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LIST OF FIGURES

LIST OF ACRONYMS

BAS	Bioanalytical Systems
CCE	Catalyst Coated Electrode
CCM	Catalyst Coated Membrane
CV	Cyclic Voltammogram
DI-water	Deionized water
DM	Direct Methanol
DMFC	Direct Methanol Fuel Cell
DOE-NE	Department of Energy, Office of Nuclear Energy
EDAX	Energy Dispersive Spectroscopy
EDL	engineering Development Laboratory
EIS	Electrochemical Impedance Spectroscopy
EW	Equivalent Weight
FTIR	Fourier Transform Infrared Spectroscopy
GES	Giner Electrochemical Systems
HyS	Hybrid Sulfur
ILS	Integrated Lab-Scale
LSV	Linear Sweep Voltammogram
MEA	Membrane Electrode Assembly
NHI	Nuclear Hydrogen Initiative
OCP	Open Circuit Potential
OPM	Oxford Performance Materials
PA	Phosphoric acid
PAFC	Phosphoric Acid Fuel Cell
PBI	Poly-Benzimidazole
PEM	Proton Exchange Membrane and Polymer Electrolyte Membrane
PEMFC	Polymer Electrolyte Membrane Fuel Cell
PFSA	Perfluorinated Sulfonic Acid
RT	Room Temperature, 25 °C
SCUREF	South Carolina Universities Research and Education Foundation
SDAPP	Sulfonated Diels-Alder Polyphenylenes
SDE	Sulfur Dioxide-depolarized Electrolyzer
SEM	Scanning Electron Microscopy
SHE	Standard Hydrogen Electrode
SNL	Sandia National Laboratory
SPEK	Sulfonated Poly-Etherketone
SPEKK	Sulfonated Poly-Etherketone-ketone
SRNL	Savannah River National Laboratory
SRS	Savannah River Site

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1.0 INTRODUCTION

The primary objective of the DOE-NE Nuclear Hydrogen Initiative (NHI) is to develop the nuclear hydrogen production technologies necessary to produce hydrogen at a cost competitive with other alternative transportation fuels. The focus of the NHI is on thermochemical cycles and high temperature electrolysis that can be powered by heat from high temperature gas reactors. The Savannah River National Laboratory (SRNL) has been tasked with the primary responsibility to perform research and development in order to characterize, evaluate and develop the Hybrid Sulfur (HyS) thermochemical process. This report documents work during the first quarter of Fiscal Year 2009, for the period between January 1, 2009 and March 31, 2009.

The HyS Process is a two-step hybrid thermochemical cycle that is part of the "Sulfur Family" of cycles. As a sulfur cycle, it uses high temperature thermal decomposition of sulfuric acid to produce oxygen and to regenerate the sulfur dioxide reactant. The second step of the process uses a sulfur dioxide depolarized electrolyzer (SDE) to split water and produce hydrogen by electrochemically reacting sulfur dioxide with H_2O. The SDE produces sulfuric acid, which is then sent to the acid decomposer to complete the cycle. The DOE NHI program is developing the acid decomposer at Sandia National Laboratory for application to both the HyS Process and the Sulfur Iodine Cycle. The SDE is being developed at SRNL.

During FY05 and FY06, SRNL designed and conducted proof-of-concept testing for a SDE using a low temperature, PEM fuel cell-type design concept. The advantages of this design concept include high electrochemical efficiency, small footprint and potential for low capital cost, characteristics that are crucial for successful implementation on a commercial scale. During FY07, SRNL extended the range of testing of the SDE to higher temperature and pressure, conducted a 100-hour longevity test with a 60-cm^2 single cell electrolyzer, and designed and built a larger, multi-cell stack electrolyzer. During FY08, SRNL continued SDE development, including development and successful testing of a three-cell electrolyzer stack with a rated capacity of 100 liters per hour.

The HyS program for FY09 program will address improving SDE performance by focusing on preventing or minimizing sulfur deposition inside the cell caused by SO_2 crossover, reduction of cell voltage for improved efficiency, an extension of cell operating lifetime. During FY09 a baseline technology development program is being conducted to address each of these issues. Button-cell (2-cm^2) and single cell (60-cm^2) SDEs will be fabricated and tested. A pressurized button-cell test facility will be designed and constructed to facilitate addition testing. The single cell test facility will be upgraded for unattended operation, and later for operation at higher temperature and pressure.

Work will continue on development of the Gas Diffusion Electrode (GDE), or Gap Cell, as an alternative electrolyzer design approach that is being developed under subcontract with industry partner Giner Electrochemical Systems. If successful, it could provide an alternative means of preventing sulfur crossover through the proton exchange membrane, as well as the possibility for higher current density operation based on more rapid mass transfer in a gas-phase anode.

Promising cell components will be assembled into membrane electrode assemblies (MEAs) and tested in the single cell test facility. Upon modification for unattended operation, test will be conducted for 200 hours or more. Both the button-cell and modified single cell facility will be utilized to demonstrate electrolyzer operation without sulfur build-up limitations, which is a Level 1 Milestone.

Work performed on NHI Work Package N-SR07TC0301 during the second quarter of FY09 is presented in the following sections of this report.

2.0 TECHNICAL PROGRESS

The major SRNL tasks in this Work Package for FY09 performed during the second quarter and reported herein include:

- Electrolyzer Component Development
- Development of Gas Diffusion Electrode
- Single Cell Performance Testing
- Project Management

2.1 ELECTROLYZER COMPONENT DEVELOPMENT

2.1.1 Membrane Testing\

Experimental

Membrane Selection

During the second quarter, the list of the tested membranes is shown in Table 1. Although most of these membranes where tested for SO_2 transport, the new data includes electrolyzer performance. During the selection process of commercially available and experimental membranes, an array of thicknesses, equivalent weights (EWs), chemistry, and reinforcements were considered. Before testing, membranes were hydrated by immersing in deionized water for several minutes. Commercial membranes include perfluorinated sulfonic acid (PFSA) membranes. Experimental membranes prepared exclusively to reduce the transport of inert species such as dissolved SO_2. The membranes include Sandia National Laboratory (SNL) hydrated sulfonated Diels-Alder polyphenylenes (SDAPP), Case Western Reserve University stretched recast Nafion® and Nafion®/fluorinated ethylene propylene (FEP) blends, Giner Electrochemical Systems (GES) hydrated treated Nafion® 115, Clemson University perfluorocyclobutane-biphenyl vinyl ether (BPVE) and perfluorocyclobutane-biphenyl vinyl ether hexafluoroisopropylidene (BPVE-6F) polymer blends, and special blends from Dupont.

Table 1. Evaluated Commercial and Experimental Membranes

ID	Manufacturer	Classification	Thickness (μm)
Nafion® 115	DuPont	PFSA	127
Nafion® 211	DuPont	PFSA	25
SDAPP5192C	SNL	Sulfonic Diels-Alder Polyphenilyne	50-85
BPVE2	Clemson	BPVE	18
B1F11	Clemson	BPVE-6F	16
B2F13	Clemson	BPVE-6F	19
Giner 1	GES	Treated PFSA	127
Giner 2	GES	Treated PFSA	127
Case 1	Case	Stretched PFSA	55
Case 4	Case	Stretched PFSA	63
Case 60402	Case	PFSA-FEP blends	62
Case 50502	Case	PFSA-FEP blends	55
Case 45552	Case	PFSA-FEP blends	53
Dupont Sample1	DuPont	Bi-layer PFSA	140
Dupont Sample 2	DuPont	PFSA	100
Dupont Sample 3	DuPont	Treated PFSA	50
Dupont Sample 4	DuPont	Treated PFSA	90

SO_2 Transport

The membrane SO_2 transport was evaluated using a custom made permeation cell; a schematic of the cell is shown in Figure 1. The cell consists of two glass chambers joined by a Teflon™ bridge where the membrane is secured. The bridge consists of a diffusion layer in the left chamber where acid saturated with SO_2 is forced by pump A in to the membrane acid interface. Additionally, the diffusion layer presses the membrane to the working electrode which is supported by a perforated tantalum support that provides electrical connection to the working electrode. Finally, a non-conductive diffusion media separates the Ta support from the counter electrode in order to allow the flow of fresh acid pumped by pump B to the counter electrode.

During measurements both chambers were filled with the concentrated acid of interest and purged of oxygen by flowing nitrogen. A two electrode system, which included a platinum mesh working electrode and a porous carbon counter electrode was used during measurements. A potential of 1.1 V was applied across the cell.

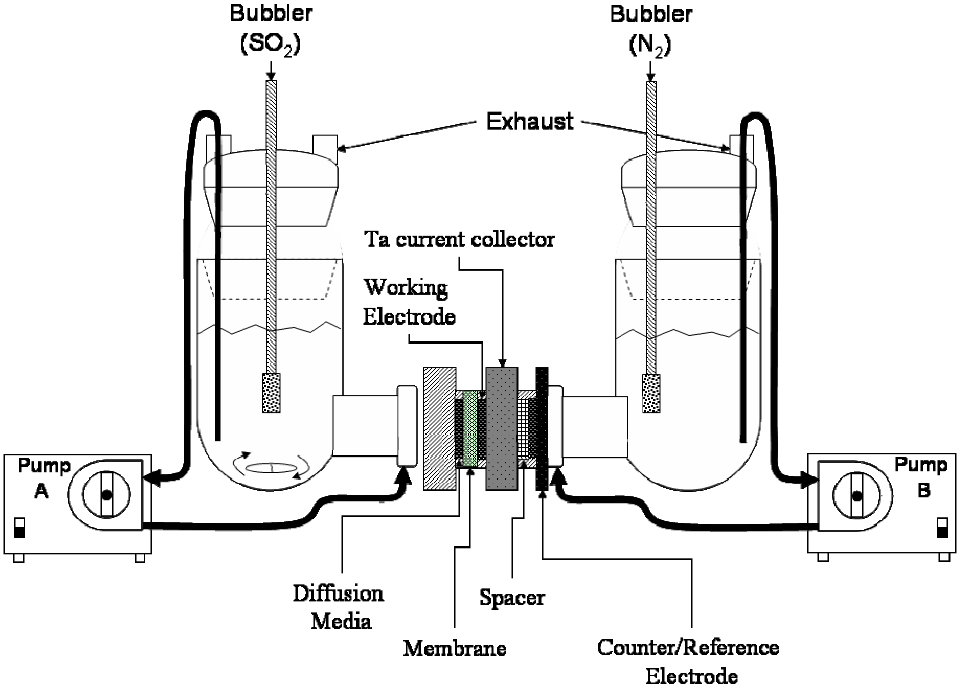

Figure 1. Simplified Schematic of the SO_2 Transport Characterization Cell

The SO_2 transport was monitored by measuring the current as a function of time while a constant potential is applied. The cell was filled with 30 wt% H_2SO_4 and purged with N_2. A constant potential of 1.1 V was applied on the working electrode while the current was measured as a function of time. Once the background current became close to zero, the flowing of SO_2 was started on the left chamber. The SO_2 permeating through the membrane was oxidized to sulfuric acid by the working electrode. A PARSTAT 2273 electrochemical analyzer was used to measure the current responses as a function of time. Experiment was run at 25 °C and atmospheric pressures.

Electrolyzer testing

Membrane performance and ionic conductivity of each membrane was measured using the HyS electrolyzer cell. Membrane Electrode Assemblies (MEAs) were prepared in order to measure these properties. An airbrush was used to spray the catalyst ink onto the surface of the membrane. Typical catalyst layers consist of 25 wt% Nafion® ionomer as a binder, and 75 wt% platinized carbon (TKK – 45.9 wt% Pt). Anode and cathode catalyst layers were targeted at 1.8 mg Pt cm^{-2} and 0.9 mg Pt cm^{-2} respectively. The prepared MEAs were assembled into the electrolyzer cell, which consisted of a simplified version of the sulfur dioxide transport cell described on Figure 1. Electrolyzer runs were performed at 67 ºC.

Results

Normally samples that show good barrier properties for sulfur dioxide, also shows a decrease in proton transport. In order to have a better comparison between the different samples and better aid in the selection of a membrane, a selectivity parameter has been selected for this comparison. This plot is of great importance since normally membranes that show good barrier properties towards SO_2 also shows increased resistance for proton transport. Figure 2 shows a summary bar plot of the selectivity of the membrane samples. The selectivity was calculated by dividing the moles of hydrogen produced under electrolyzer mode by the moles of sulfur dioxide crossing the membrane in the sulfur dioxide experiment setup. In other words, the selectivity shows the moles of hydrogen crossing the membrane per moles of sulfur dioxide crossing under rest conditions. The moles of hydrogen were obtained by applying a potential of 1 V and measuring the stable current. Using Faraday's constant the current was changed to molar hydrogen flow. The flow of sulfur dioxide used was reported in the previous report.

As can be seen in the figure, Nafion® 211 shows a selectivity of approximately 93. Only three samples tested show lower selectivity, these are the Dupont Sample 1, Dupont Sample 2 and Clemson Sample B(2). While the Dupont samples 1 and 2 show the lowest SO_2 transport, however it also affects tremendously the proton transport in the membrane. The end result is an extremely low selectivity. The Clemson Sample B(2) shows only a slight lower selectivity. All other samples show slightly higher selectivites, except for Case 45-55-2 and BASF Celtec-V. Case sample 45-55-2 shows that the blending of FEP with Nafion® is an effective process to increase the selectivity of the membrane. BASF sample Celtec-V shows the best selectivity, being around 9 times more selective than the Nafion® baseline.

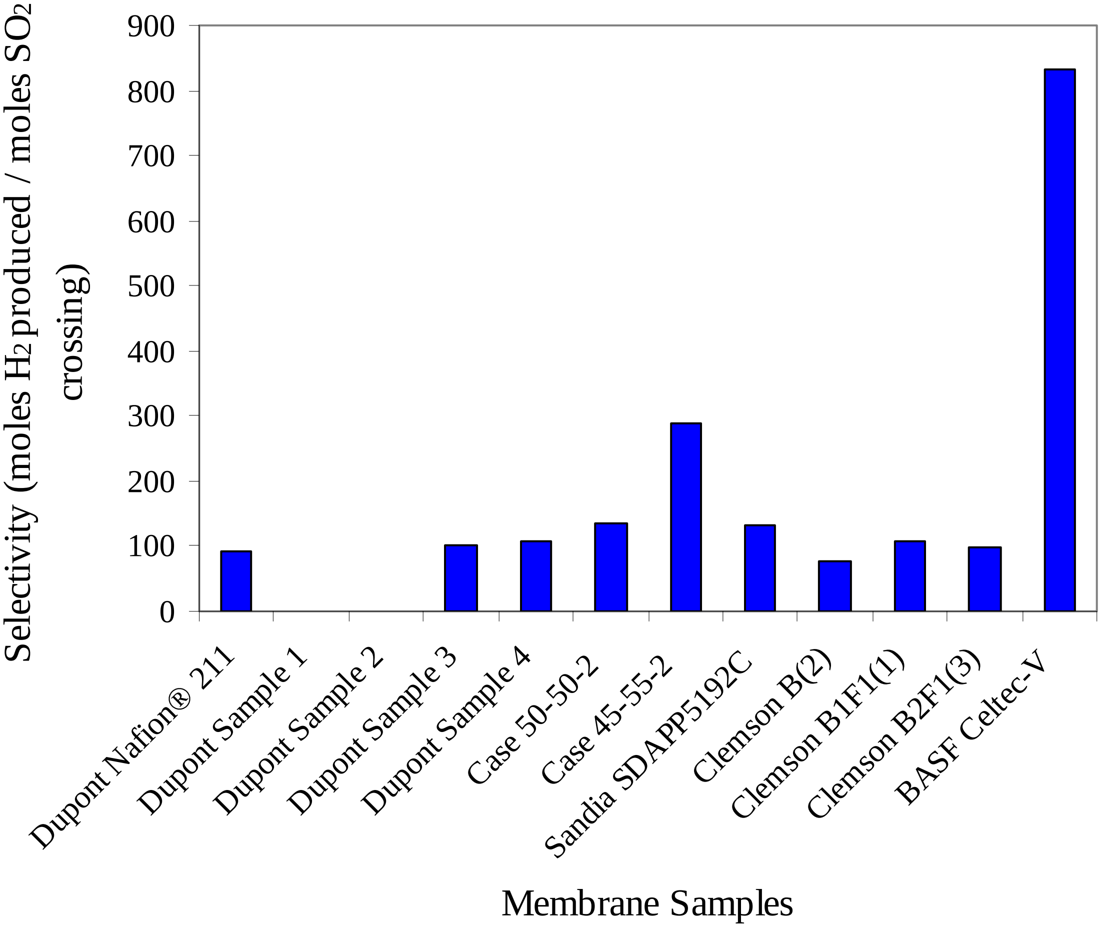

Figure 2. Selectivity of the Membrane Samples Tested. Moles of SO_2 correspond to the amount of SO_2 crossing the membrane under rest conditions at atmospheric and 25 ºC conditions. Moles of H_2 produced corresponds to the amount of H_2 produced under electrolyzer mode at 67 ºC and an applied voltage of 1.0 V

CONCLUSIONS AND RECOMMENDATIONS

The SO_2 transport at 30 wt% acid concentrations was studied on commercially and experimental membranes. It is desired for the SDE to have a membrane with a low diffusion coefficient, but high proton conductivity. The experimental membrane from Case Western (45-55-2) Reserve University (FEP blends) and BASF- Celtec-V showed the highest selectivity, followed by Clemson University (BPVE-6F) blends and Dupont samples 3 and 4. Future work will measure the catalyst activity for the cathode and anode.

2.1.2 Small Scale Electrolyzer Unattended Operation

Modifications were also completed for operating the component development test facility unattended. This included the preparation of a relay box that will close the flow of SO_2 in the event of lost of hood air flow. Unattended operation will greatly extend the test time for component development tests. Figure 3 shows the installed solenoid valve on the SO_2 cylinder and the relay box that controls the valve.

Figure 3. Relay Box and Solenoid Valve Installed for Unattended Operations

2.1.3 Small Scale Electrolyzer Pressurized Test Facility

SRNL is contracting with Giner Electrochemical to provide assistance in the fabrication of a small-scale pressurized electrolyzer system. This contract will allow SRNL to leverage Giner’s expertise in electrolyzer systems as well as their familiarity with the Hybrid Sulfur process to accelerate the fabrication of the small-scale test system. This system will allow SRNL to characterize high temperature membrane candidates for the hybrid sulfur process.

All of the major hardware has been ordered. Most of the components have been received with the exception of some of the long lead items including 2 of 4 pumps, the backpressure regulators and several smaller electronic components. A photo of the hardware assembled to date is shown as Figure 4.

Figure 4. Hybrid Sulfur Button Cell Control System under Construction

Programming the control system has also started. Giner continues to interact with SRNL to assure proper integration with the SRNL components.

2.2 DEVELOPMENT OF THE GAS DIFFUSION ELECTRODE

SRNL continues to contract with Giner Electrochemical Systems LLC to research the gas diffusion electrode (GDE). Giner has nearly completed their research on the gap-cell, GDE concept for SO_2-depolarized electrolysis and the results reported were in first quarter of FY 09. Some additional testing and preparation of the final report have been put on hold to allow Giner to maximize their efforts on the fabrication of the small-scale pressurized electrolyzer system.

2.3 SINGLE CELL ELECTROLYZER TESTING

2.3.1 Evaluation of MEAs 30 and 31 for Sulfur Formation

Testing from the 1st Quarter of FY ’09 suggested that restricting the concentration of sulfur dioxide in the anolyte had potential for reducing the formation of sulfur. Therefore, MEA 30 was run with restricted sulfur dioxide for fifty hours. This testing was on day shift only so every day of testing involved three hours of startup and 1.5 hours of shutdown. The results showed that the increase of voltage with time was greatly decreased versus previous MEA operations. Also, there was no colloidal sulfur in the Sulfur Collector and much less hydrogen sulfide odor. Although MEA 30 showed significant progress, it was surmised that startups and shutdowns affected voltage performance due to the transients involved.

An identical cell, MEA 31, was constructed and tested with nominally the same conditions, but it was operated continuously for a period of approximately fifty hours. As was reported last quarter, there did not appear to be any noticeable voltage increase or cell performance degradation over the 50-hour test period. This was a positive indication that the cell was not experiencing a sulfur layer build-up.

Post-test cell examinations were performed in the 2nd quarter of FY ‘09. Scanning electron microscope images of MEAs were conducted at Clemson University. Analysis showed reduced formation of the sulfur-rich layer in the case of MEA 30, and no visible presence of a sulfur-rich layer in the case of MEA 31. These MEAs where both tested under SO_2 limited operating conditions, which is predicted to reduce SO_2 crossover. MEA 30 ran with daily startups and shutdowns, creating situations outside the SO_2 limited region. MEA 31 ran continuously, with a single startup and shutdown.

An SEM of MEA 30 is shown as Figure 5. Several spots are identified on the photo that were evaluated.

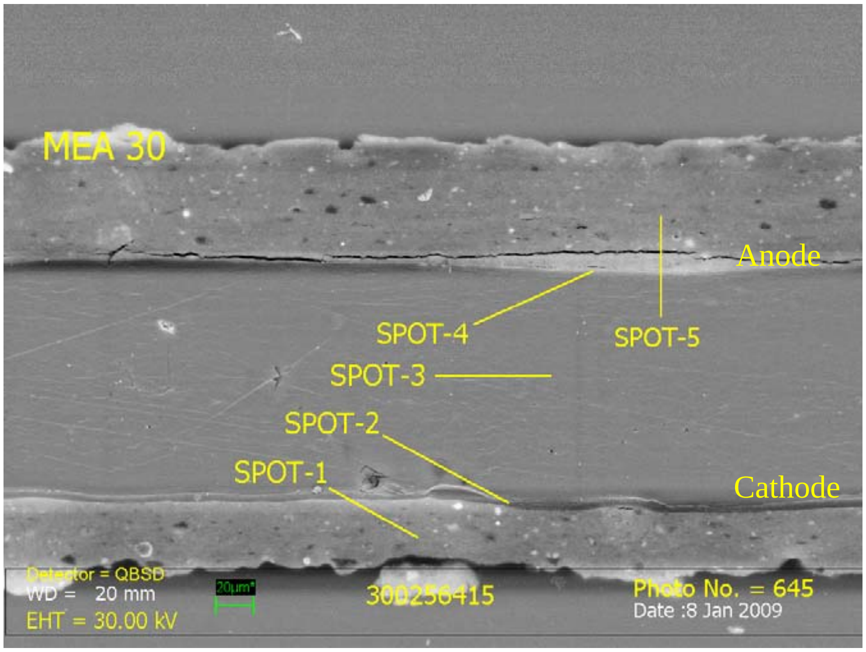

Figure 5. SEM of MEA 30 with Reduced Sulfur Buildup after 50 Hours of Operation

A region that had elevated sulfur concentrations was found between the membrane and the cathode. This area is shown as SPOT-2 in Figure 5. However, this can be contrasted with the sulfur layer formation in an example of a previously run MEA. MEA 20 is shown in Figure 6.

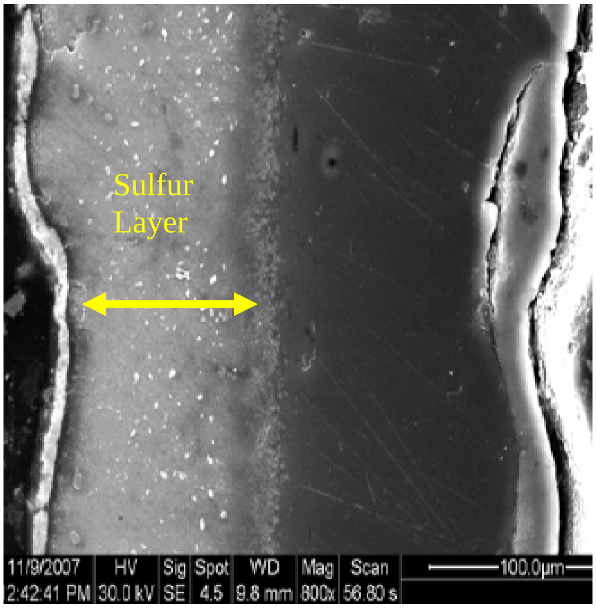

Figure 6. MEA 20 Showing Sulfur Layer after Approximately 20 Hours of Operation

As can be seen in Figure 6, the sulfur layer in the left side of the photo is almost as thick as the original membrane itself. This layer was formed in approximately 20 hours of operation for MEA 20 while MEA 30 was run for over 50 hours.

MEA 30 was tested over seven days of discontinuous operation. It was believed that the multiple starts and stops during testing while evaluating the operational protocol for SO_2 limited operations may have resulted in the formation of some sulfur. Therefore, an identical MEA, MEA 31, was prepared and tested for over 50 hours of continuous operation and a single stop and start.

After completion of testing, samples of MEA 31 were taken from 3 different locations; close to the inlet, close to the outlet and at the center. SEM analysis was performed at Clemson University. No image shows the presence of a sulfur rich layer. An extensive interrogation at the cathode membrane interface for existence of a sulfur layer using Energy Dispersive Spectroscopy (EDAX) on the location nearest to the anolyte inlet revealed the sulfur content was very small in all locations. These results suggest that sulfur is not accumulating in this MEA near the cathode-membrane interface and no evidence of a precursor stage to a sulfur-rich layer. An SEM photo of MEA 31 is shown as Figure 7.

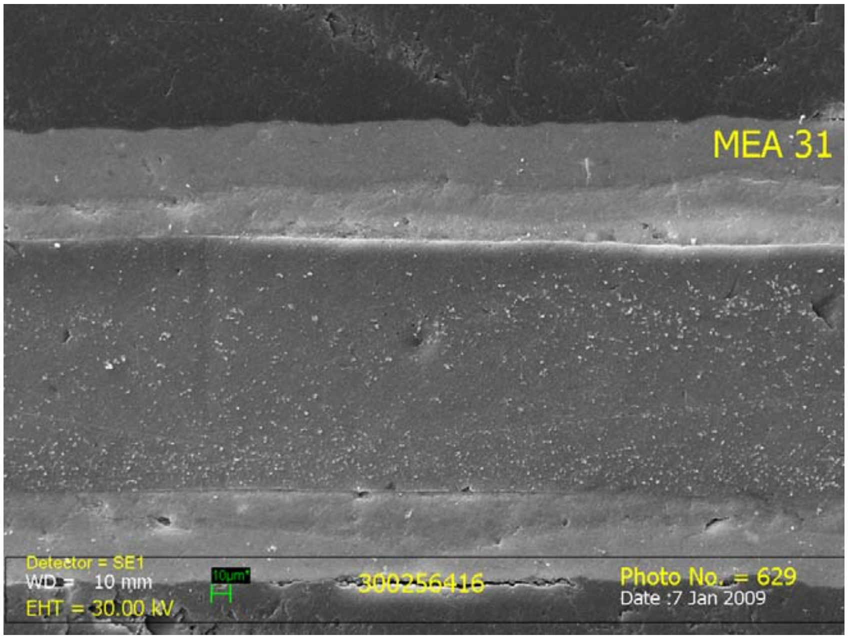

Figure 7. SEM of MEA 31 Showing No Sulfur Layer Buildup

SRNL Analytical Development obtained EDAX spectra on MEA-31 Location #1 (nearest anolyte inlet) moving from the cathode catalyst layer into the membrane. The beam energy was reduced to 10 kV and the acquisition time was limited to 30 seconds to minimize vaporization of the membrane. During the analysis, the instrument was also moved horizontally along the MEA so that the next interrogated spot would be outside of the beam-affected region from the previous spot. From the EDAX spectra, it was observed that the sulfur content was very small in all locations.

Figure 8 shows an SEM at the cathode interface for MEA 31. Figure 9 through Figure 11 show the corresponding spectra of selected interrogated spots.

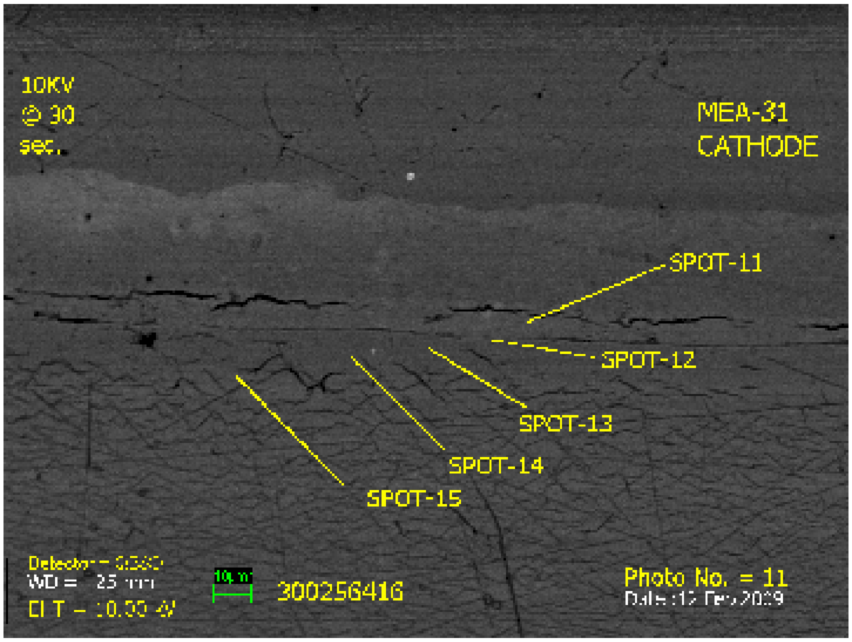

Figure 8. Scanning Electrom Microscope Photo of MEA 31

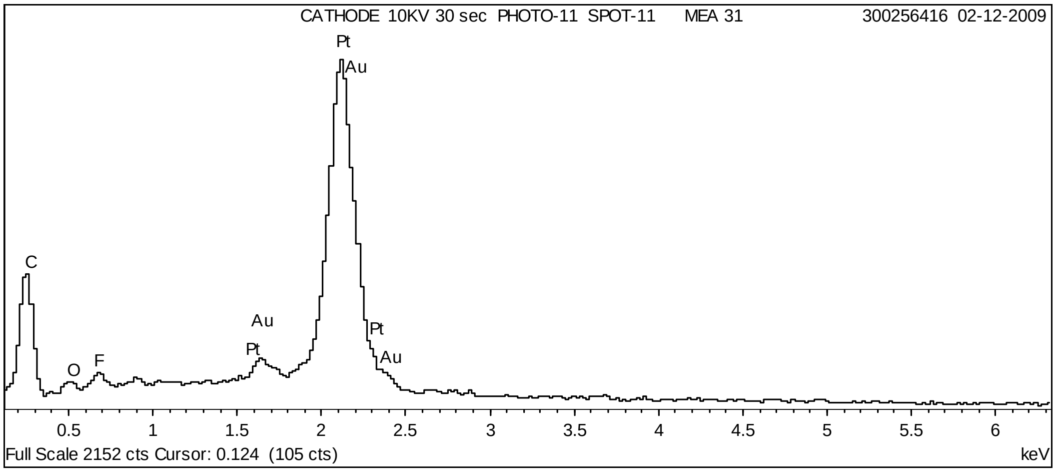

Figure 9. Spectra from MEA 31 Spot 11 of Figure 8

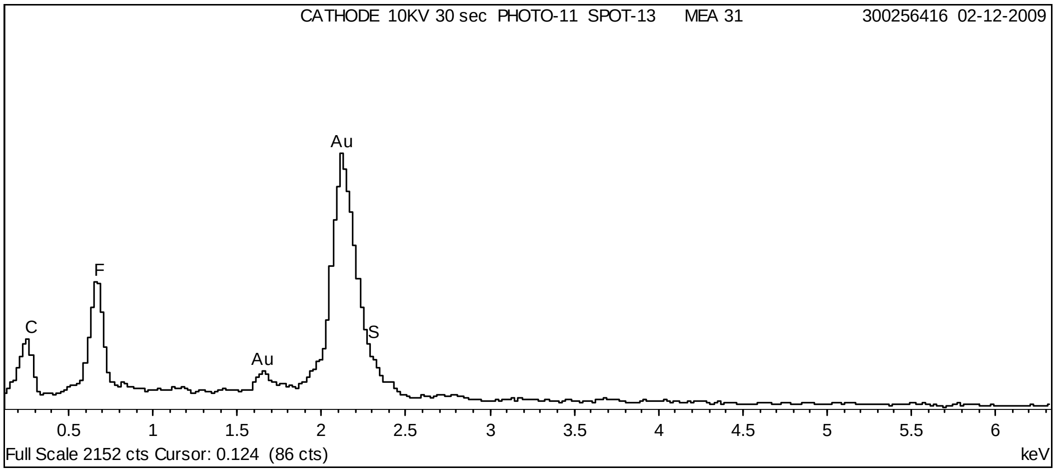

Figure 10. Spectra from MEA 31 Spot 13 of Figure 8

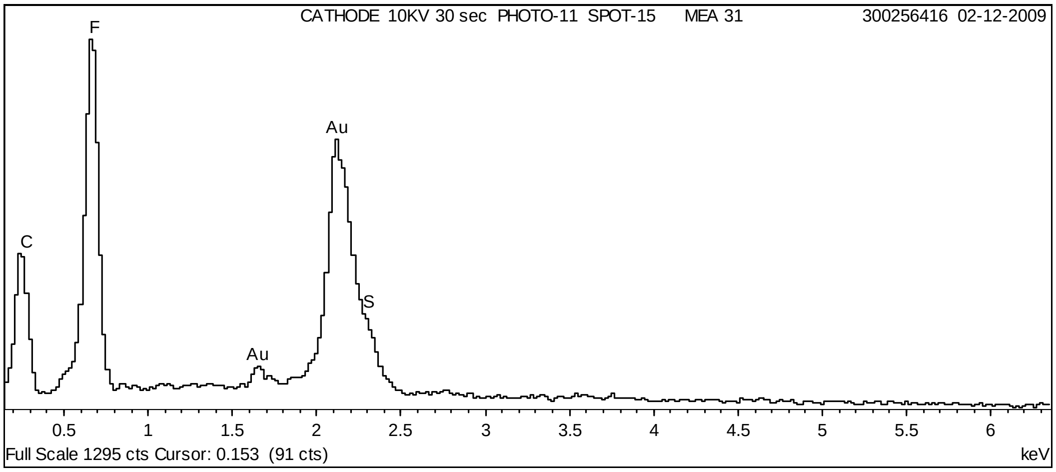

Figure 11. Spectra from MEA 31 Spot 15 of Figure 8

Note the decrease in Pt content and increase in F content across the series. These results suggest that sulfur is not accumulating in this MEA near the cathode-membrane interface and no evidence of a precursor stage to a sulfur-rich layer.

SEMs from both Clemson University and SRNL Analytical Development showed no evidence of significant sulfur formed at the cathode of MEA 31.

Transmission electron microscopy (TEM) analysis was also performed. The results verified previous analysis which showed no visible formation of the sulfur rich layer.

MEAs 30 and 31 were tested at high SO_2 utilization using SRNL's "SO_2–limited" operating procedures. The next step will be to demonstrate similar sulfur-free operation for extended operating lifetimes.

2.3.2 Upgrades to Single Cell Test Facility

The HyS single cell electrolyzer test facility in SRNL's Engineering Development Laboratory (EDL) has been shutdown to allow necessary modifications. The single cell electrolyzer is designed for operation with a sulfur dioxide depolarized anode using a liquid sulfuric acid anolyte solution. The cell has an active cell area of approximately 55 cm2, which allows production of approximately 20 lph of hydrogen. Most of the membrane electrode assemblies (MEA) tested in the electrolyzer have resulted in the formation of a sulfur layer between the cathode and membrane, which is highly undesirable. In the first quarter of FY'09 SRNL developed a methodology for preventing the formation of a sulfur layer, which was tested during two successful fifty hour runs with MEAs 30 and 31.

Due to operating requirements and safety regulations it was necessary to have at least one engineer present at all times during the testing. In order to further verify the effectiveness of the new operating method, it will be necessary to run longer duration tests. To that end, SRNL is creating the ability to operate the facility overnight unattended. A list of action items was prepared to allow unattended operation, and the process is proceeding. Some items on the list require purchasing and installing instruments, purchasing and installing actuator valves and software changes to computer that controls the experiment.

Work continued throughout the second quarter to upgrade the HyS Electrolyzer Single Cell Test Facility in the EDL for unattended operation. Modifications have been made and new equipment installed to permit test facility operation at temperatures up to 95C and pressure to 80 psig (6.3 bar). The control system has been modified so that it will permit a number of functions that previously had to be done by personnel to be conducted automatically, including: water addition and density control of the sulfuric acid; SO_2 addition; level control of anolyte tank; and syringe pump refilling. Alarms can be sent to a manned control room 200 yards away. A laser technique has been added to measure level in the Anolyte Tank so that the Data Acquisition System (DAS) can control level automatically. The old Hydrogen Collector has been replaced with a mass flowmeter. The DAS will use anolyte density measured by the density meter to control the water addition rate to the anolyte. The DAS also now controls the Anolyte Tank pressure by controlling the rate of sulfur dioxide addition. An automatic refill has been added to the heating bath for the Anolyte Tank to compensate for evaporation. The cathode flush, formerly provided by a rotameter, has been upgraded to a flow controller. The cell heaters had been upgraded by the addition of automatic controllers.

In order to provide additional analysis of the hydrogen product gas, an Agilent Model 3000a Micro Gas Chromatograph (GC) is being configured and integrated into the product stream of the single cell electrolyzer test stand. The GC will provide real time measurements from the electrolyzer identifying and quantifying impurities of the gas product stream.

All new equipment has been installed and a final safety review and approval were completed. Final checkouts are in progress, and the restart of the test facility is planned for early in the Third Quarter of FY '09.

3.0 PROJECT MANAGEMENT

The HyS electrolyzer work package was modified due to a drop in anticipated funds. The Level 2 milestone, M2NSR07TC030131_Prepare Modeling Report, due 8/15/09, was removed from the workpackage as this reduction reflects the amount of funding we had allocated to this task. A new spendout plan and schedule were included in the revised work package.

Professor Brian Benicewicz of the Department of Chemistry and Biochemistry at the University of South Carolina visited SRNL on January 13, 2009. Professor Benicewicz is a leading expert on conductive membranes, including the development of a new synthetic method for phosphate-imbibed polybenzimidazole (PBI) membranes. These PBR membranes are currently being commercialized by BASF (Germany) for high temperature fuel cell applications. This type of membrane may be a good candidate for the HyS process, particularly if a sulfate-imbibed version could be produced. Professor Benicewicz will first test the chemical stability of the phosphate-imbibed PBI membrane in concentrated sulfuric acid. If the phosphate-imbibed PBI membrane exhibits good chemical stability, he will provide a sample to SRNL to test for SO_2 transport characteristics and electrolyzer performance. These results will be compared to previous results with phosphate-imbibed PBI membranes produced by a different synthetic method. The original PBI membranes, obtained from PEMEAS, exhibited reduced SO_2 transport and good conductivity, but suffered reduced chemical stability, presumably due to the leaching of the phosphate upon contact with the concentrated sulfuric acid solution. Professor Benicewicz and his research group will also explore synthetic strategies for producing a sulfate-imbibed PBI membrane.

The Level 3 Milestone to complete a Second Quarter report was completed, and the deliverable report has been uploaded to PICS. A significant amount of planning has been accomplished for the SRNL lead Electrolyzer Workshop scheduled for April 20th and 21st.

Due to funding restrictions imposed by the Continuing Resolution, additional funding to SRNL was provided by the Idaho Operations Office under an MPO. Funding continued to be an issue throughout the Second Quarter as work had to be limited in several areas, primarily the design and fabrication of the pressurized button cell test facility.

4.0 FUTURE WORK

Work will continue during the second quarter of FY09 on the Component Development task, Gas Diffusion Electrode development, and Single Cell Performance Testing.

The Component Development task will continue to evaluate advanced cell membranes, including samples from Sandia, CWRU, Clemson, USC and DuPont. We will also design and construct a second small-scale electrochemical cell and test facility. The Pressurized Button-cell Test Facility will allow versatile cell operation at higher temperature and pressure conditions, permitting a much faster screening of new membranes and cell configurations. A workshop to address SDE performance issues will be organized and held during the third quarter. All interested parties involved in SDE development will be invited to attend.

Giner Electrochemical will continue development and testing of the Gas Diffusion Electrode cell as a means of developing an alternative cell design.

The single cell electrolyzer test facility has been reconfigured to allow unattended operation. The startup of this facility will occur early in the next quarter Tests will be conducted over longer test periods to examine the issue of sulfur crossover and to determine the effects of various operating conditions and cell configurations to support the completion of the Level 1 Milestone.