Hydrogen Fuel Cell Development in Columbia (SC)

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Title/Topic: Hydrogen Fuel Cell Development in Columbia, SC (FY 2008)

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OVERVIEW

Timeline:

- Start – September, 2008
- Finish – May, 2010
- 45% complete

Budget:

- Total DOE funding: $1,476,000
- Total funding received as of March, 2009: $44,702.60

Barriers:

- Cost – of catalysts, electrodes, & seals
- Durability of PEM & SOFC for transportation and portable power
- Performance under transient operation, and in the presence of hydrogen impurities

Partners:

- University of South Carolina

RELEVANCE

Objectives:

The **general objective** of this program is to contribute to the goals and objectives of the Fuel Cell element of the Hydrogen, Fuel Cells and Infrastructure Technologies Program of the Department of Energy by enhancing and supplementing the fuel cell research and development efforts at the University of South Carolina. The project **research activities** focus on the following **technical objectives**:

- ❑ The development of metal-free oxygen reduction catalysts to reduce cost, facilitate manufacturing, and enhance durability of fuel cells (Barriers A-C; Task 2 electrodes)

- ❑ The development of redox stable mixed ionic and electronic conductors (MIECs) for bi-electrode supported cell (BSC) symmetrical SOFC designs, to reduce cost by simplifying manufacturing, enhance durability, and greatly reduce sensitivity to thermal cycling (Barriers A-C,G; Tasks 8-portable power, 11-innovative fuel cells, 10-long term failure mechanisms)

DOE Barriers: A-Cost, B-Durability, C-Performance, D-Transport, E,F-Thermal, air mgmt., G-Transient operation

RELEVANCE

Objectives (continued):

❑ The development of durable, low cost seals for PEM stacks, through the establishment of laboratory characterization methodologies that relate to cell/stack performance (Barriers A, C; Task 6 Seals)

❑ The development of understandings and methodologies to establish hydrogen quality as it relates to PEM cell applications for transportation needs (Barriers B,C,G; Tasks 9-models for impurities, 8-portable operation)

❑ The development of a first principles multiphysics durability models based on interpretations of Electrochemical Impedance Spectroscopy (EIS) data that link the multiphysics processes, the microstructure, and the material states, with cell impedance responses and global performance, mechanistically, as a foundation for engineering durability during design and manufacture of fuel cells (Barriers A-G; Tasks 9-models, 10-long term failure mechanisms, 11-innovative fuel cell design and manufacture)

• DOE Barriers: A-Cost, B-Durability, C-Performance, D-Transport, E,F-Thermal, air mgmt., G-Transient operation

Approach - Overview

Five sub-projects were selected by DOE to address technology challenges of cost, durability and reliability, system size, efficiency, and performance of PEM and SOFC fuel cells and systems. **Specific goals** addressed include specific power and energy density, cost, cycle capability, durability, transient response, and stack technologies.

1. Work on surface modification of carbon (previous DOE program DE-FC36-03GO13108) will be leveraged to create new carbon-based, metal-free catalysts for oxygen reduction.

2. Work done under a partnership with NASA Glenn, Savannah River National Laboratory, and ENrG Inc. will be leveraged to create a new symmetrical SOFC design with greatly increased durability, efficiency, and ease of manufacturing.

3. Recent advances at the University of South Carolina (USC) in controlled hydration and temperature characterization of polymer-based materials will be used to establish a methodology for characterization of materials for seals in PEM stacks, leveraging work being done in the USC National Science Foundation Industry /University Cooperative Research Center.

Approach – Overview (continued)

4. The partnership with NREL, ANL, SRNL, LANL and investigators at other universities involved in the DOE Hydrogen Quality program at the national level will form the foundation of an effort to understand contaminant adsorption/reaction/transport/performance relationships at low contaminant levels in PEM cells.

5. Conceptual foundations laid by previous and ongoing research supported by a variety of mission agencies and companies including United Technologies Fuel Cells, ExxonMobil, and Henkel Loctite will be used to create a multiphysics engineering durability model based on electrochemical impedance spectroscopy interpretations that associate the micro-details of how a fuel cell is made and their history of (individual) use with specific prognosis for long term performance, with attendant reductions in design, manufacturing, and maintenance costs and increases in reliability and durability

PROJECT SUMMARY

The activities of the present program are contributing to the goals and objectives of the Fuel Cell element of the Hydrogen, Fuel Cells and Infrastructure Technologies Program of the Department of Energy through five sub-projects, which report significant progress since beginning in September, 2008:

- The development of **metal-free oxygen reduction catalysts** to reduce cost, facilitate manufacturing, and enhance durability of fuel cells
- The development of redox stable **mixed ionic and electronic conductors** (MIECs) for bi-electrode supported cell (BSC) symmetrical (and other) SOFC designs
- The development of durable, **low cost seals for PEM stacks**, through the establishment of laboratory characterization methodologies that relate to cell/stack performance
- The development of understandings and methodologies to establish **hydrogen quality** as it relates to PEM cell applications for transportation needs
- The development of first principles multiphysics durability models based on interpretations of Electrochemical Impedance Spectroscopy (EIS) data that form a foundation for **engineering durability during design and manufacture** of fuel cells

COLLABORATIONS

1. Member of the North American Fuel Quality Team organized by Dr. James Ohi (NREL) to addresses the impact of critical hydrogen fuel constituents as they affect the barriers of Durability, Cost, and Performance
2. Savannah River National Laboratory (SRNL) for nanocrystalline ceramic synthesis
3. Air Force Research Laboratory (AFRL) - sulfur-tolerant anode development, with support for a summer faculty research fellowship for investigator.
4. Dana and Dow-Corning – providing materials as well as their knowledge in seal materials
5. General Motors corporation – correlation with their stack testing results
6. Collaboration with ENrG Corporation on the modeling of the bielectrode supported (BSC) SOFC electrode architecture.

FUTURE WORK

1. **Hydrogen quality -** validate potential measurements by using reference electrodes and mass balance techniques to allow determination of partial current densities associated with NH_3 transport and reaction on the cathode; study the interaction of CO and NH_3 contaminants; continue work on the model for recovery of performance from contaminants; measurement isotherms.
2. **Carbon composite catalyst -** prepare using mesoporous carbon support; improve integrity of the carbon composite catalyst layer in the MEA; reduce MEA resistance by decreasing the catalyst layer thickness and by increasing the specific gravity and activity of the catalyst
3. **Hydrocarbon fuel SOFC -** Ionic conductivity of LSGMn pellets in different atmospheres at different temperatures will be measured; cell performance using the macroporous LSCF as cathode will be evaluated
4. **Gaskets and Seals -** design new compression set tests to include various compression strains and more realistic heating/cooling cycles to FC operation; develop a life prediction model
5. **Durability modeling in SOFC -** complete button cell test system and EIS test protocols; complete conductivity model of BSC electrode configuration

Sub-Project 1: Development of Carbon Composite Electro-Catalyst for the Oxygen Reduction Reaction (ORR)

Gang Liu, Xuguang Li, Branko N. Popov

Department of Chemical Engineering
University of South Carolina

Technical Accomplishments & Progress - Catalysts

OVERALL

Project objective

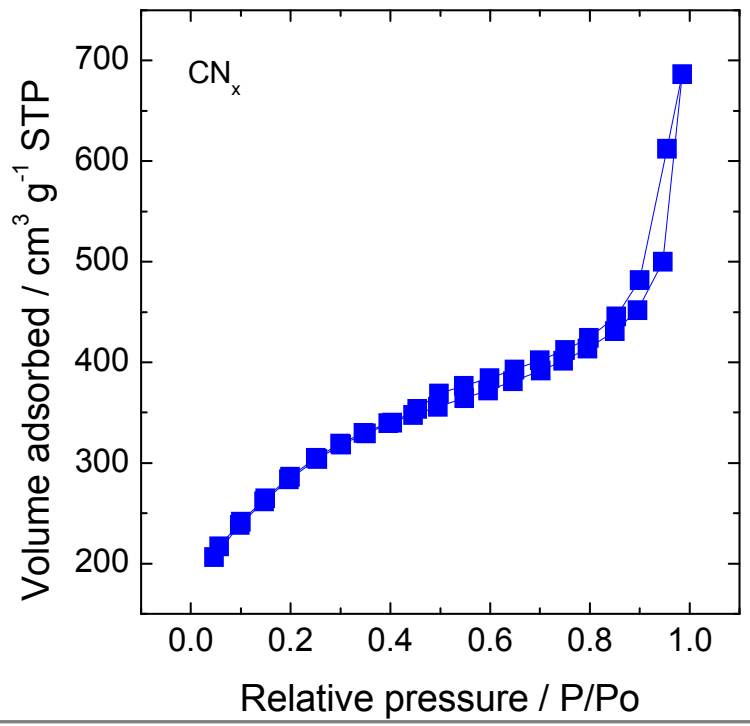
To develop non-precious metal catalysts for PEMFC with high catalytic activity, selectivity and durability which perform as well as conventional Pt catalysts with a cost at least 50% less than a target of 0.2 g (Pt loading) / peak kW

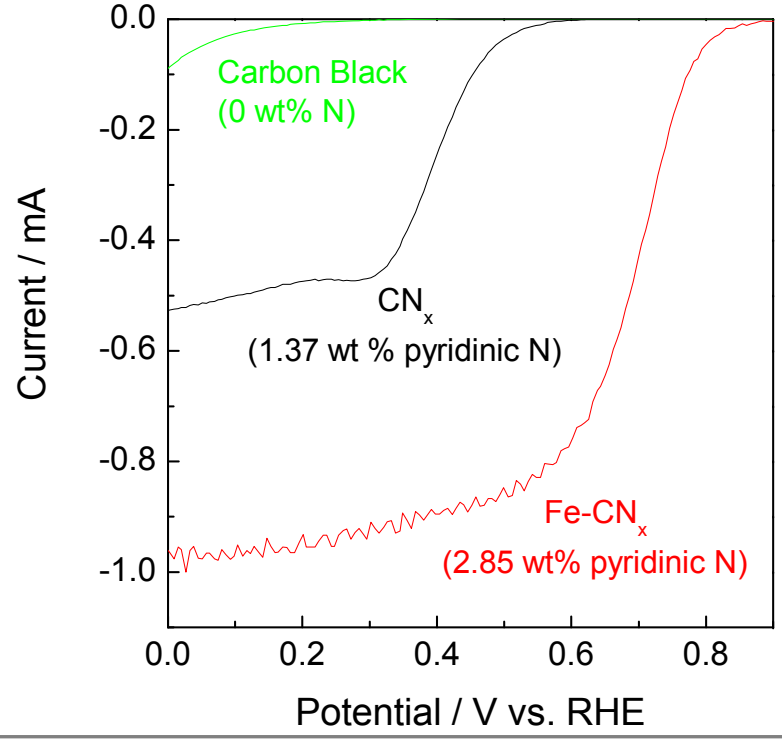
SPECIFIC FOCUS

- **Synthesize mesoporous carbon-based composite catalysts for ORR.**
- **Optimize catalytic active reaction sites as a function of carbon support, surface oxygen groups, nitrogen content, surface modifiers, pyrolysis temperature and porosity.**
- **Improve water management by controlling hydrophobicity of catalyst layer and by reducing catalyst layer thickness.**
- **Increase the fuel cell durability by improving the integrity of the carbon composite catalyst layer in the MEA.**
- **Reduce the MEA resistance by decreasing the catalyst layer thickness and by increasing the specific gravity and activity of the catalyst.**
- **Develop a low platinum loading hybrid cathode catalyst by using the carbon composite catalyst as a support.**

Technical Accomplishments & Progress - Catalysts

Mesoporous carbon-based composite catalysts

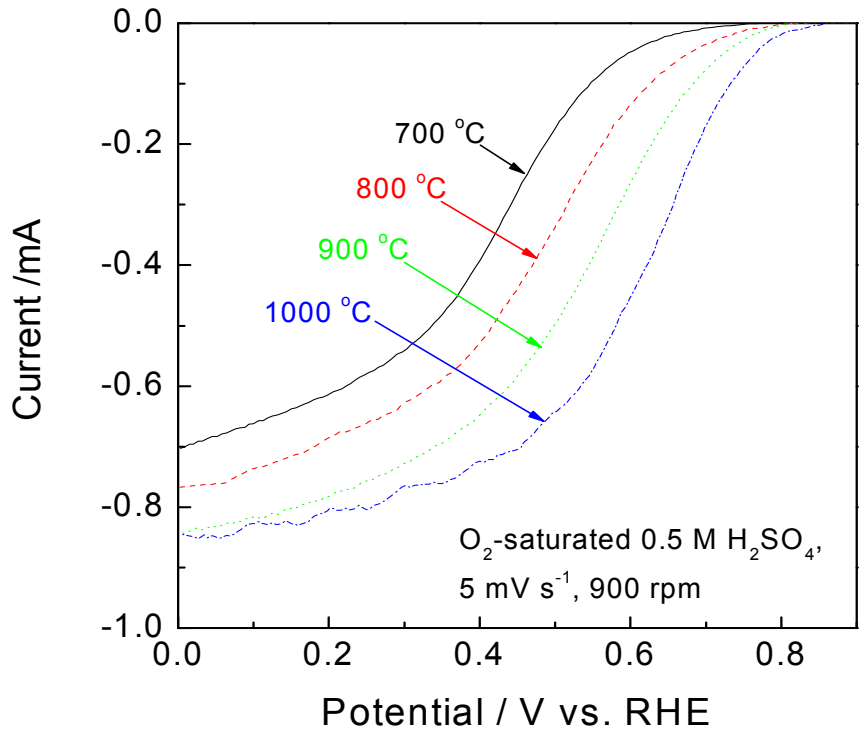


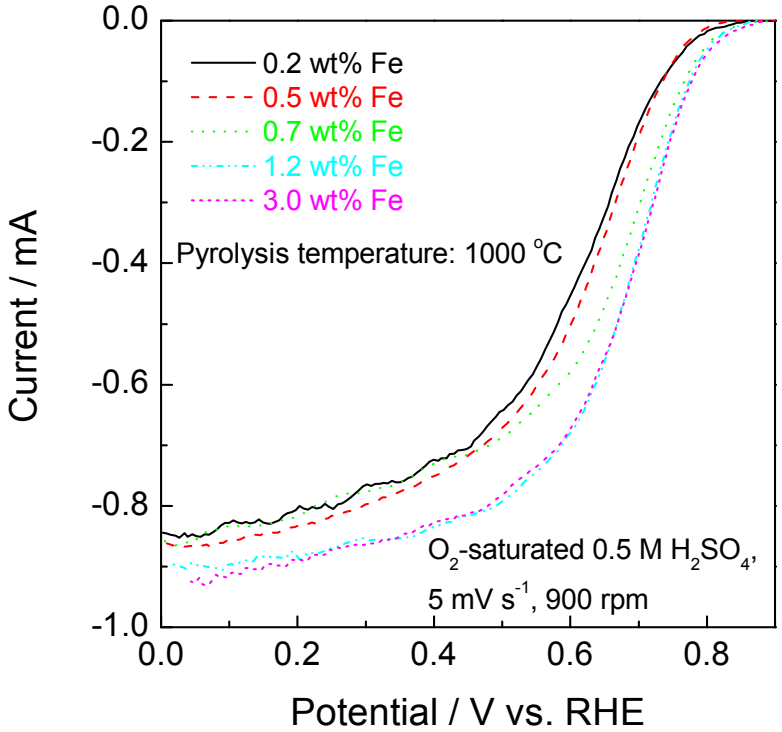


- **The adsorption-desorption isotherms indicates the nitrogen-containing mesoporous carbon (CN_x) is mesoporous with cylindrical pore channels.**
- **The onset potential for ORR is as high as 0.9 V on the mesoporous carbon-based composite catalyst ($Fe\text{-}CN_x$).**

Technical Accomplishments & Progress - Catalysts

Effect of pyrolysis temperature and iron content on activity of catalysts

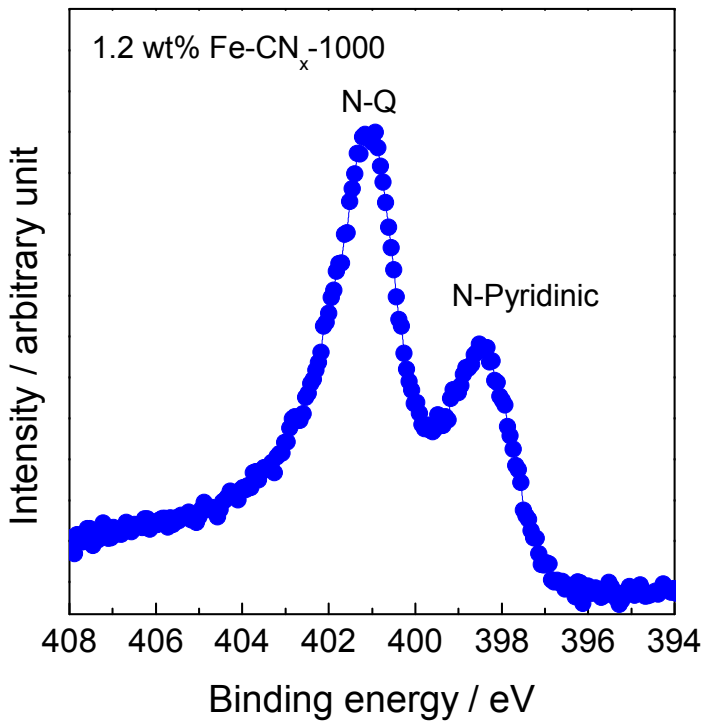


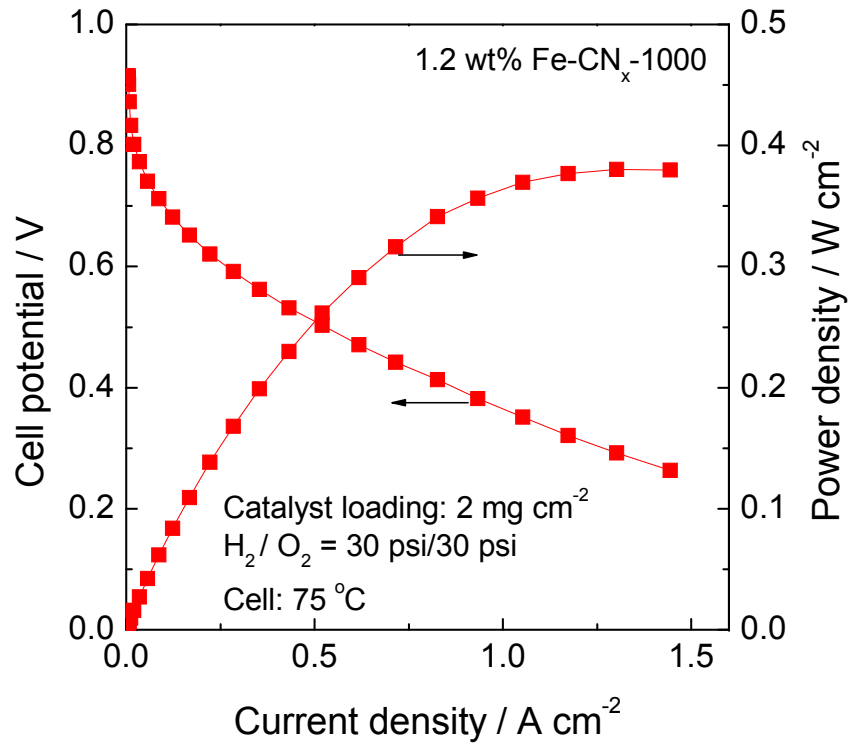


- **The activity of the catalysts significantly increases with increasing pyrolysis temperature.**
- **The activity of catalyst gradually increases with the increase of iron content up to 1.2 wt%.**

Technical Accomplishments & Progress - Catalysts

Formation of active sites and fuel cell performance





- **The high-temperature pyrolysis and iron helps incorporation of nitrogen into the graphitic structure**
- **The open circuit potential of the single cell is as high as 0.92 V. At 1.3 V, and the power density is 0.38 W cm^{-2}.**

Technical Accomplishments & Progress - Catalysts

Technical accomplishments – milestones:

- The mesoporous carbon-based composite catalyst was synthesized which is active for ORR.
  - A relationship between activity and nitrogen concentration of the composite catalysts was demonstrated.
  - The "catalyzed pyrolysis" conditions were optimized to improve the activity of the composite catalysts.

- The mesoporous carbon-based composite catalyst shows good fuel cell performance.
  - The catalytic activity is as high as 1.3 A cm^{-2} at 0.3 V for 2 mg cm^{-2} catalyst loading in the fuel cell.

Technical Accomplishments & Progress

Sub-Project 2: Hydrocarbon Fuel Powered High Power Density SOFC

Frank Chen
Department of Mechanical Engineering
University of South Carolina

Objectives / Relevance

This main **focus** of this project is to develop a high performance solid oxide fuel cell (SOFC) which can directly operate on hydrocarbon fuels and achieve high power density.

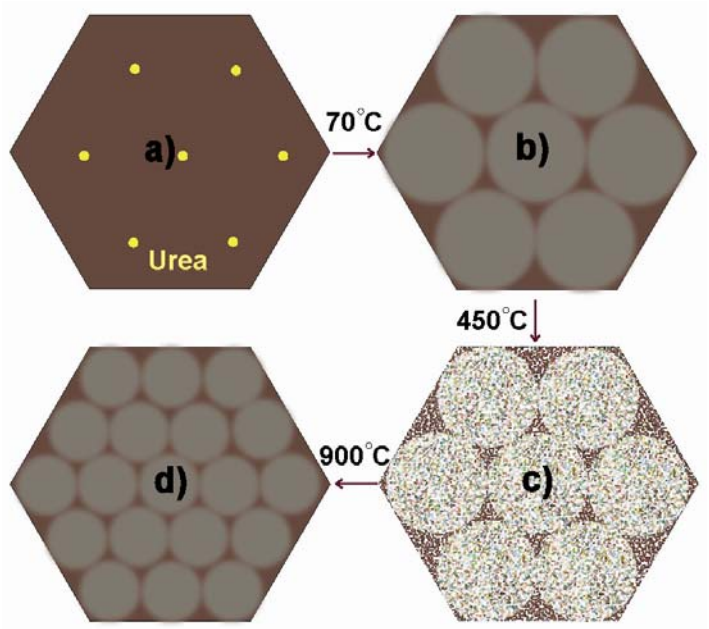
In order to meet this goal, the experiments are designed with the following **tasks**:

- Fabricate hierarchically porous electrode microstructures.
- Develop mixed ionic and electronic conducting electrode materials.
- Develop anode materials which are capable of direct utilization of hydrocarbon fuels with tolerance to carbon formation and sulfur poisoning.
- Demonstrate high power density SOFCs using hydrocarbon fuels.

Technical Accomplishments & Progress – SOFC

Approach

Hierarchically porous cathode structure to improve cathode performance by facilitating mass transport while increasing reaction sites



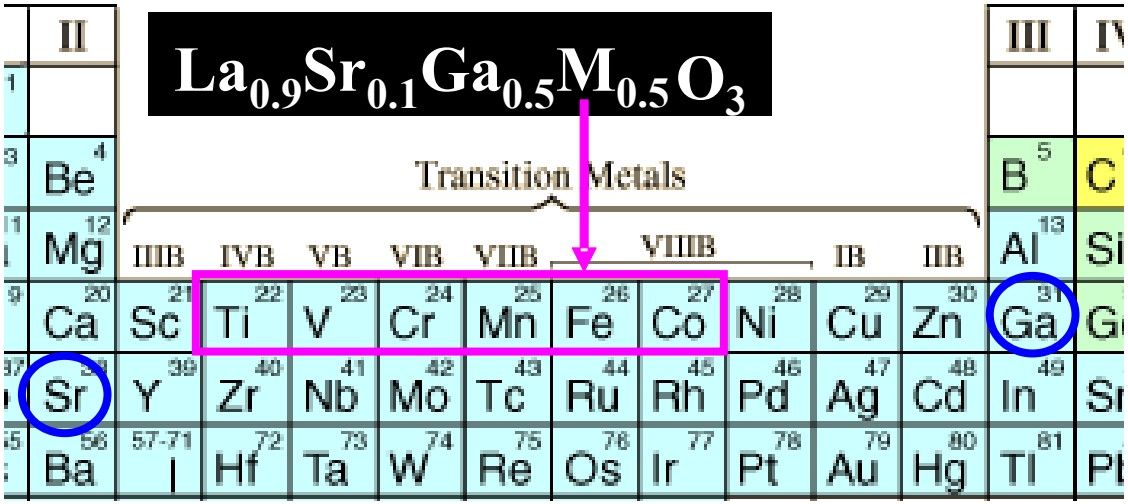
a) urea is uniformly distributed in the precursor complex;
b) urea decomposes at 70 °C and macropores are formed;
c) surfactants are burned off, leaving mesopores inside the macroporous walls;
d) upon calcination, shrinkage occurs for the macropores.

Schematic illustration of the self-rising approach to synthesize 3-dimensionally order macroporous (3DOM) cathode material.

Technical Accomplishments & Progress – SOFC

Approach

Mixed ionic and electronic conducting ceramic anode which is carbon-formation resistant and sulfur tolerant.

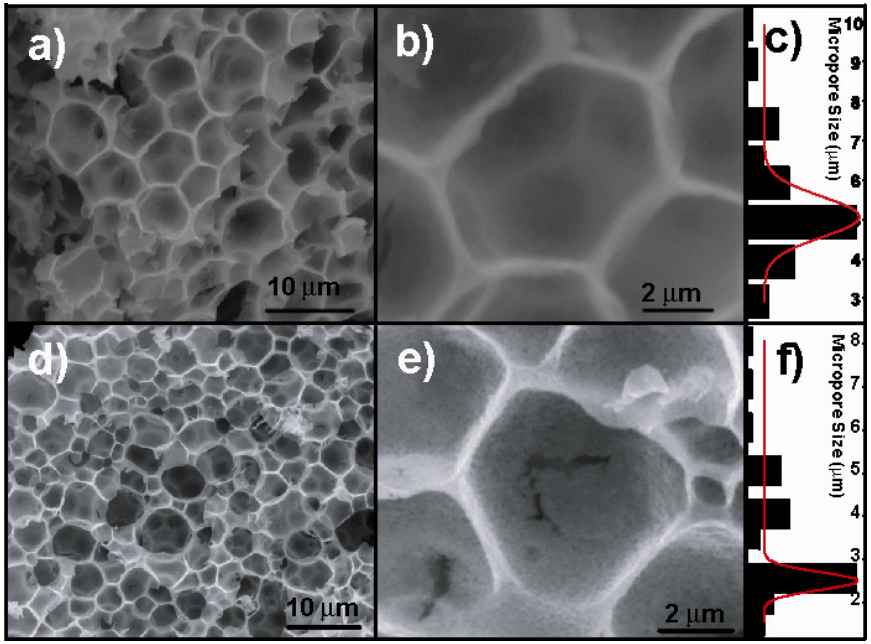


- $La_{0.9}Sr_{0.1}Ga_{0.8}Mg_{0.2}O_3$ is an excellent ionic conductor
- Introducing electronic conduction while maintaining ionic conduction

Technical Accomplishments & Progress – SOFC

Accomplishments / Milestone

Hierarchically porous $La_{0.5}Sr_{0.5}Fe_{0.5}Co_{0.5}O_3$ (LSCF) cathode synthesized from self-rising approach

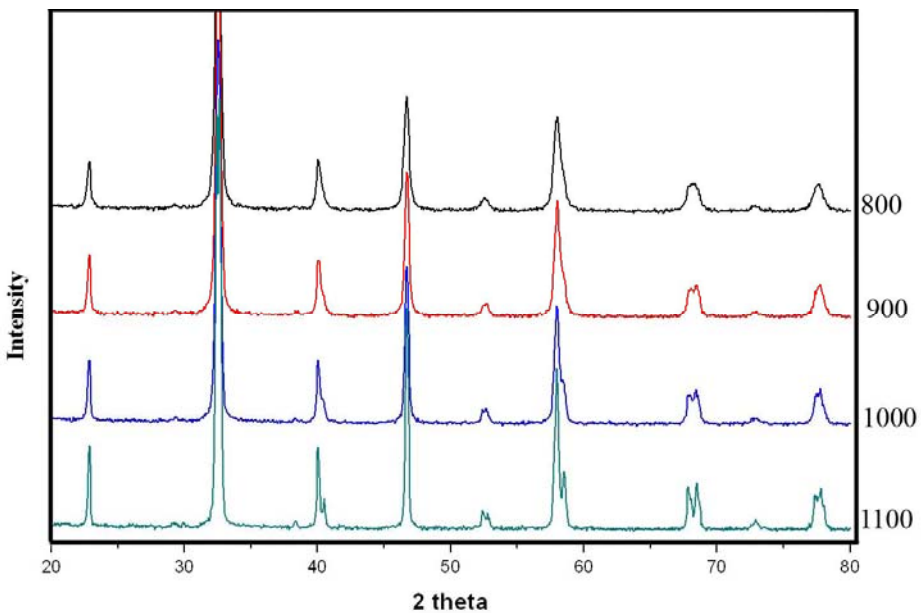


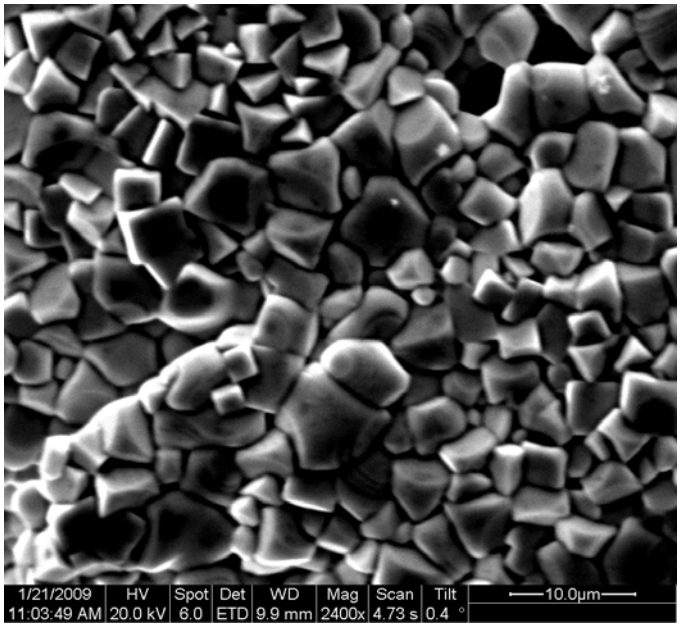
- SEM and macropore size distribution of LSCF samples. a)-c) 450°C, d)-f) 900°C.
- Q. Liu and F. Chen, "Method to Synthesize Porous Metal Oxide", US provisional patent application, USCRF#00768

Technical Accomplishments & Progress – SOFC

Accomplishments / Milestone

$La_{0.85}Sr_{0.15}Ga_{0.5}Mn_{0.5}O_3$ (LSGM) powders are prepared by glycine-nitrate combustion method.





High density LSGM samples obtained for conductivity measurement after sintering at 1500°C; Pure perovskite phase; XRD peaks split at T>900°C; understanding the cause for XRD peak splitting and effects on conductivity ongoing

Summary – Hydrocarbon Fuel SOFC

Relevance: Develop materials for a high performance solid oxide fuel cells which can directly operate on hydrocarbon fuels and achieve high power density.

Approach: Prepare hierarchically porous electrode using self-rising technique and develop mixed conducting ceramic anode based on $LaGaO_3$ system.

Technical Accomplishment and Progress: Hierarchically porous LSCF has been successfully prepared using self-rising technique; LSGM samples are prepared and shown promising conductivity in air.

Technology Transfer / Collaborations: One invention disclosure on self-rising approach has been filed. Collaborate with SRNL for nanostructured ceramic synthesis and AFRL for sulfur-tolerant ceramic anode work.

Proposed Future Research: Evaluate solid oxide fuel cell performance using hierarchically porous electrode and $LaGaO_3$-based ceramic anode.

Technical Accomplishments & Progress

Sub-Project 3: Durability of Gaskets and Seals in PEM Fuel Cells

Yuh Chao
Department of Mechanical Engineering
University of South Carolina

Objective: Develop a fundamental understanding how the degradation mechanisms of PEM gaskets affects the performance of existing and improved materials

From Company 1	From Company 2
liquid silicone elastomer (**DLS**), Fluorosilicone rubber(**DFS**), copolymeric resin(**DC**)	**EPDM**, Fluoroelastomer(**FKM**)

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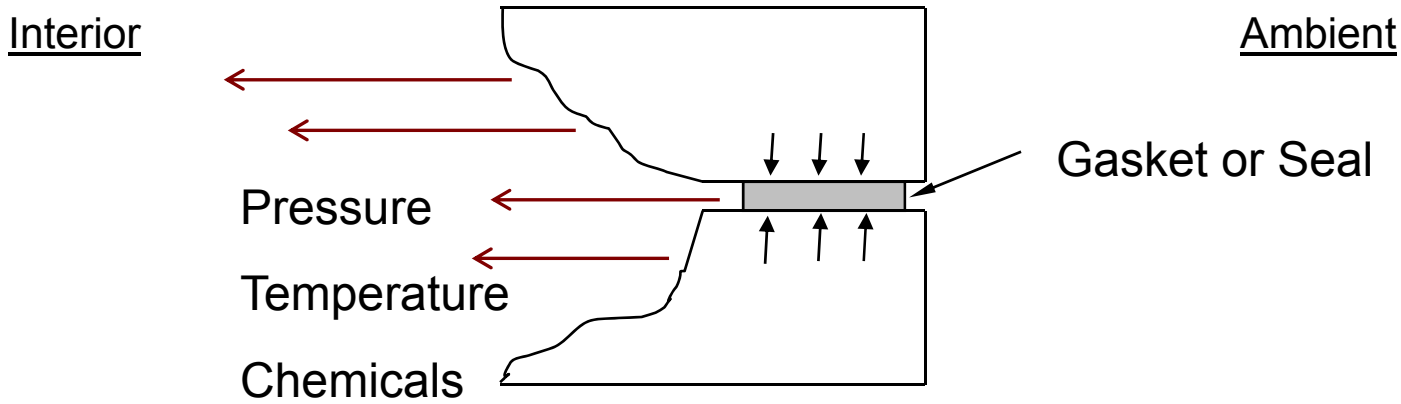
Technical Accomplishments & Progress – Seals/PEM

Relevance: Gasket/Seal as a structural member in Fuel Cells



Characteristics of gasket/seal :

Under compression, exposed to chemicals, high temperature, pressure, cyclic conditions, etc.

Loss of functionality : by cracking and /or stress relaxation

Cracking : due to corrosion under compression (Chemical stability)

Stress Relaxation : material degradation… loss its sealing ability (mechanical stability)

Leachants: detrimental sometimes (chemical stability)

Technical Accomplishments & Progress – Seals/PEM

Approach

Task 1. Selection of Commercially Available Seal Materials (95 % complete)

Task 2. Aging of Seal Materials (on-going)

In simulated regular and accelerated FC environment (ADT)
With and without stress/deformation

Task 3. Characterization of *Chemical Stability (on-going)*

FTIR, XPS, Weight loss, Atomic Absorption for leachants detection

Task 4. Characterization of *Mechanical Stability (on-going)*

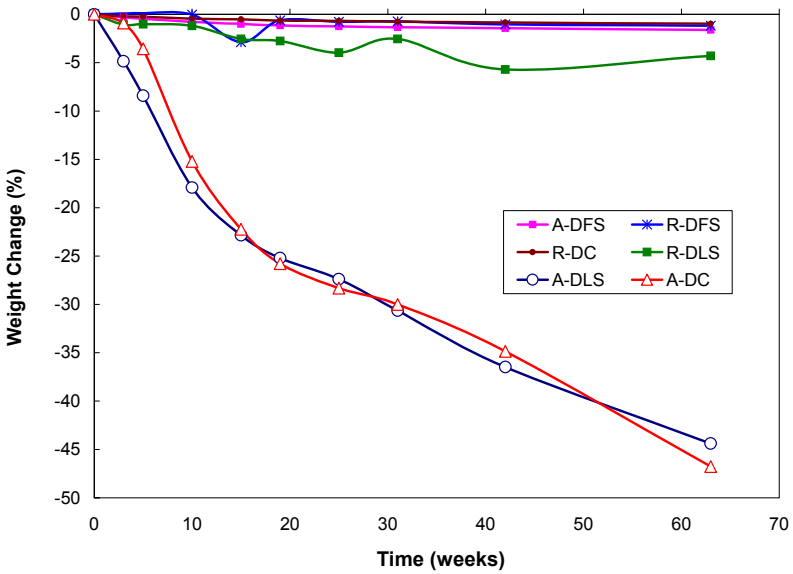
tensile strength, ductility, DMA (Dynamic Mechanical Analyzer), micro-indentation, CSR (Compression Stress Relaxation)

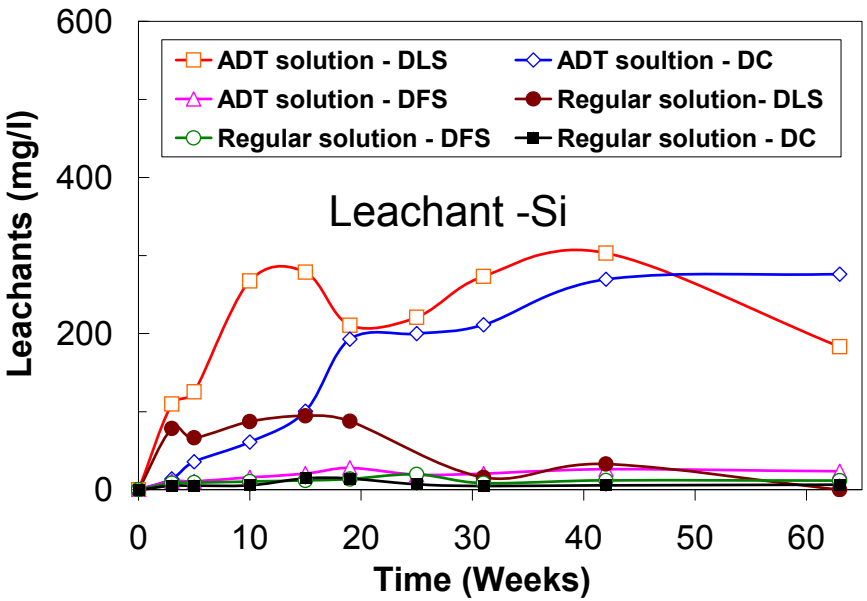
Task 5. Development of Accelerated Life Testing Procedures (on-going)

Task 6. Industrial Interaction and Presentations (on-going)

Technical Accomplishments & Progress – Seals/PEM

Weight loss and chemical leaching (63 wks study)

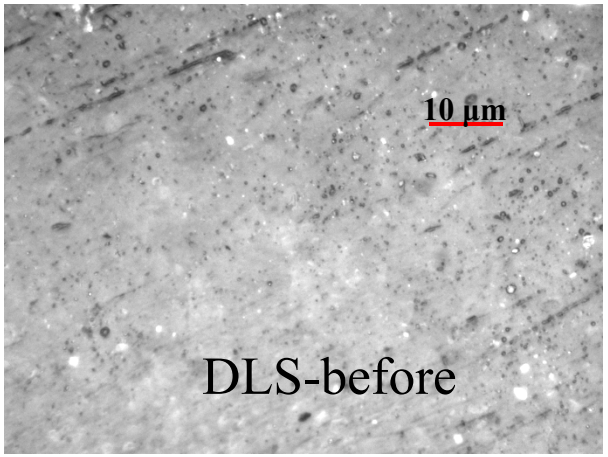


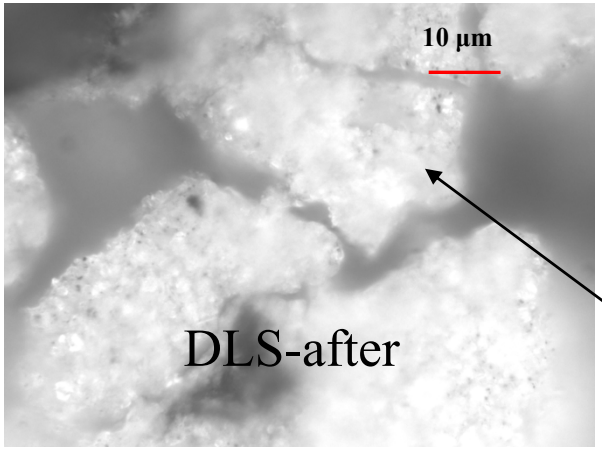


- A-DLS and A-DC → more weight loss and more Si leaching → **Lost Si is the cause of weight loss**
- Detectable Mg only in A-DLS
- The amount of Ca is negligible, except for R-DLS (0-3 mg/*l*) and A-DLS (0-12 mg/*l*)
- The amount of Si is in the range of 5-300 mg/l

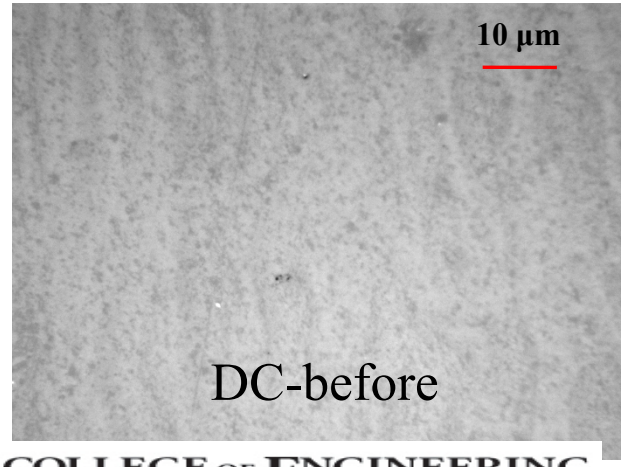
Technical Accomplishments & Progress – Seals/PEM

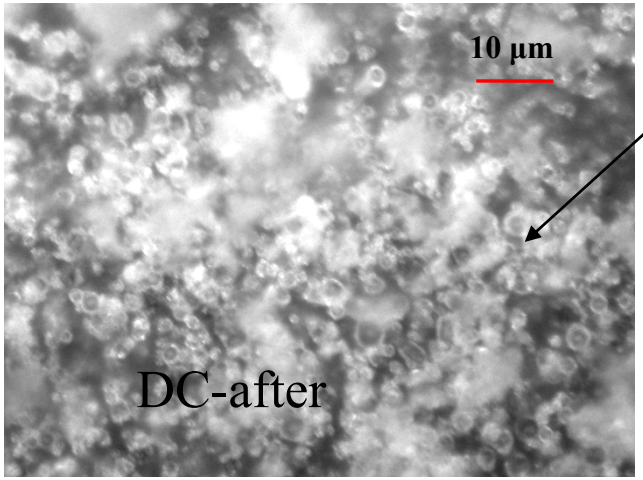
Optical image of DLS and DC before and after exposure to ADT solution for 10 weeks





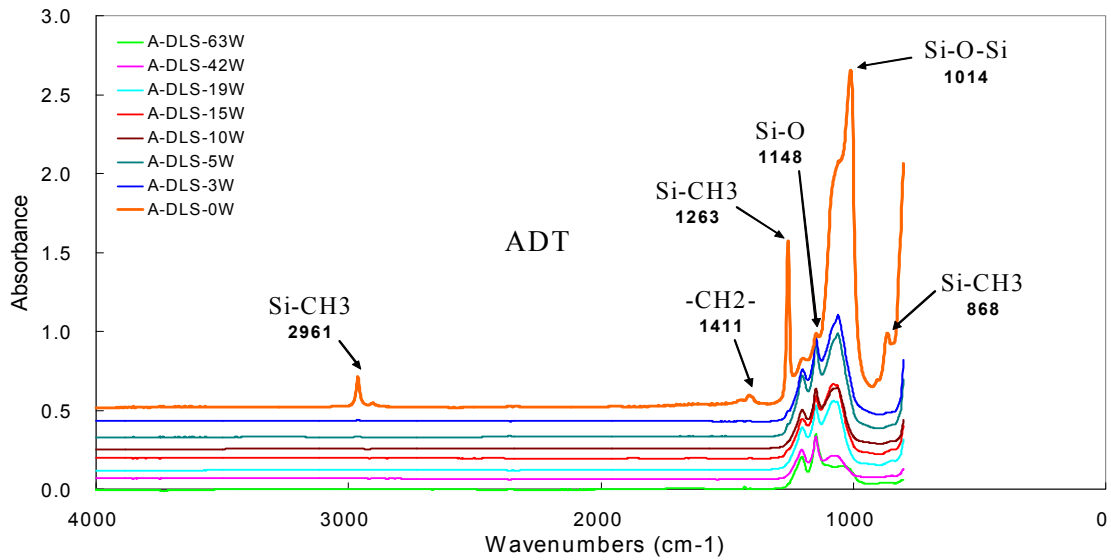
Crystal-like accumulation



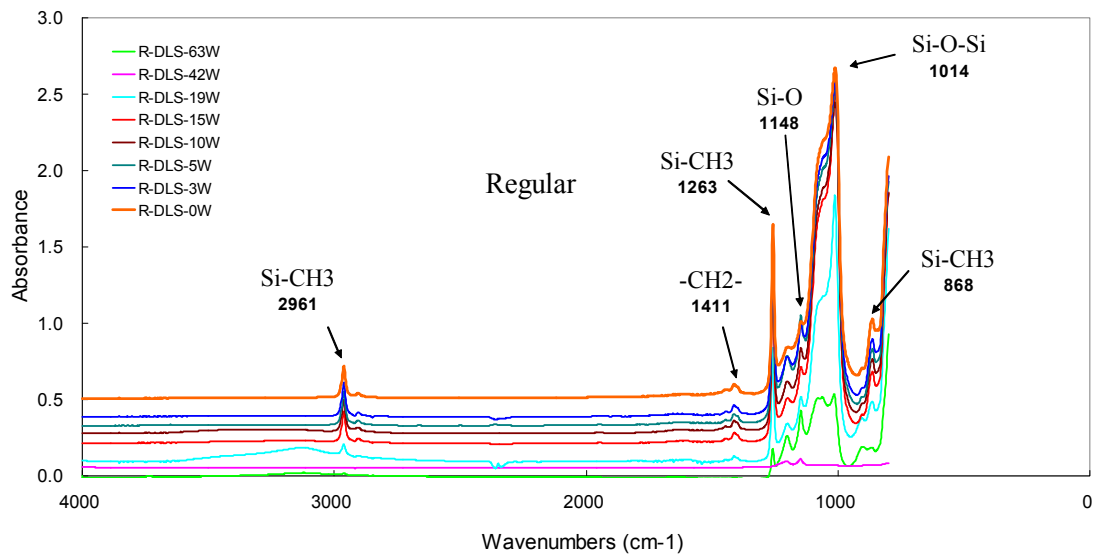


Technical Accomplishments & Progress – Seals/PEM

ATR-FTIR for DLS (ADT and Regular Solution)



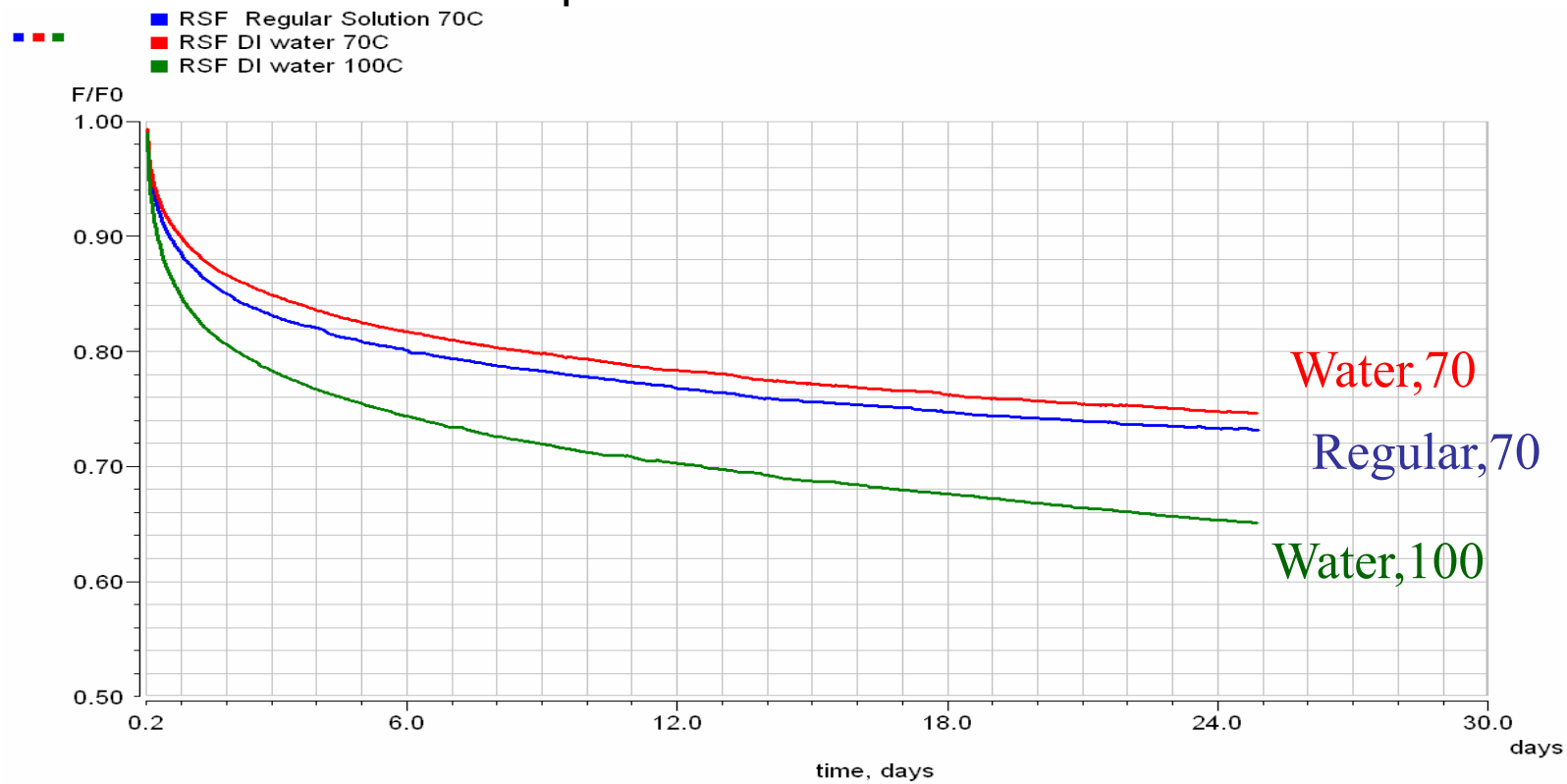
Chemical changes in backbone and crosslinked domain after 3 week exposure



No significant Chemical Changes after 42 week exposure

Technical Accomplishments & Progress – Seals/PEM

Compression Stress Relaxation curves of DLS at different temperature and different medium



- **A combination of DI water and high temperature results in dramatic reduction of the retained seal force**
- **Effect from different medium : not significant**
- **Effect from temperature : significant**

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Technical Accomplishments & Progress – Seals/PEM

Sub-Project 3: Summary- Technical Accomplishments

1. **Optical microscope** and SEM analysis to examine the degradation of surface.
2. **ATR-FTIR** test to elucidate the material surface chemical degradation.
3. **Atomic adsorption spectrometry** analysis to identify leachants from seals into the soaking solutions.
4. **Microindentation** test for assessing the mechanical properties of the gasket materials.
5. **DMA** for assessing the dynamical mechanical properties of the gasket materials.
6. **Compression Stress relaxation** test system to monitor the retained seal force under fuel cell condition
7. **New equipment purchased (2/2009):** Instron tensile testing Model 5566EH for polymeric materials
8. **Developing** life prediction methodologies.
9. **Publications** in Journal and Conferences and discussions with members in the USC NSF Center for Fuel Cells.

Technical Accomplishments & Progress

Sub-Project 4: Hydrogen Quality

John Van Zee and Jean St. Pierre,
Department of Chemical Engineering

Objective: To quantify the mechanisms of performance and durability loss resulting from contaminants in the fuel for PEMFCs by performing experiments, analyzing data, and developing models. The study will provide equilibrium and rate constants suitable for use in new and existing models, and in computer code at Argonne National Laboratory.

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Technical Accomplishments & Progress – H_2 Quality

Objectives / Relevance

- Critical constituents for H2 quality are listed in Appendix C of the 2007 Technical Plan-Fuel Cells section of the Multi-Year Research, Development and Demonstration Plan. A North American Fuel Quality Team has been organized by Dr. James Ohi (NREL) to addresses the impact of these critical constituents as they affect the barriers of Durability, Cost, and Performance that are labeled A-C on page 3.4-25 of the Technical Plan. This project supports that team by obtaining experimental data, and is part of the cross-program effort on H2 quality that addresses parts of Tasks 1-3 and 8-10 of Table 3.4.15 entitled "Technical Task Descriptions" of the 2007 Technical Plan-Fuel Cells section of the Multi-Year Research, Development and Demonstration Plan.

- Contaminants are selected and studied to be complementary to other work by national laboratories and funded universities.

Technical Accomplishments & Progress – H_2 Quality

Approach

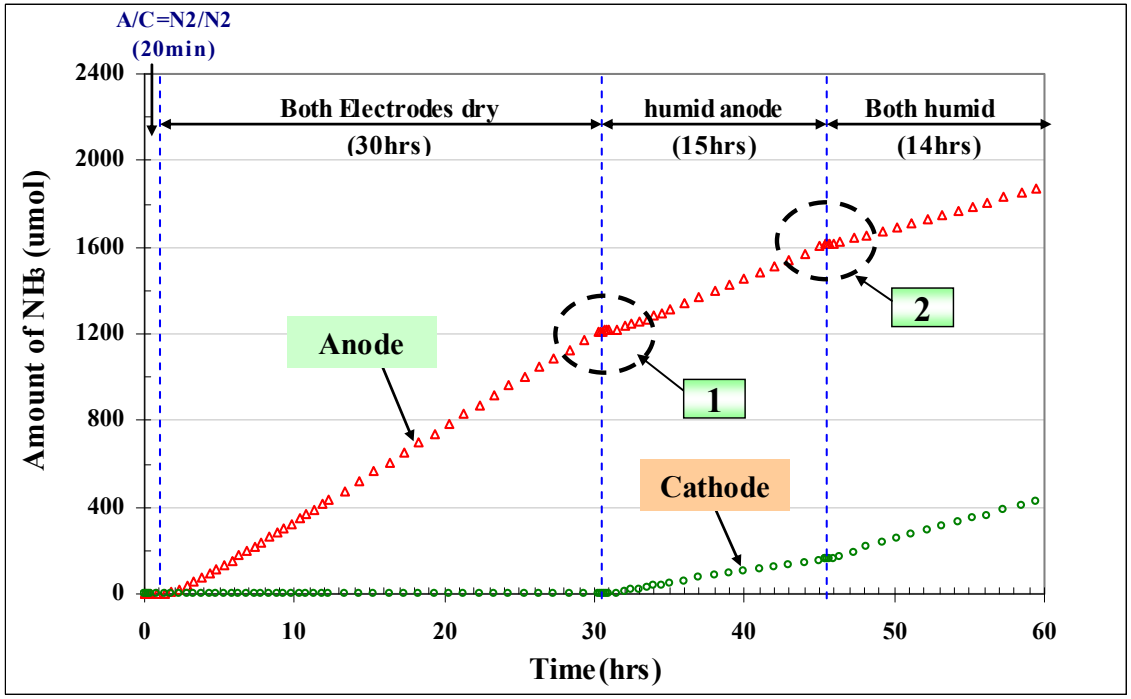
Task Number	Project Milestones	Task Completion Date				Progress Notes
		Original Planned	Revised Planned	Actual	Percent Complete	
4.1	Develop techniques to assess transport of NH_3	09/30/09			25%	On Track.
4.1	Develop techniques to assess transport of Sulfur species;	09/30/09			25%	On-Track.
4.1	Measure transport rates and assess effect on contamination	03/30/10			0%	Not started.
4.1	Develop improved activation-loss model	10/30/09			0%	Not started.
4.2	Develop techniques to measure the isotherms and rate constants of Sulfur species	06/30/10			25%	On-Track.
4.2	Develop techniques to measure ion exchange and reaction rates of NH_3	08//30/10			0%	Not started.
4.3	Publish comparison of model with performance data	06/30/10			10%	On-Track.
4.3	Disseminate the data and findings	10/31/10			12%	Ongoing.

Technical Accomplishments & Progress – H_2 Quality

Accomplishments / milestones:

We have developed techniques to quantify the transport of NH_3 from the anode to the cathode during open circuit conditions

Amount of NH_3 detected from both electrodes by the effect of anode humidity with 100 ppm NH_3/N_2 (Flow rate A/C =150 sccm, Temp.: A/C/Cell =78/73/70°C)

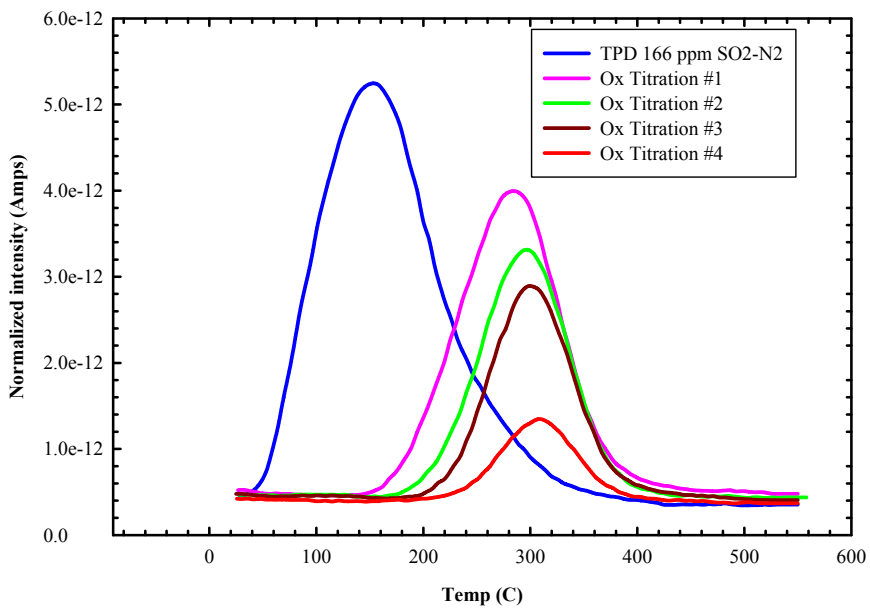


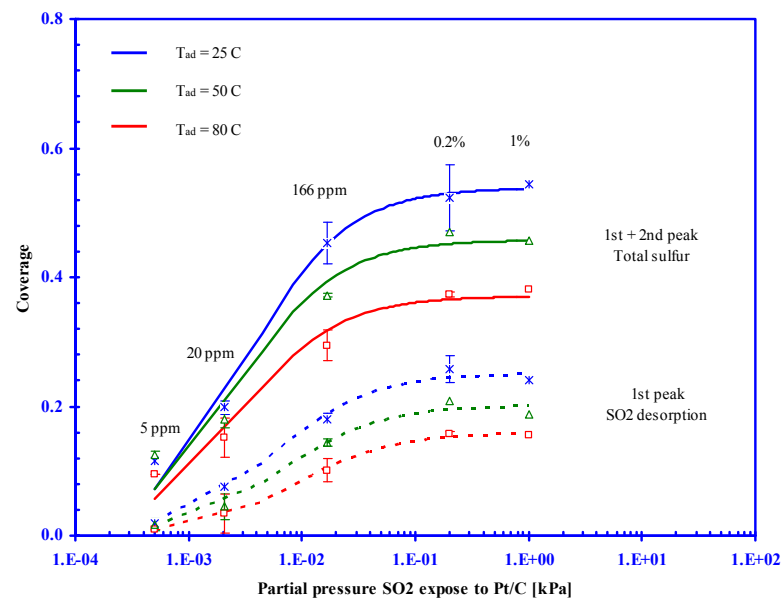
We can explain these results by considering that under humid conditions NH_3 would be dissolved in water and converted to NH_4^+ which could displace (by ion exchange?) and H+ in the ionomer of the electrode and/or the membrane

Technical Accomplishments & Progress – H_2 Quality

Accomplishments / milestones:

We have developed techniques to identify the sulfur species that adsorb on the cathode through temperature programmed desorption and reaction. We chose SO_2 as a preliminary model compound for sulfur species in the fuel that may be transported to the cathode. The strongly adsorbed species may accumulate so that dosage is a important variable.

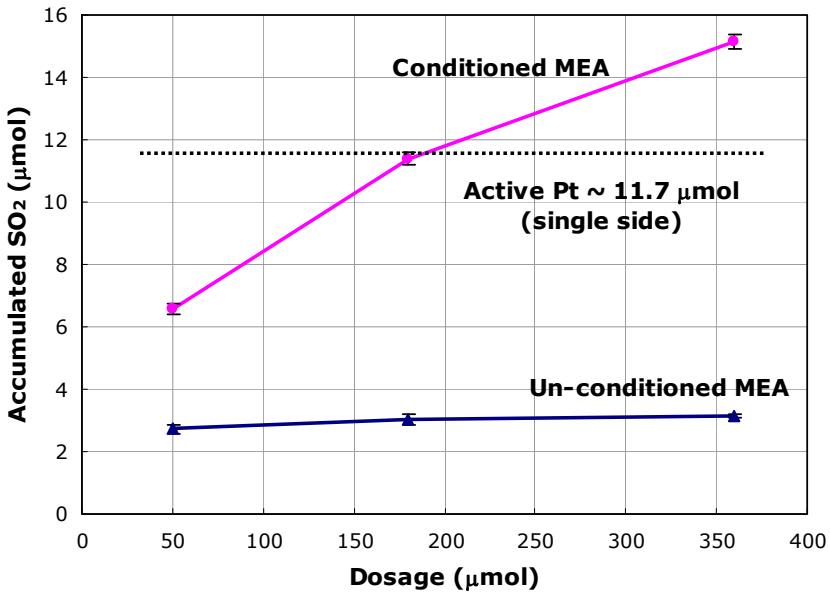


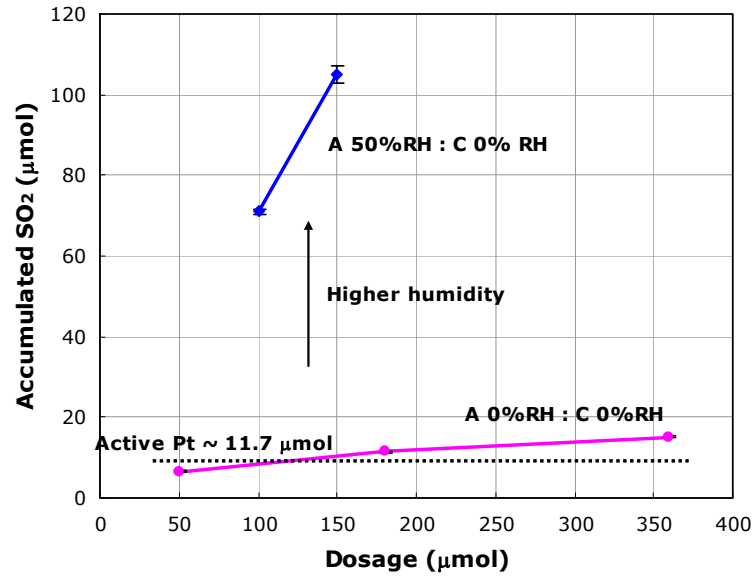


Technical Accomplishments & Progress – H_2 Quality

Accomplishments / milestones:

We have developed techniques to quantify the accumulation of SO_2 in the cell in the presence of water. We chose SO_2 as a preliminary model compound for sulfur species in the fuel that may be transported to the cathode.





We can explain these results by considering that under humid conditions SO_2 would be dissolved in water and converted to HSO_3^-. Thus it is not simply the concentration which must be understood but the dosage (total exposure of sulfur species) which must be reported to understand the performance loss.

Technical Accomplishments & Progress – H Quality

Sub-Project 4: Summary- Technical Accomplishments

- The extent of transport of NH_3 has been quantified as a function of humidity in the anode and cathode streams; a mechanism for the transport has been verified at open circuit conditions to serve as a baseline for studying transport and reaction under load.

- Ex-situ methods have been developed to identify sulfur species that remain on the catalysts and to measure isotherms for SO_2 adsorption on Pt/C catalysts using temperature programmed desorption/reaction techniques. At least two sulfur species on the surface of Pt catalysts in the presence of N_2 are indicated. Studies in the presence of O_2 and H_2O have been started. These studies have implications for sulfur species transport from fuel contaminants.

Technical Accomplishments & Progress – H_2 Quality

Sub-Project 4: Summary- Technical Accomplishments

- Work on a new model to describe partial recovery of performance indicative of sulfur and CO contamination has begun. The model accounts for the simultaneous presence of two contaminant adsorbents on the catalyst surface. During performance recovery, one of the adsorbents will desorb but the catalyst surface will still be partially covered with another adsorbate. Thus, the cell performance is only be partially recoverable.
- Work has begun on extracting rate constants from experimental data for the understanding transient performance loss and recovery when pluses of contaminants are introduced.

Technical Accomplishments & Progress

Sub-Project 5: Multi-physics Materials System Foundations for Durability Modeling in SOFC Fuel Cells and Electrolyzers

Chris Xue and Ken Reifsnider, Department of Mechanical Engineering

Objective: **To build a first principles multiphysics durability model based on interpretations of Electrochemical Impedance Spectroscopy (EIS) data that link the multiphysics processes, the microstructure, and the material states (and their changes), with cell impedance responses and global performance mechanistically.**

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Technical Accomplishments & Progress – Durability

Objectives / Relevance

- **Durability** is one of the most prominent barriers cited by DOE (Barriers A-G; Tasks 9-models, 10-long term failure mechanisms, 11-innovative fuel cell design and manufacture). **First principles models** are especially needed to establish a bridge between the science that makes fuel cells possible and the engineering that makes them work. **Manufacturing of nanostructures**, a rapidly developing discipline, also requires the guidance of science-based models**.**

Approach

- The authors are leveraging prior work on several DOD programs to create a first principles multiphysics **durability model based on** interpretations of Electrochemical Impedance Spectroscopy **(EIS) data** that link the multiphysics processes, the microstructure, and the material states, with cell impedance responses and global performance, mechanistically, as a foundation for **engineering durability during design and manufacture of fuel cells.**

Technical Accomplishments & Progress – Durability

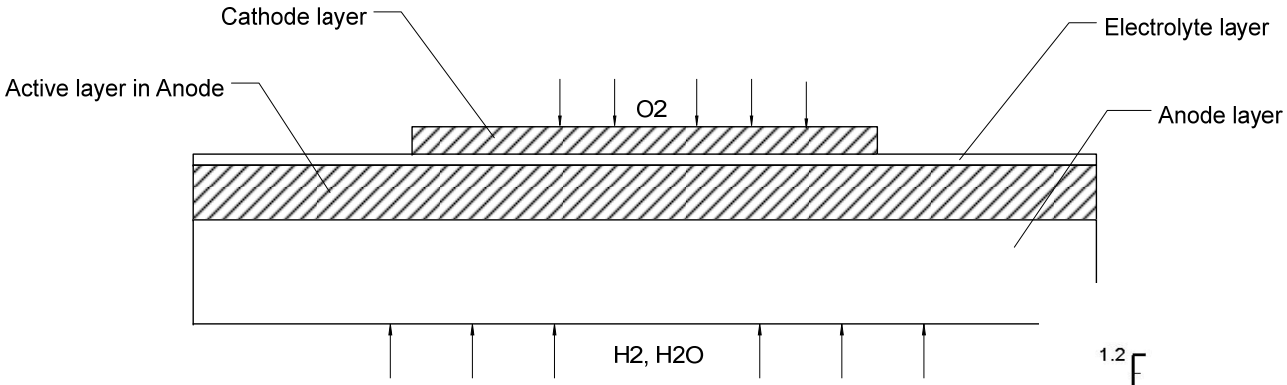
Approach

Task Schedule

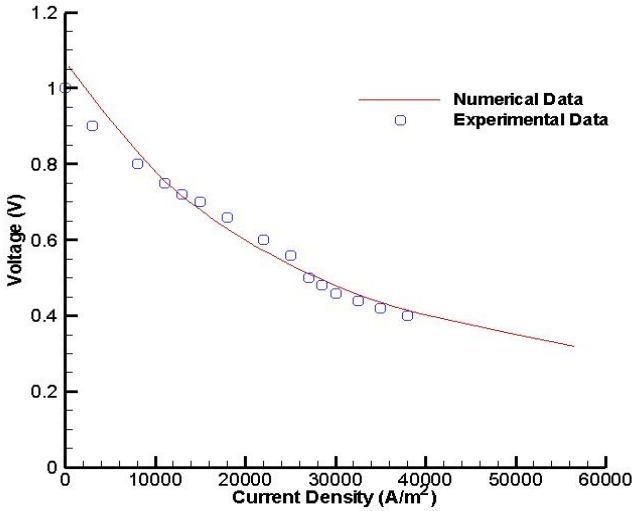
Task Number	Project Milestones	Task Completion Date				Progress Notes
		Original Planned	Revised Planned	Actual	Percent Complete	
1	Literature Review	11/15/08		11/15/08	100%	Complete.
2	EIS multi-physics modeling	09/30/10			20%	On-Track.
3	Multiphysics Interpretations of half cells	09/30/10			0%	Not started
4	Multiphysics Interpretations of button cells	09/30/10			10%	On-Track.
5	Multiphysics Interpretations of BSC stacks	09/30/10			10%	On-Track.
6	Project Management	09/30/10			5%	Ongoing.

Technical Accomplishments & Progress – Durability

Accomplishments / milestones:



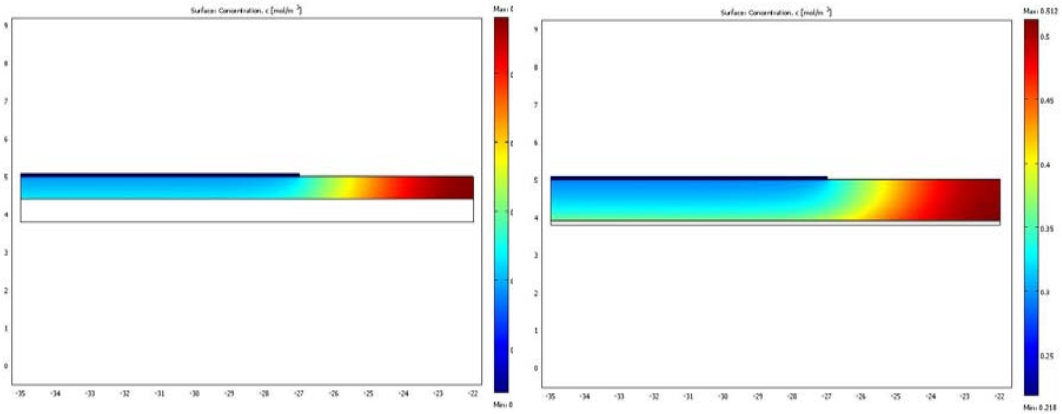
Using COMSOL, a multiphysics model of the construction and performance of button cells has been constructed and validated



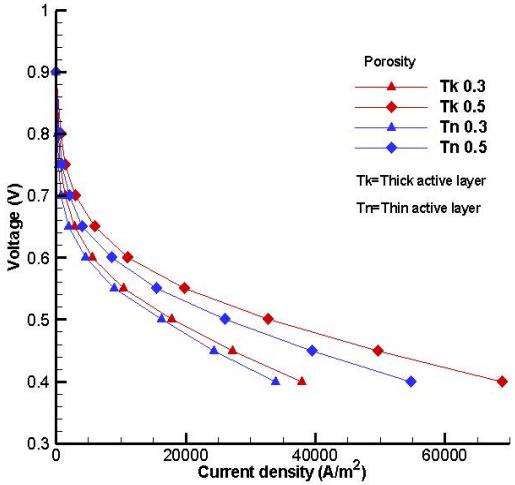
Comparison between numerical predictions and experimental data

Technical Accomplishments & Progress – Durability

Accomplishments / milestones:

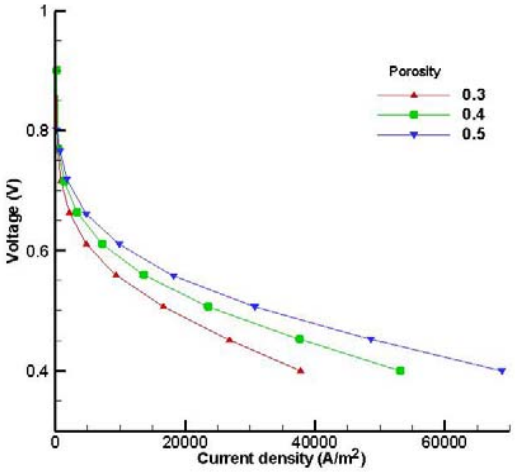


Ionic potential distribution of different thickness of active layer



Combing (active layer and porosity) effect on SOFC performance

The multiphysics model was used to study the effect of micro-geometry and porosity on performance and local species distributions



Porosity effect on SOFC performance

Technical Accomplishments & Progress – Durability

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Accomplishments / milestones:

Models of internal nano-structure will be constructed to predict EIS results, e.g.

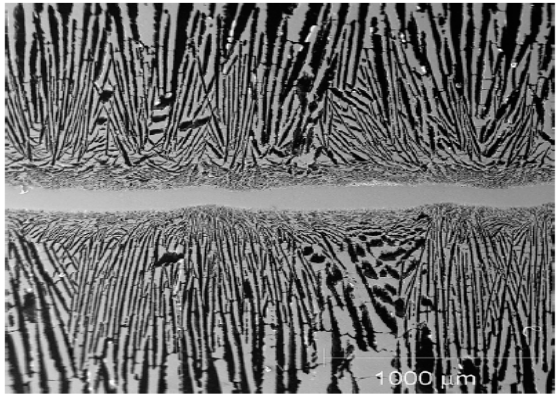


Fig. 6. Cross-section of sintered BSC prior to electrode infiltration

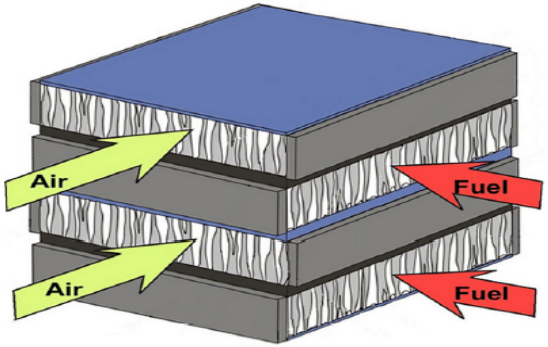
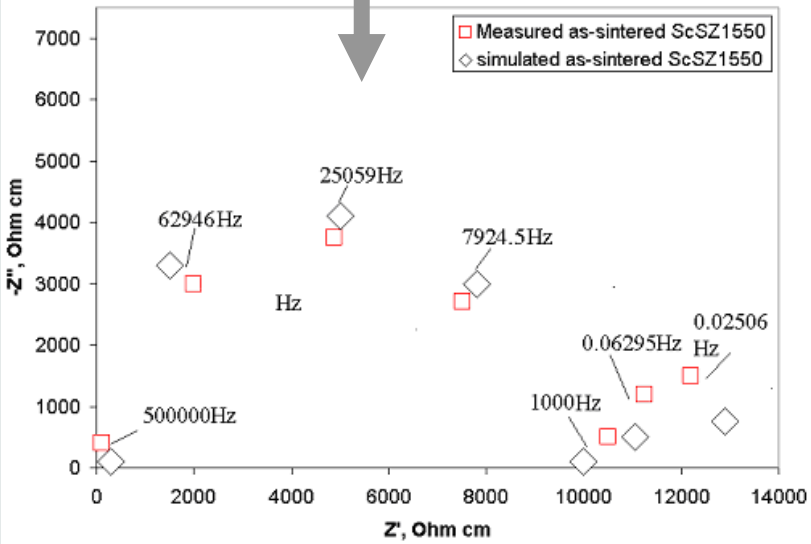


Fig. 3. BSC cross-flow stack.

T.L. Cable, S.W. Sofie / Journal of Power Sources 174 (2007) 221–227



Multiphysics models of a novel architecture are being constructed in preparation for durability modeling of next-generation SOFCs

R. Solasi, K. Reifsnider, et al., *Journal of Power Sources,* v.**167**, 2007, 366-377.

K. Reifsnider, et al., J. Fuel Cell Sci.& Tech.,2004, 35-42

Technical Accomplishments & Progress – Durability

Sub-Project 5: Summary- Technical Accomplishments

- A multiphysics model of button cells has been constructed, validated, and used to study the effect of local details related to how cells are manufactured on cell performance
- A multiphysics nano-structure model of conductivity in bi-electrode supported SOFC fuel cells (BSC) is being formulated to enable impedance spectroscopy interpretations (EIS)
- Test equipment is under construction for button cells and for BSC stacks
- A durability model that uses EIS data interpretations to follow mechanistic material state changes as a basis for durability and prognosis predictions is under construction, and on-track