2011 Internal Conference
COLORADO CENTER FOR ADVANCED CERAMICS
Thursday & Friday, August 18-19

PROCEEDINGS
PROGRAM & PROCEEDINGS

COLORADO CENTER FOR ADVANCED CERAMICS
3rd Annual Meeting

Estes Park, Colorado
AUGUST 18-19, 2011

Organized by
Prakash Periasamy
Liangju Kuang
Taylor Wilkinson
Sarah McMurray
Welcome to the meeting,

It is with a great pleasure I welcome you all for this year’s CCAC 2011 conference in a beautiful location, Estes Park – the home of the Rocky Mountain National Park. This is the 3rd edition of this conference. As an organizing chair, I’m very delighted to introduce major changes to the conference this year. For the first time, we expanded the conference to a two-day event, mainly to accommodate more active discussions between the participants. In addition we have introduced a tutorial on electrochemical impedance spectroscopy (EIS). This Tutorial is one of a kind effort and is prepared and will be taught by graduate students to graduate students. In this tutorial a unique treatment is given to EIS and is targeted towards beginners and assuming no precedence of participant’s knowledge on this subject.

All these wouldn’t be possible if not for the trust in us by our Center Director, Prof. Ivar Reimanis. During the early stages of conference planning, when this idea was proposed to him, he encouraged and supported our new ideas. I’m very thankful to REMRSEC (Renewable Energy Materials Research Science and Engineering Center), CFCC (Colorado Fuel Cell Center), GSA (Graduate Student Association, CSM) and NSF (National Science Foundation) for their generous financial support in sponsoring this conference. In particular I’m very thankful to Prof. Craig Taylor and Prof. Neal Sullivan for their guidance and support during our fund generation process. My sincere thanks to Archana Subramaniyan, Tatiana Reyes, James Horan and Edwin Widjinarlo for agreeing to be a part of the EIS tutorial preparation team. On behalf of the EIS tutorial preparation team, I would also like to thank Prof. Jianhua Tong, Dr. Jian Li, Prof. Tom Furtak and Prof. Ryan O’Hayre for their help during this preparation.

My thanks to Alice Jensen for her help with all the tedious financial and travel issues. My special thanks to Kim Walker of the “YMCA of the Rockies” for patiently
working with me on the lodging and other arrangements at the YMCA facility. My personal thanks to Harvey Guthrey and Marc du Merac for agreeing to be the social event organizers. My thanks are also to Subramanian Ramalingam and Badri Narayanan for agreeing to be the official photographers to capture the important moments in this conference. My thanks to all the graduate students for presenting their research work in this conference and their active response during this entire planning stage. Last but not the least, my personal thanks to my fellow organizing members: Liangju Kuang, Sarah McMurray and Taylor Wilkinson. In particular, I thank Liangju for voluntarily helping me with some of the laborious preparation. Thanks to Sarah and Taylor for their work on the design and format on this proceedings. If not for the full support and help from the organizing members, it would be highly difficult to organize this conference.

Overall, I had personally learnt and enjoyed organizing this year’s conference and I sincerely hope that you will all enjoy the conference in the same spirit. Without your active participation, this conference will not be a complete one. Please help us make this conference a great success.

Looking forward to seeing all in Estes Park. I hope you enjoy this beautiful venue and find the conference very valuable.

Warm Regards,
Prakash Periasamy
Conference Organizing Chair, CCAC 2011 Conference.
Thursday’s Overview

10.30a Opening remarks from the Chair

10.40a CCAC Introduction, Ivar Reimanis

10.55a EIS Tutorial

Prakash Periasamy and Archana Subramaniyan

11.25a EIS Q&A

11.30a Lunch Break

12.30p Poster Showcase

Archana Subramaniyan, Stefan Nikodemski, Jason Fish, Daniel Clark, Sarah McMurray, Matthew Craft, Brian Hansford, Badri Narayanan, Harvey Guthrey, Robert Pasquarelli, Prakash Periasamy

1.00p Poster Session

3.00p Energy Harvesting

Ann Deml, Kevin Wood, Liangju Kuang

3.45p Break

4.00p Energy Harvesting

Grant Klafehn, April Corpuz

4.30p Networking & Dinner

Dinner served from 5-7.30p in common dining room.
Friday’s Overview

8.00a  EIS Tutorial
      Tatiana Reyes, James Horan
      Edwin Widjinarko

9.00a  EIS Demo, Q&A

9.15a  Break

9.30p  Oral: Processing & Behavior
      Subramanian Ramalingam,
      Amy Morrissey, Taylor
      Wilkinson, Prakash Periasamy

10.30a Break

10.45p Oral: Processing & Behavior
      Marc du Merac, Wan Zheng,
      Yi Ke

11.30a Vote of Thanks

11.45a Closing Remarks

12.00p Conference Adjourned
Effect of BaO on the Electrical Conductivity of the Prototypical Proton-Conducting Ceramic BaCe$_{0.8}$Y$_{0.2}$O$_{3-\delta}$

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20 mol% yttrium doped barium cerate (BCY20) is a pervoskite, well-known for its proton conductivity under wet or hydrogen atmosphere. They are important candidate materials for electrochemical applications such as fuel cells, sensors, and membranes. However, the evaporation of barium during sintering of BCY20 can result in: (a) barium deficiency at bulk and (b) BaO accumulation as a second phase at the grain boundaries, which can both decrease the proton conductivity. While the deficiency of barium at the bulk has already been studied in the literature, the effect of BaO has not yet been understood despite the hypothesis that it has a negative effect. Direct experimental observation of BaO in the grain boundary region is never been reported in the literature. In this work, we successfully fabricated BCY20 pellets with varying concentration of BaO second phase at the grain boundary region. The phase composition, microstructure, and electrical conductivity were characterized in detail using XRD, FE-SEM with EDS, and EIS, respectively. Using these samples, the effect of BaO on the grain boundary conductivity is being studied. In order to understand the effect of BaO, specific grain boundary conductivity (which excludes the geometrical effect) has to be calculated from the impedance measurements. This work explains in detail the existing complexities (due to the unusual microstructure, Figure 1) to calculate the specific grain boundary conductivity, based on the brick layer model. A possible modification of the brick layer model to suit this microstructure (Figure 1) is also proposed.
Metal nanoparticles were introduced in the proton-conducting ceramic \( \text{BaCe}_{0.6}\text{Zr}_{0.3}\text{Y}_{0.1}\text{O}_{3-d} \) (BCZY63) via internal reduction to generate an electronic space charge layer at the metal/ceramic interface. This study seeks to exploit the formation of a percolating network of the aforementioned space charge layers in order to enhance the ionic contribution to conductivity. This ionic hybrid material can result in an entirely new form of proton conductor which has potential energy conversion applications including batteries, capacitors, sensors, and fuel cells.

High-quality BCZY63 and BCZY63-M (M = metal dopant) powder was first synthesized by polymeric sol-gel method. The powder was subsequently pressed into pellets and sintered at different temperatures to produce both fully dense and porous ceramic pellets. The crystal structure, composition, morphology, and grain size of the BCZY63 ceramic pellets were characterized by XRD and FESEM. After that, the electrical conductivities were investigated using AC impedance in the temperature range of 100\(^\circ\)-650\(^\circ\)C under different atmospheres in order to determine if the metal dopant yielded a significant enhancement in ionic conductivity.
Development and Characterization of Mixed Protonic-Electronic Conductors for Hydrogen Separation Membranes

J S Fish¹ S Ricote² N Bonanos² and R O’Hayre¹

¹CCAC, Colorado School of Mines, Golden, CO, ²Fuel Cells and Solid State Chemistry Division, Risø DTU National Laboratory for Sustainable Energy, Roskilde, Denmark

Mixed ionic-electronic conduction is an important property for various fuel cells and gas separation membranes. Designing materials with simultaneously significant electronic and ionic conductivity allows for the current collector (electron transport), electrode (electrochemical reaction), and electrolyte (ion/mass transport) to be merged into one component, and/or to create better chemical and thermal stability in a multi-component system. The goal of the current work is to synthesize, optimize, and characterize novel perovskite oxide materials with mixed protonic-electronic conductivity for use as hydrogen separation membranes. Two material systems are being studied: 1) SrTiO₃ co-doped on the Ti⁴⁺ site with an acceptor (Al³⁺, Fe³⁺, Sc³⁺) and a donor ion species (Nb⁵⁺), and 2) a composite of Nb-doped SrTiO₃ (STN) and Y/Zr-substituted BaCeO₃ (BCZY). Initial synthesis results for solid-state reaction and sol-gel methods and phase characterization will be presented for both systems. The future plans for the project will also be discussed. In particular the effects of microstructure scaling (between nanosize and micron-size) on mass and electrical transport properties will be highlighted, along with detailed analysis with a variety of characterization methods such as cone electrode testing, conductivity studies, symmetrical cell testing, and hydrogen flux measurements. This work is being conducted jointly with researchers in the solid-state protonics group of the Fuel Cells and Solid State Chemistry Division at Risø DTU National Laboratory for Sustainable Energy in Denmark.
BaCe$_{0.7}$Zr$_{0.1}$Y$_{0.1}$Yb$_{0.1}$O$_{3-d}$: From Fundamental to Fuel Cell

D Clark$^1$ J Tong$^1$ and R O’Hayre$^1$

$^1$CCAC, Colorado School of Mines, Golden, CO

The co-ionic conductor of BaCe$_{0.7}$Zr$_{0.1}$Y$_{0.1}$Yb$_{0.1}$O$_{3-d}$ (BCZYYb) shows high coking and H$_2$S poisoning resistance and looks to be promising for applications such as SOFC anodes and electrolytes, steam/hydrogen/oxygen permeation membranes, and sensors etc. Three main efforts have been made in order to understand and use this material to make high-performing fuel cells. BCZYYb has been synthesized via a high purity sol-gel route in order to examine the fundamental transport phenomena happening within the material. The BCZYYb will undergo in-depth AC conductivity measurements in order to understand the co-ionic transport taking place. Additionally, 1 wt. % NiO has been added (BCZYbNiO1) in order to take advantage of solid-state reactive sintering (SSRS) and decrease the cost of the material by a factor of ten while still having a high-quality sintered material. Using TEM, the effect of the nickel on the BCZYbNiO1 conductivity will be examined before and after reduction and compared to the BCZYb data in the fundamental transport study. Finally, a fuel cell will be built using pulse laser deposition (PLD) to demonstrate the performance of the BCZYb as an electrolyte (BCZYbNiO1) and an anode. The anode is made of 35 wt. % BCZYb and 65 wt. % NiO that is reduced to produce a porous cermet. Using the cathode Ba$_{0.5}$Sr$_{0.5}$Co$_{0.8}$Fe$_{0.2}$O$_{3-\delta}$ (BSCF), the fuel cell will be microstructurally engineered via PLD and its performance will be examined.
TEM and Atom Probe Investigation of Calcium Carbonate Precipitation in Seawater
S G McMurray¹ B P Gorman¹ and D Diercks¹
¹CCAC, Colorado School of Mines, Golden, CO

Upper ocean waters experience a rise in pCO2 as it simultaneously rises in the atmosphere. The result is a decrease in the oceanic concentration of carbonate ions. This, in turn, leads to a decrease in the saturation state of seawater with respect to carbonate minerals. CaCO3 is not well understood regarding both precipitation and dissolution reaction kinetics and solubility behavior. In the last 50 years, it has been observed that aragonite precipitation acts in an unpredictable manner in relation to simple thermodynamic models. Indirect evidence suggests that there may be a surface phase with different properties of solubility, causing this unusual behavior.

In order to investigate this behavior, calcite and aragonite substrates were soaked for various times in different saturations of seawater. SEM analysis of the resulting precipitations shows an evolution of carbonate growth morphology that does not follow the Terrace Ledge Kink model for crystal growth. Previous work shows that there appears to be an amorphous calcium carbonate (ACC) surface layer nm’s thick above the overgrowth; and that the overgrowth itself seems to be growing epitaxially to the substrate.

FIB/SEM was used to prepare site-specific specimens for cross-correlative atom probe and TEM analyses. Low angle and low current FIB specimen preparation was combined with minimum laser pulses in the atom probe to reduce damage to the specimen surface. Results from this initial experiment on field evaporating CaCO3 are promising, though they reveal that much research needs to be done to optimize atom probe experimental parameters. A representative mass spectrum clearly shows the presence of thermal tails, which are most likely due to excess heat buildup in the specimen during laser pulsing. This excess thermal energy is also most likely to the cause of inaccurate stoichiometry in the bulk composition analyses. Using a laser pulse energy of 0.1 nJ, a temperature of 20 K the concentrations of each element are as follows: Ca: 20%, C: 11%, and O: at 69%, whereas the theoretical composition is 20%, 20% and 60% respectively. We suggest that a significantly lower laser power or a longer laser wavelength should be utilized.
Low Cost Sensor for Early Detection of Catalyst Coking

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Direct conversion of a hydrocarbon feedstock (e.g. natural gas) to electricity in a fuel cell system, either by pre-reforming or direct reforming in a solid oxide fuel cell (SOFC), is a major objective of the fuel cell community. However, carbon formation and the resultant catalyst deactivation of reforming catalysts is a significant problem in hydrocarbon reforming and direct reforming of hydrocarbon gases in SOFC systems. Carbon formation on catalysts has been studied extensively for many years and much effort is being put into developing catalysts that are resistant to carbon formation. Although many promising catalysts have been developed, catalyst deactivation by carbon formation remains a major obstacle to developing robust SOFC systems operating on natural gas and logistics fuels.

The objective of this project is to develop a low cost sensor for detection of carbon formation (coking) on reforming catalysts in SOFC systems. The device will seek to detect the onset of carbon coking before it begins to take place on the reformer; preventing damage and/or reduced effectiveness of the catalyst. The sensor will detect the presence of carbon formation by measuring the electrical impedance across a thin catalyst layer. Carbon formation on the catalyst layer increases electrical conductivity across the catalyst, leading to a response from the sensor that alerts the system operator or control system that a coking condition exists. Prototype sensors have been built and demonstrated in the Colorado Fuel Cell Center (CFCC). Once developed and initially tested, the sensors will be tested in SOFC reforming environments at the CFCC.

The current sensor design employs a Nickel catalyst supported by a partially stabilized zirconia substrate. The substrate is uniaxially pressed into a circular disk, and a Dimatix “drop on demand” printer is used to deposit the catalyst layer onto the substrate surface. These prototype sensors are then inserted into a tube furnace for conductivity measurements, where both the temperature and gas environment can be precisely controlled.
TRISO Coated Fuel Durability under Extreme Conditions

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Since the early 2000’s concern over ever increasing fossil fuel prices and stricter restrictions on greenhouse gas emission has helped in the revival of the nuclear power industry. This “nuclear renaissance” aims to provide clean domestic electrical power to cities in nuclear capable nations. However, high profile commercial nuclear power plant accidents over the last 30 years have led to concern for public safety. In order to change poor public perception, unfavorable economic environment, and worry over nuclear weapon proliferation research in the nuclear field continues.

As part of the ongoing nuclear resurgence, ceramics for use as structural materials in nuclear fuel assemblies are at the forefront of the research field. Ceramics have been used as structural materials for a long period in human history due to their high strength in compression, chemical inertness, and ability to tolerate elevated temperature environments. These properties along with others have made them of vital interest to the nuclear industry.

The current research aims to validate the use of ceramics as part of the nuclear fuel design by mechanical testing of silicon carbide hemispheres from surrogate nuclear fuel particles. These tri-isostructural (TRISO) fuel particles are a layered spherical fuel assembly undergoing validation testing for use in next generation nuclear reactors. As received SiC hemispheres as well as hemispheres exposed to CO and CO2 environments at different temperatures and times are being examined. Crush tests of the different varieties of silicon carbide hemispheres provides fracture stresses data for the different test environments. Finite element analysis is also being done to examine the differences in hemisphere geometry and verify the crush test.
Lithium aluminum silicates are an important class of engineering materials mainly because of their low (near-zero or slightly negative) thermal expansion coefficient and exceptional thermal stability. β-eucryptite (LiAlSiO₄) is a prominent member of this class with applications ranging from heat exchangers to ring laser gyroscopes and telescope mirror blanks. Recently, solid solutions based on β-eucryptite have been recognized to be promising materials for blankets and fuel coatings in breeder reactors. It is, therefore, essential to gain a fundamental understanding of the damage induced by incident radiation in β-eucryptite. It is well known that LAS ceramics become amorphous under irradiation; however, the nature and extent of the structural modification depends both on the type of material and the type of incident radiation. In the present work, we have employed classical molecular dynamics to study collision cascades in ordered β-eucryptite (a unit cell is shown in figure above) by imparting initial kinetic energy (up to 10 keV) to a primary knock-on atom (PKA). A reactive force field (ReaxFF) for Li-Al-Si-O systems, developed based on density functional theory calculations, was used to describe interatomic interactions. We determined the threshold energies required to cause atomic displacements for each atom species (i.e., Li, Al, Si and O) and compared them with those available from the literature for Al₂O₃. Higher energy cascades were then investigated to study the nature of radiation-induced defects, their accumulation and the mechanisms underlying structural modifications under irradiation. These mechanisms provide a plausible explanation for the amorphisation of β-eucryptite foils under a convergent electron beam (even at small spot sizes and low temperatures) as observed by Xu et al. [H. Xu, P.J. Heaney, H. Böhm, Phys. Chem. Min. 26, 633 (1999)]. The results have been discussed in the context of developing radiation tolerant materials based on β-eucryptite.
Understanding the Effects of Defects and Impurities in Poly-Si Wafers for PV Cells Using a Multi-Scale Characterization Approach

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As demand for poly-Si photovoltaics (PV) increases, so does the need for understanding the effects that defects and metallic impurities have on the conversion efficiencies of poly-Si PV cells. In order to reveal the true nature of the dependence of conversion efficiency reduction on defects and metallic impurities, it is necessary to use a multi-scale characterization approach. Regions that exhibit qualities known to be associated with conversion efficiency reduction must first be identified using macro-scale techniques capable of providing information about the entire wafer. The properties studied at this level include the spatial distributions of minority carrier lifetimes, defect densities, breakdown sites, and luminescence phenomena. Once an area has been identified as a likely limiter of conversion efficiencies the region is isolated so that smaller scale techniques can be performed. The smaller scale techniques provide information about impurity concentration, localized defect densities, recombination strength of specific features, localized orientation relationships, and defect structure. Combining the information gathered at the macro-scale, micro-scale, and nano-scale can provide an understanding of how individual features act to limit conversion efficiencies as well as how they originate with respect to processing conditions. This study demonstrates the benefit of using multi-scale characterization techniques to understand the factors limiting conversion efficiencies in poly-Si wafers used for PV cells.

When analyzed using small scale techniques the regions identified by macro-scale techniques were found to contain both structural defects and metallic impurities which together act to limit conversion efficiencies in poly-Si PV cells.
Solution Processing of Nb-doped TiO₂ and Ti-doped In₂O₃ Transparent Conductors

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Transparent conducting oxide (TCO) thin-films play a critical role in many current and emerging opto-electronic devices due to their combination of high transparency in the visible region of the spectrum and tunable electronic conductivity. Nb-doped anatase TiO₂ deposited by physical vapor deposition methods and on lattice-matched substrates has demonstrated potential as a replacement for indium-based TCOs such as indium tin oxide. Atmospheric-pressure solution deposition is an attractive alternative to these conventional vacuum-based TCO deposition techniques due to its ease and potential to lower device manufacturing costs. Here we report on 5 at% Nb-doped TiO₂ films deposited from solutions of Ti(OEt)₂(acac)₂ and Nb(OEt)₅. The desired anatase phase formed from an amorphous state on glass substrates at 500°C without the need of lattice-matching or a seed layer. Films were achieved with conductivities of 20–60 S/cm with 5x10²⁰ carriers/cm³ and mobilities ranging 0.1–0.6 cm²/V-s. Processing conditions had a significant effect on the final electronic properties and will be presented. The Ti precursor was also used in combination with the novel low-temperature indium precursor, In₃O(OPr°)₁₃, to prepare 3 at% Ti-doped In₂O₃ films. Undoped films have demonstrated conductivities of 100–230 S/cm with mobilities as high as 10 cm²/V-s.
Depth Controlled Point-Contact MIM diodes via Nanoindentation

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Metal-Insulator-Metal (MIM) diodes are candidate components for applications ranging from optical rectennas for harvesting sunlight to infrared detectors. One configuration that is popular due to its simplicity and ease of fabrication is the point-contact diode. In this study, precise control over the contact area and penetration depth of an electrically conductive tip pressed into a metal/insulator film combination is achieved using a nanoindenter with in-situ current-voltage measurement capabilities. A diamond probe tip, degeneratively doped with boron (b-Diamond) for conductivity, serves both as the point contact and as the second ‘metal’ of the MIM diode. Taking advantage of the precise penetration depth offered by the nanoindentation method, the influence of the native oxide and/or other contamination on diode performance is studied for four different metal (Nb, Ag, Pt and Au) surfaces. Nb, Ag and Pt were deposited as thin films (~200 nm) on glass substrate, whereas Au was in a pellet form. Atleast six I-V curves are acquired for each metal and are repeated on different days to examine repeatability. Excellent repeatability in the measured I-V curve performance is observed in all metals, indicating a uniform contact and device area (Figure 1). Pt/adsorbed layer/b-Diamond exhibited good diode behavior (Figure 1), with asymmetry and nonlinearity values of 56 and 11, respectively, at 1 V and a contact depth of 3 nm. The other metals exhibited ohmic behavior across the same bias range. XPS analysis was carried out to understand the nature of the surface on Pt and Ag films. XPS analysis on the Pt film (diode response) and the Ag film (ohmic) clearly indicated an increased concentration of C and O in the Pt film (C+O = 56 at%) compared to the Ag film (36 at%). The increased C based adsorbed layer (C+O) observed on the Pt sample may explain this material’s superior diode performance if the layer is acting as an insulator. This finding have a wider impact on expanding the search horizon for the insulator layer beyond oxides (in-organic) to organic insulators. In addition, the nanoindentation based approach provides highly repeatable data at a depth resolution that was previously unattainable.
Computational Thermodynamic Screening of Perovskites for Solar Thermochemical H$_2$ and/or CO Production

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Global efforts to develop secure, low-carbon energy systems have generated interest in hydrogen as a clean energy carrier and carbon monoxide as a hydrocarbon fuel precursor. One promising technology to produce these compounds by a clean, renewable process is the solar thermochemical (STC) dissociation of H$_2$O and/or CO$_2$, a process which utilizes concentrated solar radiation to thermochemically produce H$_2$ and/or CO. This attractive approach offers high fuel production rates and efficiencies by utilizing the entire solar spectrum and operating at high temperatures[1].

In a typical two-step thermochemical redox cycle, a metal oxide is reduced at high temperatures and then reoxidizes upon reacting with H$_2$O and/or CO$_2$ producing H$_2$ and/or CO, respectively. Several material systems have been demonstrated as effective in this application; however, these systems commonly exhibit slow reaction rates, material degradation, limited fuel production capacity, and/or complex cycling requirements[2]. Alternatively, perovskite oxides are well-known in the fields of solid oxide fuel cells and oxygen permeable membranes to exhibit good structural stability and tolerance of high oxygen nonstoichiometry which permits considerable oxygen release and uptake. Perovskites also possess high oxygen vacancy diffusivity, high oxygen surface exchange, good catalytic properties, and tolerance of extensive doping. As such, perovskite oxides hold significant promise for STC H$_2$ and/or CO production due to their promising kinetic properties and potential for favorable thermodynamic properties.

Here, we present an approach for the computational investigation of perovskites for STC H$_2$ and/or CO production. Our initial phase of investigation aims to evaluate and screen perovskite oxides for favorable thermodynamic properties using density functional theory (DFT) calculations as established by Meredig et al.[3]. Enthalpy and entropy differences between the oxides’ reduced and nonreduced states determine the thermodynamic performance of those materials and thereby indicate promising material systems. Correlation of these computational results with experimental studies will provide for an accelerated evaluation of materials and will promote progress toward renewable H2 and CO supplies.

Tuning Fuel Cell Catalyst Supports Though Fluorine, Iodine, and Hydrogen Functionalization

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Surface modification of highly ordered pyrolytic graphite (HOPG), a model catalyst support for fuel cell systems, have been shown to effect catalyst performance and durability as a function of dopant and implantation parameters. Nitrogen, nitrogen with hydrogen passivation, fluorine, and iodine were all investigated to ascertain the dopants ability to enhance both the performance and durability of the supported electrocatalyst. Raman spectroscopy and X-ray photoelectron spectroscopy (XPS) were used to study the structural and chemical modifications that various dopants induce across a wide range of ion implantation conditions.

These HOPG samples were then sputtered with consistently dispersed, equally sized, and alloyed nanoparticles of Pt/Ru. TEM, XPS, and Cyclic Voltammetry (CV), were used to characterize the changes in activity, morphology, coverage, and migration before and after potential cycling of the model catalyst system. As compared to an unmodified support, supports functionalized with nitrogen and fluorine showed dramatically improved Pt/Ru stability. All dopants investigated in this study, showed wide ranges of effectiveness as a function of dosage and improvement of the electrocatalyst support (durability and activity) was only seen after the dopant was incorporated into the structure of the surface. Effective exploration of the dopant contribution was seen at high dosages. Meanwhile low iodine dosage levels show negative effects on the nanoparticle stability. Various results depending on dosage and nitrogen with a hydrogen passivated surface.
Composite Nanoparticle-Directed Cost-Effective Microalgae Harvesting for Biodiesel Conversion

Microalgae are prokaryotic or eukaryotic photosynthetic microorganisms (most have sizes 2-50μm) that can grow rapidly and live in harsh conditions due to their unicellular or simple multicellular structures. Many microalgae species can be induced to accumulate substantial amount (up to 90% dry weight) of storage lipids that are only one-step away (e.g. a transesterification reaction) from transportation fuels. Various analyses based on production rate and ecological considerations have suggested that microalgae-derived biofuels may be the only realistic substitute to eventually release our reliance on fossil fuels. However, due to their low cell density, small cell size, low specific gravity (especially those species with high oil content), and like-charged cell surface, economic approach for large-scale microalgae harvesting remains as the most overwhelming barrier between microalgae in the farms and biodiesels at the pumps.

We consider that the microalgae dewatering problem can be understood in the context of colloidal stability, and directed, cost-effective microalgae harvesting through materials engineering is possible. We report here a composite nanoparticle design that combines two known colloidal destabilization agents (e.g. nanoparticle and polymer) into one system, and employs an external field (e.g. magnetic force) to not only modulate inter-algae pair potentials, but also facilitate retrieval of the coagulation agents to be reused after algal oil extraction. Both fresh water and marine microalgae species are tested to show high coagulation efficiency. Preliminary results on the composite nanoparticle preparation, structure-dependent microalgae coagulation performance, and the re-usability of the novel solid-phase coagulation agents for continuous microalgae farming and harvesting will be discussed.
Creating a variable temperature/humidity environment for testing mechanical properties of PEM fuel cell electrolytes

G W Klafehn and C E Packard
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As new fuel cell technologies are developed, they must also have an acceptable lifetime to be commercially adopted. Currently, in order to test fuel cell materials, prototypes containing samples of test materials must be fabricated, tested for an extended period of time, and dissected to conduct a failure analysis. If the internal conditions of a fuel cell can be experimentally simulated, the response of polymer electrolyte membranes to humidity-induced swelling may be characterized directly to efficiently estimate the strength and durability of a material.

Recent advances in high temperature nanoindentation have developed an understanding of the instrumentation and testing practices needed to conduct accurate nanoscale measurements under such conditions. In this presentation, we describe our efforts to build on that work by equipping a commercial nanoindentation system with a controlled temperature and humidified environment for high-resolution fuel cell membrane mechanical property measurement. Management of thermal stability, humidity regulation, and condensation in the system will be described.

Work toward understanding the mechanical properties of membrane materials as a function of temperature and humidity may one day allow for accelerated testing and the prediction of failure. The completed system will also allow for characterization of deformation processes in other materials that are sensitive to changes in temperature and humidity, such as fracture and delamination.

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Nitrogen-doping effects on PtRu/C anodes: Fundamental Study

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For fuel cell technology to become commercially viable advances in the area of electro-catalysts are necessary and it has been proposed that a methanol economy would have several advantages [1]. Specifically, for the commercialization of polymer electrolyte membrane (PEM) direct methanol fuel cells (DMFCs) catalyst activity needs to be increased by an order-of-magnitude [2]. To achieve this, much research has been done on nitrogen doping of the anode and cathode catalysts. Typically, carbon-supported platinum (Pt/C) is used as the cathode material and carbon-supported platinum ruthenium alloy (PtRu/C) is employed as the anode material. Nitrogen-doped carbon has shown increased activity in both the oxygen reduction reaction (ORR) on the cathode and the methanol oxidation reaction (MOR) on the anode in comparison to naked carbon [3,4]. In addition, platinum and platinum ruthenium alloys on nitrogen-doped carbons have shown increased activity in ORR and MOR respectively [2,4]. While this increased activity has been linked to several positive effects of nitrogen-doping, including smaller metal sizes and distributions, positive platinum-nitrogen interactions, and increased metal immobilization [2, 5-8], there is still much to be learned about the metal-nitrogen-carbon interface and its effect on ORR and MOR. All previously reported studies on PtRu/N-doped carbon have examined the effects of incorporating the metals after nitrogen doping. In order to better understand the role of nitrogen in N-doped PtRu/C, a series of nitrogen-doped in-house and commercial catalysts were studied: in-house Vulcan sputtered with PtRu, in-house nitrogen-doped Vulcan sputtered with PtRu, commercial PtRu on carbon, and nitrogen post-doped commercial PtRu on carbon. The catalysts were compared by creating inks and hand-painting anodes onto Nafton 117. These anodes, along with standard Johnson Matthey cathodes, were electrochemically tested for performance in methanol:air polarization curves and methanol anode polarization. They were also electrochemically characterized by determination of the active surface area (both anode and cathode). Additionally, diffuse reflectance infrared Fourier transform spectroscopy was employed to study the methanol decomposition reaction on the catalysts as well as model catalysts utilizing highly ordered pyrolytic graphite (HOPG) as the carbon support.
Utilizing Pressure-Induced Transformations for Toughening of Ceramics

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A novel mechanism for transformation toughening of ceramic composites using pressure induced transformations is proposed. b-eucryptite (LiAlSiO₄) is one of the prominent members of the family of lithium aluminum silicate ceramics owing to a number of attractive properties. It has a highly anisotropic coefficient of thermal expansion (CTE) (aₐ = aₐ = 7.26 x10⁻⁶ /°C and aₐ = -16.35 x 10⁻⁶ /°C), which leads to a negative crystallographic average CTE. Being an open crystal structure, b-eucryptite structure collapses under sufficiently high, applied hydrostatic pressure. It exhibits a pressure induced phase transformation to b-eucryptite at about 0.8 GPa. This transformation is characterized with nanoindentation and in-situ diamond anvil cell–Raman spectroscopy. Micromechanical modeling is presented that captures the amount of toughening and anti-toughening in zirconia/b-eucryptite composites. Toughening results from the dilation during the e -> b transformation, while the anti-toughening is due to the presence of high thermal residual tensile stresses developed due to thermal expansion anisotropy between zirconia and b-eucryptite. The model shows that a net gain of toughening is possible. To validate these results, composites of ZrO₂ with eucryptite of various particle sizes and volume fractions are made by hot pressing. This research identifies the key parameters required for achieving transformation toughening using pressure induced phase transformations.
Synthesis and Characterization of Nano-Ni, Co, and Fe Y2O3-Stabilized ZrO2

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Metal (M = Ni, Co, Fe) doped Y₂O₃ stabilized ZrO₂ (YSZ) has important applications in electrochemical devices. Controlled synthesis and subsequent thermal processing enables the development of nano-structural features which can enhance the electrochemical performance. An EDTA sol-gel method was developed to synthesize M-doped YSZ. Subsequent thermal treatments were used to develop nano- and micro-structural features of the ceramic powders. The high sensitivity of SQUID magnetometry is useful for studying the effects of low M-dopant concentrations in YSZ. Additional characterization techniques were employed to quantify the nano- and microstructure features of the M-doped YSZ powders. The objective is to identify the processing parameters which produce specific nano- and microstructural features and to correlate these physical characteristics to a magnetic response. The results indicate the magnetometry is useful for characterizing nano- and microstructural features of M-doped YSZ.
Characterizing the Environmental Degradation of Concentrating Photovoltaic Lenses through Nanoindentation

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Concentrating photovoltaic lenses increase the amount of power generated from a photovoltaic cell. Polymeric materials are currently under investigation for use in CPV systems, because they offer a light-weight alternative to silicone on glass technology that promises reduced installation costs and system complexities. The resistance of polymeric lenses to weather-induced aging (i.e., sand/soil ablation, hail, snow loads, moisture and UV exposure) must be evaluated to understand the consequences of material substitution. For this degradation study, poly(methyl methacrylate) (PMMA) samples are used.

As-received and aged PMMA samples of two different commercial formulations that have very different optical aging characteristics are provided by NREL. The aged samples are exposed to simulated accelerated UV degradation in a Ci4000 Weather-Ometer (ATLAS Material Testing Technology LLC) (with controlled temperature (60 °C) and relative humidity (60%)) for the cumulative duration of 12 months. Though the aging acceleration factor is not yet known for polymeric concentrating photovoltaic lenses, the conditions used will provide at least an 8x acceleration factor.

The samples are analyzed using quasistatic nanoindentation. This technique applies small loads with a diamond tip while tracking the displacement of the tip into the sample (within the nanometer range). The loading and unloading curves (a function of force and displacement) allow for the modulus and hardness to be calculated for the sample. Data is collected from the surface and cross-sections of all samples. The impact of aging (relative to the unaged references) on mechanical properties is compared for both PMMA formulations. Using this information together with optical data, manufacturers may be able to develop improved polymer formulations for concentrating photovoltaic lenses.
I-V Response of Planar MIM Diodes at Liquid Nitrogen and Room Temperatures

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MIM structures are promising candidates for high frequency rectification applications such as infrared cameras, THz communications and optical rectenna devices. This is owing to the fact that MIM structures, if designed properly, conduct electrons via tunneling – an ultra fast process ($10^{-15}$ s) which scales with the frequency ($10^{12}$ to $10^{15}$ Hz) of the target radiation to be rectified. Hence, for high-frequency applications, it is necessary to harness tunneling as compared to other conduction processes such as thermionic emission. In this work, the I-V response of four different MIM diodes based on Nb/Nb$_2$O$_5$ as metal 1/insulator blankets and Cu, Ag, Pt and Au as metal 2 electrodes are presented. I-V curves are collected at two different temperatures: 78 K and 298 K. At 78 K, the thermionic contribution is significantly eliminated and only the tunneling contribution is expected to be responsible for conduction, whereas at 298K, both contributions will be present. Nb/Nb$_2$O$_5$ blankets are prepared by DC sputtering and anodic oxidation to a nominal thickness of 100 and 15 nm, respectively. The top electrodes are deposited by DC sputtering and are patterned to yield a device size of 60 μm$^2$. I-V response was collected for a minimum of nine different devices for each MIM combination (Figure 1) to facilitate quantitative comparison with enough statistical confidence. Rectification performance was analyzed using two figures of merit: the asymmetry (ratio of forward to reverse bias current) and non-linearity (first derivative of current and voltage in the forward bias) calculated from the I-V response. It is preferred to have a greater contribution from tunneling than from thermionic emission (which is a slower conduction process). The relative contribution of thermionic versus tunneling conduction processes will be presented for each of the four devices. In all four devices, the asymmetry of the devices dropped considerably when measured at 78 K compared to 298K indicating a significant thermionic contribution at room temperature. Among the four devices, the Nb/Nb$_2$O$_5$/Cu MIM diode, exhibited the highest asymmetry and nonlinearity values at 78 K. This result gains significance since the above MIM combination is never been reported in the MIM research community.
Electro-optical Properties of Transparent MgAl$_2$O$_4$
Hot Pressed with LiF

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Magnesium aluminate spinel has an excellent combination of physical properties suited for use as transparent armor, IR-transparent domes, and possibly UV-transparent windows. However, it is difficult to sinter to the near-theoretical density required for transparency. Use of LiF additive and expensive, non-automatable processing, consisting of cold isostatic pressing/sintering or hot-pressing, followed by hot isostatic pressing are typically required to achieve transparency. Unfortunately, this processing methodology results in reduced mechanical properties and scattering at shorter wavelengths. An approach based on electrochemistry and characterization with transmittance analysis, IR-ellipsometry, Raman spectroscopy, and impedance spectroscopy is used to try and understand how defects affect the sintering process and optical properties. The relationship between sintering additive, sintering atmosphere, temperature and pressure profile, defects, and optical properties will be discussed.
Biocompatible Nanocarriers to Deliver Hydrophobic Therapeutics with High Loading Efficiency and Stimuli-Responsive Release Capability

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Controlled delivery of insoluble therapeutic agents in a dose-, time-, and site-specific manner across aqueous physiological pathways remains an outstanding challenge. This challenge is particularly troublesome in the fighting of cancers since most chemotherapy agents for the treatment of localized and metastasized cancers are fairly hydrophobic and have narrow therapeutic indices. We report here a nanocarrier design based on biocompatible chitosan and inorganic mesoporous silica. Chitosan is the N-deacetylated derivative of chitin, a naturally abundant polysaccharide with excellent biocompatibility, biodegradability, and biosorption properties. Mesoporous silica holds great promise as drug carriers due to its high structural stability, large specific surface area, tunable pore sizes (2-30nm), and abundant surface functionalization options. The nanocarriers we are developing have desirable size range (50-200nm) for prolonged circulation, high loading efficiency for hydrophobic pharmaceuticals, and smart drug release profiles in response to physiological stimuli. Preliminary results on nanocarrier design and preparation, prototypical payloads encapsulation, and stimuli-responsive drug release profiles will be discussed.
The Origin of the Electrical Property Deterioration with Increasing Mg Content in ZnMgO:Ga

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This work aimed to understand the microscopic causes of the reductions in conductivity/carrier concentration with increasing band gap (increasing Mg content) in the Ga-doped ZnMgO alloy. Based on the studies undertaken in this work, wide band gap ZnMgO:Ga thin films of world record conductivity were achieved. In addition, the detailed comparison of epitaxial versus polycrystalline films strongly indicated that both the intrinsic effects of increased Mg alloying as well as structural factors associated with crystal quality reduction contributed to a deterioration in the electrical properties of ZnMgO:Ga thin films. The results enable a strategic approach to a TCO material whose conductivity (or carrier concentration) and band alignment can be independently tuned. Such material would provide new ways to optimize the performance of a variety of optoelectronic devices that incorporate TCO contacts.

Specifically, transparent conductive Ga-doped Zn$_{1-x}$Mg$_x$O (ZnMgO:Ga) films were epitaxially grown via Pulsed Laser Deposition (PLD) on sapphire by optimizing the substrate temperature and other parameters of deposition. Zn$_{0.68}$Mg$_{0.31}$Ga$_{0.01}$/sapphire films deposited at 400°C have a Hall mobility ($\mu$) of $9.2 \pm 0.5$ cm$^2$V$^{-1}$s$^{-1}$ and a free electron density (n) of $1.79 \times 10^{20} \pm 0.06 \times 10^{20}$ cm$^{-3}$, yielding one of the highest reported electrical conductivity values ($s = 262 \pm 22$ S/cm), for doped ZnMgO films at similar Zn/Mg ratios. Zn$_{0.90}$Mg$_{0.09}$Ga$_{0.01}$/sapphire films, deposited under the same growth conditions, have similar crystalline quality, but significantly better electrical properties ($s=1450 \pm 10$ S/cm$^2$V$^{-1}$s$^{-1}$, $\mu = 24.5 \pm 2.5$ cm$^2$V$^{-1}$s$^{-1}$, n = $3.81 \times 10^{20} \pm 0.20 \times 10^{20}$ cm$^{-3}$). This comparison provides the evidence of electrical property deterioration in doped ZnMgO bulk material with increasing Mg content, independent of crystalline quality. Electrical properties of ZnMgO:Ga are further deteriorated by decrease of the crystalline quality. Polycrystalline Zn0.90Mg0.09Ga0.01O/a-SiO$_2$ samples deposited under the identical conditions on amorphous silica substrates had both inferior crystal quality and inferior transport properties ($\mu = 2.5 \pm 0.2$ cm$^2$V$^{-1}$s$^{-1}$, n = $2.04 \times 10^{20} \pm 0.20 \times 10^{20}$ cm$^{-3}$, s= $80 \pm 8$ S/cm) compared to their epitaxial counterparts.
CCAC News

Awards, Honors & Achievements

Professor Reimanis is a candidate for the Board of Directors for the American Ceramic Society.

Professor Reimanis is the Chair of the Program Coordinating Committee for the 2011 MS&T conference in Columbus, OH, October 16-20, 2011 (link: www.matscitech.org).

PhD student Marc Rubat-du Merac and Professor Ivar Reimanis summited Mt. Rainier in Washington State on July 30, 2011, with Marc’s wife Melanie and Professor Brian Flinn from the Materials Science Department at University of Washington.

Sarah McMurray received a Distinguished Scholar Award from the Micronalysis Society for attending the 2011 Microscopy and Microanalysis conference. The award is based on her submitted abstract and letter of recommendation; and covers registration as well as associated travel and accommodation expenses.

Prakash Periasamy, PhD student working with Prof. Ryan O’Hayre was awarded a doctoral scholarship for a four-month research stay (extendable up to 12 months) as a visiting PhD student at TU-Darmstadt, Germany under the mentorship of Prof. Andreas Klein. The scholarship was awarded based on a five-page proposal submitted by the applicant to perform a detailed XPS/UPS study on metal/insulator interfaces. The scholarship covers a monthly stipend with allowances and related research expense during the entire research stay.

Prof. Ryan O’Hayre was one of the four conference chairs for the Materials Research Society (MRS) Spring Meeting that was held between April 25-29, 2011 in San Francisco, California.

Prof. Ryan O’Hayre was awarded “Frontiers of Science Kavli Fel-
low” for the year 2011 by the Kavli Foundation.

Prof. Ryan O’Hayre was the “Symposium Organizer” for “Fuel Cells/Energy Conversion” at the European Materials Research Society Annual Meeting, Nice, France, May 2011.

Prof. Ryan O’Hayre was the Guest Editor of a Special Issue on Fuel Cells/Energy Conversion (2011) for the journal “Solid State Ionics”.

Archana Subramaniyan, Masters student working with Prof. Ryan O’Hayre was a finalist for the Max Planck Institute Graduate Fellowship Award to pursue PhD studies at Max Planck Institute of Solid State Research at Stuttgart, Germany.

Prof. Jianhua Tong was promoted to Research Associate Professor

Jason Fish, PhD student working with Prof. Ryan O’Hayre will begin a one-year PhD research fellowship at Riso DTU in Denmark.

Prof. Brian Gorman was awarded tenure in March.

New Equipment

The CCAC acquired a diamond anvil cell (DAC) with temperature stage this spring. It enables the study of materials at elevated temperature and pressure with techniques such as Raman spectroscopy and neutron diffraction.

Helios FIB / SEM was acquired as multi-user instrument.

Brian Gorman acquired 2 Cameca atom probes (one with STEM, electron diffraction) and a Zeta potentiometer donated from CoorsTek.

THE JEOL 6320 FESEM moved from the EMLab to 312 and Prof. Gorman will add nanomanipulators in the next couple months.
Prof. Gorman acquired JEOL T330 SEM from Missouri – Rolla and will install an X-ray tomography system in August / September.

Prof. Gorman acquired Philips EM430 300 keV TEM from Missouri – Rolla that will be dedicated to examining radioactive materials.

The Packard Research group has recently acquired a Hysitron TI950 Nanoindenter. Its capabilities include quasistatic and dynamic mechanical testing from small volumes (<500nm) of material. This machine is fully automatable and ideally suited for measuring the mechanical properties of thin films and from within single grains of material. Nanoscratch and wear testing are also available. Several tips and transducers are available for use depending on the needs of your application. Please contact Corinne Packard <cpackard@mines.edu> to inquire about contract testing.

**New Researchers**

New Research Assistant Professor has joined Gorman's group. David Diercks is a graduate of UIUC and UNT. He has 7+ years of experience with SEM, FIB, TEM, and Atom Probe tomography. His materials expertise ranges from ceramic processing to Si microelectronics. He will be working on the Dynamic Atom Probe primarily.

Amy Morrissey is a new PhD student working on the magnetometry of doped oxides under Prof. Ivar Reimanis. Amy received her BS in Materials Science and Engineering from Purdue University in May 2006, then worked four years in Materials Technology at Rolls-Royce Corporation before attending CSM.

John Moseley will be joining Prof Reimanis’s research group as a PhD student working on cracks near interfaces in toughened, transparent ceramics. John received a BS from the MME Department at Mines this past May.

Professor Corinne E. Packard joined CCAC last fall as an Assis-
tant Professor in the Metallurgical and Materials Engineering Department at CSM and holds a joint appointment at the NREL in the National Center for Photovoltaics. She earned her Ph.D. in Materials Science & Engineering from MIT for her research in nanomechanics and high temperature nanoindentation. At Mines, she explores the fundamentals of plasticity and stress-assisted failure mechanisms in materials, especially those used in energy applications.

Grant Klafehn is a PhD candidate in the Materials Science program with a focus in electronic materials. He received his Bachelor’s in Mechanical Engineering from Kettering University, a co-op focused school, in 2008. His Masters research focuses on creating an apparatus to allow high humidity, elevated temperature nanoindentation to mimic the environment inside a fuel cell.

Taylor Wilkinson graduated from New Mexico Institute of Mining and Technology in December 2010 with a Bachelor’s of Science in Materials Engineering. She is currently pursuing a Master’s of Science in Materials Engineering with a focus on the mechanical properties of fuel cell membranes.

Publications


T. Holme, Y.K. Zhou, R.M. Pasquarelli, R. O’Hayre; First principles


P. Periasamy, J. Berry, A. Dameron, J. Bergeson, D. Ginley, R. O’Hayre, and P.Parilla; Fabrication and Characterization of MIM Diodes Based on Nb/Nb2O5 Via a Rapid Screening Technique;


