

**UNIVERSITY OF FLORIDA**  
*An Integrated Sustainable Transportation System*

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**Description:** The proposed vehicle, operating on biofuel while in transit and charged by the sun while parked, is the ultimate sustainable transportation system operating completely on renewable American energy resources. Moreover, the use of solid oxide fuel cells (SOFCs) rather than an IC engine in this hybrid vehicle results in a dramatic improvement in efficiency and reduction in emissions. SOFCs are the most efficient technology for converting energy from hydrocarbon fuels to electricity on a “well to wheels” basis. In contrast, the more conventional fuel cells require hydrocarbon fuels to first be converted to H<sub>2</sub>, with resultant efficiency losses, followed by losses due to H<sub>2</sub> transport and storage. Therefore, on a system-basis SOFCs hold the potential for producing the least CO<sub>2</sub>/kWh from conventional fuels, and if designed to operate on biofuel would in effect be carbon neutral and operating on a renewable resource. *If developed this vehicle would be a transformational change in transportation technology.*

**Budget:** \$594,000

**Universities:** UF

**External Collaborators:** Solid-State Energy Technology, Inc., Lynntech, Inc., Planar Energy Devices, Inc., CFX Battery, Inc.

### **Progress Summary**

We have made significant gains in the science of energy conversion, from fundamental studies of the atomistic underpinning for materials properties to the engineering of the highest performance solid oxide fuel cells in the literature.

Fundamental studies of the two most promising materials for solid electrolytes, using computational modeling, enabled us to determine the optimal potentials to use (the Gotte potential) in predicting materials properties for oxides of interest in this field.

Moreover, from a molecular dynamics study of bismuth oxide we were able to confirm that dopant polarizability was the key feature in determining oxygen vacancy mobility in the fluorite system. Undergirded by these results we are now positioned to use our computational tools to further optimize the material properties of known oxygen ion conductors as well as develop new ones with superior performance.

This year we have also expanded the application of our previously developed continuum-level electrochemical model to describe and predict the performance of SOFCs as a function of electrolyte thickness. We were able to show that, for maximum power density, mixed conducting electrolytes had an optimal thickness below which they were rapidly overwhelmed by electronic conduction and above which their ohmic losses grew. We also are the first ones to predict the drop in open circuit potential with decreasing thickness for these materials.

Our development of record conductivity materials also continued with the highest conductivity ceria based and bismuth oxide based materials reported. We further studied their stability and optimal temperature window for operation. In so doing we have established our institution as the clear leaders in this area.

We are also leading in the area of anode development as the first to develop SOFCs with anode functional layers, which enabled a quantum step in SOFC performance. By comparison, our work on cathode performance is set to take off. We have already developed one of the highest conductivity cathodes (a bismuth ruthenate-bismuth oxide composite) in the literature. However, our fundamental studies on the oxygen reduction reaction (ORR) have positioned us to break further ground towards making a significant performance jump using conventional cathode materials. We have used multiple

techniques to investigate the factors governing the ORR and the results of those studies point to new electrode architectures that should both improve performance and increase stability (especially, with respect to chrome poisoning and secondary phase formation).

Finally, we have also found time to complete our research efforts in hydrogen production, which is critical for the realization of a future hydrogen economy. In addition, we moved forward with our work on sensors, by indentifying the factors affecting the sensing process and developing high selectivity sensors with high signal-to-noise ratios. This has garnered interest from industry, leading to collaborations with General Electric (GE) and Energy Management Solutions (aka EPS).

## 2010 Annual Report

Several accomplishments were made over the past year, these are summarized below according to category:

### Solid oxide Fuel Cells (SOFCs)

#### *Performance*

High performance SOFCs were developed for lower temperature operation using anode functional layers, single- and bi-layered electrolytes, and novel cathodes. GDC single-layer electrolyte-based SOFCs were prepared from four different fabrications and exhibit maximum power densities ranging from 0.338 to 1.03 W/cm<sup>2</sup> at 650°C. At each fabrication stage, an ESB layer was applied to form a bilayer electrolyte. Erbium-stabilized bismuth oxide (ESB) was deposited by a range of techniques including colloidal deposition and pulsed laser deposition. The result confirms that depending on a fabrication route, the bilayer electrolyte can reduce the total area specific resistance (ASR) 33–49% and increase the maximum power density 44–93%. By using a combination of the materials and fabrication routes, a maximum power density of 1.95 W/cm<sup>2</sup> and 0.079 cm<sup>2</sup> total cell ASR was achieved at 650°C for a bilayer cell.

#### *Electrolyte Development*

A new ceria-based electrolyte (Sm<sub>0.075</sub>Nd<sub>0.075</sub>Ce<sub>0.85</sub>O<sub>2-δ</sub>) was developed with the highest conductivity in the ceria system. Solid oxide fuel cells containing these electrolytes were fabricated and they produced very high power densities. At 650 °C, the maximum power density of the cell reached an exceptionally high value of 1.43 W cm<sup>-2</sup>, with an area specific resistance of 0.105 Ω cm<sup>2</sup>. Impedance measurements show that the power density decrease with decrease in temperature is mainly due to the increase in electrode resistance. The results confirm that Sm<sub>0.075</sub>Nd<sub>0.075</sub>Ce<sub>0.85</sub>O<sub>2-δ</sub> is a promising alternative electrolyte for intermediate temperature solid oxide fuel cells.

In a related study, comparing the grain ionic conductivity, Nd<sub>0.10</sub>Ce<sub>0.90</sub>O<sub>2-δ</sub> exhibited high ionic conductivity in the doped ceria system. The grain ionic conductivity is around 17% higher than that of Gd<sub>0.10</sub>Ce<sub>0.90</sub>O<sub>2-δ</sub> at 500°C, in air. From the lattice expansion data at high temperatures, the minimal elastic strain due to the presence of dopant was observed in Dy<sub>0.10</sub>Ce<sub>0.90</sub>O<sub>2-δ</sub>. However, Nd<sub>0.10</sub>Ce<sub>0.90</sub>O<sub>2-δ</sub> exhibited larger elastic lattice strain than Dy<sub>0.10</sub>Ce<sub>0.90</sub>O<sub>2-δ</sub> with better ionic conductivity at intermediate temperatures. Therefore, it was shown that the previously proposed crystal structure–ionic conductivity relationship based on minimum elastic strain is not sufficient to explain the ionic conductivity behavior in ceria-based system.

Stabilized bismuth oxide based electrolytes—(DyO<sub>1.5</sub>)<sub>x</sub>(WO<sub>3</sub>)<sub>y</sub>(BiO<sub>1.5</sub>)<sub>1-x-y</sub> (DWSB)—were also developed with the highest measured conductivity attained thus far. Studies were conducted to optimize conductivity and temporal phase stability. The conductivity of DWSB electrolytes increased linearly as the total dopant concentrations decreased with fixed dopant ratio. Overall, DWSB has a closer inherent structure to pure δ-Bi<sub>2</sub>O<sub>3</sub> than any singly doped compositions. Both lattice parameter and conductivity

linearly extrapolate with total dopant concentration to that of pure  $\delta$ -Bi<sub>2</sub>O<sub>3</sub>, resulting in the ability to stabilize  $\delta$ -phase at lower dopant concentration, and thus achieving higher conductivity.

We found that (DyO<sub>1.5</sub>)<sub>0.08</sub>(WO<sub>3</sub>)<sub>0.04</sub>(BiO<sub>1.5</sub>)<sub>0.88</sub>, 8D4WSB, was the highest conductivity composition but underwent the greatest conductivity degradation at 500 °C due to its low total dopant concentration. The effect of dopant composition on conductivity behavior with time at 500 °C demonstrates that there is a trade-off between initial conductivity and long-term stability at this temperature. Therefore, it is necessary to find an optimal total and relative concentration of dopants to provide the enhanced long-term stability needed to make this DWSB electrolyte system feasible for 500 °C operation. To this end, it was found that (DyO<sub>1.5</sub>)<sub>0.25</sub>(WO<sub>3</sub>)<sub>0.05</sub>(BiO<sub>1.5</sub>)<sub>0.70</sub>, 25D5WSB, maintained a conductivity of 0.0068 S/cm without appreciable degradation after annealing at 500 °C for 500 h. Moreover, since bismuth oxide-based electrolytes do not exhibit any grain boundary impedance, the total conductivity of 25D5WSB is significantly higher than that of alternate electrolytes at this temperature.

Several DWSB compositions with the same 2:1 dopant content ratio (Dy:W) were annealed to determine the conductivity behavior in the IT range with time. All DWSB compositions maintained their initial conductivity at 700°C, but underwent conductivity degradation at  $\leq 600^\circ\text{C}$ . The effect of total dopant concentration on the conductivity degradation behavior was investigated at 600° and 500°C. It was also found that 8D4WSB, which is the highest conductivity composition, has promise as an electrolyte for an isothermal operation above 650°C or below 400°C.

#### *Anode Development*

Anode supported solid oxide fuel cells (SOFCs) with various NiO content in the anode function layer (AFL) were fabricated and investigated. For AFLs with lower NiO content, the observed open circuit potential was higher. However, the highest maximum power density of 1.15 W/cm<sup>2</sup> at 650 °C was achieved at 60 wt% of NiO in the AFL, which is a 1:1 volume ratio of Ni to GDC phase. The optimal composition was valid in the intermediate temperature range from 450 to 650 °C. The effect of the optimal composition on power density was retained for 200 hours with high power density of 1.1 W/cm<sup>2</sup>. Electrochemical impedance tests supported this result by showing the lowest electrode area specific resistance at 60 wt% NiO, which has a linear relationship with maximum power density.

#### *Cathode Development*

The development of high performance solid oxide fuel cells is dependent upon the fundamental understanding of the oxygen reduction process at the cathode surface. Isothermal isotopic switching is a promising technique used to reveal the mechanism of oxygen exchange on (La<sub>0.8</sub>Sr<sub>0.2</sub>)<sub>0.98</sub>MnO<sub>3- $\delta$</sub>  (LSM) and La<sub>0.6</sub>Sr<sub>0.4</sub>Co<sub>0.2</sub>Fe<sub>0.8</sub>O<sub>3- $\delta$</sub>  (LSCF) powders. Various temperatures, pO<sub>2</sub> and sample sizes were explored. The rate of oxygen exchange for LSM was determined to be insensitive to changes in pO<sub>2</sub> and strongly dependent on temperature. The opposite is observed in LSCF, which was insensitive to changes in temperature between 600–800 °C and strongly dependent on the pO<sub>2</sub>. This behavior indicates that LSM is limited by incorporation of adsorbed oxygen atoms into the lattice and that LSCF is either gas phase diffusion limited or dissociative adsorption limited under operating conditions.

A 2-step mechanism was used to model the isotopic exchange. Rate constants and simulated profiles were obtained using an iterative program to fit parameters from experimental measurements. The fit for LSCF was good for both surface and bulk behavior, however, bulk conversion for LSM did not agree with the predicted behavior. LSCF fit the behavior expected for a surface coverage limited reaction, where the surface reaction occurs more rapidly than the mass transport of the reactants to the surface. The conversion for LSM was slower than predicted by the model, suggesting that the diffusion of oxygen from the particle core to the surface is the actual rate-limiting step. Degradation in LSCF samples was observed to occur after 20+ switching cycles; the reactivity difference was due to the reduction in the turnover frequency and not to a change in mechanism.

The impact of  $\text{La}_2\text{Zr}_2\text{O}_7$  (LZO) on interfacial resistance ( $R_p$ ) at the  $\text{La}_{0.78}\text{Sr}_{0.20}\text{MnO}_3$ -Ytria-stabilized zirconia interface was studied upon isothermal sintering at  $1200^\circ\text{C}$  for 2–25 h. Quantification of triple phase boundary length was performed by applying focused ion beam/scanning electron microscopy (FIB/SEM) serial-sectioning techniques and classical stereology. Electrochemical impedance spectroscopy was used to characterize  $R_p$ . The effect of LZO formation on microstructural models for  $R_p$  was analyzed with respect to previous research that did not include this effect. We found that LZO formation modifies the TPB length, rapidly increases  $R_p$ , and needs to be controlled in analytical microstructural  $R_p$  models.

$\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-\delta}$  (LSCF) displays mixed ionic electronic conductivity at high temperature that makes it suitable for solid oxide fuel cell cathodes, ceramic oxygen generators, and oxygen permeation membranes. However, upon changes in temperature and oxygen partial pressure LSCF exhibits changes in volume, termed chemical expansion, which can result in mechanical failure of the aforementioned devices. The chemical expansion change with temperature also complicates thermal expansion modeling. We measured and modeled the chemical expansion as a function of oxygen partial pressure ( $10^{-4}$ –1 atm  $\text{O}_2$ ) and temperature ( $700^\circ$ – $900^\circ\text{C}$ ) using our previous defect equilibria model to determine the chemical expansion coefficient per oxygen non-stoichiometry ( $\delta$ ) of 0.031. The thermal expansion was also measured and modeled using the Morse potential and Debye frequency distribution. The results were combined to yield a model describing the thermo-chemical expansion of LSCF.

$\text{Y}_{2-x}\text{Pr}_x\text{Ru}_2\text{O}_7$  ( $0 \leq x \leq 2$ ) pyrochlore powders were prepared using a precipitation method, which allowed control of their composition and morphology. Materials structure and morphology were characterized by X-ray diffraction analysis and field emission scanning electron microscopy, respectively. All the synthesized powders exhibited a single pyrochlore phase with particle size depending on the Pr content. Powders with Pr content smaller than 25 mol% were nanometric. X-ray photoelectron spectroscopy analysis revealed a mixed oxidation state of both Pr and Ru, and a variation of the oxidation state of the elements in response to oxygen partial pressure changes. Electrical conductivity measurements were performed by dc 4-probe method at several temperatures, showing that increasing the Pr content in the A site of the pyrochlore structure increased the oxide electrical conductivity.

To better understand the surface structure of cathode materials at high temperatures and thereby improve the SOFC cathode performance, the chemical and structural variations of  $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-\delta}$  (LSCF) surfaces under oxidizing environment were investigated. Cr contamination free LSCF showed the formation of submicron-sized  $\text{SrO}_x$  precipitates on the grain surface. This caused the reduced concentration of transition metals in B sites. The addition of cobalt oxide on the surface of LSCF was found to improve the cathode performance. In contrast, Cr vapor deposition caused the formation of larger  $\text{SrCrO}_4$  particles on the LSCF surface along grain boundaries, and Sr-deficient matrix. Structural analysis identified the phase transition from rhombohedral to cubic perovskite in due to Sr deficiency. A defect chemistry model was presented based on observed phenomena. Electrical conductivity relaxation, AC impedance spectroscopy and temperature programmed isotopic exchange (TPX) were conducted to study the impacts of Cr contamination on the cathode performance. It was found that Cr contamination was a chemical process for Co-Fe based materials and an electrochemical process for Mn based materials. Unstable  $\text{Co}^{4+}$  and  $\text{Fe}^{4+}$  lead to the formation of a nucleation agent for the chemical reaction. By contrast, stable  $\text{Mn}^{4+}$  does not lead to the formation of a nucleation agent. At last, a new hypothesis for the vaporization of Sr from LSCF was developed based on previous observations, and evidence for Sr vaporization was obtained.

A novel approach using isotope exchange was developed called isothermal isotope exchange (IIE) to study the oxygen reduction behavior on lanthanum ferrites and manganites. Two common kinetic parameters have been used to characterize the ability of a material to exchange oxygen: the tracer diffusion coefficient ( $D^*$ ) and the surface exchange coefficient ( $k^*$ ).  $D^*$  is a measure of the bulk diffusivity while  $k^*$  is a measure of surface exchange. Both aspects are involved in the oxygen reduction reaction (ORR), but in particular  $k^*$  can be difficult to measure. Techniques used to measure  $k^*$  currently require dense, thick samples that restrict testing to the diffusion controlled regime. IIE has the capability



of testing powder materials which allows for accurate measurement of  $k^*$  in the surface exchange controlled regime.  $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-\delta}$  (LSCF) was shown to have low activation behavior of  $k^*$  between 500-800 °C indicating the surface is catalytically unchanged within the temperature range. On the other hand  $k^*$  for  $(\text{La}_{0.8}\text{Sr}_{0.2})\text{MnO}_3$  (LSM) was observed to increase with decreasing temperature. This behavior is consistent with a precursor-mediated mechanism in which there is no energy barrier for chemisorbed dissociative adsorption. Composite cathodes were tested and results showed that when a pure electronic conductor is combined with a pure ionic conductor,  $k^*$  increases to higher values than either of the pure materials alone.  $k^*$  for different A- and B-site stoichiometries of lanthanum ferrites, cobaltites, and manganites were also measured. The mixed ionic and electronic conductors were shown to exhibit higher  $k^*$  values than the pure electronic conductors indicating the importance of having both electrons and oxygen vacancies present for ORR. These results provide guidance for improving  $k^*$  of SOFC cathodes and suggestions for optimization were made.

### Modeling

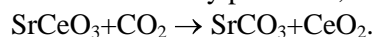
The materials fidelity of six interatomic potentials for ceria, based on predicted lattice constants, thermal expansion, chemical expansion, dielectric properties, oxygen migration energy and mechanical properties was assessed. While, no potential reproduced all fundamental properties, the Gotte (2007) and Grimes potentials displayed the combination of highest fidelity with the widest range of applicability. The simulations showed that sub-stoichiometry led to a significant softening of the elastic constant, which is consistent with the experimental results. Similar results were observed for doped-ceria systems.

The effect of polarizability of cation dopants on oxygen diffusion in  $\delta\text{-Bi}_2\text{O}_3$  was determined using molecular-dynamics simulation in which the polarizability of the ions was treated within the shell model. It was found that the magnitude of the oxygen polarizability had no effect on diffusion. However, the high cation polarizability, associated with the lone pair of electrons in Bi, was found to be the key to achieving sustained oxygen diffusion. Consistent with earlier experimental results, the oxygen diffusion path was found to be between oxygen equilibrium sites, which are displaced from the 8c oxygen sites of the fluorite lattice.

Our previously developed continuum-level electrochemical model was used to investigate the dependence of open-circuit voltage (OCV), and maximum power density on electrolyte thickness for solid oxide fuel cells (SOFCs) with mixed conducting electrolytes. Experimental results confirmed the models predictions that OCV decreases monotonically with decreasing electrolyte thickness due to increased permeation flux. The model was further extended to show that there exists an optimal electrolyte thickness at which maximum power density occurs for mixed conducting electrolytes. As expected, for electrolyte thicknesses greater than optimal, losses from ohmic overpotential reduce cell output. However, when the electrolyte thickness is lower than optimal losses from an increasing electronic “leakage” current reduce cell output.

### Hydrogen Production

$\text{SrCeO}_3$  and  $\text{SrCe}_{1-x}\text{Eu}_x\text{O}_3$  have been reported as promising materials for hydrogen separation membranes. However, their stability needs to be improved under hydrocarbon gas conditions. We investigated the effect of Zr doping in  $\text{SrCe}_{1-x}\text{Zr}_x\text{O}_3$  on chemical stability. We found that  $\text{CO}_2$  was the main species to cause the stability problems, and that decomposition occurs by the following reaction:



By using Zr doping, we subsequently demonstrated that hydrogen permeation through a tubular  $\text{SrCe}_{0.7}\text{Zr}_{0.2}\text{Eu}_{0.1}\text{O}_3$  membrane is stable under water gas shift reaction conditions.

### Sensors

Lanthanum ferrites and manganites have been shown to be excellent catalysts for use as sensing electrodes for potentiometric gas sensors to detect  $\text{CO}$ ,  $\text{CO}_2$ , and  $\text{NO}_x$  gases and for use as cathode materials for solid oxide fuel cells (SOFC) to reduce oxygen. However, these materials have complex

surfaces that are not well understood. For further optimization, more understanding of the surface behavior is needed. NO<sub>x</sub> adsorption behavior on LaFeO<sub>3</sub> (LFO) and LaMnO<sub>3+□</sub> (LMO) was characterized using temperature controlled methods and mass spectrometry. Temperature program desorption revealed decomposition of complex surface species formation when NO or NO<sub>2</sub> was adsorbed on LFO and LMO. LFO exhibited higher adsorption capacity for NO<sub>x</sub> species than LMO and was shown to be more active for NO<sub>x</sub> surface conversion. Both effects were attributed to the different B-site cations, with iron in LFO in the 3+ valence state, and manganese in LMO in the 3+ and 4+ valence states. Results from diffuse reflectance infrared spectroscopy were used to identify specific nitrite and nitrate species that are formed on the surfaces of LFO and LMO at room temperature. Temperature programmed reaction revealed a complex NO<sub>2</sub> decomposition mechanism to NO and O<sub>2</sub> for LFO and LMO in which the formation of nitrite and nitrate species serve as intermediates below ~600 °C. NO<sub>x</sub> sensing mechanisms were considered and predicted based on the types and quantities of surface species formed.

## Papers

1. Dependence of open-circuit potential and power density on electrolyte thickness in solid oxide fuel cells with mixed conducting electrolytes  
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2. A critical assessment of interatomic potentials for ceria with application to its elastic properties  
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3. Performance of anode-supported solid oxide fuel cell using novel ceria electrolyte  
*Journal of Power Sources, Volume 195, Issue 8, 15 April 2010, Pages 2131-2135*  
Jin Soo Ahn, Shobit Omar, Heesung Yoon, Juan C. Nino, Eric D. Wachsman
4. Isotopic-switching analysis of oxygen reduction in solid oxide fuel cell cathode materials  
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C.C. Kan, E.D. Wachsman
5. Effect of total dopant concentration and dopant ratio on conductivity of (DyO<sub>1.5</sub>)<sub>x</sub>-(WO<sub>3</sub>)<sub>y</sub>-(BiO<sub>1.5</sub>)<sub>1-x-y</sub>  
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9. Stability of SrCe<sub>1-x</sub>Zr<sub>x</sub>O<sub>3</sub>- under Water Gas Shift Reaction Conditions  
*J. Electrochem. Soc. 157, B383 (2010)*  
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11. Effect of ionic polarizability on oxygen diffusion in δ-Bi<sub>2</sub>O<sub>3</sub> from atomistic simulation

*Ionics, 2010, Volume 16, Number 4, Pages 297-303*

Dilpuneet S. Aidhy, Susan B. Sinnott, Eric D. Wachsman and Simon R. Phillpot

12. Enhanced long-term stability of bismuth oxide-based electrolytes for operation at 500 °C

*Ionics, 2010, Volume 16, Number 2, Pages 97-103*

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13. Stability of  $\text{SrCe}_{0.9}\text{Eu}_{0.1}\text{O}_{3-\delta}$  and  $\text{SrZr}_{0.2}\text{Ce}_{0.7}\text{Eu}_{0.1}\text{O}_{3-\delta}$  under  $\text{H}_2$  atmospheres

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Takkeun Oh, Jianlin Li, Heesung Yoon and Eric D. Wachsman

Thermo-Chemical Expansion in Strontium-Doped Lanthanum Cobalt Iron Oxide

*Journal of the American Ceramic Society 3 SEP 2010*

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Novel  $\text{Y}_{2-x}\text{Pr}_x\text{Ru}_2\text{O}_7$  ( $x = 0-2$ ) Pyrochlore Oxides Prepared Using a Soft Chemistry Route and their Electrical Properties

*Journal of the American Ceramic Society, Volume 93, Issue 7, July 2010, Pages: 1970-1977, Chiara*

Abate, Vincenzo Esposito, Keith Duncan, Juan C. Nino, Daniele Mirabile Gattia, Eric D. Wachsman and Enrico Traversa

Effect of Annealing Temperature and Dopant Concentration on the Conductivity Behavior in  $(\text{DyO}_{1.5})_x-(\text{WO}_3)_y-(\text{BiO}_{1.5})_{1-x-y}$

*Journal of the American Ceramic Society, Volume 93, Issue 5, May 2010, Pages: 1384-1391, Doh Won*

Jung, Keith L. Duncan, Matthew A. Camaratta, Kang Taek Lee, Juan C. Nino and Eric D. Wachsman

### **Patents**

Novel Conductive Glass Seal for SOFC Stack and Novel SOFC Fabrication Methods\_UF\_13478 (Yoon).

### **Industry Contacts**

General Electric

Energy management Solutions