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# 1.0 ABSTRACT

The following report details the results under the DOE SECA program for the period July 2006 through December 2006. Developments pertain to the development of a 3 to 5 kW Solid Oxide Fuel Cell power system for a range of fuels and applications.

This report details technical results of the work performed under the following tasks for the SOFC Power System:

- Task 1SOFC System Development
- Task 2Solid Oxide Fuel Cell Stack Developments
- Task 3Reformer Developments
- Task 4Development of Balance of Plant Components
- Task 5Project Management
- Task 6System Modeling & Cell Evaluation for High Efficiency Coal-Based Solid<br/>Oxide Fuel Cell Gas Turbine Hybrid System

The next anticipated Technical Progress Report will be submitted July 2007.

# 2.0 EXECUTIVE SUMMARY

## 2.1 System Development Executive Summary

SECA Phase II is a continuation of the core hardware development activities begun in Phase I. The systems efforts in Phase II are more application-driven as Delphi moves this technology closer to pilot and production releases. The Phase II program will support and address two markets that Delphi has selected to pursue with the SOFC technology it has been developing with the assistance of the DOE. The stationary market and the transportation market have unique demands, and the development tasks must address the specific values that are the economic drivers of the design and application.

Two power system products are the focus for SECA Phase II:

- **Stationary:** Small Residential Combined Heat and Power (CHP)
- Transportation: Commercial Vehicle Auxiliary Power Unit with CHP

Stationary power systems will be referred to as Stationary Power Units (SPU) and are the general product classification that applies to a power generation device that is permanent and fixed in a location with a structure that provides primary or back-up power to an occupant. The intended market will have the device installed with nearly continuous operation. There will be few thermal cycles over the expected lifetime of nearly 10 years or approximately 80,000+ hours.

Power systems for the transportation market will be referred as Auxiliary Power Units (APUs) and are the general product classification that applies to a power generation device that is not fixed in location but can be mounted to a vehicle or transported to a site for temporary power. The device will be mounted in a vehicle or portable frame and operated for short periods or cyclic, based on the usage time profile. The expected lifetime will be nearly 10 years or approximately 15,000 hours.

## 2.2 SOFC Stack Development Executive Summary

Delphi has continued to build and test 30-cell Gen 3.1 stack modules. Key lessons have been learned from the performance of these modules and their post-test autopsy analysis. The next iteration in stack design (Gen 3.2) is complete and prototype parts are currently being validated.

Fundamental development has focused on the development of low cost coatings for interconnects, development of robust seals, development of sulfur tolerant anodes and understanding the failure mechanisms within the cell and stack during thermal cycling and long term continuous durability tests.

Process development and improvements have also been key focus areas for development. This included development of more robust and cost effective formulations and processes for cell manufacturing, and optimization of cassette fabrication and stack assembly processes to improve first time quality of stacks.

## 2.3 Reformer Developments Executive Summary

#### 2.3.1 Reformer – Subsystem Executive Summary

Focused work on diesel (US07) endothermic reforming was conducted this period. The benefit of recycle as a reactant to diminish inlet coking was demonstrated. A comprehensive comparison of NCV (non contact vaporizer) versus CV (contact vaporizer) was conducted and the CV approach was better. The CV concept / hardware were subsequently demonstrated to 500hr with no carbon buildup effecting performance. The Gen 5T reactor continues to be evaluated versus the DPS3000D/NG requirements. Initial enthalpy balances and heat exchange analysis revealed areas of improvement in heat management. These Gen 5T deficiencies are being addressed thru a Gen 5.1T brazed core design and a more substantial re-design for Gen 8 (for DPS3000NG SPU – Mar '07). Sources of reforming variation between reforming paths (tubes of Gen 5T reactor) have been investigated and quantified through both empirical and modeling efforts. A natural gas cracking reactor has been developed to crack or reform C2s to make possible the benefits of internal reforming of Natural Gas. This reactor awaits validation.

#### 2.3.2 Reformer Catalyst – Component Level Executive Summary

Although deactivation was observed, Generation 3.5 reforming catalysts had more than sufficient activity to meet system requirements for significantly more than the 4600 hours of testing completed. Progress continues to be made in optimizing catalyst formulations and processes to meet natural gas pre-reforming product requirements. Comparison of several different substrate configurations reveals that high-cell count ceramic monoliths are most effective for partial oxidation reactions, while moderate cell count ceramic foams are most effective for endothermic reactions. Improvements to liquid fuel vaporization and mixing permitted extensive durability and performance testing using diesel fuel for endothermic reforming. Performance characterization of diesel fuel endothermic reforming indicates that all system operating points can be met, within requirements, using existing reformer designs. Durability testing of diesel endothermic reforming catalysts indicates nearly the same rate of deactivation as catalyst tested for methane partial oxidation. Reforming with recycle at 0 % stack utilization has no impact on feed conversion to reformate

#### 2.4 BOP Components Development Executive Summary

Additional durability hours were accumulated on existing PAMs installed in systems. A new six valve PAM assembly was designed, fabricated and assembled along with new modular control valve blocks.

Cast Inconel Integrated Component Manifolds were received from the supplier and process development brazing the castings has been kicked-off.

Recycle pump robustness has been improved by re-orienting the pump, changing pump bearings, and modifying a recycle cooler.

Igniter failure analysis continued. An igniter test was developed for screening out potential igniter solutions.

Significant progress was made in the area of desulfurization. Prototype Natural Gas Desulfurizers were specified, purchased, and placed on test. This completed the Phase II Milestone: running a system on line Natural Gas. A designed experiment/robust engineering test of sorbents for a Hot Reformate desulfurizer continued, and resulted in sorbent selection for a non-regenerating desulfurizer.

#### 2.5 Coal-Based SOFC Gas Turbine Hybrid System Executive Summary

United Technologies Research Center (UTRC) has initiated an effort since August 2006, as part of Delphi's ongoing SECA project, to investigate solid oxide fuel cell (SOFC) system configurations utilizing gasified coal and evaluate SOFC cells that are provided by Delphi. The objective of the system study is to formulate highly efficient SOFC-based hybrid system configurations and establish an optimized conceptual design of SOFC stack and stack-module. The objective of the cell evaluation effort is to determine the compatibility between Delphi's cell and UTRC's stack design and identify gaps between the current Delphi cell technology and the cell stack requirements for the MW-scale Hybrid Power System.

Effort during the past several months have been focused on understanding power system requirements and coal-based fuel composition, conducting initial assessment of various power generator configurations and CO<sub>2</sub> capture options, conceptual design of stack module for MW-scale power plants, and Delphi cell performance evaluation. Detailed technical progress is reported in the following sections.

# 3.0 TASK 1: SOFC SYSTEM DEVELOPMENT

#### 3.1 System Development Executive Summary

SECA Phase II is a continuation of the core hardware development activities begun in Phase I. The systems efforts in Phase II are more application-driven as Delphi moves this technology closer to pilot and production releases. The Phase II program will support and address two markets that Delphi has selected to pursue with the SOFC technology it has been developing with the assistance of the DOE. The stationary market and the transportation market have unique demands, and the development tasks must address the specific values that are the economic drivers of the design and application.

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Power systems for the transportation market will be referred as Auxiliary Power Units (APUs) and are the general product classification that applies to a power generation device that is not fixed in location but can be mounted to a vehicle or transported to a site for temporary power. The device will be mounted in a vehicle or portable frame and operated for short periods or cyclic, based on the usage time profile. The expected lifetime will be nearly 10 years or approximately 15,000 hours.

## 3.2 System Design

## 3.2.1 Define System / Subsystem Requirements

The primary development initiative during this period was a three kilowatt, natural gas fueled, stationary power system. The overall system requirements were generated, leading to requirements for stack and reformer subsystems, as well as the balance of plant components. Additional hardware in this system will include an inverter, an energy storage system to aid in transient load handling, a pump to boost the pressure of the natural gas, a gaseous fuel desulfurizer, and an endothermic reformer rather than a catalytic partial oxidation (CPOx) reformer.

The Quality Functional Deployment process was used to determine the most important requirements, allowing each of the system requirements to be ranked based on relative

importance. **Table 3.1.1-1** presents the sixteen system needs and how they were ultimately ordered. The system requirements were generated once the order was established utilizing the system operating metrics. The highlights of these requirements include a gross stack power output of 4400 watts, a reformer output power capability of 8800 watts, an overall system efficiency of 45% at its rated output, and the ability of the system to handle sulfur laden fuel.

Customer System Requirements	
Prototype Cost Target (k\$/unit - Assuming 10 Units)	
Degradation Rate [% Rated/500 hours]	
Fuel Sulfur Tolerance [ppm]	309
Rated Output Power [kW]	307
Thermal Cycles (-10% of Rated)	288
NOC Fuel to Elec Efficiency	286
NOC Power	277
Rated Fuel-to-Elec (60Hz, single phase AC) Electrical Efficiency [%]	
Max Ambient Operating Temp [deg C]	254
Package Size [Liters] (not including fuel tank, no dimension greater than 24")	231
Mass Limit [kg]	231
Noise Output [Rated Load: SPL @ 3m, dBA]	181
B10 Life @ 90% Confidence [hours] (Includes load transients and thermal cycles d	164
Gaseous Fuel Supply Pressure [kPa]	
Start-Up Time (25 Ambient soak to Rated Power) [minutes]	
Surface Temp Rise (from Ambient) [deg C]	90
Min Ambient Operating Temp [deg C]	47

 Table 3.2.1-1
 Customer System Requirements Rankings

Further definition is being developed for the other major system components such as the process air blower and the cathode air heat exchanger. Special attention is being given to the cumulative system pressure drop with regard to these major system components specifically, as well as other secondary components in the system.

## 3.2.2 Develop System Mechanization

The system mechanization for the DPS-3000-NG system has similar characteristics to the mechanization of the Phase I development system, SPU-1B. However, some marked changes have been made in order to reshape the efficiency curve over the output power range, as well as augment the thermal management of the high temperature components. Though many of these changes have only been supported by modeling, the design of this hardware will take place in the next period, with the integration and testing to occur in the subsequent period.

The most notable change to the system comes from the replacement of the CPOx reformer with an endothermic reformer. This change boosts the system efficiency in the middle of the power range, as well as cause a significant rerouting of flow through the system.

## 3.2.3 Perform System Analysis

The modeling results generated this period were the output of a system model similar to that used in the generation of SPU1B performance forecasts. It contains a stack model with a simply scaled polarization curve, and does not make performance adjustments based on operating parameters such as temperature. However, this model has been reconfigured to align with a set of fuel processing equations that are generic with respect to the hardware configuration and fuel input. This means that a natural gas or diesel system, with a CPOx or endothermic reformer, can be analyzed with the same model. The only adjustment occurs in the calibration targets, providing a means for comparing many systems without significant model changes.

Though this model is adequate for steady state operating characteristic predictions, a more detailed transient stack model will be integrated in the next period. This will allow for better predictions of performance to be made with respect to system operating characteristics in transient modes, as well as across a range of operating conditions for a given steady state load.

The initial system modeling is focused on two discrete points, the rated power point (RATED), and the normal operating condition (NOC) point. The notable differences in the operation of the two are shown in **Figure 3.1.3-2**. The first difference is that the fuel processing efficiency is 35% higher at RATED operation than at NOC, primarily due to the increase of internal reforming.

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		FORECAST	FORECAST
Stationary Power Unit (SPU)		DPS 3000	DPS 3000
Steady State Power Analysis		RATED	NOC
REFORMER			
Fuel Power	kW	7.67	5.07
Fuel Massflow	g/sec	0.153	0.101
Anode Tail Gas Recycle Fraction	%	<b>50%</b>	37%
Primary Fuel Fraction (□ <sub>1</sub> )	%	1%	100%
Reformer (Fuel Processing) Efficiency	%	172%	137%
STACK			
Stack Fuel Utilization	%	60%	44%
SOFC Stack Conv Efficiency	%	32%	<b>29%</b>
System Effective Air Utilization	%	28%	11%
Cell Operating Voltage	v	0.70	0.86
Stack Output Power	kW	4.177	1.974
Stack Current	Α	149.2	57.5
Estimated Internal Reforming Effect	kW	2.300	0.000
PARASITIC LOSSES			
Parasitic Load Efficiency	%	<b>90%</b>	83%
Electric Parasitic Load (-)	kW	-0.424	-0.344
DC/AC Inverter Efficiency	%	92%	92%
NET SYSTEM OUTPUT			
Net System Efficiency @Rated Power	%	45.0%	29.6%
Rated Output Power	kW	3.45	1.50

#### Figure 3.2.3-2 Static System Model Results

The second difference between the two points is the fuel utilization, being 60% at RATED and 44% at NOC. The stack is the primary need, while the reformer, operating endothermically at the NOC point, requires a burner with adequate energy available to contribute to the reaction. That burner energy need is not as great at rated power as the full reforming load is carried by the stack, and the duty of the reformer is low.

The third notable difference is the air utilization. While operating at the NOC point, the system air utilization is only 11%. This is due to the fact that the cathode air provides the only means of cooling to the stack. When the RATED power point is evaluated, the system air utilization is increased, to 28%. That increase is possible as the effect of the internal reforming reaction reduces the heat removal duty of the cathode air.

The system airflow characteristics, which aid in understanding the dramatic air utilization increase, are shown in **Figure 3.2.3-3**. The NOC point lies at 50% of the total application

load, and requires nearly eleven grams per second of total air, with nine grams per second being cathode air used in cooling the stack. The RATED point lies at 100% of the total application load, twice the electrical output of the NOC point, yet requires total system airflow of ten grams per second, still with nine grams per second of cathode air required to maintain the stack temperature.



Figure 3.2.3-3 System Airflow Characteristics

# 3.2.4 Develop Power Architecture and Electronics Design

(No update this period.)

# 3.2.5 Develop System Design and Perform Optimization

The design layout effort this period was focused on the three kilowatt system. The layout of this system is a vast departure from the previous generation, with the primary goal being optimal control over the thermal environment of each component. The testing that took place during Phase I, and that has taken place thus far in Phase II, suggests that the thermal environment of all components is critical to their performance. This includes thermal communication not only to the ambient environment, but also communication between components. As the system attempts to achieve higher and higher overall

efficiencies across the power band, the ability to isolate and possibly control these component environments becomes a greater concern.

The design effort to date produced the layout shown in **Figure 3.2.5-1.** The notable additions to this system include the desulfurizer beds, the pump used to boost the natural gas pressure, and the hydrocarbon cracker. These components represent necessary content additions to operate the system on commercially available natural gas, versus the chemically pure methane used today.



Figure 3.2.5-1 System Model Concept

The stack module has been upgraded to include an external cooling jacket, as well as an independent insulation enclosure, aiding in the control of the stack thermal environment. The CPOx reformer has been replaced with an endothermic reformer, which, as previously mentioned, aids in boosting the fuel processing, and thus system efficiency during the middle of the operating range. The process air module has also been upgraded to include a commercially available blower and utilize six valves, rather than the four used in the current unit.

Several hot zone components have been given package volume for insulation enclosures in order to aid in the control of the thermal environments. The semi-transparent enclosures that exist around the stack (light blue), the hydrocarbon cracker (light gold), the anodecathode equalizer (light gold), and the hot zone base (light blue), represent the individual enclosures.

There are three more pieces of hardware to be added to the system, they include an inverter, a system to enable the handling of random load applications, and the internal power electronics. These components will enable the system to supply power to typical appliances, rather than the simple direct current power output available today.

## 3.2.6 Develop System Controls

Activities within this task involve optimizing and finalizing the system control strategy and safety and diagnostic system software algorithms. This will be accomplished through algorithm development and validation. Validation of system control algorithms and safety and diagnostic algorithms will be achieved through a process which incorporates virtual dynamic system models, hardware-in-the-loop simulation and finally system tests. Delphi will virtually test and tune the algorithms using Matlab/Simulink/Stateflow tools, and validate the algorithms using an OPAL-RT Testdrive HIL (Hardware-In-The-Loop) simulator. Once confidence in the algorithm and calibrations is established, the algorithms will be implemented on the real system hardware. The current control algorithm and safety and diagnostic system are nearly complete and fully automatic, but in order to progress to a production ready robust solution, a significant amount of validation work needs to be completed.

# 3.2.6.1 Safety and Diagnostics

A major accomplishment has been reached in the safety and diagnostics portion of the control algorithm during this reporting period. The second level S&DII, which includes state based sensor monitoring, has been implemented, tested, and calibrated successfully. The implementation also includes sensor warning levels and provides intermediate safety and diagnostics states to indicate the current "health" of the system as well as a user defined timer limit before complete shutdown. The advantages of this implementation has already been seen during system testing with failures caused by slow fuel leaks through fuel lines, as well as defining the operation range of the GPC temperature. This achievement brings the control algorithm a step closer in finalizing production development. Future implementation will have individual timers based on either severity of the fault or the type of the overall fault.

# 3.2.6.2 Hardware-in-the-Loop Simulations

The OPAL-RT HIL simulator has been validated on a real-time dynamic model of the Process Air Module currently used in the SPU01 systems. This is also a monumental accomplishment which validates the HIL setup and verifies the reliability of the process. As individual system models are created and prepared, they too shall be validated individually on the system finally becoming a fully integrated dynamic model. This will allow for control algorithm verification and validation of the system model, system hardware design, system response to control software, controller response to system dynamics. All of these are essential for efficient system development and validation.

#### 3.2.6.3 Version 2 SOFC Software Design

Despite successful implementation of current software on the latest SOFC system, it remains convoluted and cumbersome in many aspects. The original model was created over eight years ago with little knowledge of an SOFC system. There have been many improvements to the software to facilitate required operation and constancy in the algorithm, however all of this is limited to the original structure. A new software algorithm is currently being designed to focus on the following:

- Modularity To support multiple platforms and fueling schemes
- Efficiency Eliminating control loop dependencies on intermediate variables as well as unnecessary cascade PI loops, etc.
- Advanced Safety & Diagnostics Instilling safety & diagnostics at subsystem levels for simple creation of fault codes, creating foundation for possible mitigation action if required, and EEProm management for fault signals.

The new software design is currently being specified with initial programming scheduled to begin towards the end of the first quarter of 2007

## 3.3 System Integration and Testing

## 3.3.1 Subsystem/Supplier Verification to Requirements

(No updates this period.)

#### 3.3.2 System Assembly and Manufacturing Process Development

#### 3.3.2.1 Development of Inventory System

The previous inventory system was not functioning as efficiently as it should due to the number of systems being assembled and disassembled.

Part tracking was relatively uncomplicated in the past when there was only one or two systems to build at time. Currently there are multiple systems being built and tested while others are being disassembled. This increase in testing has strained the old phase I prototype part tracking system. To accommodate the increased workload, an inventory system has been developed as described below:

- The parts are organized and staged for builds as shown in **Figure 3.3.2.1-1.**
- A manual sign out sheet is currently used to track the parts. This will eventually be replaced by an electronic barcode system which will be linked to the central parts database.

• Parts will undergo a conformance measurement phase where they could be rejected or accepted based on key product characteristics (KPC's).

It is an ongoing process to improve the inventory system for accessibility as well as ease of use. Constant improvement will result in a centralized system designed to streamline the assembly and disassembly processes.



Figure 3.3.2.1-1 Part / Inventory Staging Area

## 3.3.2.2 Redesign of SPU1B for Manufacture

There is a constant drive at the system level to design for manufacturability and ease of assembly. The latest design iteration illustrates two key examples of design for assembly:

- The fuel interface on the Application Interface Module has been redesigned to allow the technician to tighten fuel fittings before installation -see **Figure 3.3.2.2-1**
- The stack mounting screws now have an access panel. This allows the technician to remove the stacks while all other components remain undisturbed see **Figure 3.2.2.2-2.**

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Figure 3.3.2.2-1 Fuel Interface



Figure 3.3.2.2-2 Stack Access Panel

Because the system is so tightly packaged, every effort has to be made to make the assembly process as easy as possible. These changes have greatly reduced the assembly time while improving system analysis during teardown and autopsy.

#### 3.3.3 System Performance Testing

#### 3.3.3.1 Primary Integration Test Stand Capability

The Primary Integration Test Stand is utilized in all stages of component testing where isolation of one component is necessary. The versatility of the equipment is what sets it apart from standard test stands. The differences are listed in the following sections. This test stand is capable of testing individual system components as well as determining the effect on the entire system when changing the nature of a single component.

#### 3.3.3.2 Stack Isolation Module

The performance test stand was designed such that the stacks could be isolated from all other system components - **Figure 3.3.3.2-1**. The isolation module allows the engineer to analyze the performance of individual cells in two separate stacks under different environmental conditions. This assessment allows a better understanding of environmental effects on different areas of the stack. The module will also allow confirmation of modeling more effectively by changing one variable at a time.



Figure 3.3.3.2-1 Stack Isolation Module

#### 3.3.3.3 Fuel Blending Capability

The performance test stand has the ability to blend the following gases:

• H<sub>2</sub>

- N<sub>2</sub>
- CO
- CO<sub>2</sub>
- CH<sub>4</sub>
- Simulated natural gas (without Sulfur)
- Simulated natural gas (with Sulfur)
- Utility supply line natural gas

By blending the fuels, any simulated reformate can be chosen. This allows a surrogate hydrocarbon fuel to be employed for testing before a means of producing reformate is available. The blending capability allows the engineer to make fueling-based decisions quickly and cost-effectively - **Figure 3.2.3.3-1**.



Figure 3.3.3.3-1 Blend Fuel Module

# 3.3.3.4 Desulfurized Natural Gas

The performance test stand is capable of testing system components with desulfurized natural gas. Natural gas is used from the local utility to carry out the tests. Once it has entered the building, a lift pump compresses the natural gas to 55 psig. It is then run through a point-of-use desulfurizer where it can be utilized as a primary fuel source. The

desulfurized natural gas is a requirement to testing system components on a readily available fuel - see **Figure 3.3.4-1**.



Figure 3.3.3.4-1 Natural Gas Desulfurizer

# 3.3.3.5 Performance Testing Results

## 3.3.3.5.1 Methane Fuel with Internal Reforming

Performance testing is done to determine the maximum power and efficiency that the system can achieve without causing damage to the system components. With the current design, the performance test also allows the engineer to test the internal reforming mode of operation. Performance testing was executed on a methane-fueled SPU1D system to determine the maximum power and efficiency while operating in the internal reforming mode. The results of a performance test can be seen on **Figures 3.3.3.5.1-1** and **3.3.3.5.1-2**. Test result highlights include:

- Rated net power: 2.2 kW
- Peak net power:2.6 kW
- Peak System efficiency: 36%
- Run time: 150 hours (Figure 3.3.3.5.1-3)-currently running
- Fully automatic warm up in <4 hours







Figure 3.3.3.5.1-2 SPU1D Peak Performance



Figure 3.3.3.5.1-3 SPU1D Durability Testing

SPU 1D systems were also tested to verify that modifications to the stack exhaust recycle circuit would perform as expected. The changes requiring validation include:

- Reorienting the recycle pump 90 degrees to prevent problems with the water.
- Decreasing the surface area of the recycle loop heat exchanger to decrease the temperature drop of the recycle gas stream.

Both changes have been tested in the system and are operating as expected. Further long-term testing is needed to verify durability of the recycle pump with the modification.

# 3.3.4 System Reliability Testing

## 3.3.4.1 Natural Gas Fueled Durability Test

The ability of the system to operate on natural gas is one of the outlined requirements. It is imperative that the system is able to run on a readily available fuel source for widespread commercialization. Reliability testing was done with line natural gas as the fuel source in order to show that the system is ready for a widely available fuel source. The utility company delivers low pressure natural gas to the Delphi facility. Once inside the facility, a compressor boosts the fuel to 55 psi. The pressurized fuel passes through a point of use desulfurizer next to the test stand where it is consumed by the system - see above **Figure 3.3.3.4-1**.

The results of the natural gas fueled constant current testing can be seen in **Figure 3.3.4.1**. **1**. Note that the power degradation of 2.1% per 500 hours is the same with natural gas as it is with previous methane fueled testing. The details are described below:

- 5.4% degradation over 1280 hours
- 2.1% degradation per 500 hours
- Power at beginning of test= 1.2 kW



Figure 3.3.4.1-1 SPU1D Durability Testing

The desulfurizer is projected to last 1500 hours at a flow rate of .22 g/s of natural gas with 20 ppmv sulfur (DMS). The current flow rate is .13 g/s and the average total sulfur level is 4.1 ppmv. Further testing will confirm the actual lifespan of the unit under normal operating conditions of the system.

There is a focus to determine the effects of natural gas on the reliability of the SPU1B system. At present, there is no notable difference between running the system on methane or desulfurized natural gas.

# 3.3.4.2 Assess System Capability Gaps

Several advancements will be required in order to achieve the desired targets. The first advancement will be in the ability of the system to tolerate sulfur laden fuel. The handling will be enabled through two paths, the first being the passive desulfurization beds used at

the fuel inlet, and the second will be the ability of the stack to handle reasonable sulfur breakthrough.

The second advancement will be the development of an endothermic reforming system. This will be necessary in order for the system to achieve higher efficiency targets at the low and middle ranges of the power band. The testing of an endothermic reformer will begin in the next period, with the intent to integrate the hardware with a system.

The third advance will be in the system's ability to control the thermal environment, specifically around the stacks. Testing has demonstrated that the stack performance depends greatly on their thermal state, and that state is terrifically affected by the environment. This thermal dependence may also prove to exist with regards to the endothermic reformer, and the reduction of thermal losses in all components will only serve to benefit the system.

The fourth advancement will be in the ability of the system to navigate transient load application cycles. In order for the system to tolerate sudden applications of load, an energy storage system will be necessary. This energy storage system will provide the additional current needed for the load until the system fueling actuators are able to provide the necessary air and fuel to the stacks, allowing for the entire load to be sourced.

# 4.0 TASK 2: SOFC STACK DEVELOPMENT

#### 4.1 SOFC Stack Development Executive Summary

Delphi has continued to build and test 30-cell Gen 3.1 stack modules. Key lessons have been learned from the performance of these modules and their post-test autopsy analysis. The next iteration in stack design (Gen 3.2) is complete and prototype parts are currently being validated.

Fundamental development has focused on the development of low cost coatings for interconnects, development of robust seals, development of sulfur tolerant anodes and understanding the failure mechanisms within the cell and stack during thermal cycling and long term continuous durability tests.

Process development and improvements have also been key focus areas for development. This included development of more robust and cost effective formulations and processes for cell manufacturing, and optimization of cassette fabrication and stack assembly processes to improve first time quality of stacks.

#### 4.2 SOFC Stack Development Experimental Approach

Typical stack testing is carried out using a test stand that has a hot furnace, electrical load bank and gas mixing cabinet. Cells are fabricated at Delphi. A typical experiment involves the measurement of standard polarization curves and power densities at constant load.

## 4.3 SOFC Stack Development Results and Discussion

#### 4.3.1 Design Stack

Delphi has continued to demonstrate the viability of the Gen 3.1 stack design. 30-cell stacks of this design were tested in the system and met the SECA Phase 1 program requirements. **Figure 4.3.1-1** shows an exploded view of the different parts that constitute a cassette repeating unit in the Gen 3.1 design.



Figure 4.3.1-1 - Generation 3.1 Repeating Units in a Stack

## 4.3.2 Model Stack Under Steady-State Conditions

Delphi has acquired the Marc model from SECA Core and is currently working to get it functional for the Delphi design.

## 4.3.3 Develop High Performance Cathode

No activity this period

## 4.3.4 Develop High-Performance Anode

The anode development task has continued to focus on understanding the H<sub>2</sub>S poisoning of the current anode. The primary focal point during this reporting period has been to understand effects of testing parameters such as voltage, current, and temperature on sulfur poisoning. All H<sub>2</sub>S poisoning testing has been done in button cells with  $(La_{0.60}Sr_{0.40})_{0.95}Co_{0.20}Fe_{0.80}O_3$  cathode (LSCF- 6428 A/B = 0.95) and standard Ni/YSZ anode using test stands which employ a 1" diameter circular cell sealed to an alumina tube. This schematic has been detailed in previous progress reports.

It has been reported that sulfur poisoning is influenced by operating voltage in electrolytesupported cells [1]; that for a given concentration of sulfur in fuel, lower operating voltages will result in lower power loss. To characterize the effect of voltage on the degree of sulfur poisoning, Ni-YSZ anode supported cells with LSCF cathode were tested following the test cycle shown in **Figure 4.3.4-1**. Each cell was subjected to various operating voltages from OCV to 0.5 V for 10 hours in each testing cycle to see the effect of voltage on sulfur poisoning. After 10 hour operation at various voltages, the cell was kept at an open circuit

voltage (OCV) for 3 minutes, and then voltage was maintained at 0.7 V for 2 minutes to check the degree of sulfur poisoning at the same voltage before the cell was returned to its particular operating voltage for another 10 hour cycle.



## Figure 4.3.4-1 - A Test Cycle to Characterize the Effect of Voltage on Sulfur Poisoning

The power retention of cells operated at different voltages after introduction of 1 ppm of  $H_2S$  is shown in **Figure 4.3.4-2**. In this plot, the power retention was measured at the particular operating voltage for each cell. As reported in the previous publication, the overall trend is that measuring at lower voltage shows higher power retention (or lower power loss). The cell operated and measured at 0.5 V retains 82% of power at 50 hours after  $H_2S$  introduction, while the cell tested at 0.8V exhibits only 49% of power at the same point. The power retention measured at 0.7V however, is almost identical for all of the cells (see **Figure 4.3.4-3**). This result indicates that the degree of sulfur poisoning is fundamentally the same regardless of operating voltage even though the power retention measured at different voltages does vary. In other words, sulfur poisoning is possibly a thermal process rather than electrochemical process and the degree of sulfur poisoning at the given concentration of  $H_2S$  and the given temperature is basically unchanged by operating voltages between OCV and 0.5 V.

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Figure 4.3.4-2 - Power Retention of the Cells Operated at Various Voltages after H2S Introduction (Power Retention for This Plot was Measured at the Various Operating Voltages)



Figure 4.3.4-3 - Power Retention Measured at 0.7 V: from the Cells Operated at Various Voltages

To understand the effects of measurement methods on apparent power retention after H2S exposure, measurements were made using the IV curves that were collected every 10

hours for the cell tested at the constant voltage of 0.7V. The initial IV curve (blue) and three of the IV curves representing 50 hour intervals after H2S was introduced are shown in **Figures 4.3.4-4** and **4.3.4-5**. Lines are drawn at various constant voltages in **Figure 4.3.4-** to show how the power retention was determined. Similarly, lines are drawn at various constant current levels in **Figure 4.3.4-5**. **Figure 4.3.4-6** shows the results of these IV curve measurements. These calculations, all based on the same set of IV curves, show much less apparent power retention at constant current than at constant voltage. Thus, constant voltage measurement is much more sensitive to sulfur poisoning (more power loss) than constant current measurement. It is necessary to maintain the same conditions for measurement is preferable in detecting sulfur poisoning since power loss under this condition is more sensitive than constant current measurement.



Figure 4.3.4-4 - IV Sweep Curves with Time: Power Retention Calculation at Constant Voltage



Current Sweep

Figure 4.3.4-5 - IV Sweep Curves with Time: Power Retention Calculation at

**Constant Voltage** 



Figure 4.3.4-6 - Power Retention Calculated from IV Sweep Curves: Constant Voltage vs. Constant Current

Second stage degradation is another noticeable effect of current and voltage. The rate of second stage degradation (a flatter region after a rapid drop up to 20 hours after  $H_2S$  introduction) is more sensitive at lower voltage or higher current. Thus, measurement at constant voltage of 0.7 V can provide more sensitive information on sulfur poisoning in terms of first and second stage degradation compared to constant current measurement or constant voltage measurement at higher voltage.

Impedance analysis conducted at OCV and with 0.2 V bias also shows the sensitivity of measuring voltage on second stage degradation (see Figure 4.3.4-7). Polarization resistance (represented by arcs) drastically increases after the introduction of H<sub>2</sub>S, while ohmic resistance (represented by X intercepts) does not significantly change due to the sulfur poisoning, is a general trend observed in impedance analysis. Therefore, a main effect of sulfur poisoning on power loss is increased polarization resistance reflecting changes in catalytic activity of Ni rather than an increase in electrical resistance. The impedance measured at OCV is much larger than that measured with a 0.2 V bias, which is close to the operating voltage. Furthermore, polarization resistance measured at OCV during the second stage degradation (refer to curves collected at 140, 200, and 300 hrs) does not show a consistent trend in polarization resistance in the second stage degradation while polarization resistance measured with 0.2 V bias shows an increase in polarization resistance with time, which is associated with power loss during the second stage. This result also confirms that lower voltage (or higher current) gives more sensitive results in terms of second stage degradation of sulfur poisoning.



Figure 4.3.4-7 - Impedance Data Measured at (a) OCV and (b) with 0.2 V Bias

In order to confirm the validity of power retention calculated from IV sweep curves, the calculated values are compared with data directly collected from the cells operated at constant current and constant voltage., Points represented by hollow shapes in **Figure 4.3.4-8** are calculated from the IV sweep curves of the cell operated at 0.7V, and solid lines are real data collected from the cells operated under either constant current (0.65A/cm<sup>2</sup>, initial voltage: 0.8V) or constant voltage (0.8V, initial current: 0.63A/cm<sup>2</sup>) conditions.

Basically, the values calculated from IV curves are well matched with data directly collected from the cells operated under the conditions compared. Thus, periodic IV sweeps taken from one cell can provide representative data on sulfur poisoning at the given concentration of  $H_2S$  and the given temperature.



Figure 4.3.4-8 - Power Retention from Direct Measurement vs. Calculation from IV Sweeps

**Figure 4.3.4-9** shows the effects of operating temperature on the power retention of Ni/YSZ anode supported cells at 0.7V after exposure to 1 ppm  $H_2S$ . Higher operating temperatures produce higher power retention. Power retention at 850°C was 23% at 50 hours after 1 ppm  $H_2S$  exposure, while only 51% of power was retained at 650°C. It is not certain at this point whether high power retention at high temperature is due to reduced sulfur poisoning or enhanced power (or current) associated with high temperature. Nonetheless, a similar rate of second stage degradation was observed at various operating temperatures.

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Figure 4.3.4-9 - Effect of Operating Temperature on Power Retention

# 4.3.5 Develop Cell Fabrication Techniques

The cell fabrication process development continues to be a key focus of ongoing efforts in the development of the SOFC system. Significant progress has been made in ceramic tape formulation development, scaling up material improvements with industrial suppliers, and efficiently producing more consistent cells with regard to flatness and dimensional control.

## 4.3.6 Develop Separator and Support Components

No update in the period.

## 4.3.7 Develop Seals

Delphi – Battelle continues to make progress in seal development. A reinforced glass seal has been developed that is robust to >15 thermal cycles.

## 4.3.8 Fabrication Developmental Stacks

Single cell testing of both intermediate size cells (33 cm<sup>2</sup> active area) and full size Gen 3 cells (105 cm<sup>2</sup> active area) has been used extensively to identify problems and evaluate potential solutions.

## 4.3.9 Evaluate Stack Performance

Evaluation of 30-cell stacks included polarization tests at  $750^{\circ}$ C on standard fuel consisting of 48.5% H<sub>2</sub>, 3% H<sub>2</sub>O, rest N<sub>2</sub>. The IV tests were terminated after applying electrical load up to 60 Amperes. Overall power density and individual cell voltages were measured to evaluate performance. **Figure 4.3.9-1** shows data from such a recent test.



Figure 4.3.9-1 - Typical IV Curve from a 30-Cell Stack Module with H<sub>2</sub> Fuel

The power density at 60 Amps (570 mA/cm<sup>2</sup>) is 475 mW/cm<sup>2</sup> at 25 Volts (average cell voltage of 0.83 Volts, 30% fuel utilization). Fuel utilization studies showed minimal (< 1%) lowering of power going from 30% to 70% fuel utilization. Stacks have also been successfully integrated and tested in the system. Two 30-cell stacks were run for 4600 hours in an SBU system. The degradation observed was 2% per 500 hours.

# 4.4 SOFC Stack Development Conclusion

Delphi has successfully continued to develop its Generation 3 stack design. Improvements have been implemented in design, process and fundamental materials. Work has focused on understanding and solving critical issues like durability, thermal cycling, and sulfur contamination to generate a more robust product.

# 5.0 TASK 3: REFORMER DEVELOPMENTS

## 5.1 Reformer Developments Executive Summary

## 5.1.1 Reformer – Subsystem Executive Summary

Focused work on diesel (US07) endothermic reforming was conducted this period. The benefit of recycle as a reactant to diminish inlet coking was demonstrated. A comprehensive comparison of NCV (non contact vaporizer) versus CV (contact vaporizer) was conducted and the CV approach was better. The CV concept / hardware were subsequently demonstrated to 500hr with no carbon buildup effecting performance. The Gen 5T reactor continues to be evaluated versus the DPS3000D/NG requirements. Initial enthalpy balances and heat exchange analysis revealed areas of improvement in heat management. These Gen 5T deficiencies are being addressed thru a Gen 5.1T brazed core design and a more substantial re-design for Gen 8 (for DPS3000NG SPU – Mar '07). Sources of reforming variation between reforming paths (tubes of Gen 5T reactor) have been investigated and quantified through both empirical and modeling efforts. A natural gas cracking reactor has been developed to crack or reform C2s to make possible the benefits of internal reforming of Natural Gas. This reactor awaits validation.

# 5.1.2 Reformer Catalyst – Component Level Executive Summary

Although deactivation was observed, Generation 3.5 reforming catalysts had more than sufficient activity to meet system requirements for significantly more than the 4600 hours of testing completed. Progress continues to be made in optimizing catalyst formulations and processes to meet natural gas pre-reforming product requirements. Comparison of several different substrate configurations reveals that high-cell count ceramic monoliths are most effective for partial oxidation reactions, while moderate cell count ceramic foams are most effective for endothermic reactions. Improvements to liquid fuel vaporization and mixing permitted extensive durability and performance testing using diesel fuel for endothermic reforming. Performance characterization of diesel fuel endothermic reforming indicates that all system operating points can be met, within requirements, using existing reformer designs. Durability testing of diesel endothermic reforming catalysts indicates nearly the same rate of deactivation as catalyst tested for methane partial oxidation. Reforming with recycle at 0 % stack utilization has no impact on feed conversion to reformate

# 5.2 Reformer Developments Experimental Approach

# 5.2.1 Reformer Subsystem Testing Setup

# 5.2.1.1 Reformer Lab Air Handling Improvement

Reformer test lab air handling was changed-over from a scrubber to a dilution type system. Previously, each of the 3 test stands had a dedicated catalytic exhaust gas converter (i.e., scrubber) responsible for exhaust gas conversion. Scrubbers required temperature control and monitoring to ensure exhaust stream mole percentages remain within safety standards for each gas constituent. Scrubber control capability was limited and required limiting
endothermic reformer testing to approximately 18kW (on a single test stand) in order to maintain exhaust stream safety standards.

A dilution system was sized to sufficiently dilute the exhaust gas stream of each test stand ensuring unburned hydrocarbons remain well below the required 4% LEL level and that carbon monoxide remains well below the required 1800ppm LD50 level. The capacity of each test stand was also upgraded to allow for the operation of three (3) endothermic Reformers simultaneously to 30kW. As a result, 3,400CFM dilution air was required per test stand.

## 5.2.1.2 Hard Plumbed Natural Gas

A 1 inch NG line service was added to the Reformer test lab. Each test stand has a ½ inch supply line. Pressure is centrally boosted to 55 psig throughout the Delphi Metro Park lab. This enables direct and economical NG fueled reformer testing.

## 5.2.1.3 Furnace for Contact Vaporizer Tube Testing

Currently, contact vaporizer design development verification is performed on a single performance test stand with an operating combustor. To expedite development, an eight-sample, single fuel pass apparatus was constructed using an oven to provide heat. Diesel fuel flow metering to each ½ inch O.D., U-shaped tube sample is provided by a Multec II injector (Delphi Production). Vaporized diesel fuel exits the oven chamber after approximately 36 inches exposure. The vaporized diesel fuel is fed into a heat exchanger where it is re-condensed, collected and properly disposed. In-situ monitoring of outlet fuel temperatures and pressure is achievable. The latter is critical to monitoring tube sample pressure drop, and serves as the primary response for determining deposit formation. Experimental factors include, but are not limited to:

- Tube Sample Material
- Tube Sample Wall Thickness
- Tube Sample Pre-Oxidation Treatments
- Tube Sample Coatings (I.D.)
- Temperature (Oven)
- Fueling Flow Rate
- Air Carrier Flow Rate
- Water Vapor (added to Carrier Air)

Efforts should lead to the optimum combination of CV tube material, coating or treatment, carrier air flow rate and schedule to prevent the chemical bonding of carbon to the tube I.D.

#### 5.2.1.4 Portable POx Cart

All reformers will be tested in the Reformer Lab before and after System Lab testing to characterize Start of Test (SOT) and End of Test (EOT) performance. Previously these characterization tests required the removal of a current reformer test from a test cart every time. Change over time cost days of lost test time and man hours to perform. A small test cart was built in order to perform characterization tests without displacing the test setup on a performance test stand in the Reformer Lab. The POx test cart can be placed next to a Reformer Performance test stand in the Reformer Lab with minimal intrusion or setup time. The characterization tests are programmed to run a set profile encompassing the range of test points used in system testing. Programmed testing allows the user to start the test and then perform other lab activities while it is running, increasing productivity, since constant monitoring is not needed.

The POx test cart contains an EDU system controller as well as air and methane flow controllers. Gas connections and power are shared with the Performance test cart in the lab to keep setup time and costs down. Connections for all reformer related hardware are accommodated making the test cart easy to operate. An insulated box on top of the test stand houses the reformer assembly.

The POx test cart also has the capability to perform calibration of the Fuel Metering Manifold used in system testing making it even more useful and versatile. Two flow meters and a fuel pressure sensor calibration need to be performed for each reformer subsystem. These calibrations are then supplied to the Systems test group along with the reformer subsystem.

## 5.2.2 Catalyst Test Improvements

## 5.2.2.1 Diesel Fuel Reforming

Improvements were made throughout the bench test system for catalyst evaluation to permit improved operations with diesel fuel. Modifications were made to the fuel vaporizer, the reactor configuration, and handling of products. Good fuel vaporization and mixing while preventing carbon formation is required in order to evaluate catalysts for reforming performance in the absence of hardware interactions. A good vaporization system will yield smaller liquid droplets for more uniform mixing with air or other reactants. Better mixing results in a more even temperature profile in the catalyst, and uniform oxygen to carbon ratio in the catalyst.

Issues with vaporization of heavy fuels include wide boiling point distributions with significant fraction of the components boiling over 325 °C. At the highest temperatures required, resins and tars can be deposited due to pyrolysis and thermal cracking, leading to increased pressure drops in the vaporizer and degrading reforming catalyst

performance. Good mixing, efficient heat exchange, turbulent flow in the vaporizer fuel tubing, and short residence times will all improve fuel vaporization.

The older version liquid vaporizer consisted of fourteen feet of stainless steel tubing coiled in metal heat media. Metal is used for increased contact and heat transfer; using zinc as the media permitted operating the vaporizer up to 420 °C, the melting point of zinc. The heat media cannot be operated in molten state due to safety considerations. The vaporizer is electrically heated with four 750 watt plastic injection molding heaters. Approximately 25 pounds of zinc was required. Temperature control is provided by a thermocouple encased in the zinc.

The new design uses a metal tube wrapped around a cartridge heater, with the external surface heavily insulated. Temperature control is provided by monitoring the outlet temperature. While stainless steel tubing was initially employed, aluminum was found to work better, leading to minimal pressure drops and low-carbon formation operation. Other materials that could be used include copper, glass-lined stainless steel, or fused-silica coated stainless steel. This configuration eliminates the upper temperature limit of the old design, reduces residence time, and prevents adverse reaction between the heat media and the tubing.

The reactor consists of a steel or Inconel tube, approximately 14" long and 1" or 0.5" inside diameter, held inside of a tube furnace. In the previous configuration, an assembly of heat retainer monolith, flame arrestor paper, catalyst or catalysts, and heat retainer were place in the longitudinal center of the tube. The heat retainers are 45 ppi 1" length zirconia-toughened alumina reticulated foams. The design resulted in too much energy entering the inlet of the reactor, especially for endothermic reactions where the catalyst temperature was required to be greater than 700 °C. This resulted in fuel decomposition and precombustion in the space in front of the catalyst assembly.

The new configuration moves the catalyst assembly close to the front end of the reactor tube, eliminating the empty space in the inlet section, leaving a small volume for open-space mixing. The diameter is reduced to 0.5 inches, increasing superficial velocity in the system. Flame arrestors are placed in front of the inlet heat retainer monolith and two between the inlet heat retainer and the catalyst.

Reformate is passed through an exhaust particulate filter to eliminate problems with carbon formation as the reformate temperature decreases to ambient. The exhaust particulate filter is about a one liter vessel in which the gas flows over an inert silica fiber particulate trap and heat exchanger. The complete system is illustrated in **Figure 5.2.2.1-1**.



Figure 5.2.2.1-1 - Bench Test Configuration for Lab-Scale Testing of Diesel Fuel Reforming Processes

# 5.3 Reformer Developments Results and Discussion

## 5.3.1 Develop Endothermic Reactor

The requirement for capable Endothermic Reformers has not diminished despite the advent of internal reforming for NG SPUs. **Figure 5.3.1-1** shows the progression of requirements from POx, to POx w/ Internal reforming, to Endothermic Reformers for both NG and diesel. During this reporting period the majority of testing was in the area of endothermic reforming of US07 Diesel fuel. While the next system under development is a Natural Gas SPU (DPS3000NG) it was necessary to focus efforts on the more difficult of the 2 applications. The burden of fuel processing efficiency must be supported by an endothermic reformer because a diesel fueled APU cannot take advantage of internal reforming. The work done during this reporting period on Gen 5T Reactor fits appropriately on the path of meeting DPS3000D requirements as it has shown to be capable to approximately15kW reformate level. Efforts are being focused to address areas where deficiencies in actual performance exist relative to the DPS3000D requirements.

		Fuel 🗸	Туре 🖵	Reqts# 👻	Unique Title 👻	EWO for Data 🗸
		СНИ			NG	US07 Diesel
		REQTS		REQTS-eet	REQTS	REQTS
		3	4	7	7C	15C
		SPU 1 pre LIR: Rev 6/11/04	3PU 18 LIR: Rev 3/1405	8PU 2 ee NGIS/DPS 3000NG(DS): Rev 8/1608	0P83000-A-NG- S1: Rev 10/12/08	0P83000-0: Rev 11/20/06
Mode •	Data 🗸	(bienić)	9	ecaled if SPU2 3/1406 col	SOFC System Requirements Warkbook_Rev16 CMM 091808.xte	DP83000- D_Reted_Simulin k_emi(01.xde
SWU	Average of Ref Pwr Celc (kW)	16.00	16.00	9.80		
	Average of Recycle - Fuel Util (Inect)	0.00	0.00	0.00	0.40	
	Average of Recycle - recycle frection (hect)				0.00	
	Average of Gross Ref EE, Bef Pwr / Fuel Pwr	0.86	0.86	0.86		
	Average of Net Ref Ell: Bef Pwr/ (Fuel+Rec Pwr)					
ide	Average of Ref Pwr Celc (KW)	4.30	1.60	1.98	2.12	2.43
	Average of Recycle - Fuel Util (Inect)	0.13	0.13	0.12	0.20	0.17
	Average of Recycle - recycle fraction (hect)				0.00	0.00
	Average of Gross Ref EE: Bef Pwr / Fuel Pwr	0.86	0.86	1.33		0.72
	Average of Net Ref EE Bef Pwr/ (Fuel+Rec Pwr)					
Min	Average of Ref Pwr Calc (KW)	4.30	1.60	1.98		
	Average of Recycle - Fuel Util (Inect)	0.13	0.13	0.12		
	Average of Recycle - recycle fraction (hect)					
	Average of Groce Ref EE Sef Pwr / Fuel Pwr	0.86	0.86	1.33		
	Average of Net Ref EE: Bef Pwr/ (Fuel+Rec Pwr)					
NOC	Average of Ref Pwr Calc (kW)	4.80	1.70	4.66	8.79	6.91
	Average of Recycle - Fuel Util (Inect)	0.50	0.50	0.80	0.48	0.60
	Average of Recycle - recycle frection (hect)				0.40	0.45
	Average of Grose Ref EX The Pwr / Fuel Pwr Average of Net Caf ER Ref Cur / Evals Car Curb	0.86	0.86	1.33		1.31
Reted	Average of Ref Per Celc II/VI	11.40	6.30	13.32	3.25	13.81
	Average of Recycle - Fuel Util (Inect)	0.50	0.50	0.60	0.60	0.60
	Average of Recycle - recycle frection (frect)				0.50	0.45
	Average of Grose Ref EE Bef Pwr / Fuel Pwr	0.86	0.86	1.33		1.31
Marra	Average of Net Ref EX Tef Pwr/ (Fuel+Rec Pwr)	13.50	6 70	44 70		
	Average of Recycle - Fuel Lttl. Bacti	0.50	0.50	14.20		
	Average of Recycle - recycle fraction (fract)	0.00	0.00			
	/warage of Groee Ref EE. Bot Pwr / Fuel Pwr /warage of Net Ref EE. Bot Pwr / (Fuel+Rec Pwr)	0.86	U.86	1.44		

#### Figure 5.3.1-1 - Requirement for Reformer Power & Efficiency - Chronology

#### 5.3.1.1 Endothermic Reforming Fundamentals

#### 5.3.1.1.1 Enthalpy Balance Gen 5T

An enthalpy analysis was computed using several reformer data sets in order to properly understand heat transfer performance and losses to ambient. **Figure 5.3.1.1.1-1** shows the enthalpy balance for the Gen 5T reactor on NG at 12 kW reformate power.

Reactor	5T 💌		
	Ref_out_Power_w_CH4_kw F2-[kW] Rp 0	Name	interpretation
Dε▼	12.05		
Average of Fuel + Air + Recycle LHV-[kW]	12.35		chemical enthalpy in reforming stream @ entrance to CV
Average of RefOut LHV w/ CH4 total-[kW] Rp,0	12.04		chemical enthalpy in reforming stream @ exit of CV
Average of del LHV Ref (Rm temp implied) - Net-[kW] Rp,0	-0.32		chemical enthalpy decr of reforming stream
Average of delH sensible RefOut (Rm temp to RefOut Temp)-[kW] Rp.0	2.21		sensible enthalpy in reforming stream
Average of Qref-[kW] (+) = heat to CV (-) = heat fr CV	1.90	Qref	enthalpy incr of reforming stream
Average of Comb In - LHV total-[kW] Cr,0	19.19		chemical enthalpy in comb/hx stream @ entrance to CV
Average of Comb Out LHV-[kW] Cp,0	0.00		chemical enthalpy in comb/hx stream @ exit of CV
Average of del LHV comb (Rm temp implied) - Net-[kW] Cr,a,RT -> Cp,0,RT	-19.19		chemical enthalpy decr of comb/hx stream
Average of del H comb Rm Temp to Rm Temp-[kW] Cr,a,RT -> Cp,0,RT	-19.22		same as above but calc via mdotCpdeIT method
Average of abs H comb in fuel + air-[kW] Cr,0	-0.93		total enthalpy in comb/hx stream @ entrance of combustor
Average of abs H Comb Out at Comb out temp (TC14)-[kW] Cp,0	-3.84		total enthalpy in comb/hx stream @ exit of combustor
Average of del H comb Rm temp to CombOut Temp-[kW] Cr,a,RT -> Cp,0,TC14	-2.91	Qloss-Combustor	enthalpy loss at comb (to amb)
Average of del H sensible Comb Out Rm temp to CombOut Temp-[kW] Cp.0 RT-TC14	-16.31		sensible enthalpy in comb/hx stream at combustor exit
Average of del H Reactor CHx in to out-[kW] Cp,1-2	-4.11	Qchx	enthalpy decr of comb/hx stream (some goes to losses; some becomes Qref)
Average of Qchx - delH sensible CHxin - CHx Out-[kW (mdotCpDelT)]	-4.14		same as above but calc via mdotCpdelT method
Average of Qloss to amb-[kW] (+) = heat to CV (-) = heat fr CV	-2.21	Qloss-Reactor	enthalpy loss at Reactor
	-5.1	Qloss (Reactor+Combustor)	total enthalpy loss by full combustor/reformer system

Figure 5.3.1.1.1-1 - Gen 5T Enthalpy Balance

Most concerning is the fact that less than 50% of the heat transfer from the combustion gas stream arrives as heat transfer into the reforming stream. This shows up as the 2.21 kW Q loss of the reactor. Also of concern is the 2.91 kW loss (out of 16.91 kW evolved by the combustor) from the combustion gas stream prior to arriving at the reactor proper. In total, 5.91 kW is loss from approximately 31kW entering the system (reformer + combustor). **Figure 5.3.1.1.1-2** shows a diagram of Gen 5T and the location and condition nomenclature used to track enthalpy changes. Follow-up work has involved better insulation methods and better understanding of heat loss paths.



Figure 5.3.1.1.1-2 - Gen 5T Enthalpy Balance Notation

# 5.3.1.1.2 Heat transfer evaluation

Testing was conducted to better understand the heat transfer performance of the reactor without the presence of reforming reactions in order to address the above enthalpy balance results. Figure 5.3.1.1.2-1 shows the locations of temperature data to determine heat exchanger characteristics. The initial test involved the insulating the reactor per Delphi's then standard lab practice of locating the reactor inside a zircar board "doghouse" which was then stuffed w/ Kaowool insulation (Alumina). **Figure 5.3.1.1.2-2** is an x-section at the reactor body of this configuration. Test results are given in the lower portion of **Figure 5.3.1.1.2-3** (lines 4, 5 and 6).



Figure 5.3.1.1.2-1 - Gen 5T Reactor + NG1 Mixer TC locations



Figure 5.3.1.1.2-2 - MG733C265 Insulation Cross Section

ON	EWD		Test Point ID	T2 Ref Path Inlet Temp [deg C]	T3 alt (ref out tube ave) [deg C]	T3 (reactor reforming path outlet) [deg C]	delta T3alt - T3 [deg C]	T6 (actual) reactor comb/ht exh inlet) [deg C]	TC16 (reactor comb/ht exh outlet) [deg C]	Effectiv eness	UA [W/C]	Heat Transfer (Comb/Hx) [W]	Heat Transfer (Ref Path) [W]	Ht xfer Ratio
1	M3730C2/0	without	ide 230	331	629	472	157	756	574	0.83	0.51	-924	87	0.09
2	MG730C270	without	NOC 5550	256	645	571	74	747	555	0.86	2.33	-1398	433	0.31
3	MG739C2/0	without	Flated 86100	234	532	505	27	614	482	0.86	4.59	-1956	647	0.33
4	MG733C285	wio insul	ide 669	309	538	379	159	674	485	0.79	0.43	-952	64	0.07
6	MG739C285	wio insul	NOC 8860	229	551	487	63	667	474	0.82	1.98	-1396	350	0.25
0	M0730C285	<b>we insu</b>	Fatel 35100	183	531	501	30	660	473	0.80	3.61	-2808	739	0.26

Figure 5.3.1.1.2-3 - Gen 5T Heat Exchange Evaluation

Noteworthy findings from this test are that UA values are surprisingly small and need to be investigated further. Most concerning is the low heat transfer ratio which indicates how much of the heat lost by the combustion gas stream is received as heat input to the reforming stream. Subsequent testing aimed to improve the insulation R value by applying a wet-lay up form fitting low thermal conductivity insulation layer directly to the reactor casing. **Figure 5.3.1.1.2-4** is a cross-section of this new configuration at the reactor body. Test results for this new "w/insulation" configuration are given in the upper portion of **Figure** 

**5.3.1.1.2-3** above (lines 1, 2, and 3). While the heat transfer ratio, UA, and heat exchange effectiveness all improved, the improvement was less than anticipated. The more prominent heat loss path is potentially via conduction to the relatively large flanges and port structures. A more rigorous thermal evaluation is now underway.



Figure 5.3.1.1.2-4 - MG733C270 Insulation Cross Section

# 5.3.1.1.3 Gen 5T Evaluation: Requirements vs. Actual

Gen 5T was evaluated under several tests. Two of the tests involve sampling of reformate from individual reforming tubes to detect differences in reforming between tubes.

# 5.3.1.1.3.1 Tube Sampling - #1

Testing was conducted with individual tube sampling while the Gen 5T reformer was fueled with JP8 surrogate fuel.

Results from this test are shown in **Figure 5.3.1.1.3.1-1** and can be summarized as follows:

- Quality reformate was made by all tubes (Quality Reformate requires <1 mol% CH4 and <0.1 mol% C2s+)</li>
- C2s+ were negligible for all tubes
- Product CH4 varied by tube and follows tube temperature



• Tube Temperature variation was evident (~ 70 deg C range)

Figure 5.3.1.1.3.1-1 - MG733C246 JP8 Gen 5T Tube-Tube Variation in Reforming Composition

The second objective of this test was to determine if this tube composition/temperature variation was a product of feed stream preparation. To test this, the FDU was "clocked" through four distinct orientations with parallel data collected at each orientation. The effect of FDU orientation versus reforming temperature is shown in **Figure 5.3.1.1.3.1-2**. It is apparent that no big effect is at work and so data was plotted by tube in **Figure 5.3.1.1.3.1-3**. This data repeats the response seen in **Figure 5.3.1.1.3.1-1** in that tube temperature varies in sequence from low to high by tube number as 2,1,3,7,6,5,4.



Figure 5.3.1.1.3.1-2 - Effect of FDU Orientation on Gen 5T Tube-Tube Variation in Reforming Composition



Figure 5.3.1.1.3.1-3 - Effect of FDU Orientation on Gen 5T Tube-Tube Variation in Reforming Composition

# 5.3.1.1.3.2 Tube Sampling - #2

A second test (MG733C261) involving individual tube sampling was conducted as a test to requirements for DPS3000D (US07 fueled APU). **Figure 5.3.1.1.3.2-1** shows the results of this testing in which eight data points (representing reformate power conditions ranging

from 2.3 to 17.3 kWt) are averaged together. A very similar pattern of temperature by tube is evident and nearly identical to that indicated in the previous test. The pattern from low to high temperature by tube is nearly identical to previous test results (2, 1, 3, 7, 6, 4, 5). **Figure 5.3.1.1.3.2-2** shows this data expanded to present data at all reformate power levels. Tubes 7 and 4 meet quality reformate requirements up to approximately14 kWt. Other tubes showing C2 inflections at much lower power levels indicates that conditions are appreciably different between tubes. A diagnostic tree was employed to better discuss and explain the variation sources and is shown in **Figure 5.3.1.1.3.2-3**. The status of the tree as more investigations were conducted will be revisited later in this report. The FDU orientation testing discussed above suggests removing box #4 as a source of the variation.



Figure 5.3.1.1.3.2-1 - MG733C261 US07 Gen 5T Tube-Tube Variation in Reforming Composition



Figure 5.3.1.1.3.2-2 - Gen 5T Tube-Tube Variation in Reforming Composition



Figure 5.3.1.1.3.2-3 - Diagnostic Tree - Reformate Composition Variation between Tubes

# 5.3.1.1.3.3 Delta P by Tube

The three viable Gen 5T reactors were subjected to individual tube delta P testing given the concern over tube-tube variation. The results shown in **Figure 5.3.1.1.3.3-1** indicate that substantial flow variation could exist between tubes based on the delta P data. This data manipulation involves collecting delta P data with room temperature air at fairly low flows. Data is regressed to create an equation for that particular tube and a flow prediction at a nominal pressure drop (as would be seen in operation) is determined.



Figure 5.3.1.1.3.3-1 - Predicted Flow Variation Due to Tube Delta P Variation

# 5.3.1.2 Produce Endothermic Reactor Prototypes

# 5.3.1.2.1 Gen 5.1T Manufacturing Development: Brazed Core effort

It was determined that cycling a wash coated reactor assembly through a nickel braze process would be detrimental to the activation and/or adhesion of the wash coat material. This finding, coupled with the required location of the wash coat material in the reactor, affected the manner in which the originally designed Gen 5T assembly was performed. The original design was assembled by TIG welding the reactor components around a prewash coated reactor core. The welding process resulted in high stress points in the reactor shell that resulted in warping and cracking during testing. The endothermic reactor was redesigned to be a brazed assembly in an effort to eliminate some of the manufacturing challenges of previous reactor builds. The redesigned components enabled the brazed Porvair core assembly to be secondarily brazed into the remaining reactor assembly. The redesign separated the flow distribution cone assembly from the reactor assembly by incorporating an additional flanged joint. The reactor assembly would be wash coated after braze followed by the cone assembly being attached using bolts. This activity is ongoing and the redesigned components are in the process of being ordered. **Figure 5.3.1.2.1-1** is a view of the redesigned assembly shown below.



# Figure 5.3.1.2.1-1 - Gen 5.1T – Brazed Core Reactor – X-Section View

Braze joints were created where the TIG welded joints cracked in the previous design. The base plate was sectioned into three separate plates to alleviate the bucking that occurred in the previous design due to over constraining caused by the single base plate.

## 5.3.1.2.2 Gen 7P: Stamped Planar Design Development

Work in this area is currently being performed under another government funded program (EMTEC) and is also being summarized in a separate technical report. Portions of the other report are included below as a means of disclosing the activity performed in this area.

## 5.3.1.2.2.1 Summary

It was believed that laser welding and brazing the thin stamped plates into reformers would be the major challenge of this project and would require the most development work. Edison Welding Institute (EWI) is a contractor in this effort and is involved in developing the high speed weld capability. However, once the initial reformer plate design was completed and run through the stamping simulation software, the results predicted that stamping Haynes 230 (H230) would become a major technical challenge. The remaining discussion goes into the detail of the work that was performed to attempt to stamp the H230 material. Ultimately, due in part to the technical challenges and the project timing combined with a project scope change within the Delphi reformer team, this development project was taken off the critical development path in favor of current concept development (i.e. Gen 5T, 5.1T and derivatives).

Delphi and Edison Welding Institute (EWI) are preparing a revised statement of work (SOW) for submission to EMTEC. The revised statement of work would involve a project timeline extension and a reallocation of the project tasks that would utilize EWI's stamping expertise with the development of the stamping process. EWI would also continue to be responsible for laser welding development. Delphi will provide design requirements and plate designs based on the collaboration with EWI's stamping experts.

# 5.3.1.2.2.2 Material Selection & Procurement

Delphi has identified the plate material required to provide robust catalyst wash coat adhesion and product durability in the planar reformer design. The material identified is a high nickel alloy called Haynes 230 (H230). This material was procured in sheet form with a thickness of 0.020 inch for use in trials and plate manufacture.

# 5.3.1.2.2.3 Created Flow field based Plate Design

The initial plate design was revised based on CFD flow modeling to provide adequate pressure drop and adequate between plate flow distribution. This resulted in significantly larger flow ports for the combustion/heat exchange gas but did not significantly alter the reforming ports. **Figure 5.3.1.2.2-1** depicts the "flow field based" design from which stamping analysis and trials were to be based.



Figure 5.3.1.2.2.3-1 - Flow Field Derived Stamped Plate Design

#### 5.3.1.2.2.4 Stamping Development

#### 5.3.1.2.2.4.1 Initial Material Stamping Trials:

A trial was performed on an existing tool that was designed to run 0.0215 inch thick 430 stainless steel in order to understand the room temperature formability of H230. The initial trials suggested the material could be formed successfully into small round parts at 25 °C. A photo of the trial parts can be seen below in **Figure 5.3.1.2.2.4-1**. It was decided to investigate the formability of specific features required by the stamped plate design based on the positive initial results of the material trials.



Figure 5.3.1.2.2.4.1-1 - H230 Stamped Samples Using Available Tooling

#### 5.3.1.2.2.4.2 Stamping Simulation/Modeling:

As part of the stamping development process, Delphi stamping experts input the part design shown above in Figure 5.3.1.2.2-1 into the simulation software, which is used to help create more robust tool designs. In order to perform this simulation, the software required longitudinal and transverse strain values (n and r values) for H230 in the intended gage thickness of 0.020 inch. An independent material-testing lab was contracted to perform the necessary testing because these strain values were not readily available through the material manufacturer. The strain values were then used in the simulation software to identify the high stress areas of the design that were at risk of tearing and/or cracking. The software predicted the highest risk of tearing was on the inside radius of the racetrack feature. For this reason, a small stamp coupon was created to validate the simulation model and gain practical stamping knowledge about H230. A sample of the simulation software output showing the thinning fraction (local reduction in material thickness as a fraction of pre-stamped material thickness) is shown in Figure 5.3.1.2.2.4.2-1. As a general rule, thinning fractions above 25% lead to fracture. High thinning (and therefore high stress) areas are clearly evident and suggest where the part would likely fail (fracture) during stamping.





## 5.3.1.2.2.4.3 Stamped Coupons:

Physical trials using a version of the same stamped features used for simulation were conducted in order to gain practical experience and correlate the modeling results. The coupon design shown in **Figure 5.3.1.2.2.4.3-1** incorporates the same high stress "racetrack" feature of the design shown in **Figure 5.3.1.2.2-1** above. The stamped coupon was formed in a two-step process that is shown in **Figure 5.3.1.2.2.4.3-1** below. The actual part made on the prototype coupon stamp tool is shown in **Figure 5.3.1.2.2.4.3-2**.

Although the part initially looked acceptable, further evaluation determined there was cracking/tearing on the inside radius of the formed racetrack feature.



Figure 5.3.1.2.2.4.3-1 - Part Geometry for Stamping Trial



Figure 5.3.1.2.2.4.3-2 - Actual Stamped Part from Trial Tooling

## 5.3.1.2.2.4.4 Coupon Testing/ Simulation Correlation:

The actual coupon stamping results correlated with the simulation software and validated the prediction model. From there, radii and other high stress features of the part design

were modified, within the software, in an effort to eliminate the predicted tearing while maintaining the function of the part design. It became clear from the simulation results that forming the necessary design features in the reformer plates with H230 at room temperature were not going to be possible. Further research by Delphi's Materials team, along with Haynes International, determined that the material would need to be stamped at a temperature of 400°C in order to eliminate the tearing that occurs from cold working the material. The simulation software has the ability to perform simulations of material at elevated forming temperatures provided the necessary material properties for the material at temperature exist. The elevated temperature strain values for H230 did not exist and none of the outside material test labs contacted could perform the necessary strain testing at 400°C. Haynes International agreed to perform the testing for us to input into the simulation software. The elevated strain values for H230 at 400°C have just recently been completed but not yet input in to the simulation software.

## 5.3.1.2.2.4.5 Stamping Plan Forward

- Continue the stamping simulation work with stamping of H230 at 400°C and present part geometry
- Based on results of above and any needed part geometry modifications, build a prototype tool and test the hot stamping process to validate the model.

## 5.3.2 Task 3.3 Develop Reforming Catalysts

The objective of this task is to deliver a catalyst that meets reformate quality, durability, turndown, and sulfur tolerance requirements. Reforming catalyst development will extend findings concerning fuel-reforming catalysts, processing, and modeling developed for natural gas, gasoline, and diesel fuel-based SOFC power systems. Catalyst process optimization will develop the steps necessary to build a catalyst in a manufacturing environment.

## 5.3.2.1 Catalyst Formulation Optimization

Catalyst formulations for natural gas and diesel fuel reforming will be optimized for resistance to both thermal degradation and sulfur inhibition using design-of-experiments methodology.

Several new formulations were identified for natural gas pre-reforming processing. These remain in the family of rare-earth element oxides discussed previously. The catalysts were tested at one of two conditions, summarized in **Figure 5.3.2.1-1**. These represent either the most rigorous system operating point, SS84 operating mode, or a more fuel-rich operating point, at a fuel to recycle ratio of 0.801. The fuel rich operating mode is used to reduce methane conversion. Compositions of simulated anode recycle and simulated natural gas used for these evaluations are also listed in **Figure 5.3.2.1-1**.

space velocity (1/hr)	~33,200 - 66,400
catalyst inlet temperature (°C)	~300 – 750
fuel / recycle ratio (vol. /vol.)	0.140 (SS84 operating mode) 0.801
P6584C-33	

feed compositions (mole fraction)					
	simulated simulated				
	natural gas	anode recycle			
H <sub>2</sub>	0	0.1059			
H <sub>2</sub> O	0	0.1992			
CO	0	0.1106			
CO₂	0	0.1296			
$N_2$	0	0.4507			
CH4	0.9300	0.0040			
$C_2H_6$	0.0499	0			
$C_3H_8$	0.0201	0			

#### Figure 5.3.2.1-1 - Processing Parameters and Feed Gas Compositions for Natural Gas Pre-Reforming Performance Testing

## 5.3.2.2 Substrate Development

The applicability and design of ceramic, metallic, and cer-met substrates will be evaluated to ensure they are robust to the application environment and meet performance needs. For example, selection of a foam, foil, or monolithic substrate will depend not only on system requirements, such as pressure drop, but also on washcoat adhesion, thermal properties, and mechanical durability. Environmental oven aging and durability testing will be performed to assess currently employed substrates and selected concepts.

#### 5.3.2.3 Washcoat Adhesion

This section addresses washcoating parameters that specifically impact washcoat adhesion. This includes slurry parameters and chemistry, as well as substrate pre- and post-coating handling. These parameters will be optimized for washcoat adhesion from a catalyst performance and durability perspective.

A work plan was developed to study methods for improved washcoat adhesion and improved coating of substrates. This plan will be executed during the first part of the next reporting period. Methods developed for testing adhesion on automotive three-way catalysts will be used. The primary experiment is to evaluate adhesion based on resistance to air-knife exposure. The first set of experiments will use an L18 DOE methodology to investigate solid percent content of slurry, particle size, and pH on adhesion. In addition, slurry rheology and particle physical parameters will be characterized.

Washcoating uniformity will be improved through the use of a semi-automated coater. Parts were designed and ordered for the use of fuel reforming substrates on existing equipment. However, due to supplier mistakes, this work has been delayed, and the parts will not be available until during the next reporting period. Coater parameters such as vacuum strength, slurry pull and push times, and substrate pre-treatment will be investigated. Washcoat uniformity will be measured by X-ray density measurements and scanning electron microscopy analysis.

#### 5.3.2.4 Reforming Process Optimization

The reforming process can significantly impact catalyst performance and durability. Flow rates, air/fuel ratios, and catalyst geometric aspect ratio can all affect how the catalyst performs, ages, and resists process upsets. This task will expand the understanding of fuel-reforming kinetics, with the goal of developing design rules for predicting catalyst performance over the lifetime of a reformer.

#### 5.3.2.4.1 Natural gas pre-reforming process development

Natural gas pre-reforming sensitivity to space velocity, process temperature, and feed / recycle ratios were investigated using the base processing conditions and feed compositions listed in **Figure 5.3.2.1-1** above. The goal of pre-reforming is to convert a hydrocarbon feed, containing non-methane hydrocarbons, into a methane-rich feed to the anode. The methane-rich feed must be essentially free of non-methane hydrocarbons. Specific requirements are listed in **Table 5.3.2.4.1-1**. The required natural gas ( $C_2H_6$  and  $C_3H_8$ ) conversions for each system operating mode are given in **Table 5.3.2.4.1-2**. The goal of these sensitivity studies is to identify which processing trends can best meet these requirements.

H2	conserve
CO	conserve
non-methane hydrocarbons	less than 0.1 %
CH4	no stated requirement
H <sub>2</sub> O/CH <sub>4</sub>	no stated requirement

# Table 5.3.2.4.1-1- Reformate Quality Requirements to be Met by Natural Gas Pre-<br/>Reforming Processes

system operating mode	fuel / recycle volume ratio	C <sub>2</sub> H <sub>6</sub> conversion target	C <sub>3</sub> H <sub>8</sub> conversion target
SS54	0.000	n/a	n/a
SS65	0.057	0.722	0.769
SS84	0.184	0.904	0.920
SS100	0.308	0.936	0.947

Table 5.3.2.4.1-2 - System operating modes, reformer feed ratios, and non-methane hydrocarbon conversion targets for natural gas pre-reforming processes. SS54 operating mode is full anode recycle, containing no non-methane hydrocarbons. C3H8 conversion target assumes that 75 % of non-methane hydrocarbons in the reformate product are C2's.

## 5.3.2.5 Catalyst Performance Testing

Catalysts will be evaluated using laboratory-scale and full-scale reforming reactors under conditions designed to meet or exceed application requirements. Adiabatic tubular or isothermal planar reactor configurations will be tested, for time frames covering initial catalyst performance through to end-of-life performance. Additional testing using well-characterized laboratory aging and rapid degradation methods, combined with failure mode analysis, will be used to understand underlying mechanisms of catalyst performance changes.

# 5.3.2.5.1 Characterization of catalysts from methane partial oxidation system-level durability testing

A series of catalysts retrieved from system-level durability testing were retrieved for performance characterization. These catalysts were tested from between 150 and 4692 hours. For comparison, a fresh catalyst was also tested. Tests consisted of natural gas partial oxidation, conducting at either 19,600/hr or 49,000/hr space velocity, using air as an oxidant, at a total O/C mole ratio of 1.30. The composition of the natural gas employed is given in **Table 5.3.2.5.1-1**. Catalysts were evaluated by adjusting catalyst center temperature over a range of about 880 to 1080 °C, usually in seven steps. Outlet H<sub>2</sub>, CO, CO<sub>2</sub>, CH<sub>4</sub>, and N<sub>2</sub> concentrations were measured using a mass spectrometer. No other species were detected. H<sub>2</sub>O was determined using an oxygen-based mass balance. Catalyst radial- and axial-center and back-face temperatures were recorded.

	average mole
	percent
methane	95.655
ethane	1. <b>99</b> 0
propane	0.220
n-butane	0.038
isobutane	0.029
isopentane	0.010
n-pentane	0.007
hexanes plus	0.006
nitrogen	1.323
carbon dioxide	0.719

## Table 5.3.2.5.1-1 - Composition of natural gas used in performance testing.

#### 5.3.2.6 Catalyst Characterization

Reforming catalysts will be characterized with the use of advanced analytical tools, such as high-resolution electron microscopy and in-situ surface and chemical analysis of reforming processes. These tools provide insight into the activity and deactivation mechanisms of the catalysts, and test results will be used to guide continued development and improvement.

The following is a summary of work performed at Oak Ridge National Laboratory as a joint project with Delphi:

Local species and temperature profiles were measured along a honeycomb monolith catalyst during methane and propane partial oxidation and validated with a microkinetics-based reactor model using the obtained data. Combined experimental and computational results have been used to elucidate underlying mechanisms.

Partial oxidation experiments were performed over an Rh-containing wash-coated 400 cpsi monolith in a horizontal quartz reactor heated by an electric furnace. Spatially Resolved Capillary Inlet Mass Spectrometer (SpaciMS) ( Choi, J.-S., Partridge, W.P., and Daw, C.S., Appl. Catal. A 239, 24, 2005) was used for species measurements and fiber-optic thermometry and K-type thermocouples were used for temperature measurements. Both species and temperature probes are minimally invasive both in terms of physical size and sample flow rate (see **Figure 5.3.2.6-1**).



#### Figure 5.3.2.6-1 - Position and relative size of capillary probes and microthermocouple used for composition and temperature profiling. Substrate is a 400 cpsi honeycomb monolith with approximately 0.04" diameter channels.

The kinetic calculations were performed using both the gas-phase kinetic (325 reactions) and surface kinetic (56 reactions) models with the Plug Flow Reactor (PFR) driver program in the CHEMKIN Collection v. 4.1 suite programs.

This approach provided useful information under realistic reformer conditions. In particular, the combined intra-channel species and temperature measurements gave a clear picture of how POX process evolves axially along the catalyst (see **Figure 5.3.2.6-2**). At the front portion of the monolith, a fraction of the CH<sub>4</sub> undergoes total oxidation consuming the available oxygen and producing mainly CO<sub>2</sub>, H<sub>2</sub>O and a significant exotherm. Some H<sub>2</sub> and CO were also observed in this "combustion" zone. Further downstream, endothermic reforming of the remaining CH<sub>4</sub> became predominant, consuming H<sub>2</sub>O, CO<sub>2</sub> and the released heat resulting in major H<sub>2</sub> and CO formation ("reforming zone"). Future work of this study will be to improve reformer catalysts, since with the understanding generated here, considering gradients in catalyst loading or other modifications in the catalyst to match more specialized applications of reformer output, from solid oxide fuel cells to diesel exhaust after treatment could be entertained.



Figure 5.3.2.6-2 - Experimental and calculated species and temperature profiles for a methane partial oxidation, using 27.6% CH4, 15.1% O2, Ar balance; 47000/hr space velocity; no external heating.

# 5.3.2.7 Chemical Reaction Modeling

Natural gas and diesel fuel reforming chemical kinetics modeling based on elementary surface-phase and gas-phase reactions will be used to predict behaviors such as tendency toward carbon formation and unwanted reaction products. The models will be tuned with data from performance testing and catalyst characterization to permit understanding of which design factors have the greatest impact on producing quality reformate. Modeling will characterize chemistry kinetics over the lifetime of the reformer and under differing reforming processing conditions and fuel qualities.

# 5.3.2.7.1 Carbon Formation Propensity Modeling

A detailed gas-phase kinetic model has been developed for single component fuels from  $C_1$  to  $C_{12}$ . The mechanism has been derived from various detailed combustion chemistry reported in the literature; these mechanisms have been validated rigorously to a certain extent. The present kinetic model has been applied for fuels consisting of aromatics resembling those in liquid fuels, i.e., gasoline, diesel fuel, or JP-8 surrogates. This report focuses on the simulations of steady-state partial oxidation of a simple gasoline surrogate that consists of 75 mole % iso-octane and 25 mole % toluene. The simulation was done

with the CHEMKIN fluid-flow model PLUG for predicting spatial variation in gas-phase species composition along the reactor and the coking propensity in the catalyst and the downstream of the catalyst. PLUG simulates the behavior of a plug-flow chemical reactor and is designed to model the non-dispersive, one-dimensional flow of a chemically reacting ideal-gas mixture in a conduit of essentially arbitrary geometry. The catalytic zone is modeled with both the gas-phase and the surface kinetics whereas the region downstream of the catalyst is modeled with the homogeneous kinetics. The carbon formation propensity is represented by the fractional carbon content (Fc) which is defined as the ratio of the number of carbon atoms in deposit precursor species to the total number of carbon atoms in hydrocarbon fraction. All species with five or more carbon atoms and cyclic or polycyclic structure are considered to be indicators of propensity to form deposits.

#### 5.3.3 Develop NG FDU

#### 5.3.3.1 NG metering device development

Work began on developing a more productive metering device for natural gas. The current device, while reasonably accurate, is heavy, large and expensive. The initial effort is to determine what devices currently exist and whether they meet the SPU requirements. The device will need to function at low system pressures, meet the flow range specified (minimum flow =0.03 g/s of NG, maximum flow = 0.15 g/s NG), and have automotive type durability. A current production NG injector used on Honda Civic for Compressed Natural Gas operation (Honda Part Number 06164-PDN-J00) was identified and ordered for evaluation. Figure 5.3.3.1-1 shows initial low pressure flow curves for several periods. These initial tests demonstrate flow metering capability using air. Additional testing is planned for 2007 with natural gas.



#### HONDA CNG injector Dynamic Flow Sweep in Air

Figure 5.3.3.1-1 - Evaluation of Fuel Injector for DPS-3000-NG System

#### 5.3.4 Develop Diesel FDU

## **5.3.4.1** Non-Contact (NCV) vs. Contact vaporizer (CV) comparative Testing The objectives of testing were as follows:

- Determine reformer start-up characteristics
- Measure O:C variation (mixing quality) across back face of catalyst
- Measure O:C variation (mixing quality) after a minimum of 300 hrs run time
- Document presence of contamination within the mixing zone

These objectives were to be carried out on each of the two vaporizer configurations as adapted to a Gen 4.2T reformer.

Atomization is further enhanced through the use of heated fuel. The contact vaporizer hardware consists of Multec 3 injector feeding into a vaporization (heat exchange) tube. For all testing, sampling of reformer product gas at the back face of the catalyst was employed as shown in **Figure 5.3.4.1-1**. A typical output of this set up is shown in **Figure 5.3.4.1-2** where the output of interest is local O/C.



Figure 5.3.4.1-1 - Reformer Product Gas Sampling Location (The Back Face of the Catalyst)



Figure 5.3.4.1-2 - O:C Ratio Sampling Set-Up

Evaluation began with start-up characteristics and results are shown in Figure 5.3.4.1-3. The graph indicates the temporary O/C excursion that occurs with the NCV setup as a result of fuel puddling on insufficiently hot chamber walls. Hydrogen production is stunted as a result. This problem is not evident with the CV setup. Figure 5.3.4.1-4 summarizes the performance of the 2 configuration at 0 hours at identical O/Cs. The CV performance is slightly better than that of the NCV. Testing also involved catalyst back face sampling for O/C variation. Data for this technique at several fueling levels is shown in Figure 5.3.4.1-5. The O/C range values for CV configuration are significantly better than the NCV value and in addition the NCV setup suffered from pre-combustion. Figure 5.3.4.1-6 shows similar data except after 300 hours of operation. The trend is the same with CV having better O/C Figure 5.3.4.1-7 shows that the O/C range advantage is evident in range values. reformate compositions and subsequently in reforming efficiency values. Figures 5.3.4.1-8 and 5.3.4.1-9 show the resulting deposit formation for each of the 2 configurations. Both configurations caused deposits with the NCV setup resulting in significant accumulations immediately upstream of the catalyst. The CV configuration on the other hand caused a lower amount of deposits and they occurred on the exterior surface of the mixing cone. This cone sees the blend of vaporized fuel and reformer air circulate around its exterior before entering the 2 tangential "mixing ports". The mixing ports are not active as the vaporized fuel enters just outside the mixer housing (i.e. external "mixing"). While the streams initially meet external to the mixer housing, we know that the homogeneity is poor until the have traveled thought the mixer geometry proper.



Figure 5.3.4.1-3 - Test Parameters

Reformer output 0 hrs @ 0.100 (g/sec) fueling						
	H2 CH4 CO Refefficienc % % % %					
NCV	18.7	0.2	22.2	74		
CV	20.2	0.12	23.6	76		

Figure 5.3.4.1-4 - Start-Up Performance NCV vs. CV Vaporizer

	Start - up		Measured OtC ratio across back face			
	Performance		of catayist at			
	Pre-Combustion	Fuel rate	0.06 (g/sec)	0.10 (g/sec)	0.20 (g/sec)	
NCV	yes	O:C ratio	n/a	1.0 - 1.26	n/a	
		range		N 26		
CV	no	O:C ratio	n/a	1.11 - 1.12	n/a	
		range		0.01		

Figure 5.3.4.1-5 - MG733C258 Reformer Output Performance – 0 hrs

	Measured O:C ratio across back face of catayist at 300 hrs					
	Fuel rate	0.06 (g/sec)	0.10 (g/sec)	0.20 (g/sec)		
NCV	O:C ratio	0.92 - 1.39	0.99 - 1.28	0.53 - 1.27		
	range	0. <b>4</b> 2	0.29	0.74		
CV	O:C ratio	1.11 - 1.13	1.16 - 1.17	1.04 - 1.07		
	range	0.02	0.01	0.03		

Figure 5.3.4.1-6 - A/F Mixing Performance - 0 hrs

		Reform 0.060 g/se	ner output ac fuel @ 3	00 hrs.	
	H2 %	CH4 CO Refefficien			
NCV	17.6	0.43	21.2	69.0	
CV	19.5	0.38	22.5	75.0	

	Reformer output 0.100 g/sec fuel @ 300 hrs.					
	12 %	CH4 %	۶0 م	Refefficiency %		
NCV	18.5	0.4	22.3	74.0		
CV	18.8	0.06	22.5	73.5		

	Reformer output 0.200 g/sec fuel @ 300 hrs.					
	H2 %	CH4 %	со %	Refefficiency %		
NCV	17.2	0.73	21.1	64.0		
CV	21.2	0.38	24.3	79.0		

Figure 5.3.4.1-7 - MG733C262 A/F Mixing Performance - 300hrs



Figure 5.3.4.1-8 - Non Contact Reformer Output - 300 hrs



Figure 5.3.4.1-9 - Non Contact Vaporizer 300hrs.

## 5.3.5 Develop NG Endothermic Reformer

No development efforts specific to NG Endothermic reforming were conducted during this period.

# 5.3.6 Develop NG POx Reformer

During this reporting period efforts were focused more on endothermic reforming and little development occurred on the Gen 4.2T POx reformer. Four (4) additional reformers were however built to support systems activities. Efforts for NG POx reforming were focused on development of the NG Cracking Reactor and catalyst.

## 5.3.6.1 Development of NG Cracking Reactor

#### 5.3.6.1.1 MG733H1 Requirements

**Figure 5.3.6.1.1-1** represents key operating points for the NG Cracking Reactor. The function of this reactor is to ingest an Anode Tail Gas (ATG) -like stream and together with a secondary reactant of Natural Gas create a product streams that is essentially free of C2s and higher HC's+. As a secondary requirement it is desirable that the reactant CH4 be left largely un-reformed. Current system approach is to have the cracking reactor downstream of the NG Endothermic Reformer such that the ATG-like reactant is the product stream of the reformer, whether or not it is actually reforming (i.e. being fueled) or only serving as a heat exchanger to conduct the stream. A key aspect of this process is that no heat is to be brought to the process other than the sensible heat present in the reactants (assumes adiabatic Cracking reactor). The catalyst for such a reactor is discussed in section 5.3.2. Here we will discuss the reactor development, specifically the modeling conducted to insure adequate mixing at reasonable pressure drop.

	DPS3000A- NG Precat operating points_rev5 .xls			
	Regta			
Data	SS54	SS65	SS84	SS100 (Rated)
Average of atg mdot [g/sec]	0.720	0.500	0.580	0.690
Average of atg temp [deg C]	700	700	700	700
Average of atg O/C [-]	1.298	1.522	2.663	3.313
A∨erage of sfmdot [g/sec]	0.000	0.036	0.096	0.160
A∨erage of sf temp [degC]	250	250	250	250
Average of react O/C all []	1.298	1.268	1.652	1.650
Average of cat SV @ stp,vold vol [hr-1]	17,321	13,938	12,943	14,281
Average of cat SV @ stp,react vol [hr-1]	13,857	11,150	10,354	11,425

Figure 5.3.6.1.1-1 - NG Cracking Reactor Reactant Specifications

## 5.3.6.1.2 Flow Modeling

As a starting point for design a reactor shape (cylindrical) size (2"OD) and substrate (Cordierite) were selected based on prior experience with reforming processes. Based on expected pressure drop characteristic of the catalyst, an allocation for the delta P of the mixing section of reactor of 0.75 kPa was selected. **Figure 5.3.6.1.2-1** shows several configurations for reactor feed mixing. Secondary fueling (CH4 for this modeling) is shown as the red stream and the blue represents the ATG stream. Mixer performance was

measured by the CH4 mass fraction at catalyst face and the delta P from Anode Inlet to catalyst (monolith) face. **Figure 5.3.6.1.2-2** shows the results for both metrics of this modeling. The "rev3 modified" design was selected as it was the best compromise between pressure drop and good mixing.



Figure 5.3.6.1.2-1 - NG Cracking Reactor Mixer Designs & Results



Figure 5.3.6.1.2-2 - NG Cracking Reactor – Distribution Modeling Results

## 5.3.6.2 System SOT Characterization and SOT/EOT test development

During this reporting period a number of POx reformers were evaluated either for the start of systems testing or as EOT. In particular the reformer that ran 4600hrs in phase I was evaluated. Unfortunately systems data collected for the reformer during testing was found to be less than 100% accurate. Additionally a full characterization was not collected for all POx reformers put into service for Phase I. As a result, in order to determine Reformer Performance at EOT (end of test) a separate EOT characterization was conducted in the reformer lab. Concurrently we were preparing a number of POx reformers for SOT (start of test) and had noticed some short term performance changes, specifically CH4 changes. A standardized test regimen was created and used to investigate "de-greening" phenomena and is shown in **Figure 5.3.6.2-1**.


SPU 1B Reformer (w/ LIR) Characterization Test Schedule



Two reformers were evaluated:

- T42-3 build date 8/17/06
- SOT characterization
- New catalyst

Two tests run on consecutive days to show "de-greening":

- T42-5 build date 10/24/05
- EOT characterization
- ~4600 hours of run time in systems lab to meet SECA deliverable

**Figure 5.3.6.2-2** shows Data for temperature through these runs. It is apparent that SOT1 data is fairly different than the SOT2 data during the initial SWU mode (same conditions run sequentially over 2 days). Similar behavior is shown in **Figure 5.3.6.2-3** for product CH4. A long thermal equilibrium time especially on power decrease events is evident and even when thermal equilibrium is reached in test it was noted that CH4 concentration would continue to drift down slowly. This is suspected to still be a temperature effect as it is theorized that inner catalyst temperature is still not in equilibrium, both radially and axially,

despite fairly stable reformate temperatures. The de-greening phenomenon has been theorized as possibly being the effect of Rh oxide converting to metal oxide. Future characterization tests will involve 2 passes of the schedule shown in **Figure 5.3.6.2-1**. The 1<sup>st</sup> pass will be for "de-greening" while the 2<sup>nd</sup> will be to collect viable data.



Figure 5.3.6.2-2 - SOT/EOT Characterization Test Schedule



Figure 5.3.6.2-3 - SOT/EOT Characterization Test Schedule

Catalysts from selected EOT reformers are being evaluated separately from the POx Reactor and are discussed in the Catalyst section of this report.

#### 5.3.7 Develop Diesel Endothermic Reformer

#### 5.3.7.1 Test to DPS3000D requirements

The objective of the test was to evaluate the reformer to current DPS3000D requirements. Reformer configuration was a Gen 5T Reactor with NG1 mixer and internally mixed single tube contact vaporizer with ~500 hrs of operation logged. Much of the data from this specific test was discussed earlier in this report as it also involved individual reformer tube sampling. Additionally flow rates at rated and NOC power modes, scaled up and down, were run to see the limits of reformer performance. **Figure 5.3.7.1-1** shows results for some key measures. While full requirements for DPS3000D are still being formulize a it is anticipated that reformate quality (driven by stack requirement) and efficiency will be in line with current DPS3000NG requirements. Reforming Efficiencies near 130% and C2s+ at less than 0.1 mol% are expected. Given this, this reformer configuration did not meet these levels. Given the rough service that this particular sample has undergone and as yet unresolved concerns over heat loss management, our intentions are to re-test with a fresh reactor as soon as the opportunity presents itself.





#### 5.3.7.2 Durability w/ CV (contact vaporizer) - Carbon Deposits

The objective of this test was to evaluate a revised mixing strategy referred to as 'externally mixed' on a full reactor and log additional test time w/ the Gen 5T reactor on US07 diesel fuel. The earlier test used a contact vaporizer / mixing strategy that required periodic cleaning of the CV tube that inadvertently also had the benefit of removing potential carbon

deposits in the FDU & reactor inlet. The intent of this test is also to compare the effect of recycle as a reactant on the carbon formation that typically occurs in a FDU when run with a POx reactor. **Figures 5.3.7.2-1** and **5.3.7.2-2** show the difference in carbon formation that occurs when recycle is added as a reactant. In all other respects the performance of the reformer was similar to that in the previous test, indicating that the "external mixing" approach appears to be on par with "internal mixing".



Figure 5.3.7.2-1 - EOT NG1 Mixing Cone



# Pox Test shows significantly more carbon deposition

Figure 5.3.7.2-2 - EOT Mixer Housing

#### 5.3.8 Develop Diesel POx Reformer

No activity this reporting period.

# 5.4 Reformer Developments Conclusion

#### 5.4.1 Reformer Subsystem

The contact vaporizer (CV) approach to processing diesel fuel continues to show promise. Sufficient advantage was shown that work on the NCV approach will cease.

Recycle based reforming in addition to boosting Gross reforming efficiencies has a clear and demonstrated benefit in reducing FDU (reformer inlet) coking.

Gen 5T reforming of US07 has shown significantly more C2 slip than with JP8. The causes of this are being investigated.

Gen 5T reactor + Combustor configuration has thermal losses that have been identified and are now being addressed.

#### 5.4.2 Catalyst

Performance characterization of catalyst taken from system level durability show that the catalyst had more than sufficient activity through out all of the testing to meet reformate quality requirements even though significant loss of activity was observed. Detailed physical characterization will be required to understand the underlying mechanisms of catalyst deactivation. While a combination of catalyst formulation and processing

conditions can be found to meet natural gas pre-reforming product requirements, further optimization and durability testing are needed to most efficiently meet these requirements. FeCrAlloy cer-met substrates could be the most useful for endothermic reforming reactions. However, their durability and effectiveness must be improved by increasing cell count and improving gas-washcoat interactions. Diesel fuel reforming can now be accomplished in the absence of carbon-derived catalyst deactivation. However, carbon deposition at lower operating temperatures continues to be a problem that needs to be resolved. While all system operating points can be met while meeting requirements for diesel fuel endothermic reforming, operating at too low of temperatures or operating with low quality mixing will result in rapid degradation of catalyst performance; otherwise, durability and performance similar to that obtained for methane partial oxidation can be expected.

# 6.0 TASK 4: DEVELOPMENT OF BALANCE OF PLANT (BOP) COMPONENTS

#### 6.1 Development of BOP Components Executive Summary

Additional durability hours were accumulated on existing PAMs installed in systems. A new six valve PAM assembly was designed, fabricated and assembled along with new modular control valve blocks.

Cast Inconel Integrated Component Manifolds were received from the supplier and process development brazing the castings has been kicked-off.

Recycle pump robustness has been improved by re-orienting the pump, changing pump bearings, and modifying a recycle cooler.

Igniter failure analysis continued. An igniter test was developed for screening out potential igniter solutions.

Significant progress was made in the area of desulfurization. Prototype Natural Gas desulfurizers were specified, purchased, and placed on test. This completed the Phase II Milestone: running a system on line Natural Gas. A designed experiment/robust engineering test of sorbents for a Hot Reformate desulfurizer continued, and resulted in sorbent selection for a non-regenerating desulfurizer.

#### 6.2 Development of BOP Components Experimental Approach

#### 6.2.1 Process Air Subsystem

A new design air flow meter called the Small Bore Air Meter was created. It is shown in **Figure 6.2.1-1**. This meter was based on the latest generation, Delphi production air meter. It was assembled on existing production equipment. Because of this, it is much more reliable, has much greater part-to-part repeatability and has zero leakage to atmosphere.



Figure 6.2.1-1 Small Bore Air Meter

A new PAM manifold, shown in **Figure 6.2.1-2**, has been designed. This configuration increases the number of air outlet ports from 4 to 6 in order to support changes to the SOFC system architecture. This PAM is referred to as the 6-Pack PAM. It also incorporates the new Small Bore Air Meter mentioned above.



Figure 6.2.1-2 6-Pack PAM

Additionally, further re-design of the PAM subsystem was done to separate the airflow control and distribution function of a central manifold design to individual Airflow Control Blocks as shown in **Figure 6.2.1-3**. These airflow control blocks each contain one air control valve and one Small Bore Air Meter. They are intended to be located anywhere within the system piping and allow for greater packaging flexibility.



Figure 6.2.1-3 Airflow Control Block

Along with the manifold redesigns, an effort was undertaken to identify a commercial source for the PAM air pump. The component requirements document, CRD, was revisited and modified. A commercially available air pump was then purchased and tested.

Test stand equipment specifications were written for a PAM environmental durability test stand. Supplier sourcing was initiated and quotations were received in late December. The purchase and delivery of this test stand is expected to be completed in the first half of 2007. The stand will be able to test multiple PAM systems over a full range of ambient inlet air temperature and humidity conditions.

# 6.2.2 Integrated Component Manifold

Cast Integrated Component Manifolds were received in December 2006. The manifold was split into an upper and lower casting that will be brazed together with a mid-plate. The first two upper and lower castings look exceptionally nice for Inconel 625 castings (see **Figure 6.2.2-1**).



Figure 6.2.2-1 Cast ICMs

When these parts were received it was noticed that there may still be some dimensional issues. The parts are currently being checked/inspected in order to determine whether or not they will be usable and to determine what changes need to be made in the mold cavities to avoid this shrinkage/porosity in future castings.

The current ICM design, made via conventional prototype manufacturing processes, continued to perform without incident increasing our confidence in its durability. During some system teardowns however, machining chips were identified inside passages of the manifold. A post machining cleaning process is currently being developed to eliminate this in the future.

#### 6.2.3 Igniters

Early life igniter failures have been a recurring issue in our system. The igniter is used to ignite the Anode Tail Gas prior to exiting the system (see **Figure 6.2.3-1**).

The heat release from this combustion is used to indirectly heat incoming Cathode air.



Figure 6.2.3-1 Current Igniter

An igniter durability test was developed and consists of the following:

- Measure and record resistance of igniters at room temperature using a calibrated multi-meter;
- Place igniters in a calibrated thermal chamber at the desired test temperature;
- Audit at daily intervals and recording resistance;
- Plot the resistance change over time and increased temperatures.

#### 6.2.4 Recycle System

#### 6.2.4.1 Pump/Controller

The basic design of the NE-2 Recycle Pump and Controller has not changed since the last report. A new sourcing study was started to identify additional commercial sources for a pump and controller based on revised system requirements.

#### 6.2.4.2 Recycle Cooler

As reported previously, the recycle coolers were over cooling the anode tail gas during system tests. A predictive model was used to determine the required changes in thermal performance needed to increase the temperature of the Tail gas at the heat exchanger exit. A method to re-work exiting hardware was developed. A single piece was modified and characterized on the heat exchanger test stand. During this reporting period the modified piece was installed in a system and tested for final verification. **Figure 6.2.4.2-1** shows the original recycle cooler and the modifications in-process.



Figure 6.2.4.2-1 Recycle Coolers Pre- and Post-Modification

# 6.2.5 Desulfurization

Delphi has developed two different fuel desulfurization strategies for a gaseous fuel such as natural gas and for a liquid fuel such as diesel fuel.

# 6.2.5.1 Natural Gas Desulfurization

#### 6.2.5.1.1 Testing of prototype natural gas desulfurizer

Prototype NG desulfurizers were purchased from TDA Research in order to meet one of the phase II milestones: demonstrate the operation of SOFC using NG for 1500 hours. These desulfurizers are single-bed devices containing two sorbents, namely SulfaTrap-R3, and R5. The prototype desulfurizers were installed and connected in-line with System Set 5. Performance testing of the system with line NG and the prototype desulfurizers was conducted. Sulfur analysis of the cleaned reformate outlet was measured daily using a Jerome 631-X H2S analyzer.

Using this test fixture we were able to flow synthesized reformate containing  $H_2S$ . We are able to control operation temperature within the desired ranges and can measure flow rates and composition of the gas feed stream. Another Jerome 631-X from Arizona Instruments was used to measure  $H_2S$  in the ppbv range.

Delphi used 0.5 to 1.5 standard liters per minute (slpm) gas flow rate through the test fixture and from 1.8 grams to 3.0 grams of sorbent material. This resulted in test space velocities of 5,000 hr<sup>-1</sup> to 15,000 hr<sup>-1</sup>. These space velocities are in the range expected to be run in single use non-regenerating beds in systems operating at 3.5 kW.

#### 6.3 Development of BOP Components Results and Discussion

#### 6.3.1 Process Air Subsystem

The Process Air Module (PAM) motors from Stationary Power Units (SPU) sets 3 and 4 which failed during previous testing (see SECA Tech 8 for details) were rebuilt with new original design bearings. The motors were operated and vibration measurements were taken. The levels measured were acceptably low.

Since the last report 3 PAMs were put into service. One PAM operated for approximately 1450 hours, the second operated for about 400 hours and the third for 275 hours. During this time a different PAM that had run for about 650 hours experienced a failed motor bearing. The bearing was replaced, vibration measurements were taken and the PAM readied for service.

A Computational Fluid Dynamics (CFD) analysis of the newly designed Airflow Control Blocks was started. Problems in the flow model have prohibited receiving meaningful data from this analysis. The model is expected to be working early in 2007. In addition, stereo lithographic flow control blocks were fabricated and an Airflow Control Block was assembled. Testing of this will also occur early in 2007.

#### 6.3.2 Integrated Component Manifold

Brazing of cast Inconel 625 will be the next challenge in ICM manufacturing. Unusable scrap from the casting house was obtained for braze cycle development. The effect of casting porosity on the braze-joint strength is currently unknown. Some of the wall thicknesses have also increased which may cause the braze cycle time to increase in-order to allow the materials to come to temperature at a uniform rate. The Gas Phase Combustor, GPC, which is currently removable, will now be permanently located inside the cast and brazed ICM. Brazing the combustor fuel tubes internally will be an additional challenge, due to the number of them.

#### 6.3.3 Recycle System

#### 6.3.3.1 Pump/Controller

One NE-2 recycle pump with the new mounting configuration was installed in an operating SOFC system. This pump ran for approximately 360 hours without any failure.

#### 6.3.4 Desulfurization

#### 6.3.4.1 Natural Gas

The Set 5 SOFC system, complete with the NG desulfurizer, was tested for up to 1423 hours. The power degradation performance of the NG-fueled SOFC seems to be comparable to those reported for Set 2 and 3 systems, while powered by the bottled methane. The detailed test data will be reported in the system development section. Separately, the desulfurization performance will be discussed below.

It was important to first understand the sulfur species and the sulfur concentration present in local line NG, in order to establish the requirements for the prototype NG desulfurizer. The pipeline NG at Delphi's Metro Park test facility is supplied by RGE (Rochester Gas & Electric Corp.). RGE also has a local analytical laboratory and assisted with initial sulfur characterization in gas samples drawn from wall source inside the lab at Metro Park. Using the weekly collected samples, measurements of six samples showed that the average sulfur was 4.07 ppmv with a standard deviation of 0.20 ppmv. Ten sulfur species were present in the line NG. The main impurities were  $H_2S$  and COS and the predominant odorant was TBM (t-butyl mercaptan).

A recent characterization of the line NG (made using Delphi's newly purchased Gas Chromatagraph equipped with a Sulfur Chemi-luminescent Detector, GC/SCD) showed that the sulfur concentration was 3.49 ppmv (see **Table 6.3.4.1-1**).

Suldur species	ppmv
Hydrogen sulfide	0.355
Carbonyl sulfide	0.065
Methyl Mercaptan	0.125
Ethyl Mercaptan	0.048
Dimethyl sulfide	0.047
Iso-Propyl Mercaptan	0.571
T-Butyl Mercaptan	2.163
N-Propyl Mercaptan	0.083
Butyl Mercaptan	0.000
Thiophene	0.037
Total sulfur	3.494

 Table 6.3.4.1-1
 Sulfur Species in Line NG

This sulfur level, although lower than the earlier data measured at RGE, is still within the 3 sigma range. The predominant odorant species is t-butyl mercaptan of 2.16 ppm. The GC/SCD chromatogram of the line NG feed displaying the sulfur component distribution is shown in **Figure 6.3.4.1-1**.



Figure 6.3.4.1-1 Sulfur Component Distribution in the Natural Gas Feed

# 6.4 Development of BOP Components Conclusions

# 6.4.1 Process Air Subsystem

Since the last report 3 PAMs have operated for over 2100 hours and as of this report they are still operating. A fourth PAM experienced a catastrophic front motor bearing failure and was repaired and readied for service.

The new design Small Bore Air Meter has greatly improved part-to-part variation over the previous design. This will eliminate the need for individual calibrations for every air meter. With the new meters, one calibration curve can be used for all parts. Also, these new meters are completely sealed and have zero leakage to atmosphere.

The new Airflow Control Block design will improve the layout of components in the Stationary Power Unit system. The modular design eliminates the need for reserving a large fixed space for the air distribution manifold, and frees up that space for other components.

The Ametek (model 117417-01) regenerative blower meets the revised system requirements. This blower is commercially available, and will be used in a future system build.

#### 6.4.2 Integrated Component Manifold

Developing the process of casting Inconel 625 will greatly reduce the manufacturing and material cost of producing the current hot zone components. The current manufacturing processes discard too much material and this material waste is extremely undesirable considering Inconel's high cost. Delphi will be able to eliminate much of the wasted material in many of the hot zone components while also simplifying the manufacturing process.

#### 6.4.3 Igniters

Finding a robust solution for igniters is very important to the system. Every time an igniter fails a significant portion of the system needs to be torn down for replacement. These failures make system thermal cycling impossible and igniter failures must be resolved before the next generation system is manufactured. Development with igniter suppliers is on-going as well as investigating alternative locations for igniter placement in future generation systems.

#### 6.4.4 Recycle System

#### 6.4.4.1 Pump/Controller

The NE-2 recycle pump installed in an operating system accumulated over 360 hours of run time since the last report.

Delphi is awaiting the shipment of 5 new NE-2 recycle pumps with the new bearings. These pumps will be installed into operating systems to determine if the new bearing eliminates the water related failures.

#### 6.4.4.2 Recycle Cooler

Anode tail gas temperatures were within the desired range during system tests with the modified recycle cooler. This verified the modifications to the recycle cooler. Additional recycle coolers will now be modified and newly built recycle coolers will incorporate the reduced fin content/design change.

#### 6.4.5 Desulfurization

#### 6.4.5.1 Natural Gas

The NG testing of the Set 5 system with an in-line desulfurizer clearly demonstrated the success of desulfurizer function. This system delivered power degradation performance at least comparable to that of Set 2 & 3 systems when powered using sulfur-free bottled methane.

# 7.0 TASK 5: PROJECT MANAGEMENT

Delphi and its subcontractors have managed the project including integration of the work among task leaders and consultants to provide consistency and management best practices. Delphi held multiple meetings with the Department of Energy's COR. Subtasks included:

- Prepared and delivered presentations
- Management of work breakdown structure and statement of work
- Prepared required reports and revisions to reports per DOE COR comments
- Prepared and delivered monthly project highlights report
- Project costing and budgeting activities

# 8.0 TASK 6: SYSTEM MODELING & CELL EVALUATION FOR HIGH EFFICIENCY COAL-BASED SOLID OXIDE FUEL CELL GAS TURBINE HYBRID SYSTEM

### 8.1 Coal-Based SOFC Gas Turbine Hybride System Executive Summary

United Technologies Research Center (UTRC) has initiated an effort since August 2006, as part of Delphi's ongoing SECA project, to investigate solid oxide fuel cell (SOFC) system configurations utilizing gasified coal and evaluate SOFC cells that are provided by Delphi. The objective of the system study is to formulate highly efficient SOFC-based hybrid system configurations and establish an optimized conceptual design of SOFC stack and stack-module. The objective of the cell evaluation effort is to determine the compatibility between Delphi's cell and UTRC's stack design and identify gaps between the current Delphi cell technology and the cell stack requirements for the MW-scale Hybrid Power System.

Effort during the past several months have been focused on understanding power system requirements and coal-based fuel composition, conducting initial assessment of various power generator configurations and CO<sub>2</sub> capture options, conceptual design of stack module for MW-scale power plants, and Delphi cell performance evaluation. Detailed technical progress is reported in the following sections.

#### 8.2 Modeling of MW-scale IGFC Hybrid Power Systems

#### 8.2.1 Task Overview

The modeling effort is comprised of several sub-tasks that are geared towards establishing optimal design concepts and parameter selection for high-efficiency coal-gas fueled solid oxide fuel cell power plants with  $CO_2$  capture. The global objectives of the modeling task are as follows:

- 1. Formulate system design requirements (plant size, coal-gas composition, etc.)
- Develop conceptual designs for a 100 MW utility-scale and a 1 MW demonstrationscale integrated gasification fuel cell (IGFC) hybrid power plant which incorporate CO<sub>2</sub> capture schemes.
- 3. Evaluate at least two stack and stack-module conceptual designs that are generated under Task 6.2 and understand the impact of cell footprint and stack module arrangement on system efficiency and performance.
- 4. Employ the IGFC system-level models to define preliminary stack goals with input from Delphi on cell and stack performance.

#### 8.2.2 Further Work

The remainder of the systems analysis effort will focus on the following:

- 1. Wrap-up of SOFC stack model calibration against both Delphi spreadsheet model and short stack experimental data on simulated coal-gas.
- 2. Parameter optimization of down-selected system designs for both demonstrationand utility-scale systems.
- 3. Completion of Task 3 relating to the evaluation of stack and stack-module concepts as previously described Section 8.2.1.

## 8.3 Conceptual Design of SOFC Cell Stack and Stack-Module

#### 8.3.1 Task Overview

The principal goals of this task are to generate and evaluate several SOFC stack and stack module (array of stacks) concepts that are intended for multi-megawatt stationary power generation. The main areas of focus are the impact of cell size and manifold design, but other aspects will be included such as interconnects. The concepts have been divided in to categories to help identify the key issues associated with solid oxide fuel cells at this scale. This decomposition has evolved as the project has progressed and greater knowledge of the problems has been acquired. Initially the main classification of concepts was for small cells (<120cm<sup>2</sup>) and large cells (>350cm<sup>2</sup>). This has subsequently been amended to use 400cm<sup>2</sup> as the baseline cell size in part due to the large number of cells required for multi megawatt production and also due to the encouraging preliminary results that Delphi has had in scaling up the cell size they are capable of manufacturing. This task will still incorporate the effects of cell size but it won't be used as a classification of concepts. Currently the main classification is for pressurized and unpressurized designs.

### 8.3.2 Further Work

Near future emphasis of the effort is on CFD modeling to determine more detailed information on the size requirements for adequate flow distribution at acceptable pressure drops. Updating / calibration of the electrochemistry models to function with Delphi cells and coal gas compositions are underway and this will be incorporated into the CFD software by way of user defined functions. The combination of CFD with electrochemistry will enable trade studies to be performed on the effects of cell areas and aspect ratios. It will also provide a means of comparing flow schemes and their impact on system efficiency. Once a better understanding of these parameters is achieved it will be used to down select the concepts and produce more detailed information for the candidate designs.

## 8.4 Cell Evaluation and Benchmarking

## 8.4.1 Task Overview

The objective of this task is to evaluate Delphi's cell technology for its compatibility with the current UTRC stack components and design and feasibility for the Hybrid Power System. Specifically, UTRC examines key cell performance including power density, long term stability, thermal cycling capability, mechanical integrity, and chemical compatibility with other stack components. Delphi provides UTRC with cells of two different geometries and two different current-collecting configurations for performance characterization. The cells are fabricated with Delphi's current material selection and fabrication processes. UTRC, with participation from Delphi, has developed the required test protocols for the performance testing of Delphi's cells and initiated electrochemical tests using one-cell radial module and one-cell rectangular stack. Test results are discussed in detail in the following sections.

# 8.4.2 Test Configurations

In the radial module configuration, the anode interconnect/cell/cathode interconnect is held concentrically between two loading metal plates. It is essentially a seal less design with unused fuel exhaust at the periphery of the module. The radial module test is being used to evaluate cell, interconnect, protective coating material, and interfacial material performance, as well as chemical compatibility among the various materials. In the 1-cell stack configuration, the anode interconnect/cell/cathode interconnect is held between two loading metal plates with gases in cross-flow (or other flow configuration) via internal manifolding. A non-contaminating compliant seal is used. 1-cell stack test is used to study the effect of the fuel composition and utilization on cell performance, evaluate flow-field configuration and sealing materials performance.

# 8.4.3 Test Procedures

Test articles are assembled following standard procedures established at UTRC. For 1-cell stack tests, leaking check is performed prior to heat up. For both the radial module and 1-cell stack configurations, heating up is typically carried out at 1°C/min. Leak check is performed again at the operating temperature prior to the electrochemical testing.

For all the tests, an initial polarization curve (V-I curve) is collected once the open circuit voltage of the module is stabilized. V-I curve is normally measured at fixed fuel and air flow rate, by scanning the current while monitoring the voltage. The cell current is varied from zero to an upper current limit at a rate of 20 mA/sec with 1 data points collected every 2 seconds. The upper current limit depends on the cell performance and the operating temperature. The current where the cell potential reaches 0.55 volts is typically used as the upper current limit for the V-I measurement. Alternatively, the V-I curve can be collected at a fixed fuel utilization, where the fuel flow rate is varied at different applied currents. The typical fuel composition is a  $H_2$ - $N_2$  mixture, that is used to simulate a reformate gas stream.

For steady-state performance evaluation, a constant current is applied and the voltage output is recorded as a function of time. The test is conducted with a fixed fuel flow rate, corresponding to constant fuel utilization. A nominal fuel utilization of 20-30% is used for the radial module tests while a fuel utilization of 50% or higher is utilized for the 1-cell stack tests. One test segment typically lasts for a few hundred hours until a linear voltage behavior is observed. Performance degradation is defined as a percentage of voltage drop per 1000 hours.

Thermal cycling performance is examined after the cell performance has quasi-stabilized. A thermal cycle is initiated by removing the current load and ramping the furnace temperature down at 3°C/min to 500°C followed by a natural cooling period until the temperature reaches 80°C. The test article is heated up at 3°C/min back to the operating temperature. After reaching the operating temperature, the cell is held at open circuit for 2 hours, before a constant current is applied. The duration of the constant current is normally 10 hours between successive thermal cycles. Cell / stack power output at the end of each 10-hour steady state operation is used to quantify the thermal cycling performance over multiple thermal cycles.

#### 8.4.4 Delphi Cell Performance Evaluation

#### 8.4.4.1 Cell Polarization Characteristics

Three radial modules (RM-D-1, RM-D-2 and RM-D-3) and one 1-cell stack (SM-D-1) have been assembled and are currently being tested. The voltage current characteristics (V-I curves) of the four tests measured at 700°C and 750°C are shown in. For comparison, the V-I curve of a Delphi's 30-cell stack tested at 750°C is also shown in the same plot. All four tests have demonstrated consistent polarization performance. The cell performances are comparable to that of the Delphi's 30-cell stack at lower current density. At higher current density, the cells tested at UTRC have exhibited a slightly lower voltage compared with that of the Delphi's 30-cell stack, likely due to variation in local temperature distribution as well as ohmic resistance. Overall, the cells performed well at 750°C, exhibiting a power density greater than 0.45W/cm<sup>2</sup> at 0.75V.

#### 8.4.4.2 Steady State Performance under Constant Current Load

The module voltage as a function of time at constant current load for the radial module (RM-D-1). The module voltage showed an initial degradation rate of <1%/1000hrs when

tested at 700C with a constant current load of 0.3A/cm2. When the operating temperature was raised to 750C, the module demonstrated excellent stability with a constant current load of 0.46A/cm2. No change in module voltage was observed over 360 hours.

The test was carried out at 750°C with a constant current load of 0.46A/cm<sup>2</sup>. The module voltage decayed rapidly initially with 2% degradation over the first 50 hours. However, the degradation rate decreased over time and stabilized after ~150 hours with a rate of 3.7 %/1000hrs, of which half was due to the IR\_free voltage degradation and half to increase in ohmic resistance. Degradation in IR\_free voltage reflects reduction in cell's catalytic activity while increase in ohmic resistance implies either deteriorating of electric contract or formation of less conductive reaction product. Post test analysis will be carried to facilitate understanding of the observed degradation.

A one cell stack test was carried out with the square module test configuration. The 1-cell stack was initially operated at a constant current load of 0.47A/cm<sup>2</sup> at 750C using 50-50 H2-N2 as fuel and 56% fuel utilization. A high performance degradation was observed and the performance degradation was mainly caused by IR\_free voltage drop. This test was terminated due to the observed high performance degradation and post analysis is currently under way to study the causes of the observed high performance degradation. High degradation rate was also observed in the third radial module with slightly different material configurations. The cell was tested at several different temperatures and different current density. It was noticed that the module voltage degradation rate decreased with decreasing current density when the temperature was held constant. At a constant load, the module voltage degradation came from the drop of the module IR\_free voltage and the increase of the module ohmic resistance.

# 8.4.4.3 Cell Performance under Thermal Cycling

For the radial module (RM-D-1) using UTRC's paste as the cathode bonding materials, the module voltage showed a drop during the first thermal cycle and the module voltage drop slows down with further thermal cycling. Current interruption results show that the voltage drop during thermal cycling is due to ohmic resistance increase. The module IR\_free voltage, which reflects the cell intrinsic performance, is essentially unchanged during thermal cycling.

The radial module (RM-D-2) with Delphi's paste has demonstrated excellent thermal cycling capability. The module voltage showed no degradation after 10 thermal cycles.

#### 8.4.5 Future Work

The electrochemical testing will continue through rest of the project. The first three radial module tests and the 1-cell stack (square module, SM-D-1) have all been terminated and post test analysis is being conducted to understand chemical compatibility between the various materials employed in the tests and the causes for the difference in the observed

performance stability under different testing conditions. New radial modules will be constructed to duplicate RM-D-1 to examine the performance repeatability of Delphi's cell.

#### 8.5 Program Management

The project was initially kicked off via a telephone conference between the Delphi team and the UTRC team. Subsequently, a face-to-face kick off meeting was held at the UTRC facility in East Hartford, Connecticut on August 9<sup>th</sup>, 2006. During the kick off meetings, overviews of UTC, UTC Power, UTRC, as well as SOFC stack and system development effort at UTRC were presented.

During the course of the project, bi-weekly telephone conferences have been held for status update and results reporting. In September, Ellen Sun, John Yamanis and Robert Braun attended the 6<sup>th</sup> SECA Annual Workshop organized by DOE and held in Philadelphia, PA. In October, Ellen Sun, John Yamanis, Justin Hawkes, and Fanglin Chen from UTRC visited the Delphi cell fabrication facility in Flint, MI and the stack development / testing facility in Rochester, NY. Discussions were held during the visit on technical approaches to develop cells and stacks with large footprints.

It is expected that the current effort will be finished on schedule and on budget by the end of February, 2007. A final report will be prepared and delivered at the end of this effort.

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# 10.0 LIST OF ACRONYMS AND ABBREVIATIONS

A	
AE	Application Enclosure
AIM	Application Interface Module
APU	Auxiliary Power Unit
ASR	Area Specific Resistance
Atm	Atmosphere
AT%	Atomic Weight %
В	
BOP	Balance Of Plant
BEC	Bussed Electrical Center
С	
CAN	Controller Area Network
CARB	California Air Resouces Board
ССР	CAN Communication Protocol
CFD	Computational Fluid Dynamics
CH <sub>4</sub>	Methane
CHEX	Cathode Heat Exchanger
CNG	Compressed Natural Gas
СРОх	Catalytic Partial Oxidation
Cr	Chromium
CTE	Coefficient of Thermal Expansion
D	
DC	Direct Current
Demo	Demonstration
DFMEA	Design Failure Mode and Effects Analysis
DMA	Dynamic Mechanical Analysis

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DOE	Department of Energy
E	
ECU	Engine Control Units
EDU	Engineering Development Unit
E-Stop	Emergency Stop
F	
FCPP	Fuel Cell Power Plant
FDU	Feedstream Delivery Unit
FEA	Finite Element Analysis
FMEA	Failure Mode and Effects Analysis
FMM	Fuel Metering Manifold
G	
GC	Gas Chromatography
Gen	Generation
GHSV	Gas Hourly Space Velocity
GPC	Gas Phase Combustor
GPCM	Gas Phase Combustor Module
Н	
HEX	Heat Exchanger
HIL	Hardware in the Loop
HSC	High-Shear Compaction
HZM	Hot Zone Module
I	
ICM	Integrated Component Manifold
I/O	Input / Output
IRU	Interconnect Resistance Unit
L	

|--|

LCD	Liquid Crystal Display
LED	Light Emitting Diodes
LHV	Lower Heating Values
LIR	Light Internal Reforming
LNF	Lanthanum (L <sub>a</sub> ) Nickle (N <sub>i</sub> ) Ferrite (F <sub>e</sub> )
LSCF	Lanthanum (L <sub>a</sub> ) Strontium (S <sub>r</sub> ) Cobalt (C <sub>o</sub> ) Ferrite (F <sub>e</sub> )
М	
MAF	Mass Air Flow
MRF	Mass Recycle Flow
MSE	Measurement System Evaluation
Ν	
NETL	National Energy Technology Laboratory
NG	Natural Gas
NOC	Nominal Operating Condition
1100	
0	
0 0P	Open Porosity
0 0P 0/C	Open Porosity Oxygen to Carbon
0 0P 0/C P	Open Porosity Oxygen to Carbon
0 0P 0/C P PAM	Open Porosity Oxygen to Carbon Process Air Module
O OP O/C P PAM PFD	Open Porosity Oxygen to Carbon Process Air Module Process Flow Diagram
O OP O/C P PAM PFD PNNL	Open Porosity Oxygen to Carbon Process Air Module Process Flow Diagram Pacific Northwest National Laboratory
O OP O/C P PAM PFD PNNL PO	Open Porosity Oxygen to Carbon Process Air Module Process Flow Diagram Pacific Northwest National Laboratory Pre-Oxidized
O OP O/C P PAM PFD PNNL PO POx	Open Porosity         Oxygen to Carbon         Process Air Module         Process Flow Diagram         Pacific Northwest National Laboratory         Pre-Oxidized         Partial Oxidation
O OP O/C P PAM PFD PNNL PO POx PSM	Open Porosity         Oxygen to Carbon         Process Air Module         Process Flow Diagram         Pacific Northwest National Laboratory         Pre-Oxidized         Partial Oxidation         Plant Support Module
O OP O/C P PAM PFD PNNL PO POx PSM R	Open Porosity         Oxygen to Carbon         Process Air Module         Process Flow Diagram         Pacific Northwest National Laboratory         Pre-Oxidized         Partial Oxidation         Plant Support Module
O OP O/C P PAM PFD PNNL PO POx PSM R R	Open Porosity         Oxygen to Carbon         Process Air Module         Process Flow Diagram         Pacific Northwest National Laboratory         Pre-Oxidized         Partial Oxidation         Plant Support Module         Rhodium

RTI	Ragen Technologies, Inc.
S	
SECA	Solid State Energy Conversion Alliance
SEM	Scanning Electron Microscope
S&D	Safety & Diagnostics
SOFC	Solid Oxide Fuel Cell
SPU	Stationary Power Unit
SVM	System Verification Manager
Т	
TGR	Tail Gas Recycle
V	
Vol %	Volume %
Х	
XRD	X-Ray Diffraction
Z	
ZTA	Zirconia Toughened Alumina