

REPORT DOCUMENTATION PAGE			Form Approved OMB NO. 0704-0188		
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1. REPORT DATE (DD-MM-YYYY) 09-04-2008		2. REPORT TYPE Final Report		3. DATES COVERED (From - To) 17-Jan-2007 - 16-Jan-2008	
4. TITLE AND SUBTITLE Direct Logistic Fuel JP-8 Conversion in a Liquid Tin Anode Solid Oxide Fuel Cell (LTA-SOFC)			5a. CONTRACT NUMBER		
			5b. GRANT NUMBER W911NF-07-C-0032		
			5c. PROGRAM ELEMENT NUMBER 622705		
6. AUTHORS Thomas Tao, Jeff Bentley, Mark Koslowske, Linda Bateman, Bill McPhee			5d. PROJECT NUMBER		
			5e. TASK NUMBER		
			5f. WORK UNIT NUMBER		
7. PERFORMING ORGANIZATION NAMES AND ADDRESSES CellTech Power LLC CellTech Power LLC 131 Flanders Rd. Westborough, MA 01581 -1031				8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army Research Office P.O. Box 12211 Research Triangle Park, NC 27709-2211				10. SPONSOR/MONITOR'S ACRONYM(S) ARO	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S) 52303-CH-DRP.1	
12. DISTRIBUTION AVAILABILITY STATEMENT Approved for Public Release; Distribution Unlimited					
13. SUPPLEMENTARY NOTES The views, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy or decision, unless so designated by other documentation.					
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15. SUBJECT TERMS Fuel Cells, SOFC, Direct Carbon, Direct JP-8, JP-8					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT SAR	15. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON Thomas Tao
a. REPORT U	b. ABSTRACT U	c. THIS PAGE U			19b. TELEPHONE NUMBER 508-898-2223

Report Title

Direct Logistic Fuel JP-8 Conversion in a Liquid Tin Anode Solid Oxide Fuel Cell (LTA-SOFC)

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List of papers submitted or published that acknowledge ARO support during this reporting period. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Number of Papers published in peer-reviewed journals: 0.00

(b) Papers published in non-peer-reviewed journals or in conference proceedings (N/A for none)

Number of Papers published in non peer-reviewed journals: 0.00

(c) Presentations

Number of Presentations: 0.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts): 0

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts): 0

(d) Manuscripts

Number of Manuscripts: 0.00

Number of Inventions:

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Names of Post Doctorates

<u>NAME</u>	<u>PERCENT SUPPORTED</u>
FTE Equivalent:	
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FTE Equivalent:

Total Number:

Sub Contractors (DD882)

Inventions (DD882)

TECHNICAL REPORT
DARPA- ARMY
ARO

Direct Logistic Fuel JP-8 Conversion in a Liquid Tin Anode Solid Oxide Fuel Cell (LTA-SOFC)

Prepared By

**CellTech Power, LLC,
131 Flanders Road, MA, 01581**

April, 2008

Final Report
Contract Number W911NF-07-C-0032

January 2007 – January 2008

Approved for Public Release: Distribution is unlimited

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				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) CellTech Power 131 Flanders Rd Westborough MA, 01581				8. PERFORMING ORGANIZATION REPORT NUMBER 700-ARO-DARPA-FINAL	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) DARPA DSO 3701 North Fairfax Drive Arlington VA 22203-1714 CERDEC Fort Monmouth NY 07703				10. SPONSOR/MONITOR'S ACRONYM(S)	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION / AVAILABILITY STATEMENT Public					
13. SUPPLEMENTARY NOTES					
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16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT UL	18. NUMBER OF PAGES 21	19a. NAME OF RESPONSIBLE PERSON
a. REPORT UNCLASSIFIED	b. ABSTRACT UNCLASSIFIED	c. THIS PAGE UNCLASSIFIED			19b. TELEPHONE NUMBER (include area code)
UNCLASSIFIED					

Table of Contents

LIST OF FIGURES	5
LIST OF TABLES.....	5
FOREWORD	6
STATEMENT OF THE PROBLEMS STUDIED.....	7
OVERVIEW OF LTA – SOFC TECHNOLOGY.....	7
METHODOLOGY	10
Ceramic Porous Separator Development	10
Cell Cap	10
Current Collection.....	11
Electrolyte	11
Cathode	11
Single Cell Test Configuration	11
Four Cell Stack Direct JP-8 conversion.....	13
SUMMARY OF MAJOR RESULTS.....	13
Weight Reduction	13
Volume Reduction	14
Electrochemical Performance of Single Cells and 4 Cell Bundle	14
Manufacturability of Gen 3.1 Single Cell Design.....	17
ANALYSIS OF A PORTABLE 250 W SYSTEM.....	17
CONCLUSIONS AND RECOMMENDATIONS	19
REFERENCES	20

List of Figures

Figure 1: The ceramic porous separator with 65% porosity encapsulates the liquid tin around the electrolyte.....	12
Figure 2: The ceramic cap is used to connect the electrolyte–cathode assembly to the porous separator.	12
Figure 3: A resistance model for Gen 3.1 cell was generated and used in the initial design stages to evaluate the component changes.....	12
Figure 4: The Gen 3.1 LTA–SOFC cells were installed in an alumina process tube for single cell testing.....	12
Figure 5: The four cell stack was modeled using CAD before outsourcing the production of an integrated air manifold.....	13
Figure 6: The power achieved a single Gen 3.1 cell level using JP-8 as fuel was approximately two thirds of the power achieved on hydrogen.....	15
Figure 7: The Gen 3.1 cells have significantly less polarization compared to the Gen 3.0 cells, therefore resulting in a 400% increase in the power density independent of using JP-8 or hydrogen as a fuel.....	15
Figure 8: A Gen 3.1 cell under constant current conditions maintained an efficiency of 17.4% for 100 hrs with no electrochemical performance decay.....	15
Figure 9: A Gen 3.1 cell demonstrated efficiencies >40% after 100 hrs of constant current testing at 17.4% efficiency.....	16
Figure 10: A Gen 3.1 cell thermally cycled 13 times from room temperature to 1,000°C showed only slight variations in OCV.....	16
Figure 11: A small stack consisting on four Gen 3.1 cells achieved a peak power of 9.6 W using JP-8 as a fuel with no reforming or processing.	16
Figure 12: A model of a 250 W portable system based on Gen 3.1 cells, which would use JP-8 as a fuel without reforming or fuel processing.	19

List of Tables

Table 1: A breakdown of individual cell components illustrates the Gen 3.1 reduction of weight in contrast to the Gen 3.0 cell used in the previous MISER program.....	14
Table 2: A breakdown of individual cell components, cell assembly and test equipment contrasts in house fabrication and outside sourcing.	17
Table 3: The single cell design parameters used for the 250 W System Analysis.	18
Table 4: The gravimetric and volumetric system analysis for a portable 250 W system using a standard BOP layout and also for an alternative lighter BOP	18

Foreword

The *direct logistic fuel JP-8 conversion in a Liquid Tin Anode-SOFC (LTA-SOFC)* program officially commenced the 16th of January 2007. This program was proposed as an extension of the DARPA/MISER program that was concluded in the first half of 2006. The DARPA/MISER program objective was the development of a technology for direct conversion of field packaging waste into energy. Under the DARPA/MISER program CellTech Power also demonstrated the ability of the LTA-SOFC to directly convert logistic fuel, JP-8. The demonstration of direct JP-8 conversion without fuel processing or reforming was unprecedented in fuel cell technology. The DOD has a broad interest in power generation using logistic fuel only. Therefore two DOD agencies, DARPA DSO and Army CERDEC jointly funded this program. The aim of this program was to advance LTA-SOFC technology with respect to direct conversion of JP-8. A tabulated matrix of program goals and targets is presented below. The status of LTA-SOFC technology at the start of the program is represented by DARPA/MISER Gen 3.0 cell with goals for the present program represented by Gen 3.1 cells.

Metrics, per cell	Gen 3.0 Direct Plastic and Waste	Gen 3.1 Cell Direct JP-8	
	<u>Status</u>	<u>Target</u>	<u>Achieved</u>
Power (W)	3 ^α	3	✓
Specific Power (W.cm ⁻²)	0.04	0.1	✓
Electrolyte Active Length (cm)	15	10	✓
Electrolyte Active Area (cm ²)	80	30	✓
Cell Diameter (cm)	1.75	1.0	✓
Cell Length (cm)	19	11	✓
Cell Volume (cm ³)	143	35	✓
Cell Weight (g)	215	75	✓
Component Count	10	7	✓
α 3 W was the best performance of Gen 3.0 cells on hydrogen, sustainable power on JP-8 was 1.6 W			

Statement of the Problems Studied

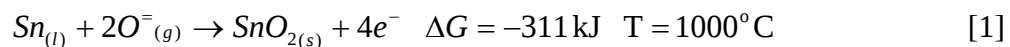
The required improvement in LTA-SOFC technology developed during the DARPA/MISER program fell into three broad areas of study. These areas were single cell form factor and weight; electrochemical performance; and, manufacturability. The problems are briefly outlined below.

- A portable system would require 3-4x reduction in volume and weight from the Gen 3.0 levels of 143 cm³ and 215 g per cell. System volume and weight metrics for a comparable 250–500 W system are outlined in the CERDEC draft battery charger specification [1].
- The electrochemical performance had to be improved to maintain at least a 3 W per cell, although the volume reduction had shrunk the electrolyte active area from 80 cm² to 30 cm². Electrochemical performance was addressed by the improvement of cathode current collection and reduction of mass diffusion limitations through the porous separator at the anode.
- The design for Gen 3.1 cells had to be appropriate for scale up to a system prototype manufacturing. This was demonstrated by statistical analysis of cell component tolerances and demonstrating the ability to fulfill a build quota of cells per month.

Overview of LTA – SOFC Technology

The LTA-SOFC is a modified SOFC consisting of a traditional cathode half cell and molten tin as the anode. Due to kinetic limitation at lower temperatures and material issues at higher temperatures, the operational range is 700–1,000°C. Tin and other p-orbital-electron metals as high temperature fuel cell anodes create an inherent duality for the devices to be batteries and fuel cells. To produce electricity as a battery oxidation of the liquid metal anode occurs directly without any additional fuel. The addition of fuels such as hydrogen, hydrocarbons, carbon and other materials that reduce the tin oxide back to tin allows the device to function as a fuel cell. The utilization of carbon and sulfur as fuels allows the LTA-SOFC to operate on common fuels like JP-8, diesel and coal. The aforementioned properties of the LTA-SOFC system have been demonstrated in multiple stacks and prototypes system including a kilowatt-class system that continuously operated for over 2,000 hours using hydrogen and natural gas [2-6].

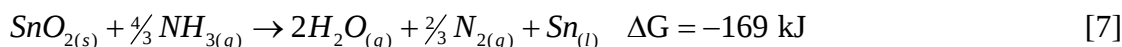
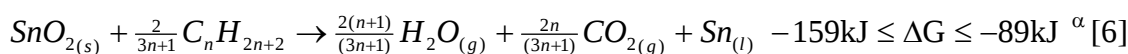
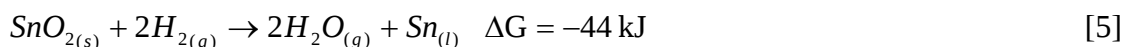
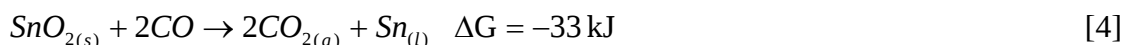
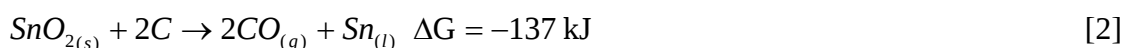
Tin has unique physical and chemical properties, including a low melting point, high boiling point and spontaneous – exothermic oxidation. Therefore the liquid tin phase is inherently stable at 1,000°C. The LTA-SOFC electrochemical reaction is based on the following thermodynamic equation.



As governed by the Nernst equation Open Circuit Voltage (OCV) is inversely proportional to temperature. The OCV of equation 1 is 0.8V at 1000°C, using an oxygen partial pressure of one. This equation gives the OCV for a LTA-SOFC functioning as a battery. The tin oxide has to be constantly reduced by a fuel for a LTA-SOFC to function as a fuel cell.

Several fuels, including carbon will reduce tin oxide at 1,000°C. The influence of temperature on the complete oxidation of carbon is very slight. In contrast, partial oxidation of carbon is directly proportional to temperature. Therefore, a majority of oxides are reduced by carbon. This relationship is the basis of pyrometallurgy and is commonly represented as the Ellingham-Richardson diagram. The temperature required for spontaneous reduction of tin oxide by carbon is about 600°C at STP.

For the hydrogen oxidation reaction, the OCV and Gibbs free energy are inversely proportional to temperature. At elevated temperatures the thermodynamics of the hydrogen oxidation reaction are dominant. Thereby, allowing the reduction of tin oxide, at temperatures above 600°C at STP. The thermodynamics properties of carbon and hydrogen oxidation allow recharging of the LTA-SOFC. A system based on LTA-SOFC technology can utilize all fuels based on carbon and hydrogen, including charcoal, wood, paper, plastics, hydrocarbons and all other carbonaceous fuels. Fuels containing nitrogen and sulfur are also useable. The chemical reactions of these fuels are represented by simplified versions below: at 1,000°C.



Equations 2–8 are thermodynamically favorable and spontaneous. Direct use of hydrocarbons as a fuel source is more complicated due to pyrolysis as the fuel enters the cell at 1,000°C.

Although fuel cells using solid carbon directly as a fuel have been attempted a number of times before, no commercial technology has emerged. Recent research and development efforts in this area are summarized in the US Army Corps' report and elsewhere [1, 7, 8]. Initial work in 1997 by CellTech Power founder Tom Tao demonstrated a 2-cell-stack using carbon, graphite and coal pellets directly charged into the liquid tin as fuel. Since then, CellTech Power has demonstrated operation using solid, liquid and gaseous fuels including coal and bio-charcoal.

LTA-SOFC operational voltage is affected by activation, ohmic and concentration losses. The cathode half cell has typical polarization characteristics to traditional SOFCs. Anodic

^a Range of ΔG values are based on saturated hydrocarbons CH_4 to $\text{C}_{32}\text{H}_{66}$,

JP-8 can be approximated as Cetane $\text{C}_{16}\text{H}_{34}$ with $\Delta G = -158 \text{ kJ}$

polarization is unique due to the molten tin anode. Anode activation polarization is governed by the kinetics of the tin oxide reduction reaction. In contrast, the concentration polarization is governed by transfer of fuel species and reaction products through the porous separator that encapsulates the liquid tin anode. In addition, diffusion of the fuel species, tin oxide and reaction products through the liquid tin also contribute to anode concentration polarization. For current densities $\leq 0.5 \text{ A.cm}^{-2}$, preliminary data indicates that oxygen transfer from electrolyte to the liquid tin anode is not a limiting factor.

The fuel cell–battery duality of the LTA–SOFC is governed by fuel utilization. A continuous fuel stream will ensure reduction of tin oxide and thus fuel cell behavior. Tin oxide reduction is fuel dependent and is typically no longer sustainable at $\geq 80\%$ fuel utilization. Deliberate systems engineering allows this phenomenon to be an advantage. Temporary fuel supply interruption can be tolerated and substantially higher peak (surge) power can be generated by allowing the LTA–SOFC to function as a battery. These features have been demonstrated in kilowatt prototypes. Battery performance depends on the amount of molten tin; and, the oxygen solubility and diffusivity through the tin. Traditional SOFCs are limited to 80% fuel utilization. Therefore the ability of the LTA–SOFC system to function as a battery above 80% fuel utilization is an inherent advantage over traditional SOFC technology.

The state of LTA–SOFC technology at the start of the Army-DARPA program was represented by the key metrics for the previous Gen 3.0 cell:

- 215 g cell weight;
- 143 cm^3 cell volume;
- 40 mW.cm^{-2} power density for a 80 cm^2 active area; and,
- 10 components per cell.

Methodology

This program built on the generic architecture of the MISER Gen 3.0 cells with component size and weight scaled down. Redesign and selection of alternative materials for some of the components was necessary. Therefore, the methodology for redesign of each component is presented. In addition, the methodology for single cell and 4 cell stack testing is also discussed.

Ceramic Porous Separator Development

The key component for a smaller lighter cell design was the ceramic porous separator, Figure 1. The porous separator performs key functions of fuel delivery to the liquid tin and encapsulation of the liquid tin around the electrolyte. The redesigned porous separator optimized both functions, which was demonstrated by increases in electrochemical performance and successful retention of the molten tin for over 40 Gen 3.1 cells tested.

Fuel delivery through the porous separator is one source of concentration polarization at the anode. Oxygen transport through the liquid tin is also a source of concentration polarization; however, it is not thought to be the dominating factor. Therefore, porosity and pore morphology of the ceramic separator was the focus of research, aimed at reduction of anode concentration polarization thereby allowing the smaller Gen 3.1 cell to obtain a four times increase in power density over earlier generations. The final porous separator design had a typical porosity of 65% and a network of interconnected pores <200 μm . The ability of this highly porous separator to encapsulate the liquid tin was due to the high surface tension of tin.

The high surface tension of molten tin from its melting point to cell operational temperature of 1,000°C is a double edged sword. The surface tension allows the tin to be encapsulated by a highly porous separator which is advantageous for fuel delivery. However, high surface tension also promotes beading of the liquid tin on the electrolyte which can lead to uncovered areas of electrolyte. The effect of surface tension was counteracted by altering the thickness of the tin between the electrolyte and the porous separator. The increase in tin thickness was offset by the negative effect of a longer oxygen diffusion path through the tin. Therefore, empirical data was used to determine an ideal tin thickness of 300–500 μm . This allowed the internal dimensions of the porous separator to be sized appropriately. Further, empirical data and modeling were used to determine the wall thickness needed to retain the liquid tin. The inhomogeneity of the pore size and morphology required a 2.5 mm wall thickness to ensure tin retention.

Cell Cap

The Gen 3.1 cell cap incorporates air delivery, air exhaust, cathode current collection, and anode current collection. The cap also provides a structural connection between the porous separator, electrolyte and cathode, Figure 2. This component was designed in house with fabrication outsourced to a local vendor. The cap design is suitable for large volume manufacturing.

Current Collection

Current collectors for the anode and the cathode were resized from the Gen 3.0 cell. No research was directed at improving the efficiency of the current collector material. Current collection methodology is proprietary.

Electrolyte

The electrolyte consisted of a closed end thin wall tube with a nominal thickness of 160–200 μm . The fabrication was outsourced. Details are company proprietary.

Cathode

A resistance model of the Gen 3.1 cell was produced, Figure 3. The cathode circumferential resistance was identified as an area that needed to be improved. A redesigned cathode minimized circumferential resistance. A close correlation was observed between the model and single cell experimental data. The cathode design is company proprietary information and therefore is not disclosed at this time.

Single Cell Test Configuration

The Gen 3.1 single cells were orientated vertically in a Carbolite furnace. The cell test configuration is shown in Figure 4. A Chroma load box and MKS mass flow controllers were interfaced to Labview software and a National Instruments field point DAQ. Dry air was supplied from an Atlas Copco GX7FF compressor. The air flow rate was kept constant during testing at 300 cc.min^{-1} . Spec Air – Specialty Gases industrial grade hydrogen (99.99% purity) was used during cell heat up and for initial performance benchmarking. The hydrogen flow rate was 300 cc.min^{-1} . Teflon tubing was used to make all the gas line connections to the cell. Liquid fuel was metered to the top of the cell using an Ivek hybrid rotary-piston pump. The fuel was ejected from the end of a 26 gauge hypodermic needle. The Chroma load box was connected using 0 gauge braided copper wire to the anode and cathode current collectors respectively. Independent 14 gauge copper voltage sense wires were also connected to the anode and cathode current collectors.



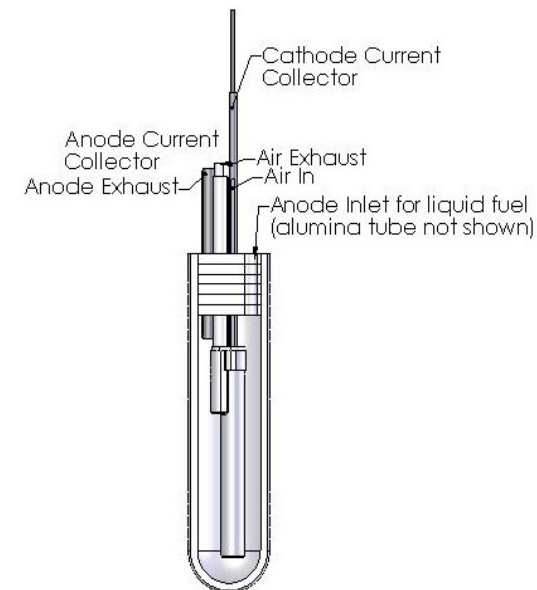
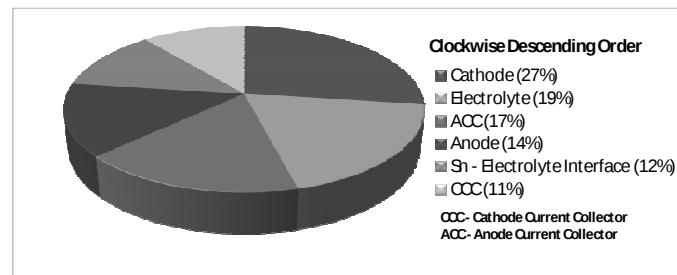
From Left to Right

Figure 1: The ceramic porous separator with 65% porosity encapsulates the liquid tin around the electrolyte.

Figure 2: The ceramic cap is used to connect the electrolyte–cathode assembly to the porous separator.

Figure 3: A resistance model for Gen 3.1 cell was generated and used in the initial design stages to evaluate the component changes.

Figure 4: The Gen 3.1 LTA–SOFC cells were installed in an alumina process tube for single cell testing.



Four Cell Stack Direct JP-8 conversion

The four cell stack was designed around an integrated air manifold. The manifold used a typical serpentine flow field to manage inlet and exhaust streams. The design was evaluated virtually before outsourcing the fabrication to a local vendor. The manifold was assembled, but not used during stack testing. A cold zone air manifold was used instead due to an unforeseen material compatibility issue with the cells. This issue complicated single cell and stack testing. Data acquisition and test stand instrumentation was interfaced using Labview as described in the single cell testing section. Fuel and oxidant flow rates are directly scalable from single cell testing.

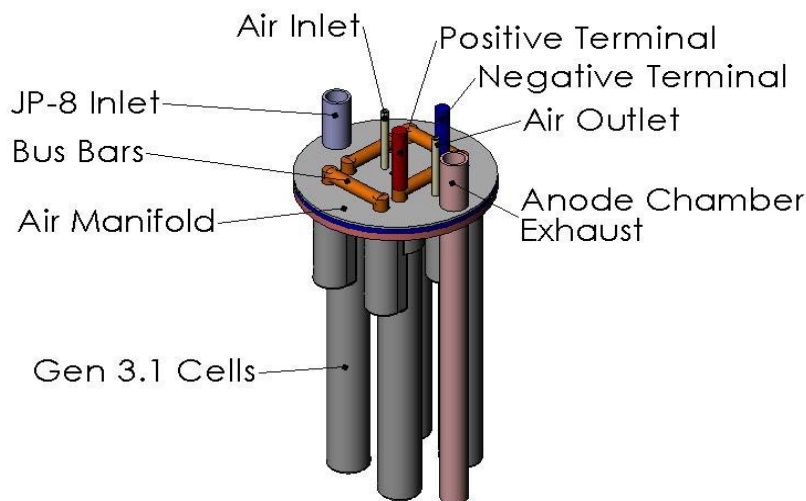


Figure 5: The four cell stack was modeled using CAD before outsourcing the production of an integrated air manifold.

Summary of Major Results

The purpose of this program was advancement of LTA-SOFC technology for direct JP-8 conversion toward the goal of using LTA-SOFC in battlefield portable and mobile power platforms. This program demonstrated a new cell design, Gen 3.1, with 3-4x volumetric and gravimetric energy densities over the previous Gen 3.0 design. The major results achieved using JP-8 directly utilized at the anode as a fuel were $>100 \text{ mW.cm}^{-2}$; $>28\%$ efficiency, >100 hours of continuous testing; >10 thermal cycles; and, demonstration of a 4-cell stack. These metrics were the proposed research objectives at the start of the program.

Weight Reduction

This program reduced single cell weight from 215 g down to 75 g by redesigning the cell components. The Gen 3.1 cell built on the generic architecture of the MISER Gen 3.0 cells. A single cell consists of a porous separator that encapsulates the liquid tin around a closed end electrolyte tube with an internal cathode. Weight reduction focused on reducing the weight of the key components and elimination of other components through cell redesign. A breakdown of the components and weights is given in Table 1. This

allowed the cell weight to be reduced by 65%. Therefore the gravimetric power density increased from 13.9 W.kg^{-1} for Gen 3.0 cells to 43.2 W.kg^{-1} for Gen 3.1 cells. This equates to a 300% increase.

Table 1: A breakdown of individual cell components illustrates the Gen 3.1 reduction of weight in contrast to the Gen 3.0 cell used in the previous MISER program.

Component	Gen 3.0 Cell	Gen 3.1 Cell
1. Porous Separator	20.0	19.0
2. Cell Cap	13	2.0
3. Anode Current Collector	4.9	4.7
4. Cathode Current Collector	19.9	11.5
5. 8YSZ Electrolyte	21.2	4.4
6. LSM Cathode	17	10.2
7. Tin Anode	87.2	23.0
8. Tin Cup	24	Eliminated
9. Air Inlet distribution tube	5.4	Eliminated
10. Air Exhaust distribution tube	2.4	Eliminated
Total Weight	215 g	75 g
Gravimetric Power Density (W.kg^{-1})	13.9	43.2
Volumetric Power Density (W/L)	21	93

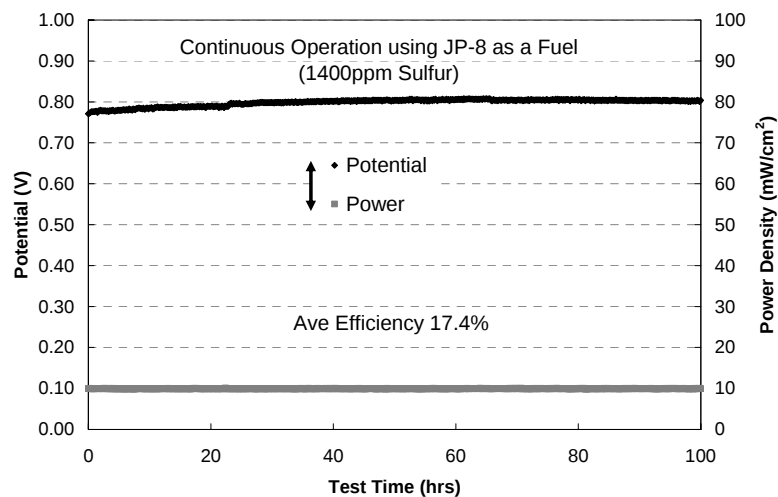
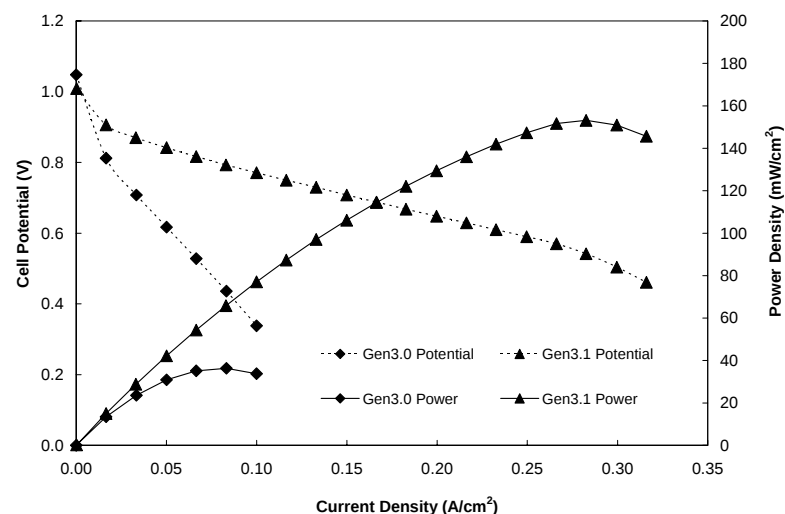
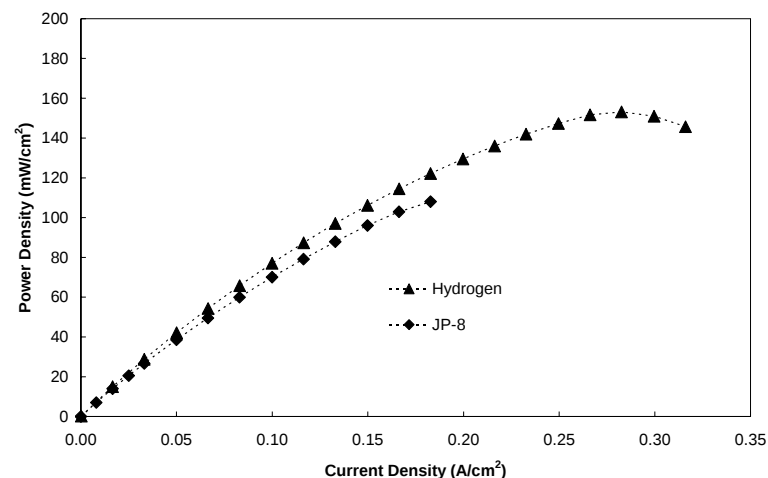
Volume Reduction

In addition to 65% weight reduction, the volume of a single cell was also reduced from 143 cm^3 down to 35 cm^3 . Both volume and weight reductions were possible since the new cell design facilitated improved power density. This allowed the cell volume to be reduced by 75%. Therefore, the gravimetric power density increase from 21 W.L^{-1} for Gen 3.0 cells to 93 W.L^{-1} for Gen 3.1 cells. This equates to a 440% increase.

Electrochemical Performance of Single Cells and 4 Cell Bundle

The Gen 3.1 cell performance using JP-8 as a fuel was approximately two thirds of hydrogen performance, Figure 6. The peak electrochemical performance of Gen 3.1 cells using JP-8 as a fuel was 3.24 W. Significant improvement from the previous generation, Gen 3.0, cells was due to the reduction of polarization losses, Figure 7. A Gen 3.1 single cell was run continuously for over 110 hrs using JP-8 as fuel. The performance under constant current conditions for the first 100 hrs showed no decay, Figure 8. The calculated efficiency was 17.4%. The cell was then run at >40% efficiency for one hour, before ~10 hrs at >30% efficiency, Figure 9. Thermal cycling on a single cell showed no significant variation in the OCV, with an average OCV of 1.048 V, Figure 10. A lack of variation in the fuel/oxidant cross over rate illustrates the integrity of the cell.

The electrochemical performance of the four cell stack using JP-8 as a fuel is shown in Figure 11. The four cells were wired in parallel with the peak power density of 81 mW.cm^{-2} and a peak current density of 0.13 A.cm^{-2} .

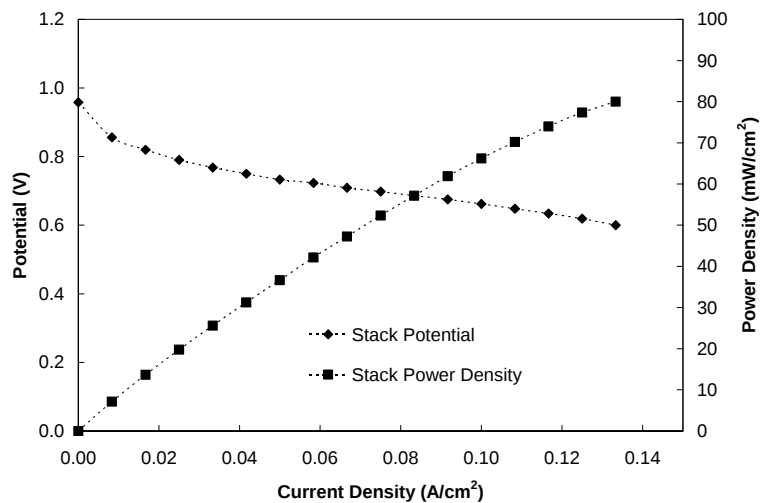
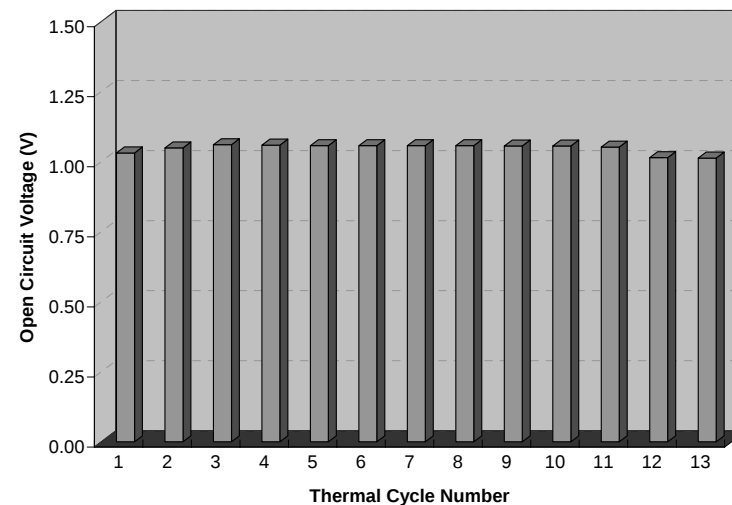
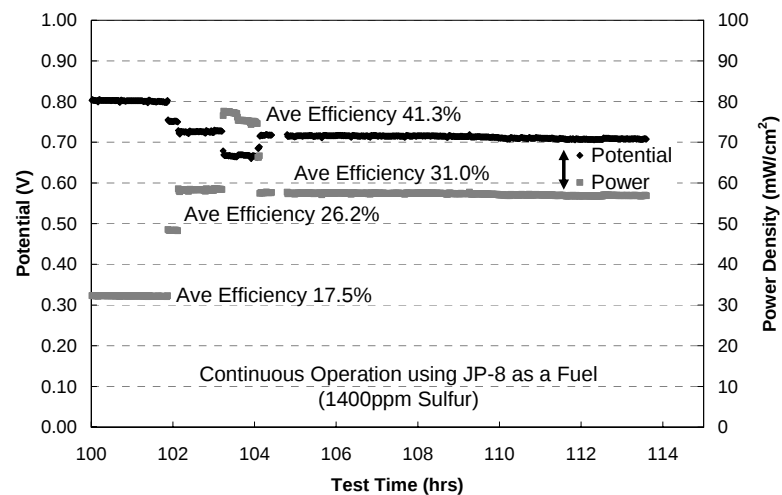


Figures From Left to Right

Figure 6: The power achieved a single Gen 3.1 cell level using JP-8 as fuel was approximately two thirds of the power achieved on hydrogen.

Figure 7: The Gen 3.1 cells have significantly less polarization compared to the Gen 3.0 cells, therefore resulting in a 400% increase in the power density independent of using JP-8 or hydrogen as a fuel.

Figure 8: A Gen 3.1 cell under constant current conditions maintained an efficiency of 17.4% for 100 hrs with no electrochemical performance decay.



Figures From Left to Right

Figure 9: A Gen 3.1 cell demonstrated efficiencies >40% after 100 hrs of constant current testing at 17.4% efficiency.

Figure 10: A Gen 3.1 cell thermally cycled 13 times from room temperature to 1,000°C showed only slight variations in OCV.

Figure 11: A small stack consisting on four Gen 3.1 cells achieved a peak power of 9.6 W using JP-8 as a fuel with no reforming or processing.

Manufacturability of Gen 3.1 Single Cell Design

After the redesign of the cell components and design of a four cell stack air manifold, CellTech Power worked with a range of vendors to ensure timely procurement of parts. Several parts that contain proprietary materials and designs were fabricated and assembled in-house to ensure IP protection, Table 2. To build 250 W prototype systems, a production model was used to predict the cell manufacturing capability needed. A fixed design and material selection for prototype production of Gen 3.1 would allow cell production to reach 100 cells per month. Current vendors and in house equipment would support 100 cells per month production level.

Table 2: A breakdown of individual cell components, cell assembly and test equipment contrasts in house fabrication and outside sourcing.

Component	In House	Sourced
Porous Separator	X	X
Cell Cap		X
Anode Current Collector	X	
Cathode Current Collector	X	
8YSZ Electrolyte		X
LSM Cathode	X	
Tin Anode		X
Single Cell Assembly	X	
4 Cell Stack Assembly	X	
4 Cell Stack Air Manifold		X
Test Stand Fabrication	X	

Analysis of a Portable 250 W System

The system analysis performed for a 250 W system was a forward looking paper study that relies on significant improvements to current cell performance and development of customized balance of plant components. The stack was the main focus of this analysis. The experimental data collected during this program suggests that Gen 3.1 cells can maintain 80% of peak performance. The design parameters are listed in Table 3.

Table 3: The single cell design parameters used for the 250 W System Analysis.

Single Cell Design Parameters	Gen 3.1
Potential (V)	0.7
Current Density (mA.cm ⁻²)	13.3
Power Density (mW.cm ⁻²)	93.3
Efficiency (%)	30
Active Area (cm ²)	30
Cell Weight (g)	75
Cell Volume (cm ³)	35
Gravimetric Power Density (W.kg ⁻¹)	37.3
Volumetric Power Density (W.L ⁻¹)	80

The copper interconnects added substantial weight to the system. Therefore, analysis was performed to minimize the weight without sacrificing performance. The cell interconnects were wired to give a stack output voltage of 14 V. Twenty cells were wired in series to form a sub-stack. Five sub-stacks were then wired in parallel to give a gross output power of 280 W at 14 V – 20 A. The potential loss due to the resistance of the copper wire at 1,000°C was analyzed for a range of different wire gauges. The gauge of wire chosen was AWG 6, which corresponded to 217 g of copper wire for the stack.

The Balance of Plant (BOP) from a previous system analysis was used to give a total system weight of 13.9 kg. A breakdown of the weight is given in Table 4 for the hot and cold zones. The volume of the system was calculated to be 20 L. A lighter BOP was also analyzed that would decrease the system weight 6.5%. A model of the proposed system is shown in Figure 12.

Table 4: The gravimetric and volumetric system analysis for a portable 250 W system using a standard BOP layout and also for an alternative lighter BOP

Gravimetric and Volumetric Metrics	Standard BOP	Alternative BOP
Sub-Total Cold Zone Mass (kg)	3.5	2.6
Sub-Total Hot Zone Mass (kg)	10.4	10.4
Total System Weight (kg)	13.9	13.0
Total System Volume (L)	20	20
Gravimetric Power Density (W.kg ⁻¹)	18.0	19.2
Volumetric Power Density (W.L ⁻¹)	125	125

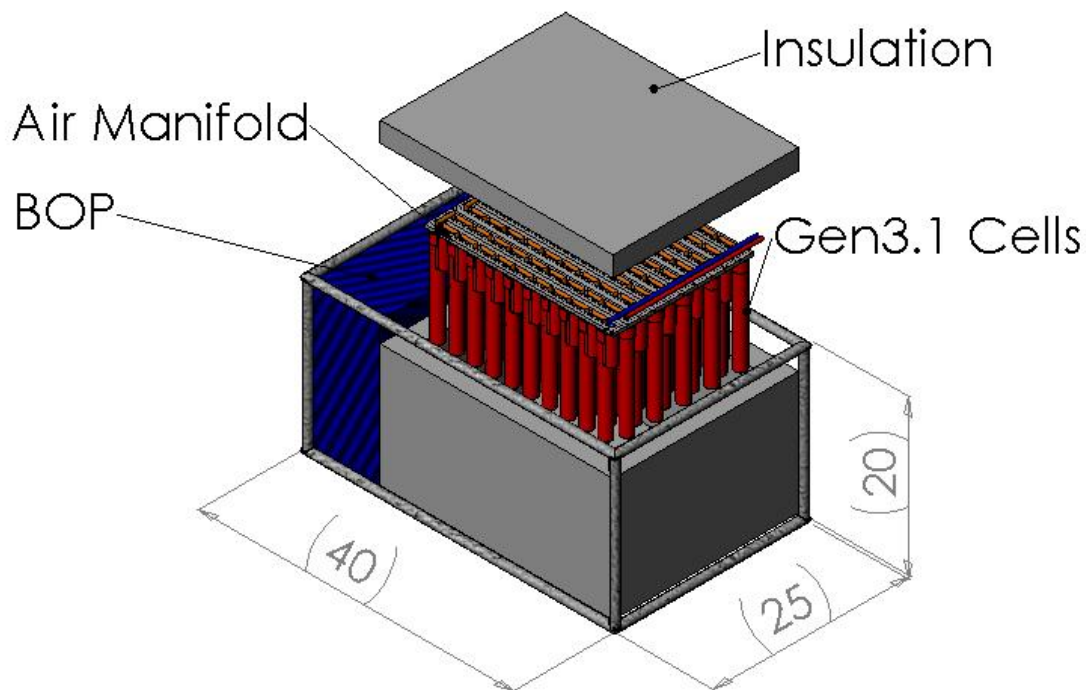


Figure 12: A model of a 250 W portable system based on Gen 3.1 cells, which would use JP-8 as a fuel without reforming or fuel processing.

Conclusions and Recommendations

In conclusion, this program further developed LTA-SOFC technology with respect to direct conversion of JP-8. The sulfur content of the JP-8 fuel used during testing was 1400 ppm. All of the programs goals were satisfied. The major accomplishments were:

1. The single cell weight was reduced 65% to 75 g.
2. The single cell gravimetric power density was increased 300% to 43.2 W.kg^{-1} .
3. The single cell volume was reduced 75% to 35 cm^3 .
4. The single cell volumetric power density was increased 440% to 93 W.L^{-1} .
5. The single cell power density was increased 300% to 120 mW.cm^2 using JP-8 as a fuel without processing or reforming.
6. A single cell was demonstrated for 100 hrs at 17.4% efficiency using JP-8 as a fuel.
7. At the completion of 100 hrs of testing the same single cell demonstrated efficiencies $>40\%$ for an hour and $>30\%$ for 10 hrs using JP-8 as a fuel.
8. A single cell was thermally cycled between ambient and operational temperature 13 times without showing significant degradation in OCV.

A four cell stack wired in parallel demonstrated 80 mW.cm^{-2} using JP-8 as a fuel. However an unforeseen material selection issue associated with the cap and electrolyte resulted in cracking. Revaluation of the cap material was undertaken at the end of the program. This work was continued after the conclusion of the program and a better materials match found. At the time of writing this report, electrolyte cracking at the electrolyte – cap interface due to thermal expansion mismatch has been resolved. Sealing materials and methods affected by the material substitution are under investigation.

The uniqueness of the LTA–SOFC technology and the technology solution it offers the U.S. military dictates its continual development to commercial prototype. To ensure judicious use of private and government funding, LTA–SOFC development in the following areas is recommended as listed below.

- Development of Gen 3.1 cell technology into small stacks and systems at brass board level to address BOP requirements for the technology.
- Research and development of Gen 3.2 cell design to simplify cell form factor, reduce weight to 48 g per cell, improve power density to 160 cm^2 , and improve longevity to >1000 hours, and >100 thermal cycles.
- In partnership with academic institutions, fundamental modeling of anode polarization losses is needed to provide a theoretical base for future cell design. Tin reaction kinetics are not currently well documented in the scientific literature.
- Balance of Plant development associated with JP-8 delivery, handling and startup for a LTA–SOFC.

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