dried. As the solvents in the pores pass from the liquid to the gas phase, they exert capillary forces on the pores that cause the pores to collapse. If the materials are not dried, the solvent blocks access to the pores. Supercritical drying can be used to eliminate capillary forces. The solvent is exchanged with liquid CO<sub>2</sub> and the CO<sub>2</sub> is brought past the critical point so that the CO<sub>2</sub> never crosses the liquid/gas boundary and never exerts capillary forces on the material. This procedure should increase hydrogen capacity of the material by drying the material and leaving the pores accessible to hydrogen.

### **Micro-Engineering of Solid Oxide Fuel Cell Anodes: Fabrication of Metallic Inverse Opal Structures on Ceramic Substrates**

Stephen K. Wilke

*Mentors: Sossina Haile and Evan Brown*

We have fabricated metallic inverse opals on electronically insulating electrolyte substrates for use as advanced anode structures in solid oxide fuel cells. These inverse opals were constructed using a multi-step sacrificial template method. A template monolayer of polystyrene microspheres (diameters: 1.9 or 3.2  $\mu\text{m}$ ) was first placed on the substrate and then etched with oxygen plasma to reduce the sphere diameter. Nickel metal was deposited on the polystyrene via thermal evaporation, and the template was then physically removed, leaving behind an ordered, porous two-dimensional metal network. Following this, several layers of new polystyrene were deposited on the network, such that the bottommost layer of spheres aligned with the existing pores in the nickel structure. Heat treatment was used to fuse adjacent spheres together. Finally, nickel was electrodeposited up through the layers of polystyrene, and the spheres were then dissolved in toluene. These inverse opal structures can serve as frameworks into which ionic conductors can be incorporated, with the objective of creating fuel cell electrodes with precisely optimized microstructural features.