



**Department of Energy (DoE)
Final Report Compilation
for the
Research and Development of
High-Power and High-Energy
Electrochemical Storage Devices**

Cooperative Agreement DE-FC26-05NT42403

Projects from July 11, 2005 through April 30, 2014

June 2014

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Acronyms and Abbreviations

12VSS	12V Start-Stop	EC	Ethylene Carbonate
ANL	Argonne National Laboratory	EDS	Electron Dispersion Spectroscopy
AMS	Advanced Membrane Systems	EOL	End-of-Life
ARC	Accelerated Rate Calorimetry	EPT	Eagle Picher Technologies
BMS	Battery Management System	ESS	Electrochemical Storage System
BOL	Beginning of Life	EV	Electric Vehicle
BOM	Bill of Materials	EVPC	Electric Vehicle Power Characterization
BPC	Battery Protection and Communications	FEA	Finite Element Analysis
BSF	Battery Scale/Size Factor	FTIR-ATR	Fourier Transform Infrared-Attenuated Total Reflection
CD	Charge Depleting	HC	High Capacity
CDC	Charge Depleting Cycle	HCMRTM	High Capacity Manganese Rich
CID	Current Interrupt Device	HEV	(Power Assisted) Hybrid Electric Vehicle
CN-SC	Carbon Nanofiber Soft Carbon	HP	High Power
COTS	Commercial Off-the-Shelf	HPPC	Hybrid Pulse Power Characterization
CPI	Compact Power, Inc.	HTMI	High Temperature Melt Integrity
CS	Charge Sustaining	HVBS	High Voltage Battery System
CTP	Coal Tar Pitch (a precursor to carbon anode materials)	INL	Idaho National Laboratory
CTQ	Critical to Quality	IPQC	In-Process Quality Control
DEC	Diethyl Carbonate	IQC	Incoming Quality Inspection
DFMEA	Design Failure Mode and Effects Analysis	JCI	Johnson Controls, Inc.
DOD	Depth of Discharge	JCS	Johnson Controls-Saft
DOE	Department of Energy	LCO	Lithium Cobalt Oxide
DSC	Differential Scanning Calorimetry		

LFP	Lithium Iron Phosphate	RPT	Reference Performance Test
LI	Lithium	SC	Soft Carbon
LIB	Lithium-Ion Battery	SEM	Scanning Electron Microscopy
LMWPE	Low Molecular Weight Polyethylene	SLMP	Stabilized Lithium Metal Powder
LSV	Linear Sweep Voltammetry	SNL	Sandia National Laboratory
MNC-OH	Manganese, Nickel, Cobalt Hydroxide	SOC	State of Charge
NCA	Nickel, Cobalt, Aluminum Oxide	SOP	Standard Operating Procedures
NCM	Nickel, Cobalt, Manganese	SOW	Statement of Work
NMP	N-Methyl Pyrrolidone	SP	Super-P
NREL	National Renewable Energy Laboratory	TAP	Technology Assessment Program
OCV	Open Circuit Voltage	TEM	Transmission Electron Microscopy
OEM	Original Equipment Manufacturer	TMA	Thermal Mechanical Analysis
PB	Polybutylene	UHMWPE	Ultra-High Molecular Weight Polyethylene
PC	Propylene Carbonate	UMT	Ultimate Membrane Technology
PHEV	Plug-In Hybrid Electric Vehicle	USABC	United States Advanced Battery Consortium LLC
PNGV	Partnership for a New Generation of Vehicles	USCAR	United States Council for Automotive Research LLC
PP	Polypropylene	U.S. DOE	United States Department of Energy
PSD	Particle Size Distribution	VCU	Virginia Commonwealth University
PVDF	Polyvinylidene Fluoride	XRD	X-Ray Diffraction Spectroscopy
R&D	Research and Development	WFW	Wound Flat Wrap
RFPI	Request for Proposal Information		

Executive Summary

The accomplishments and technology progress made during the U.S. Department of Energy (DOE) Cooperative Agreement No. DE-FC26-05NT42403 (duration: July 11, 2005 through April 30, 2014, funded for \$125 million in cost-shared research) are summarized in this Final Technical Report for a total of thirty-seven (37) collaborative programs organized by the United States Advanced Battery Consortium, LLC (USABC).

The USABC is a partnership, formed in 1991, between the three U.S. domestic automakers Chrysler, Ford, and General Motors, to sponsor development of advanced high-performance batteries for electric and hybrid electric vehicle applications. The USABC provides a unique opportunity for developers to leverage their resources in combination with those of the automotive industry and the Federal government. This type of pre-competitive cooperation minimizes duplication of effort and risk of failure, and maximizes the benefits to the public of the government funds.

A major goal of this program is to promote advanced battery development that can lead to commercialization within the domestic, and as appropriate, the foreign battery industry. A further goal of this program is to maintain a consortium that engages the battery manufacturers with the automobile manufacturers and other key stakeholders, universities, the National Laboratories, and manufacturers and developers that supply critical materials and components to the battery industry.

Typically, the USABC defines and establishes consensus goals, conducts pre-competitive, vehicle-related research and development (R&D) in advanced battery technology. The R&D carried out by the USABC is an integral part of the DOE's effort to develop advanced transportation technologies that will

significantly improve fuel economy, comply with projected emissions and safety regulations, and use domestically produced fuels.

The USABC advanced battery development plan has the following three focus areas:

1. Existing technology validation, implementation, and cost reduction.
2. Identification of the next viable technology with emphasis on the potential to meet USABC cost and operating temperature range goals.
3. Support high-risk, high-reward battery technology R&D.

Specific to the Cooperative Agreement DE-FC26-05NT42403, addressing High-Energy and High Power Energy Storage Technologies, the USABC focus was on understanding and addressing the following factors (listed in priority of effort):

- **Cost:** Reducing the current cost of lithium-ion batteries (currently about 2-3 times the FreedomCAR target (\$20/kW).
- **Low Temperature Performance:** Improving the discharge power and removing lithium plating during regenerative braking.
- **Calendar Life:** Achieving 15-year life and getting accurate life prediction.
- **Abuse Tolerance:** Developing a system level tolerance to overcharge, crush, and high temperature exposure.

This Final Technical Report compilation is submitted in fulfillment of the subject Cooperative Agreement, and is intended to serve as a ready-reference for the outcomes of following eight categories of projects conducted by the USABC under award from the DOE's Energy Efficiency and Renewable Energy (EERE) Vehicle Technologies Program:

1. Electric Vehicle (EV)
(*Section A of this report*)
2. Hybrid Electric Vehicle (HEV)
(*Section B*)
3. Plug-In Hybrid Electric Vehicle (PHEV)
(*Section C*)
4. Low-Energy Energy Storage Systems
(LEESS) (*Section D*)
5. Technology Assessment Program (TAP)
(*Section E*)
6. Ultracapacitors (*Section F*)
7. 12 Volt Start-Stop (*Section G*)
8. Separators (*Section H*)

The report summarizes the main areas of activity undertaken in collaboration with the supplier community and the National Laboratories. Copies of the individual supplier final reports are available upon request. Using project gap analysis versus defined USABC goals in each area, the report documents known technology limits and provides direction on future areas of technology and performance needs for vehicle applications. The report was developed using information such as program

plans, gap analysis charts, quarterly reports and final project reports submitted by the developers.

The public benefit served by this USABC program is that it continues the development of critical advanced battery technology that is needed to make electric, hybrid electric, and fuel cell vehicles attractive to a wide segment of the vehicle market. This will allow for a substantial savings in petroleum fuel use as these vehicles are introduced into the nation's transportation system. It will also allow a sharp reduction in automotive air pollution emissions in critical areas that are currently classified as non-attainment by the Environmental Protection Agency. This program will also help ensure the long term health and viability of the U.S. Battery and Ultracapacitor Manufacturing Industry.

The goals of eight categories of projects follow and summarization of each of the project's accomplishments are in sequence of the list above.

USABC Goals for Advanced Batteries for EVs

Parameter	Mid Term	Long Term
Power Density (W/L)	460	600
Specific Power – Discharge, 80% DOD/30 sec (W/kg)	300	400
Specific Power – Regen, 20% DOD/10sec (W/kg)	150	200
Energy Density – C/3 Discharge Rate (Wh/L)	230	300
Specific Energy – C/3 Discharge Rate (Wh/kg)	150	200
Specific Power/Specific Energy Ratio	2:1	2:1
Total Pack Size (kWh)	40	40
Life (years)	10	10
Cycle Life – 80% DOD (Cycles)	1,000	1,000
Power & Capacity Degradation (% of rated spec)	20	20
Selling Price - 25,000 units @ 40kWh(\$/kWh)	<150	100
Operating Environment (°C)	-40 to +50 20% Performance Loss (10% Desired)	-40 to +85
Normal Recharge Time	6 hours (4 hours desired)	3 to 6 hours
High Rate Charge	20 – 70% SOC in <30 minutes @ 150W/kg (<20 min @ 270W/kg desired)	40 – 80% SOC in 15 minutes
Continuous Discharge in 1 hour – No Failure (% of rated energy capacity)	75	75

FreedomCAR Energy Storage System Performance Goals for Power-Assist Hybrid Electric Vehicles (November 2002)

Characteristics	Units	Power-Assist (Minimum)	Power-Assist (Maximum)
Pulse Discharge Power (10s)	kW	25	40
Peak Regenerative Pulse Power (10s)	kW	20 (55-Wh pulse)	35 (97-Wh pulse)
Total Available Energy (over DOD range where power goals are met)	KWh	0.3(at C ₁ /1rate)	0.5 (at C ₁ /1 rate)
Minimum Round-Trip Energy Efficiency	%	90 (25-Wh cycle)	90 (50-Wh cycle)
Cold Cranking Power at -30°C (three 2-s pulses, 10-rests between)	kW	5	7
Cycle Life for Specified SOC Increments	cycles	300,000 25-Wh cycles(7.5M-Wh)	300,000 50-Wh cycles (15 M-Wh)
Calendar Life	years	15	15
Maximum Weight	kg	40	60
Maximum Volume	I	32	45
Operating Voltage Limits	Vdc	max≤400 min>(0.55 x Vmax)	max<400 min>(0.55 x Vmax)
Maximum Allowable Self-Discharge Rate	Wh/day	50	50
Temperature Range: Equipment Operation Equipment Survival	°C	-30 to +52 -46 to +66	-30 to +52 - 46 to +66
Production Price @ 1,000,000 units/year	\$	500	800

USABC Goals for Advanced Batteries for PHEVs

USABC Requirements of End of Life Energy Storage Systems for PHEVs

Characteristics at EOL (End of Life)	units	High Power/Energy Ratio Battery	Moderate Energy/Power Ratio Battery	High Energy/Power Ratio Battery
Reference Equivalent Electric Range	miles	10	20	40
Peak Pulse Discharge Power - 2 Sec / 10 Sec	kW	50 / 45	45 / 37	46 / 38
Peak Regen Pulse Power (10 sec)	kW	30	25	25
Max. Current (10 sec pulse)	A	300	300	300
Available Energy for CD (Charge Depleting) Mode, 10 kW Rate	kWh	3.4	5.8	11.6
Available Energy for CS (Charge Sustaining) Mode	kWh	0.5	0.3	0.3
Minimum Round-trip Energy Efficiency (USABC HEV Cycle)	%	90	90	90
Cold cranking power at -30°C, 2 sec - 3 Pulses	kW	7	7	7
CD Life / Discharge Throughput	Cycles/MWh	5,000 / 17	5000 / 29	5,000 / 58
CS HEV Cycle Life, 50 Wh Profile	Cycles	300000	300000	300000
Calendar Life, 35°C	year	15	15	15
Maximum System Weight	kg	60	70	120
Maximum System Volume	Liter	40	46	80
Maximum Operating Voltage	Vdc	400	400	400
Minimum Operating Voltage	Vdc	>0.55 x Vmax	>0.55 x Vmax	>0.55 x Vmax
Maximum Self-discharge	Wh/day	50	50	50
System Recharge Rate at 30°C	kW	1.4 (120V/15A)	1.4 (120V/15A)	1.4 (120V/15A)
Unassisted Operating & Charging Temperature Range	°C	-30 to +52	-30 to +52	-30 to +52
30°-52°	%	100	100	100
0°	%	50	50	50
-10°	%	30	30	30
-20°	%	15	15	15
-30°	%	10	10	10
Survival Temperature Range	°C	-46 to +66	-46 to +66	-46 to +66
Maximum System Production Price @ 100k units/yr	\$	\$1,700	\$2,200	\$3,400

USABC Goals for High Power, Lower Energy, Energy Storage System (LEESS) for Power Assist Hybrid Electric Vehicle (PAHEV) Applications

USABC Requirements at End of Life for LEESS PA HEV

End of Life Characteristics	Unit	PA (Lower Energy)	
2s/10s Discharge Pulse Power	kW	55	20
2s/10s Regen Pulse Power	kW	40	30
Discharge Requirement Energy	Wh	56	
Regen Requirement Energy	Wh	83	
Maximum Current	A	300	
Energy Over which Both Requirements are Met	Wh	26	
Energy Window for Vehicle Use	Wh	165	
Energy Efficiency	%	95	
Cycle-Life	Cycles	300,000 (HEV)	
Cold-Cranking Power at -30°C (after 30 day stand at 30°C)	kW	5	
Calendar Life	Years	15	
Maximum System Weight	kg	20	
Maximum System Volume	Liter	16	
Maximum Operating Voltage	Vdc	≤ 400	
Minimum Operating Voltage	Vdc	$\geq 0.55V_{\max}$	
Unassisted Operating Temperature Range	°C	-30 to +52	
30° -52°	%	100	
0°	%	50	
-10°	%	30	
-20°	%	15	
-30°	%	10	
Survival Temperature Range	°C	-46 to +66	
Selling Price/System (@ 100k/yr)	\$	400	



USABC / ANL TEST PLAN

EV TECHNOLOGY ASSESSMENT TESTS

PURPOSE

This document outlines a series of tests to assess the performance, cycle life and accelerated calendar life of EV batteries made by ____developer name____. The cells are ____V and ____ Ah. A total of 18 batteries are needed for this work. The distribution of cells is as follows: 6 for cycle life and 12 for accelerated calendar life.

This work is sponsored by the USABC and contains performance and life tests using the procedures and guidelines outlined by the USABC in their Battery Test Procedures Manual. Baseline life testing is to be conducted with Dynamic Stress Test (DST) discharges to 80% DOD at an accelerated cycling rate of about 2 cycles/day. Accelerated calendar life will be conducted at 25, 35, 45, and 55°C with three batteries at each temperature.

2.0 REFERENCES

USABC Electric Vehicle Battery Test Procedures Manual, Latest Release January 1996

3.0 EQUIPMENT

Existing ANL-EADL equipment will be used to conduct these tests. Measurements will include cell voltage, current and temperature.

4.0 PRE-REQUISITES

- Assign USABC ID Numbers
- Examine Deliverables for Damage
- Record Physical Sizes & Weights

5.0 CELL RATINGS/LIMITATIONS

5.1 Ratings @ 25°C of batteries:

- Rated C₃ Capacity (V_{min} to V_{max}): ____ Ah (actual TBD)
- Energy (C/3): ____ Wh
- Peak Power at 80% DOD: ____ W/kg
- DST Reference Power: ____ W/kg
- V_{max}, pulse and continuous ____ V
- V_{min}, pulse and continuous for DST cycling ____ V
- V_{min}, pulse for peak power test ____ V
- Cell Dimensions: ____
- Cell Length: ____ mm
- Cell Volume: ____ cc
- Cell Weight: ____ kg
- Operating temperature range: ____ to ____ °C



USABC / ANL TEST PLAN EV TECHNOLOGY ASSESSMENT TESTS

5.2 Termination Conditions

- Temperature > ____ °C
- Charge termination = ____ V
- Discharge termination = ____ V or 80% DOD
- Voltage < ____ V

5.3 Charging

< state algorithm >

Maximum battery voltage: ____ V
 Maximum charge current: ____ A
 Maximum cell surface temperature: ____ °C

5.4 Discharge

Minimum cell voltage: ____ V
 Maximum discharge current: ____ A (pulse and continuous)
 Maximum cell surface temperature: ____ °C

5.5 Others

- OCAC Time: 0.5 h (Fixed to maintain same self-discharge loss)
- OCAD Time: 0.5 h

6.0 Safety Concerns

< List concerns, if any; if none, state "None" >

7.0 Tests

The cells will undergo ambient performance, thermal performance and cycle and calendar life evaluation. Bracketed "[]" numbers in the outline given below indicate USABC Battery Test Procedure test identification numbers.



USABC / ANL TEST PLAN EV TECHNOLOGY ASSESSMENT TESTS

7.1 USABC Core Tests

The six cells will undergo a series of USABC Core tests as listed below to verify rated performance. Rated performance must be achieved to continue the test plan.

- a) Constant Current Discharge [2]: Three cycles of each:
 - 1) $C_3/3$ discharge with a $V_{\min} = ___ \text{ V}$
 - 2) $C_3/2$ discharge with a $V_{\min} = ___ \text{ V}$
 - 3) $C_3/1$ discharge with a $V_{\min} = ___ \text{ V}$
- b) 100% DST Discharge Capacity [5]: One cycle
(DST discharge with $___ \text{ W/kg}$ peaks to rated $C/3$ capacity;
 $V_{\min} = ___ \text{ V}$)
- c) Peak Power ($2/3 V_{oc}$) to rated $C/3$ capacity [3]: one cycle
($V_{\min} = ___ \text{ V}$; $I_{\text{base}} = ___ \text{ A}$; $I_{\text{pulse}} = ___ \text{ A}$)
- d) 48-h Stand Test at full charge

7.2 Accelerated Calendar Tests

After successfully completing the Core Tests, accelerated calendar life will be conducted at 25, 35, 45, and 55°C with three batteries at each temperature. Each battery will be charged to full charge at the $C/3$ rate and allowed to rest in an open circuit condition at the desired temperature for 4 weeks. At the end of 4 weeks, the batteries will undergo RPTs (as described in section 7.4) at 25°C.

7.3 Cycle Life Tests [14A]

After successfully completing the Core Tests, the six batteries will be life cycle tested. Cycle life testing will be conducted until End of Life (EOL) is reached using established baseline operating conditions to assess component reliability at ambient temperatures. It is anticipated that about 2 cycles/day will be accumulated. RPTs will be conducted at the start of life testing and at 50 cycle intervals (~1/month). Life test cycling will use the following conditions:

- Discharge: DST_{xxx} to $___ \text{ Ah}$ (80% of DST Ah Rating)
- Charge: See section 5.3.
- End-of-Life: Energy less than 80% of rated energy at the $C/3$ rate ($___ \text{ Wh/kg}$) or Peak Power $< ___ \text{ W/kg}$ at 80% DOD



USABC / ANL TEST PLAN EV TECHNOLOGY ASSESSMENT TESTS

7.4 Reference Performance Tests (RPT)

These tests will be conducted initially and at intervals of ~50 cycles (about once per month) during life testing to establish a performance baseline.

- a) 3-h Rate Discharge Capacity [3]: 2 cycles to 100% DOD

($V_{\min} = \text{___ V}$)

- b) DST_{xxx} Discharge Capacity [5]: one cycle

(see above)

- c) Peak Power ($2/3 V_{oc}$) to rated C/3 capacity [6]: one cycle

($V_{\min} = \text{___ V}$; $I_{\text{base}} = \text{___ A}$; $I_{\text{pulse}} = \text{___ A}$)

8) Special Measurements – none

USABC Goals for Ultracapacitors

Attributes	12 V (TSS)		42 V (FSS)		42 V (TPA)	
Discharge Pulse, kW	4.2	2 s	6	2 s	13	2 s
Regenerative Pulse, kW	N/A		N/A		8	2 s
Cold Cranking Pulse @ -30°C	4.2	7 V min	8	21 V min	8	21 V min
Available Energy, Whr (CP@ 1 kW)	15		30		60	
Recharge Rate, kW	.4		2.4		2.6	
Cycle Life	150k		150k		150k	
Cycle Life and Efficiency Load Profile	UC 10-5		UC10-5		UC 10-5	
Calendar Life (years)	15		15		15	
Energy Efficiency on UC 10 (%)	95		95		95	
Self Discharge (72 hr from max V)	<4%		<4%		<4%	
Maximum Operating Voltage (Vdc)	17		48		48	
Minimum Operating Voltage (Vdc)	9		27		27	
Operating Temperature Range (°C)	-30 to +52		-30 to +52		-30 to +52	
Survival Temperature Range (°C)	-46 to +66		-46 to +66		-46 to +66	
Maximum System Weight (kg)	5		10		20	
Maximum System Volume (liters)	4		8		16	
Selling Price (\$/system @ 100 k units/yr)	40		80		130	

USABC Goals for Advanced Batteries for 12V Start-Stop Vehicle Applications

End of Life Characteristics	Units	Target	
		Under hood	Not under hood
Discharge Pulse, 1s	kW	6	
Max current, 0.5s	A	900	
Engine-off accessory load consider removing	W	750	
Cold cranking power at -30°C (three 4.5-s pulses, 10s rests between pulses at lower SOC)	kW	6 kW for 0.5s followed by 4 kW for 4s	
Extended Stand Test (30 days at 30°C followed by cold crank test)	kW	6 kW for 0.5s followed by 4 kW for 4s	
Min voltage under cold crank	Vdc	8.0	
Available energy (750W)	Wh	360	
Peak Recharge Rate, 10s	kW	2.2	
Sustained Recharge Rate	W	750	
Cycle life, every 10% life RPT with cold crank at min SOC	Engine starts/miles	450k/150k	
Calendar Life at 30°C, 45°C if under hood	Years	15 at 45°C	15 at 30°C
Minimum round trip energy efficiency	%	95	
Maximum allowable self-discharge rate	Wh/day	10	
Peak Operating Voltage, 10s	Vdc	15.0	
Sustained Max. Operating Voltage	Vdc	14.6	
Minimum Operating Voltage under load	Vdc	10.5	
Operating Temperature Range (available energy to allow 6 kW (1s) pulse)	°C	-30 to +75	-30 to +52
30°C – 52°C	%	100 (to 75°C)	100
0°C	%	50	
-10°C	%	30	
-20°C	%	15	
-30°C	%	10	
Survival Temperature Range (24 hours)	°C	-46 to +100	-46 to +66
Maximum System Weight	kg	10	
Maximum System Volume	L	7	
Maximum System Selling Price (@100k units/year)	\$	\$220	\$180

USABC Goals for HTML Separator

Parameter	Goal
Thickness, (μm)	20
Permeability:Gurley, sec	<20
Wettability	Wet out in electrolytes
Chemical Stability	Stable in battery for 10 years
Pore size, (μm)	<1
Puncture Strength	>300g/25.4mm
Thermal Stability	<5 % shrinkage at 220°C
Tensile Strength	<2 offset at 1,000 psi
Skew	<2mm/meter
Pin removal	Easy removal from all major brands of winding machines
HTML	220°C

Section A – Electric Vehicle

Section A – Electric Vehicle (EV) Final Reports:

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1. Development of High Performance Advanced Batteries for Electric Vehicle Applications

Performing Organization: Quallion

Project Duration: 10/5/2010 – 8/16/2012

1.1 Executive Summary

In order to achieve commercial targets for electric vehicle price and driving range, significant improvements are needed to improve the capacity and power of electric vehicle batteries. Quallion LLC's (Quallion) work under a two-year USABC award demonstrates significant progress towards these ends through development of novel battery materials and new battery architecture. Quallion developed new anode materials for high power using carbon nanofibers impregnated into soft carbon particles. The nanofibers act as conductive bridges to help electrons move more quickly through the material. The power achieved for these materials is 8,700 W/kg at the materials level. The material has been evaluated in pouch cells and has achieved 324 W/kg in a small proof of concept cell. Quallion developed new anode materials for high capacity using silicon nanofibers within soft carbon particles. The program has demonstrated an energy density of 59 Wh/kg using small proof of concept pouch cells.

Quallion sought to combine the best of both worlds by demonstrating a battery composed of both high capacity and high power cells and modules. Quallion's module testing confirmed predictions that while a battery made entirely of high capacity cells could meet most performance targets, some of the high power modules are needed to meet cycle life and temperature requirements. Quallion's development and evaluation of electric vehicle battery packs contributed to the understanding of the performance, reliability, and cost associated with a modular battery design.

1.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

Project Objectives, Approach and Outcomes:

The purpose of Quallion's effort was to demonstrate opportunities for improvement in the performance and cost effectiveness of lithium ion batteries for electric vehicle applications. Currently, electric vehicle technology state of the art falls short of key performance and cost targets, thereby limiting the market adoption of electric vehicle technology. This project sought to improve upon the current state-of-the-art towards USABC performance and cost goals. Specifically, Quallion sought to develop a high power negative active material, a high energy negative active material, and a proof of concept Matrix™ battery modules using both commercial and custom cells. Additionally, Quallion sought to assess the economic viability of its proposed approach.

In order to achieve commercial targets for electric vehicle price and driving range, significant improvements are needed to improve the capacity and power of electric vehicle batteries. Quallion's work demonstrates significant progress towards these ends through development of novel battery materials and new battery architecture. Improving power capabilities of electric vehicle batteries translates into improved acceleration, faster charging time, and smaller batteries for hybrid vehicles. Quallion developed new anode materials for high power using carbon nanofibers impregnated into soft carbon particles. Improving the energy density of electric vehicle batteries means smaller, lighter and cheaper batteries with longer range for plug in hybrid and electric vehicles. Quallion developed new anode materials for high

capacity using silicon nanofibers within soft carbon particles. Quallion sought to combine the best of both worlds by demonstrating a battery composed of both high capacity and high power cells and modules.

Specifically, in the current effort, Quallion built Li-ion modules with High Capacity (HC) Commercial-Off-the-Shelf (COTS) 18650 cells, High Power (HP) COTS 18650 cells, and with Quallion's HP cells in a patented Matrix™ design. These Matrix™ batteries were characterized, tested, and compared against the United States Advanced Battery Consortium's goals. The intent was to demonstrate the proof-of-concept for a Hybrid Matrix™ battery design that outperforms traditional large format batteries intended for the electric vehicle market particularly with respect to low temperature performance (-40°C), high temperature calendar life (+50°C), and EV and PHEV cycle life. Quallion simultaneously developed new high energy density and high power nanowire impregnated carbon technology and produced test cells to demonstrate the technology. Quallion also conducted a cost study for mass production of the Matrix™ Battery with COTS and Quallion HP cells.

The project was executed in five distinct phases: (A) Cell Electrochemistry Development; (B) Cell Development; (C) Module Development; (D) Battery Development; (E) Testing.

Phase (A): Cell Electrochemistry Development:

The following two tasks were undertaken.

Task 1 – High Power Anode Material

The development of a high power negative is necessary to meet USABC power requirements. The approach for Task 1 was to incorporate high surface area soft carbon material and carbon fibers into its negative electrode. The use of fibers yields an ultra high conductivity for the electrode, thereby increasing power.

In summary, Quallion evaluated electrochemical performance data with half cells to study the effects of CN amount and carbonization temperature. Quallion determined that increasing CN concentration improves the power and rate capabilities, and higher carbonization temperature, especially above 900°C also improves the power performance. Quallion demonstrated that the new CN-SC material carbonized at 1,000°C with CN 4% outperformed MCMB 6-28. In full cell testing, higher than 3,000W/kg capability was calculated both for charge and discharge. The material has been evaluated in pouch cells and has achieved 324W/kg in a small proof of concept cell.

Task 2 – High Capacity Anode Material

The development of a high capacity negative material is based on inclusion of metal nanofibers into carbon particles. Under consideration for the high energy materials were combinations of tin and silicon nanowires. Silicon nanowires with much higher capacities (4,200mAh/g) over tin nanowires (980mAh/g) were selected to be combined with soft carbon. The preferred content of silicon to achieve a negative electrode capacity of 1,150mAh/g is between 15 to 27%. Electrodes were prepared using silicon nanofibers with soft carbon and subjected to electrochemical testing. The maximum capacity achieved with cycling was initially 380mAh/g, lower than the desired design capacity of 1,005mAh/g.

In summary, Quallion evaluated the role of Si integrated into carbon particles to improve capacity, and ultimately prepared QC-1 using smaller Si, which showed higher capacity and better cycle life than the previous QC-A (January 2012 sample). The Si utilization during cycling was improved with QC-1, and it was selected for small pouch cell assembly for cell deliverables. The pouch cell using NCA_QC-1 delivered over 80mAh or 59Wh/kg, corresponding to a calculated 280Wh/kg for Quallion-HP cell form factor. Quallion also

identified the formation schedule as a key contributor to cycle performance, as well as material delamination during cycling.

Phase (B): Cell Development:

Quallion's program involved both COTS lithium-ion cells and Quallion built cells as described below.

COTS Cells – Quallion began the effort by procuring and characterizing COTS cells that would be used in the COTS HC and COTS HP module assemblies. Quallion screened incoming cells and confirmed the manufacturer's stated performance. Quallion selected the Panasonic NCR 18650 cell for the COTS HC module and then Sanyo 18650-SAX cell for the COTS HP module.

Quallion HP Cells – This is the cell that was to be integrated into the Quallion HP module. The Quallion HP cell design was targeted to deliver 1.80Ah (80% of the rated capacity) at a 30°C rate, which is a higher power performance level. After building a small initial run of cells through the prototype R&D process, in July 2012 Quallion completed production of roughly 250 cells using its standard production process, including rigorous quality control inspections. As expected, production yields increased significantly through the formal production process compared to the previous R&D group's cell preparations. HP module components were kitted and assembly jigs and tools prepared in parallel to cell production and testing, and HP module assembly began following the completion of cell testing.

Phase (C): Module Development:

Quallion designed COTS HP and COTS HC modules based on the Quallion Matrix™ design. These designs were developed to provide a compact, lightweight, mechanically-stable module for proof-of-concept purposes; however, the designs were not optimized for thermal management or manufacturability.

Phase (D): Battery Development:

The COTS HC and COTS HP modules were paired to form the battery.

Phase (E): Testing:

Quallion gathered the following four sets of test data in USABC Test Manual format:

- Characterization
- Cycle life
- Calendar life
- Pulse power

The projected performance was compared to USABC goals, as shown in the Gap Chart. Red denotes values less than the desired target; Green fulfills the target.

Conclusions:

Quallion's HP negative material and cell development established performance goals of 8,700W/kg at the materials level and 3,500W/kg at the cell level. The material has been evaluated in pouch cells and has achieved 324W/kg in a small proof-of-concept cell. Quallion's high energy negative material and cell development effort established performance goals of 700Wh/kg at the materials level and 280Wh/kg at the cell level. The program has demonstrated an energy density of 59Wh/kg using small proof-of-concept pouch cells.

Quallion's module development set a variety of performance goals which are shown in the gap charts included in this report. Quallion's module and battery designs showed mixed results in meeting USABC goals. The resulting battery composed solely of COTS-HC cells failed to meet key goals related to cycle life and operating temperature, but these metrics improved when the COTS-HC module was combined with the COTS HP module, and they improved further when the COTS-HC module

GAP CHART AT CONCLUSION OF PROGRAM

Parameter(Units) of fully burdened system	Minimum Goals for Long Term Commercialization	Long Term Goal	COTS HC-COTS HC System	COTS HC-COTS HP System	COTS HC-Q HP System
Power density (W/L)	460	600	636.0	660.0	720.6
Specific Power - Discharge, 80% DOD/30 sec (W/kg)	300	400	369.5	387.3	432.5
Specific Power - Regen, 20% DOD/10 sec W/kg	150	200	228.1	230.8	268.5
Energy Density - C/3 Discharge Rate (Wh/L)	230	300	252.5	229.1	208.5
Specific Energy - C/3 Discharge Rate (Wh/kg)	150	200	146.7	134.5	125.1
Specific Power / Specific Energy Ratio	2:1	2:1	2.5	1.9	2.3
Total Pack Size (kWh)	40	40	43.4	40.3	40.6
Life (Years)*	10	10	5 to 8	10	10
Cycle Life-80% DOD(Cycles)*	1000	1000	500 to 800	1000	1000
Power & Capacity Degradation (% of rated spec)*	20	20	50	30	20
Selling Price - 25,000 units @40kWh (\$/kWh)	<150	100	784	1027	1803
Operating Environment (°C)	-40 to +50 20% Performance Loss (10% Desired)	-40 to +85	0 to +40 20% Performance Loss (10% Desired)	-20 to +50 20% Performance Loss (10% Desired)	-40 to +50 20% Performance Loss (10% Desired)
Normal Recharge Time	6 hours (4 hours Desired) 20-70% SOC in <30 minutes @ 150W/kg (<20min @ 270W/kg Desired)	3 to 6 hours	3.0	3.0	3.0
High Rate Charge		40-80% SOC in 15 minutes	<30 min	<30 min	<30 min
Continuous discharge in 1 hour - No Failure (% of rated energy capacity)	75	75	75	75	75

*Calculated Values

was combined with the Quallion-HP module. However, inclusion of the HP modules did cause the battery to fall short of the energy density targets. In every case, the Quallion

designs did not meet the aggressive cost targets established by the program, and this was more pronounced when HP modules were included in the design.

Quallion identified a benefit from using HP modules in the design; however, this benefit needs to be balanced with other performance requirements to find the optimal battery design. As with many engineering efforts, battery development forces trade offs between cost and performance as well as between energy and power, and energy and life. Quallion attempted to document these parameters in order to help drive optimization of future battery systems.

Future Work:

Quallion recommends that additional research continue to support development of electric vehicle battery technology. In particular, projects should focus on scaling up of the HP materials demonstrated in this project and working to improve the cycle life of the high energy materials demonstrated in this project. Additional module development work should build on the proof-of-concept of a combination high energy-high power hybrid Matrix™ battery and transition this to a more mature, robust design suitable for commercialization.

1.2.1 Computer Modeling Work

- None reported.

1.3 Deliverables/Products Developed

Quallion built Li-ion modules with HC COTS 18650 cells, HP COTS 18650 cells, and with Quallion's HP cells in a patented Matrix™ design. The Matrix™ batteries were characterized, tested, and compared against the USABC's goals.

Quallion simultaneously developed new high energy density HP nanowire impregnated carbon technology and produced test cells to demonstrate the technology.

1.4 Technology Transfer Activities

1.4.1 Proprietary Reporting

- Final Report submitted to USABC on August 7, 2013.

1.4.2 Non-Proprietary Publications and Proceedings

- None reported.

2. Advanced High Performance Batteries for Electric Vehicle Applications

Performing Organization: CobasysSB LiMotive

Project Duration: 1/20/2011 – 2/28/2014

2.1 Executive Summary

The Cobasys contract was terminated by USABC for cause.

In February 2011, Cobasys received a USABC award to investigate the development of high performance batteries for electric vehicles. The proposed ultimate deliverable at project conclusion was a 40kWh technology demonstration Electrochemical Storage System (ESS) that meets or approaches USABC's performance and cost targets. It was determined that new cell technology would have to be developed to increase cell energy density to 200W·h/kg with 400W·h/l with additional development to improve pack performance. The system level development would focus on topics including thermal management, electronics for battery management and packaging. It was expected that a fully assembled system could achieve specific energy and energy density of 150W·h/kg and 230W·h/l, respectively. Through the above mentioned developments, reduced cost should have been realizable such that the cost of a 40kW·h pack when produced at annual volumes of 25,000 units per year approaches \$6,000. However, on September 25th, Cobasys received official notification of program termination from USABC. The following report is Cobasys' representation of the status of the technical development as of the date of termination.

2.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

Current lithium-ion battery cells and packs do not have sufficient volumetric and gravimetric energy density to permit this at a price that would not significantly increase the cost of the automobile. Therefore, the USABC has introduced its goals for battery developments that establish the technical and cost thresholds for commercial feasibility and would be applied for a fully integrated battery pack ready to be installed in an electric vehicle.

Objectives and Approach:

In response to USABC's goals, Cobasys was under award to develop new cell technology to increase cell energy density to 200W·h/kg with 400W·h/l with additional development to improve pack performance. The system level development would focus on aspects including thermal management, electronics for battery management and packaging. It was expected that a fully assembled system could achieve specific energy and energy density of 150W·h/kg and 230W·h/l, respectively. Through the above mentioned developments, reduced cost should have been realizable such that the cost of a 40kW·h pack when produced at annual volumes of 25,000 units per year approached \$6,000.

To address USABC's goals, emphasis was placed on development of cells with 200W·h/kg and 400W·h/l with this project. With conventional lithium manganese oxide (LMO) or nickel cobalt manganese materials, specific energy density near 200W·h/kg is not possible.

Therefore, a parallel strategy was applied as follows for the selection of cathode materials, 1) Moderate NCM with extreme process and 2) extreme NCM with moderate process. As a first step, materials were screened in coin cell and 18650 surrogate cell studies, and then electrode design parameters like electrode packing density, current density and voltage range were investigated using 18650 surrogate cells. Design of the cell as a full cell scale including safety devices were to be decided based on the results of the previous steps. Table 1 summarizes the approach and task areas.

Tasks:

Task 1 – Moderate NCM Cell Development

Moderate NCM (with low nickel content below 50%) is conventional material and widely used with LMO in blended compositions for cathodes in lithium-ion battery. This material is widely available from several suppliers like UMICORE, Toda, 3M, BASF and Ecopro. NCM is becoming increasingly cost effective for lithium batteries for electrified vehicles, although it is still more expensive than LMO. For high energy density EV cells, mod-NCM is a promising material for its higher specific capacity than LMO, and its lower cost than NCA. The sub-tasks involved adjusting nickel content in NCM material, screening for candidate electrolytes and screening of separators.

Table 1. Technical Approach Summary

<i>Objective</i>	<i>Target</i>	<i>Approach</i>
Energy Density	200 W·h/kg 400 W·h/l	<ul style="list-style-type: none"> • Application of Novel Active Material with larger Specific Capacity. • Raising electrode density. • Broadening available SOC range. • Application of lighter packaging material/parts.
Life	1,000 cycles 10 years	<ul style="list-style-type: none"> • Application of Surface coating technique for active material. • Optimizing electrolyte (solvent, additives). • Improvement of electronic conductivity by carbon coating or adding conductive agent. • Optimizing particle shape/ size distribution of active material. • Control of uniform electrochemical reaction in electrodes.
Cost	\$300/kW·h (Pack)	<ul style="list-style-type: none"> • Utilize low-cost, commercially available raw materials • Minimize excess capacity to achieve EOL targets. • Leverage existing supplier base to acquire components, subsystems at high-volume pricing.
Safety	L3	<ul style="list-style-type: none"> • High Temperature separator (Resin with higher Melting point, Composite material-dual layers with a different material such as polyimide and polyethylene) • Ceramic coating technique and optimization of electrode structural design (not to concentrate the heat on a limited area of electrode, we will control porosity, electrode density, coating technique, etc). • Polymerizing shut down material. • Optimization of cell design for uniform heat transfer (change cell size and inner cell space for better heat dissipation). • Mechanical features to improve safety.

Task 2 – Extreme NCM Development Cell Development

The main technical obstacles of Ext-NCM material development that must be overcome are poor rate performance due to low material electron-conductivity (10⁻⁷ S/cm) and poor life performance during cycling and storage. In order to study the main causes of poor life performance of these cells, extensive postmortem evaluation on the 18650 surrogate cells has been performed. According to the analysis results, the capacity fade of the cell during cycle or storage was due to formation of a thick film layer on the anode surface and blocking of separator pores by side-reaction products exacerbated by Mn dissolution and oxidative decomposition of electrolyte at cathode and electrolyte interface. To improve rate performance of the cell Cobasys have tried to optimize the contents and types of conductive agents in the cathode formulation. Although the resistivity of the cathode after electrode formation was reduced by 50% by optimization of a conductive agent, the improvement of rate performance was not significant. Several kinds of countermeasures were tried to improve cell performance of Ext-NCM, but none of those was successful to overcome the weakness from the immaturity of material itself. Moreover, the improvement of the base materials from two major supplier of Ext-NCM material was not significant during the past two years.

Task 3 – Cell Design

Two types of cathode materials were selected for study in this program whose success was seen as key enablers to achieving the challenging targets of the program. Both Mod-NCM and Ext-NCM materials were investigated to verify the possibility of applying these advanced materials along with advanced processing methods and cell designs to achieve the desired performance and economic levels targeted by the USABC. In this program it was determined that cell performance of

approximately 180W·h/kg could be achieved in an EV cell, thus approaching some the specific energy goal of the USABC. However, in addition to specific energy, other performance criteria had to be considered to ensure a result that was well balanced and commercially viable. To determine this, nine criteria for a decision matrix were selected:

- Thermal stability
- Calendar life
- Cycle life
- Energy density
- Rate performance
- Resistance (power/regen)
- Rate capability
- Low temperature performance
- Cost and availability

According to these criteria, three new cathode materials were compared to each other and to today's conventional LMO cathode. Although Ext-NCM appears to have high potential for a high energy density cathode material, its low maturity as evidenced in low life performance, poor electrical conductivity and reduced thermal stability resulted in its elimination as a candidate material for this project. High Ni content Mod-NCM was selected due to its good overall performance the criteria in most categories, while identifying the significant challenges remaining in calendar life and cost. Therefore this material was selected for continuation in this program.

Sample cell delivered in this year as A-sample is not verified and validated how much this cell achieves USABC goals. Two or three rate performance testing (RPT) results during cycle and calendar show good performance of life, but long term performance of life is not clear at present. And as a second step, improvement of safety of cell is main plan for B- sample 1.0 System.

The Cobasys USABC high voltage battery system (HVBS) was designed to maximize energy density while minimizing volume, mass, and cost. The HVBS was composed of 297 mod-NCM Li-ion cells arranged in a 3Px99S configuration. The HVBS consisted of 16 12 and seven 15-cell modules for a total of 23 modules. This configuration was designed to meet the 40kW·h capacity target while achieving the power levels required for the program. Pack mass and volume were considered in order to meet specific energy and energy density requirements.

Task 4 – Module Development

The module's primary function is to retain cells over the life of the entire pack. It is also to prevent cells from moving while in operation and restrict cells from expanding during charge. It also needs to provide mechanical features that allow it to be secured to the housing as well as electrical features that allow them to be interconnected within the pack. The preliminary design of the module was completed with FEA analysis and shock and vibration tests completed on early prototype designs. During those tests, modifications were made to the two plastic side plates to prevent cracking found in earlier tests.

Task 5 – Housing

The function of the housing is to contain all components of the battery pack and to provide mounting points and protection for these components during storage, transport, testing and operation. Under the USABC award, the design of the housing was completed with component drawings generated, except for the

base tray due to a late design change of manufacturing process from injection molding to structural foam. Design optimization for the structural foam process had been started with FEA performed on static and shock loads with preliminary positive results. The housing has met the cost target and is within reach of the mass allocation.

Task 6 – Thermal Management

The purpose of thermal management for the USABC battery pack was to maintain the cell temperature within a temperature range that optimized cell performance, cell life and equalized temperature differences between cells. The system was required to raise cell temperature at during low ambient temperature to improve discharge and charge power performance, reduce cell temperature during usage at high ambient temperatures to improve cell life, and to balance cell temperatures to minimize variations in cell-to-cell electrical and chemistry properties.

Conclusion:

The originally proposed three-year project with Cobasys was to develop and demonstrate next generation Li-ion cells and packaging technology to meet the USABC targets of high energy density and low cost EV battery pack solutions by application of novel active materials and processing techniques. The ultimate deliverable at project conclusion was a 40kWh technology demonstration ESS that meets or approaches USABC's performance and cost targets. However, these goals were not achieved and the USABC contract was terminated for cause.

Gap Analysis Chart (from Cobasys proposal/SOW)

Parameter	Current State-of-the-Art		Proposed		USABC Goal	Gap (vs. state-of-the-art)
	Cell	Pack	Cell	Pack		Pack
Power Density (W/l)	2200	860	1500	747.4	460	None
Specific Power Dsch.- 80%DOD, 30s (W/kg)	690	485	470	358.9	300	None
Specific Power Regen. - 20%DOD, 10s (W/kg)	267	188	200	150	150	None
Energy Density C/3 (Wh/l)	230	82	400	230	230 (system)	148
Specific Energy C/3 (Wh/kg)	110	72	200	152.7	150 (system)	78
Sp. Power / Sp. Energy Ratio	6.3	6.7	2.4	2.4	2	None
Total Pack size (kWh)	-	48	-	40	40	None
Life (Years)	8	8	10	10	10	2
Cycle Life - 80%DOD(cycles)	1000+	1000+	1000+	1000+	1000	None
Power/Cap. Fade (%rated)	<20	<20	<20	<20	20	None
Selling Price - 25,000 units @ 40kWh (\$/kWh)	N/A		250	300	<150	TBD
Operating Environment (°C)	-30 to 60	-30 to 60	-40 to 50	-40 to 50	-40 to 50	10
Normal Recharge Time (hrs.)	8	8	6	6	6	2
High Rate Charge	N/A		20-70%SOC in <30min, 150 W/kg	20-70%SOC in <30min, 150 W/kg	20-70%SOC in <30min, 150 W/kg	None
Continuous Disch. 1 Hr. (%rated capacity)	90+	90+	75+	75+	75	None

Table 7 Gap Analysis Chart

2.2.1 Computer Modeling Work

- As described in the previous section, FEA was used for module and packaging design.

2.3 Deliverables/Products Developed

None, as the Cobasys contract was terminated by USABC for cause.

2.4 Technology Transfer Activities**2.4.1 Proprietary Reporting**

- Final Report was submitted to USABC dated December 14, 2012.

2.4.2 Non-Proprietary Publications and Proceedings

- None reported.

3. High Energy Lithium Batteries for Electric Vehicles

Performing Organization: Envia Systems, Inc.

Project Duration: 11/12/2010 – 5/31/2013

3.1 Executive Summary

The objective of the 30-month USABC program was to develop and integrate high capacity manganese rich (HCMRTM) layered-layered cathodes with commercial graphitic anodes and high voltage electrolytes into high capacity pouch cells (1-40Ah) to meet the minimum long-term USABC goals for electric vehicles. Throughout the project, large capacity pouch cells were to be built to evaluate the development and progress towards meeting the cell goals. Independent validation of the cells by National Laboratories was also planned to take place from the cells developed at the beginning, midpoint and conclusion of the project. The key accomplishments at the conclusion of the project were Envia developing, down-selecting and scaling-up the best cathode, anode and electrolyte formulation to be integrated into large capacity pouch cells. Envia down-selected an HCMRTM cathode after extensive material development with respect to composition, dopants and surface coatings. The down-selected cathode materials were scaled up to kilogram levels and integrated in large capacity cells to support a total of nine project cell builds. Screening and down-selection of a commercial anode was made based on optimized specific capacity, irreversible capacity loss and adhesion strength of the electrode. A new baseline electrolyte was selected based on improved low temperature performance while maintaining similar room temperature cycling stability, power and energy characteristics compared to Envia's high voltage baseline electrolyte.

The project consisted of nine cell builds where 1Ah-20Ah cells were made and tested. At the completion of the project, Envia demonstrated

meeting the gravimetric and volumetric power and energy targets from 20Ah capacity pouch cells. During the USABC project various material development, cell development and cost modeling activities took place with the final cells meeting the energy, power and temperature targets. With respect to DST cycle life and calendar life, the cells continue testing and more data is required for a robust model and prediction to be made.

3.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

On December 2010, Envia started a 2½ year project funded by the United States Advanced Battery Consortium (USABC) via the Department of Energy (DOE) to develop high-capacity and high-energy pouch cells meeting the minimum long-term USABC goals for electric vehicles (EV). The project was comprised of the development, screening and scale-up of advanced cathode, anode and electrolyte materials and the modeling, building and testing of large format pouch cells (1Ah-40Ah capacity cells).

Objectives and Goals:

The goal of the project was to integrate the best-developed layered-layered cathode material with the best down-selected commercial anode material and electrolyte formulation. These components were integrated with other commercial cell components (separator, tabs, pouch material, etc.) and made into cells to meet the aggressive USABC energy, power, cycle life, calendar life, cost and low temperature cell operation targets.

The project consisted of nine separate cell builds to evaluate new materials, cell designs and formation and testing protocols. Cells from three of the cell builds (#2, #6 and #9) were

shipped to national laboratories for independent testing. The three National Laboratories involved in the independent cell-level testing were Idaho National Laboratory testing for energy, power, cycle life and calendar life, Sandia National Laboratory conducting abuse testing and National Renewable Energy Laboratory characterizing the cell thermal properties.

Development Tasks:

The project was divided into different areas of development associated with:

1. *Cathode and Electrolyte Development* – Within the project time period, an optimized cathode composition with appropriate dopant, nanocoating and particle morphology was incorporated and the cathode material was scaled up. Identifying the best electrolyte formulation to achieve the cell target goals was part of the program. In summary, electrolyte #2 showed improved low temperature performance with similar cycling stability, power and energy characteristics when compared to Envia's high voltage baseline electrolyte. Validation of electrolyte #2 took place in multiple cell builds successfully reproducing and verifying the cell improvement. Starting in cell build #6, electrolyte #2 became the new baseline electrolyte to be used in future cell builds.
2. *Anode Material Screening* – the USABC project also involved screening commercial graphite based anodes and down-selecting the most promising to be used in the planned cell builds. As a result of the electrochemical and adhesion data, Envia will continue to use its baseline graphitic anode and water based process for all of the programs cell builds.
3. *Cathode Scale-Up* – Cathode #24 with dopant #12 with improved morphology was scaled-up to 5kg and shipped to Envia's cell fabrication facility to build 1Ah cells for the final cell build #9 of the USABC program.
4. *Cell Modeling, Development and Assembly* – This activity was undertaken to demonstrate that after material development, the cell electrochemical performance meet the USABC target specifications.
5. *Cell Testing* – Cell testing protocols used in this program closely followed USABC testing protocols. Early in the program, Envia obtained most of the test protocols from EVPC (hybrid pulse power characterization for EV's) to DST (dynamic stress test) cycling from Idaho National Laboratory. Throughout the program, various cell formats consisting of 20Ah and 1Ah capacity pouch cells were used.
6. *Cost Analysis and Cell-to-Pack Conversion Factors* – One critical goal of the program is the cell and system level cost targets in \$/kWh. The cost targets for this program were <150 \$/kWh at the system level which translated to <120 \$/kWh on the cell level. Envia firmly believes that innovative materials solutions are critical to make significant reductions in cost. There will always be engineering improvements in the cell and system level that will continue to reduce price, but the improvements will be incremental considering the current status and the aggressive USABC cost targets. In order to assess the benefits of Envia's high capacity cathode materials and high-energy cell design, with respect to system (pack) level costs, Envia contracted the system company Ricardo to understand the relationship between cell level and pack level performance.

Ricardo modeled the battery pack attributes for a 40kWh electric vehicle battery pack by using Envia's 20Ah cell data.

Gap Analysis with USABC Goals:

The gap analysis contains the USABC Systems long-term goals, minimum USABC goals for commercialization and Envia's system equivalent performance from 20Ah and 1Ah capacity pouch cells from cell build #2, #6, #8 and #9. The energy, power and cycle life data presented in the gap analysis is obtained by measuring 20Ah capacity cells with the

exception of the calendar life data (row 8 in the gap analysis table), which is obtained by measuring 1Ah capacity cells. The values presented in the gap analysis have been obtained using a voltage window of 2.2V to 4.35V as the 100% voltage window. The reported values in the gap analysis correspond to system level values where the Envia measured cell values were adjusted by the corresponding cell-to-pack conversion factors. Based on modeling by the system company Ricardo, the projected system (pack) volumetric power and energy is 58% and the gravimetric power & energy is 70% of the cell values.

System Level Gap Analysis Summarizing Data for Cell Build
(table is as submitted by supplier)

Number	Systems Performance Metrics	LONG TERM GOALS	MINIMUM GOALS FOR LONG TERM COMMERCIALIZATION	CELL Build #2 Systems Values		CELL Build #6 Systems Values		CELL Build #8 Systems Values		CELL Build #9 Systems Values	
				BOL	RPT5	BOL	RPT9	BOL	RPT7	BOL	RPT2
1	Power Density @ 80%DOD/30sec (W/L)	600	460	834	374	1881	735	587	NA	911	NA
2	Specific Power @ 80%DOD/30sec (W/kg)	400	300	491	221	1417	548	445	NA	673	NA
3	Specific Power @ Regen, 20%DOD/10sec (W/kg)	200	150	1255	1103	969	826	1088	NA	874	NA
4	Energy Density @ 3h Discharge (Wh/L)	300	230	251	211	193	174	205	NA	206	NA
5	Specific Energy @ 3h Discharge Rate (Wh/kg)	200	150	148	125	146	130	155	NA	148	NA
6	Specific Power / Specific Energy Ratio	2.2	2.2	3.3	1.8	9.7	4.2	2.9	NA	4.6	NA
7	Total Pack / Cell Size (kWh)	40	40	40	40	40	40	40	40	40	40
8	Life (Years)	10	10	NA	1.4 (1Ah cell)	NA	3.5 (1Ah cell)	NA	8 (1Ah cell)	NA	TBD
9	Cycle Life @ 80%DOD (Cycles)	1000	1000	0	450	0	810	0	NA	0	NA
10	Power Degradation (% from Target)	20	20	0	27	0	0	0	NA	0	NA
	Energy Degradation (% from Target)	20	20	0	17	0	14	0	NA	0	NA
11	Selling Price @ 25,000 units @ 40kWh (\$/kWh)	100	<150	325	325	325	325	325	325	325	325
12	Operating Environment (°C)	-40 to 85	-40 to 50	-40 to 50	-40 to 50	-40 to 50	-40 to 50	-40 to 50	-40 to 50	-40 to 50	-40 to 50
13	Normal Recharge Time (hr)	3 to 6	6	4	4	4	4	4	4	4	4
14	High Rate Charge @ 0.50W/kg in 5min	40-80% SOC	20-70% SOC	70%							
15	Continuous Discharge in 1hr No Failure @ Rated Energy Capacity	75	75	95%							
16	Battery Scaling Factor (BSF)	288 (96s, 3p)	288 (96s, 3p)	576 (96s, 3p)	576 (96s, 3p)	576 (96s, 3p)	576 (96s, 3p)	576 (96s, 3p)	11K (93s, 120p)	576 (96s, 3p)	11K (93s, 120p)
17	Battery Capacity (Ah)	40	40	20	20	20	20	20	1	20	1

Conclusion and Future Work Planned:

The project concluded with the development, screening and scale-up of advanced cathode, anode and electrolyte materials and the modeling, building and testing of large format pouch cells (1Ah-20Ah capacity cells). The

program consisted of nine cell builds integrating the best-developed layered-layered cathode material with the best down-selected commercial anode material and electrolyte formulation. Cells from all cell builds were internally tested by Envia and cells from cell

build #2, #6 and #9 were shipped to three National Laboratories (INL, SNL, NREL) for independent testing. Out of a total of nine cell builds, cell testing from cell build #6, #8 and #9 are still ongoing both at Envia and the National Laboratories.

The project saw the development of various cathode compositions, surface modifications and morphologies to engineer a material with high specific capacity, low Mn dissolution, stable voltage behavior, low resistance and good endurance characteristics. Graphite based anode and electrolyte formulations were developed and screened to down-select the best material and formulation able to meet the USABC target specifications. At the conclusion of the project, Envia demonstrated that the gravimetric and volumetric power and energy were met using 20Ah pouch cells. Low temperature energy retention was shown to meet the specifications from room temperature to -30°C. Calendar life and cycle life testing is still ongoing with the latest data from RPT9 from cell build #6 showing greater than 800 DST cycles and a prediction from RPT7 from cell build #8 predicting ~8 years of calendar life with a simple linear extrapolation. Testing will continue to be monitored to verify the predicted cell performance.

The project involved working with a system integration company to model the costs associated with the pack and determine the cell-to-pack conversion factors. The pack related costs for a 40kWh pack were determined to be \$2,626 and volumetric and gravimetric energy and power cell-to-pack multiplication factors found to be 58% and 70%, respectively. Cell cost analysis was also performed determining the cell related selling price to be 258 \$/kWh. With respect to USABC selling price targets of <120 \$/kWh for the cell and <150 \$/kWh for the pack, the targets were not met. Significant

improvements to the materials are still required to be able to reach the aggressive USABC price targets.

3.2.1 Computer Modeling Work

– None reported.

3.3 Deliverables/Products Developed

The USABC program consisted in developing and scaling-up cathode, anode and electrolyte materials and integrating them in large capacity pouch cells. The developed cells were the main deliverable for the project, where upon testing should meet the USABC targets for EVs. The developed pouch cells were tested at Envia with a few selected cells being tested in parallel by three National Laboratories. Cells from the beginning (cell build #2), mid-point (cell build #6) and conclusion (cell build #9) of the project were selected for independent testing. The three national laboratories involved in the independent cell-level testing were Idaho National Laboratory testing for energy, power, cycle life and calendar life, Sandia National Laboratory conducting abuse testing and National Renewable Energy Laboratory characterizing the cell thermal properties

3.4 Technology Transfer Activities

3.4.1 Proprietary Reporting

– Final Report submitted to USABC dated October 23, 2013.

3.4.2 Non-Proprietary Publications and Proceedings

– None reported.

4. Development of Solid-State Batteries with High Energy Density Ranges for Automotive Uses

Performing Organization: Sakti3, Inc.

Project Duration: 8/29/2012 – 2/1/2014

4.1 Executive Summary

Despite multiple requests, a final report has not been provided by Sakti3, hence this project summary was compiled from the original Proposal and Statement of Work submitted to USABC dated July 24, 2012.

Sakti3 proposed a 12-month USABC development program on solid-state batteries with high energy density for automotive use. Sakti3's fungible manufacturing process allows execution of a wide spectrum of electrochemistries. Presently the company is focused on a select number, based on careful simulations, assessments of abilities to address different markets given their needs, and cost targets. Sakti3 has designated the cells which will be tested in this program as "Gen 1" for purposes of identification. Later, they plan to use a trademarked designation for each product generation. Based on computer simulations, Sakti3 cell properties quite comfortably exceed USABC targets in several critical categories, especially in volumetric and gravimetric energy density. The objective of this USABC project is to develop high capacity cells that are capable of achieving 300mAh and energy density of at least 770Wh/l and a power density of at least 800W/l.

4.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

The proposal/SOW states that Sakti3 is commercializing a highly innovative and novel, scalable solid-state battery technology that utilizes a solid electrode and electrolyte in order

to overcome the inherent physics limitations being faced by current state-of-the-art batteries to achieve higher energy density. Sakti's innovative computational models and roll-to-roll production technologies are the basis for transferring knowledge gained from early prototypes to large-scale production.

Objectives and Goals:

The purpose of this development project with USABC funds was to develop high capacity cells that are capable of achieving 300mAh and energy density of at least 770Wh/l and a power density of at least 800W/l.

Approach:

Sakti3's approach is comprised of the following three steps:

1. Execution of a new strategy for roll-to-roll devices which enable higher throughput delivery of bulk materials in Sakti3's present production line.
2. Verification of cell-material properties in the larger configuration of cells manufactured, solved iteratively and as an inverse solution to the models developed previously at the company, while preserving the material recipe in use at the company.
3. Designating and/or hiring dedicated staff to serve the USABC contract to deliver the project reports and following contractual hardware for evaluation:
 - a. 36 1.5mAh cells to Argonne National Lab
 - b. 36 300mAh cells to Argonne.

The technology Gap Analysis is shown in the table below. Sakti3 technology is currently

Gap Analysis of Sakti3 Technology versus USABC Targets

	USABC Minimum EV Goals	Cell Simulation for Gen 1	Current Cell Experimental Performance	Final Program Cell Experimental Goals
Power Density (w/L)	460	610	517 (maximum power @ C/5)	800
Specific power - discharge, 80% DOD/30 sec (W/kg)	300	250	134 (maximum power @ C/5)	250
Specific power - regen 20% DOD/10 sec (W/kg)	150	120 (estimation)	134 (maximum power @ C/5)	120
Energy density - C/3 discharge rate (Wh/L)	230	770		770
Specific Energy - C/3 discharge rate (Wh/kg)	150	310	-	310
Specific power/specific energy ratio	2:1	1:1.24	-	1:1.24
Total pack size (kWh)	40	-	-	-
Calendar Life (years)	10	10	-	10
Cycle life - 80% DOD (cycles)	1000	3000 (Estimation)	-	3000
Power & capacity degradation (% of rated spec)	20	20 (Estimation)	-	20
Selling price - 25,000 units @ 40 kWh (\$/kWh)	< 150	<300	-	<300
Operating environment	-40 to +50	0 to +90	-	0 to +90
Normal recharge time	6 hours	6 hours	-	6 hours
High rate charge	20 to 70% in < 30 min @ 150 W/kg	20 to 70% in < 30 min @ 300 W/Kg	-	20 to 70% in < 30 min @ 300 W/Kg
Continuous discharge in 1h (% of rated capacity)	75	65	-	65

Note: * Understanding that a 40kWh pack itself will not be constructed in this project.

optimized for portable electronic applications, which comprise the first markets for their battery technology.

Tasks:

The following tasks were planned to achieve the goals of this program:

1. Cell Design
2. Scale-Up and Testing

3. Fabrication (in two rounds for delivery to ANL for testing).

Despite multiple requests, no progress reports were received from Sakti3 so the progress towards USABC goals could not be assessed.

4.2.1 Computer Modeling Work

– None reported.

4.3 Deliverables/Products Developed

None received by USABC.

4.4 Technology Transfer Activities

4.4.1 Proprietary Reporting

Despite multiple requests, a final report has not been provided by Sakti3, hence this project

summary was compiled from the original Proposal and Statement of Work submitted to USABC dated July 24, 2012.

4.4.2 Non-Proprietary Publications and Proceedings

– None reported.

Section B – Hybrid Electric Vehicle

Section B – (Power Assist) Hybrid Electric Vehicles (HEV) Final Reports:

1. Saft – FreedomCAR Performance and Cost Demonstration.....	B-1
2. CPI – Lithium Polymer Cell Technology.....	B-5
3. JCI – Lithium-Ion Development for Hybrid Electric Vehicles.....	B-9
4. A123 – Technology for Hybrid Electric Vehicle Applications.....	B-15
5. JC-Saft – Lithium-Ion Cell and System Development.....	B-19
6. EnerDel – USABC/DOE Phase 1 and 2 Projects	B-23

1. FreedomCAR Performance and Cost Demonstration

Performing Organization: Saft America, Inc.

Project Duration: 8/1/2003 – 1/31/2006

1.1 Executive Summary

The challenge of affordable electric drive has been in the forefront of the automotive industry for over a decade. Beginning with the USABC, Saft has been a proud participant in this program partnership with the DOE, Chrysler, GM and Ford, and has successfully developed a Nickel Metal Hydride module for electric vehicles as well as demonstrating the applicability of Lithium-ion technology as a viable high-energy candidate for EVs. Under the Partnership for a New Generation of Vehicles (PNGV) program, Saft's Advanced Systems Division in Cockeysville, MD created a whole new class of high power Li-ion battery technology that not only has been successfully demonstrated in National Laboratories and on OEM HEVs but is now one of the enabling technologies for the U.S. Army's new family of Hybrid Combat vehicles. Saft has incorporated many of the lessons learned from its PNGV contract in launching the first large cell Li-ion serial production program for the U.S. Army's ITAS system.

1.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

Project Objectives and Approach:

The objective was to develop an affordable, high power 42V system, which can be used in fuel cell vehicles. In response to this new challenge, SAFT synergistically leveraged the expertise of all its centers of excellence (Cockeysville MD and Bordeaux, France) to develop 42V MHEV and PHEV batteries. The program was planned to investigate the following seven key areas including:

- Electrochemistry improvement addressing (a) cost effective materials; (b) abuse tolerance; (c) cold temperature performance
- Cell development
- Development of 42V modules: (a) MHEV; (b) PHEV; (c) module pricing gap analysis
- DFMEA & PFMEA on cell and module
- Environmental study
- Life prediction modeling
- Module deliverables.

The following sections summarize the technical studies and findings conducted across six main tasks including cell hardware and liquid cooling module development.

Task 1 – Electrochemistry: Abuse Tolerance & Cost Reduction

Positive Material – NMC-NC5 in sub-scale cells: End of cycle life was reached around 300 cycles showing 22% lower than baseline with NCA for power & energy. During calendar life testing for a period of 300 days in storage @+40°C, power and available energy were found closer to baseline but the RT capacity loss was 5%.

Negative material – GrRSE in sub-scale cells: Both cycle life and regen power were shown to have improvement and the cost of this material is around 33% of baseline material.

Electrolyte – 2LowT: There was improvement in power at cold temperature.

Separator – A non-woven mat coated with ceramic powder was evaluated. The material was considered for safety. The cell made with this separator was able to withstand 150°C exposure with no shrinkage or insulation issue.

During overcharge at high rate, results were unfavorable due to lack of pressure generation required.

Task 2 – Full Scale Cell Development

NMC S-5 material was selected and implemented in making MHEV and PHEV Gen1. Cycle life results were promising but calendar life was not satisfactory. Abuse over charge testing results was below expectation. GrRSE material was accepted for Gen1 based on improved performance during cold temperature discharge. Novel salt demonstrated improved safety but performed poorly during cycling. Approximately 200 each VL20P cells were built with aluminum container with compression seal top assembly for MHEV Gen1 packs. Capacity was around 18Ah and the cell impedance was one m Ω during 250 amps pulse testing. PHEV Gen1 cell design was completed.

Task 3 – Module Development

Liquid Cool – Fifteen each MHEV Gen1 modules were made and shipped to National Laboratories for performance and safety testing as per USABC instruction.

Air Cool – A concept design was completed.

Task 4 – Life Prediction Model

A complete review of aging mechanisms was done. A preliminary model was developed for calendar and cycle life. Dissection of aged cells indicates that both negative and positive materials are stable even after four years in 50°C storage at 70% SOC.

Task 5 – DFMEA and PFMEA

FMEA was done and a preliminary report was issued. No further work was done.

Task 6 – Environmental

The major actors for Li-ion recycling were identified and life cycle analysis performed. Based on the industrial capabilities of the different recyclers, three companies were selected for further contacts: UMICORE, Falconbridge and Toxco. Concerning recycling

possibilities, UMICORE seems to be the most serious industrial way, already recycling HEV batteries and the best available technology for Ni-MH and Li-ion recycling, with a yearly capacity of 4,000 metric tons. Recycling trials of Saft Li-ion cells with UMICORE process were done.

Concerning life cycle analysis, Li-ion batteries have a low environmental impact compared to other EV and HEV battery technologies and, in addition, Li-ion batteries are more advantageous than other batteries from the viewpoint of suppression of CO₂ emission during operation because of higher energy.

UMICORE appears to be a potential vender with a capacity of 5K tons/year with a reasonable feeding system. They have prior experience in recycling non Li-ion HEV packs. UMICORE is ready to handle Li-ion HEV packs.

42V Requirements:

The following table details the mild and high power requirements for a 42V system.

42 Volt Targets (Revised August 2002)	M-HEV		P-HEV	
Discharge Pulse Power (kW)	13	2 sec	18	10 sec
Regenerative Pulse Power (kW)	8	2 sec	18	2 sec
Engine-Off Accessory Load (kW)	3	5 min	3	5 min
Available Energy (Wh @ 3kW)	300		700	
Recharge Rate (KW)	2.6		4.5	
Energy Efficiency on Load Profile (%)	90		90	
Cycle Life, Miles/Profiles (Engine Starts)	150k (450k)		150k (450k)	
Cycle Life and Efficiency Load Profile	Partial Power Assist (PPA)		Full Power Assist (FPA)	
Cold Cranking Power @ -30°C (kW)	8	21V min	8	21V min
Calendar Life (Yrs)	15		15	
Maximum System Weight (kg)	25		35	
Maximum System Volume (Liters)	20		28	
Selling Price (\$/system @ 100k/yr)	260		360	
Maximum OCV (Vdc) after 1 Sec.	48		48	
Maximum Operating Voltage (Vdc)	27		27	
Self Discharge (Wh/day)	<20		<20	
Heat Rejection Coefficient (W/°C)	N/A		>30	
Maximum Cell-to-Cell Temperature Difference (°C)	N/A		<4	
Operating Temperature Range (°C)	-30 to +52		-30 to +52	
Survival Temperature Range (°C)	-46 to +66		-46 to +66	

1.2.1 Computer Modeling Work

– None reported.

1.3 Deliverables/Products Developed

Module Deliverables – Saft delivered 17 each MHEV and PHEV baseline packs and 17 each MHEV Gen1 packs to National Labs. All packs had liquid cooling feature.

1.4 Technology Transfer Activities

1.4.1 Proprietary Reporting

- Final Report was submitted to USABC in January 2006.

1.4.2 Non-Proprietary Publications and Proceedings

- None reported.

2. Lithium Polymer Cell Technology

Performing Organization: Compact Power, Inc.

Project Duration: 2/15/2004 – 8/15/2005

2.1 Executive Summary

The goal of this program was to develop a battery for use in hybrid-electric vehicles using a widely available, less expensive and environmentally benign cathode material. Although this cathode offers several advantages, it suffers from a poor life. Thus, the principal objective of this program was to improve the life of the cells using this cathode material. A number of other issues such as the improvement of cycle life, low temperature performance, abuse tolerance, development of battery module and battery management system were also a part of this program.

A variety of approaches were pursued to improve the cycle and calendar life and improve abuse tolerance of the cell. Research carried out within this program led to the fulfillment of the cycle life target of 300,000 cycles. Using a combination of change in composition as well as additives, we have been able to improve the calendar life of the cell by several folds. The complete characterization and verification of these improvements are still in progress. More importantly, this research also led to the development of materials which will make the cells abuse tolerant, a key concern for the deployment of Lithium-ion batteries in vehicular applications. If the technologies developed in this program are confirmed in the ongoing independent tests, then this research program will have contributed significantly to the development of a high power, long-life and low-cost battery for HEV applications.

2.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

The program was aimed at improving the CPI/LG Chem's carbon/spinel Li-ion polymer cell technology previously benchmarked under a Technology Assessment Program (TAP) program undertaken in 2003. During the TAP program at ANL in 2003, over 160,000 PNGV Power Assist Cycles were obtained before the cells reached the 300Wh limit of calculated available energy. Postmortem analysis at LG Chem, using a combination of analytical tools, revealed the following key issues to be responsible for the cycle life degradation of the G3 cells.

- Decay of anode performance, as exemplified by capacity loss.
- Lithium deposition on the anode.
- Dissolution of Mn ions in the electrolyte and deposition on anode.
- Increase in cell resistance of over 100%.

Objectives and Goals:

The primary focus of this program was cell development, which was supplemented by work on battery module, FMEA and cost modeling. The major focus of the cell work was on improving the calendar as well as the cycle life of the cells.

The specific goals and accomplishments (relative to gap analysis versus USABC requirements) are summarized below:

1. *Improvement of Cycle Life over 300,000 Cycles*

Among all the parameters examined, the replacement of graphite anode with the hard carbon showed the highest impact. Hard carbons possess much better high-current charge capability than their graphite counterparts. This

minimizes the likelihood of localized Li plating, thereby significantly enhancing the cycle life.

2. *Improvement of Calendar Life (interim target of 8 years)*

Between 8-10 years per internal testing was achieved. However, this result has not been accepted by the USABC, since tests in progress at INL indicate a shorter calendar life. A number of approaches were pursued to alleviate the storability issues of LiMn_2O_4 . These include:

- Doping with cations such as Al_3^+ , Mg_2^+ , Ni_2^+ to prevent Mn_2^+ dissolution.
- Surface treatment of spinel to provide a protective coating.
- Blending of spinel with LiNiO_2 and $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$.
- Use of carbon anodes more resistant to Mn_2^+ attack than the graphite anode. This could be either a bare carbon or a carbon coated with a protective layer.
- Use of non-fluorine based salts such as LiBoB or Air Products salt.
- Use of electrolyte additives to scavenge moisture to stabilize cathode.
- Evaluation of alternative cathodes such as $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ and LiFePO_4 .

3. *Increase in Cold-Cranking Capability to 5kW at the End-of-Life (EOL)*

The result was achievement of 5kW at the Beginning-of-Life (BOL) – Needs further improvement. The important result is that the G4.1 or G4.1.1 cells can meet the 5kW target of USABC/FreedomCar. However, CPI still needs to further improve the low temperature performance to meet the cold cranking power target at the EOL. Again, a number of approaches were evaluated to improve the low temperature performance of the spinel cells.

4. *Abuse Tolerance Improvement*

Almost all of the approaches involving the addition of electrolytes, either to the electrolyte or to the electrodes, resulted in losses in initial as well as in cold cranking power. The only difference was when less-flammable solvents (fluorinated) were added to the electrolyte. Although there was some improvement in the power, especially at low temperature, the storage properties of the cells were adversely affected. Significant amounts of gassing was observed during elevated temperature storage of cells using these solvents.

The highlight of CPI's work related to abuse tolerance is the development of a separator at LG Chem which possesses low shrinkage and results in considerably higher thermal stability. The new separator has much lower shrinkage than the conventional separator. This separator is also very effective in retarding hole propagation as anticipated during the temperature rise due to internal short. Improved mechanical strength enables the cells to behave without any safety hazard during nail-penetration and thermal studies.

5. *Perform Cost Modeling*

No results were available.

6. *Perform Cell and Module-Level Design FMEA*

In addition to cell development, this program investigated module design issues that are closely intertwined with cell design choices. The three most important of these are cell interconnection, module thermal control, and Failure Mode and Effects Analysis (FMEA). Under this phase, this was a module design and analysis task, with no hardware development.

7. *Develop Battery-Management System (BMS)*

Under this task, CPI continued the development of model based, Kalman filter approaches to BMS algorithm development. As part of this effort, the development of accurate cell models is very important. This aids in the understanding of the operation of the cell and forms the basis

for system level algorithms designed to estimate SOC, SOH, available discharge and regen power, and status. As new cells became available during this program, test based cell models were developed.

8. *Develop State-of-Charge Algorithms*

During this program, the following objectives were achieved:

- Task 3.2.1: Data Collection
 - OCV versus SOC for various temperatures
 - Dynamic high-rate UDDS over range of temperatures
 - Self-discharge rate tests at different SOC's, temperatures
 - Lifetime tests including power fade, capacity fade (ongoing)
- Task 3.2.2: SOC and Maximum Power Estimation
 - SOC estimation within $\pm 3\%$
 - 10-second look-ahead power estimation
 - Algorithm developed, coded in Matlab and C
 - Sanity checks w.r.t. HPPC
- Task 3.2.3: SOH Estimation
 - SOH factor algorithm developed, coded in Matlab and C, validated.

Future Work Planned:

- Focus on power-limit estimation.
- Validate cell estimates against “gold standard” for a variety of drive cycles, temperatures, SOC's, and so forth.
- Implement on module level and validate.
- Refine, simplify, and streamline algorithms for practical implementation.

2.2.1 Computer Modeling Work

- As described in Section 2.2 (item 8) previously.

2.3 Deliverables/Products Developed

As described in Section 2.2 (item 2) previously.

2.4 Technology Transfer Activities

2.4.1 Proprietary Reporting

- Final Report dated October 27, 2005.

2.4.2 Non-Proprietary Publications and Proceedings

- None reported.

3. Lithium-Ion Development for Hybrid Electric Vehicles

Performing Organization: Johnson Controls Inc. – Automotive Group (Battery)

Project Duration: 1/1/2004 – 10/31/2005

3.1 Executive Summary

In this Lithium-ion development program for HEVs, sponsored by USABC, JCI has met many key program characterization goals during development of this program. Particularly, cycle life and calendar life data accumulated from 8/2005 through 12/2005 have yielded 120,000 cycle life and 6-year calendar life compliance.

The NMC-based chemistry did not yield the expected 300,000 cycles operation nor 15-year calendar life. Additive to the cell electrolyte and separator selection dramatically increased the cycle life three times (40,000 to 120,000 cycles) in the final few months of the program. It is further anticipated that with added electrolyte development that the potential lower material cost and inherently improved abuse tolerance advantage of NMC over NCA can be realized with additional development.

3.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

The Li-ion battery project consisted of the following main tasks:

Task 1 – Cathode and Separator Material Validation

This activity consisted of several sub-tasks:

- Delivery of Gen1 cathode and separator-NMC 1/3 materials from several suppliers have been evaluated in small cells. One type of NMC 1/3 has been made into 10Ah cells. The safety attributes of these cathode materials is still under investigation, but has shown some promising results.

- Evaluation and testing of Gen1 components – Several small Design Of Experiments were done to determine optimized cell composition. The cell composition is still not fully optimized at this time.
- Gen1 process optimization – HEV-108 was explored with TIAX and full 10Ah cells were produced in Hannover using Hannover-produced electrodes and TIAX-produced electrodes on multiple occasions. However, the 10Ah cells performed the same or slightly poorer than existing technologies in terms of cycle life and abuse tolerance testing. Processing of the electrodes proved to be challenging. An acceptable processing method was never completely developed because of gelling issues during mixing and coating process steps.
- Delivery of Gen2 cathode and separator – HEV-118 was billed as an improved version of HEV-108, actually turned out to present more processing issues than HEV-108. Even with the help of a National Laboratory, there were no identifiable advantages to this material. Hence, the Gen2 program focus switched to NMC 1/3 materials because of reported success by the ATD and from product literature which cited specific safety advantages. HEV-118 work was therefore abandoned.
- Evaluation and testing of Gen2 components.
- Gen2 process optimization – All work previously utilized for Gen1 process optimization was directly transferable to Gen2 optimization.

Task 2 – Electrode Development

The following table summarizes final cell materials.

Major Cell Component	Material Type	Supplier Information	Comments
Cathode	NMC 1/3 1.10 Li excess	HC Stark	NMC 1/3 material is a starting point.
	Conductive Additives	Timcal Supper P Li	Necessity of additives to be evaluated.
	Binder	Timcal KS-6	
	Aluminum foil – 20µm Ideally <200 ppm Fe Ideally 360 mm wide	Solvay Solef 20615 Alcan	Increased impurities linked to EOL Al foil corrosion.
Anode	MCMB	Osaka Gas	MCMB is a starting point. Other carbons through to be more effective.
	Conductive Additives	Timcal Supper P Li	
	Binder	Solvay Solef 11012	
	Copper foil – 10 µm	Schlenk	
Separator	Polyethylene (mono-layer)	Asahi 25µm	
Electrolyte	Carbonate based:	Ferro	
	EC:DMC:DEC		
	LiPF6 with VC		
	Ideally very stable		
	Ideally < 10ppm HF		

Task 3 – Cell and Module Testing

Delivery of 12 10Ah cells were made to Argonne National Labs for testing. Argonne performed cycle life and storage tests on these cells. Poor cell performance was confirmed since the chemistry of the deliverables was in preliminary stages of the Gen2 cell-chemistry optimization. To fabricate 30 cells for demonstration testing, a dry room facility of approximately 1,600 square feet was constructed within the JCI Battery Technology Center (BTC) as the cornerstone to the JCI Li-ion Battery Development Laboratory.

Task 4 – Module and System Development

The module design was based upon both the testing and handling requirements of this development program as well as the consideration of how a module or system would be designed for in vehicle use. The following high level critical-to-quality (CTQs) items were considered in this effort where asterisks indicate key areas of emphasis by JCI:

Module Design CTQs

- Provide air cooling/minimize temperature gradient*

- Ensure proper usage of the battery within recommended ranges*
- Maintain electrical isolation*
- Provide easy to use mechanical/electrical connections to allow lab testing*
- Reduce severity of high profile failure modes with a passive integrated system*
- Retain components (crash, vibration requirements)
- DFM (sub-system, vehicle level)
- Meet vehicle packaging requirements
- Meet volume/mass constraints
- Minimize cost (simplicity, fewer parts, scalable, materials, etc.).

The key outcomes of this task were:

- Cylindrical cell design selected over oval prismatic cell design due to present winding equipment limitations. NREL modeling suggests that some increased cooling capability may be feasible by using oval prismatic cell design. Verification of this would require addition

research that is beyond the scope of this program.

- A thermal model of Li-ion cells and modules was prepared by NREL. The model was used to predict the maximum temperature and the temperature uniformity expected during battery operation. The model also assisted in cooling design development.

Task 5 – Abuse Tolerance

Cell testing conducted on 12-18Ah cells through May, 2004 concluded that the cell design was not intrinsically safe with respect to overcharge, independent of active material selection. Cell entry into thermal runaway will result in explosion and/or fire. Overcharge protection was added to the cell design. Below is the overcharge abuse tolerance testing method used, based on the hazard level scale defined by EUCAR:

- Cell at 100% SOC prior to overcharge testing
- Test performed at 25°C
- Overcharge current: 2°C
- Overcharge at 2°C rate until an event occurs
- Event: Thermal runaway, flame or cell open showing zero current
- Continue recording voltage and temperature at least 30 minutes after.

Task 6 – HEV Battery System

Three 12 cell modules were assembled for overcharge testing. A 12 cell module was also assembled and tested at ZSW, Ulm, Germany for response to 1°C and 3°C overcharging. The overcharge resulted in a cell entering thermal runaway and thermal energy propagating other cells in the module. The cell protection

approach was modified to monitor all 12 cell voltages and engage an isolation relay should any cell exceed 4.6V. Re-testing of the module at ZSW, in August, 2005 successfully isolated the module during 1°C and 3°C overcharge tests.

Conclusion:

JCI has met many key program characterization goals during development of this program. Particularly, cycle life and calendar life data accumulated from 8/2005 through 12/2005 have yielded 120,000 cycle life and 6-year calendar life compliance.

The NMC-based chemistry did not yield the expected 300,000 cycles operation nor 15-year calendar life. Additive to the cell electrolyte and separator selection dramatically increased the cycle life three times (40,000 to 120,000 cycles) in the final few months of the program. It is further anticipated that with added electrolyte development that the potential lower material cost and inherently improved abuse tolerance advantage of NMC over NCA can be realized with additional development.

By virtue of an MOU for a joint venture with JCI and Saft Batteries, it is anticipated that by using Saft's NCA cell chemistry and JCI's BMS and cell overvoltage circuitry, that the cycle life, calendar life and abuse tolerance requirements can be met. Four key goals will be carried over into a subsequent USABC Li-ion Development Program Proposal:

- 1) Equipment Operational temperature
- 2) Equipment survival temperature
- 3) Production selling price
- 4) Cold cranking power at -30°C.

The final gap analysis versus USABC requirements is shown in the following table.

Johnson Controls Compliance to 40kW FREDOMCAR HEV Goals

Attribute	@	Units	Power Assist Goals	JCBG, Inc. Status
Goals Under Development				
Cycle Life	50 Wh Cycle	cycles	300,000	120,000
Calendar Life		years	15	6
Equipment Operation Temperature		Deg C	- 30 to + 52	-25 to + 45
Equipment Survival Temperature		Deg C	-46 to + 66	-35 to + 70
Production Selling Price	100,000 units/yr	\$	800	\$2,200
Cold Cranking Power at -30 Deg C	- 30 Deg C, 2 s	kW	7	4.8
Max. Round trip efficiency	50 Wh Cycle	%	90	95
Pulse Discharge Power	30 Deg C, 10 S	kW	40	40
Peak Regenerative Pulse Power	30 Deg C, 10 S	kW	35	35
Total available energy	30 Deg C, C/1	kWh	0.5	0.5
Max Weight		kg	60	48
Max. Volume		liters	45	43
Max. Operating Voltage		Volts	<400	228
Min. Operating Voltage		Volts	> Vmax * 0.55	125
Max Allowable Self-Discharge rate		wh/day	50	<10

3.2.1 Computer Modeling Work

– None reported.

Deliverable met as part of shipping 12 Cell modules to SNL6/1/05

3.3 Deliverables/Products Developed

Final deliverable Date/Comments

- Updated system cost model Transmitted to USABC
Initial cost model sent to USABC 2/04 Month 2
- Preliminary system DFMEA Meeting at USABC
Preliminary DFMEA sent to USABC 1/13/04; reviewed with USABC 6/15/04 Month 2
- Final gap analysis To USABC
Due date moved to End of Program (10/31/05). Final Gap Analysis issued 10/31/05 June 2005
- Cell and module manufacturing cost model submitted 11/30/2005 (assuming 100,000, 60-cell systems per year) December 31, 2004
- Produce 10 11.5Ah cells using Starck NMC
Cells delivered to USABC 12/31/04 December 31, 2004
- 12Ah cells for demonstration testing
Deliverable date revised to 6/1/05 (on 11/10/04) July 30, 2005

- Gap analysis and system price selling projection
June, 2005
Final Gap Analysis and projected selling price issued 10/31/05
- White paper/plan for power fade resolution
June 30, 2005
Issued Plan 6/21/05. Supplemental data issued 7/29/05
- Issue manufacturing white paper
October 31, 2005
Final Manufacturing Summary issued 10/31/05
- Pass AT overcharge test and issue summary report
October 31, 2005
AT test passed 8/23/05, Test report Issued 9/6/05
- Deliver final system FMEA to USABC
October 31, 2005
Module FMEA delivered 10/17/05 to USABC
- Deliver updated warranty model to USABC
October 31, 2005
Updated Warranty Model delivered to USABC 10/31/05
- White paper of cell design for maximum life
October 31, 2005
White Paper Delivered to USABC 10/31/05

3.4 Technology Transfer Activities

3.4.2 Non-Proprietary Publications and Proceedings

3.4.1 Proprietary Reporting

- Final Report to USABC dated 12/30/2005.

- None reported.

4. Technology for Hybrid Electric Vehicle Applications

Performing Organization: A123 Systems

Project Duration: 11/27/2006 – 12/30/2010

4.1 Executive Summary

This program funded the development of a 4.4Ah 32113 cylindrical cell which has achieved over 400k 25Wh cycles while still meeting available energy and power requirements. Continued challenges exist for achieving cold crank and cost requirements, however longer term materials sourcing and technology developments should decrease the gaps on these two targets.

This program also funded the development of an improved and cost reduced cathode production process, and the conceptualization and preliminary development of a 6Ah prismatic HEV cell, with potential for further system level cost reduction. Other novel technologies were evaluated, but could not be realized within this four year program. Several of these technologies will continue to be developed at A123, within the PHEV and HEV LEES programs.

4.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

A123 Systems was awarded funding for the development of an HEV Energy Storage System in December, 2006, in a \$15MM, 50:50 cost share program. Within the program timing and two no-cost extensions, A123 grew from a 250 to a 2,300 employee company, from a single product to a portfolio of cells and packs for multiple transportation applications and from a small startup production facility in China to international manufacturing facilities in the U.S. and Asia.

Program Goals and Objectives:

Development effort was initiated in December, 2006, on an HEV system based on A123's 26650 power cell. Preliminary evaluation of the 26650-based system indicated that requirements for power and energy could be achieved. Cost was a major barrier, however, due to the BSF required to meet performance targets. The program focus was on developing alternative technologies which would result in significant reduction in BSF, and therefore, cost. This program built upon A123's successful launch of a 26650 power cell in 2006, leveraging cathode powder in a cylindrical cell designed for exceptional high rate capability.

Early in the program, A123 efforts were refocused to the development of a newer, larger cylindrical cell as a replacement for the 26650, to improve power, energy, and reduce BSF. Initial assessments suggested that moving to a larger format 32113 cell would result in a 61% reduction in the number of cells required to meet performance targets. Development of a high quality 32113 cell became the predominant program objective, although new smart materials were incorporated into the program objectives, to improve performance and address technical challenges; cold crank was identified as a key challenge early on in the program. Two generations of 32113 cells were evaluated; the second generation was added to address initial issues with cell hardware which impacted cell calendar life. A prismatic HEV cell was also developed, primarily in the last year of the program, and was included in the program deliverables.

The program was concluded in December, 2010, following two no-cost extensions to allow for continued cycle life and calendar life testing for the Gen2 32113 and prismatic HEV cells. The program was completed on budget and all product deliverables were met. By the time the

program had completed, both Gen1 and Gen2 32113 cells had successfully completed the 25kW cycle life test.

Technical Approach:

Key technologies evaluated within the scope of this program included new artificial graphite materials, lithium titanate oxide anode powders, A123's M1x cathode powder, novel nanocomposite separator materials, redox shuttles and flame suppressing electrolyte additives and prismatic HEV cells. Although the resulting 32113 cell did not incorporate all of these new materials, new graphite anode powders, M1x cathode powder, and nanocomposite separator demonstrated sufficient promise to continue development within the USABC funded PHEV cell program. The prismatic HEV cell was added as a program deliverable, and samples were provided to the National Labs for testing. Prismatic HEV cell development is ongoing at A123 Systems.

Initial evaluation of A123's 26650 technology led to the identification of four technical challenges, which this program was designed to address:

- Improved calendar life.
- Increased cycle life capability.
- Increased power.
- Improved abuse tolerance at cell level.

Calendar life and cycle life were addressed through improving both electrode design and cylindrical cell hardware. Both improvements were incorporated into the 32113 Gen2 B1 product which is currently in production. System power was improved to reduce BSF, and was addressed through increasing the cell energy, thus decreasing current density required to support peak pulses, through improved materials and electrode design. Abuse tolerance is not typically a challenge for A123's chemistry, however this was closely monitored to ensure that the larger form factor cell would not introduce new concerns. All of the above

technical challenges were successfully addressed within the scope of this four year program.

Program technology development included: the cathode, anode, electrolyte, separator, and cell design for a cylindrical and a prismatic design.

Tests performed on the deliverable designs included: energy and power, cycle life, calendar life, and abuse tolerance.

32113 Cell Generation Nomenclature and Cell Design Modifications

	Gen-0	Gen-1	Gen-2
Capacity (Ah)	3.4 - 3.6	3.8	4.4 - 4.6
Timing	Up to Q4 2007	Late Q1 2008 Early Q2 2008	First results received DVP&R – Q3'09
Notes	Pre-DV, with original anode material	New Anode - DVP&R 95% complete PV – Q3'09	Using high-power electrode design
	Cells delivered to ANL	Design freeze, this cell validated (will be taken to limited production)	Development continuing. Cells delivered to ANL & NREL
Hardware note	Gen-0: old washer	Gen-1a: old washer (DV1 build) Gen-1b: new washer (DV2 build)	Gen-2, B0.1: new Washer, can neutral Gen-2, B1, Final: can positive

As shown in the 32113 Summary Gap Analysis, the shift from the 26650 to the 32113 design resulted in significant reduction in BSF accompanied by a reduced system pricing estimate. Although the BSF decreased over 70%, the price decrease was only 39% due to the more expensive 32113 cells, and an original underestimate of module and pack costs.

Key Accomplishments:

The 32113 was the second cell which A123 developed and progressed to manufacturing, and was launched during the timeframe of this program.

- Materials selection, electrode design, cell design, hardware and process development were conducted on three product generations, leading to a product which meets USABC performance targets, with the exception of cold crank.
- All required product testing was conducted, through 450k cycles on the

25Wh test, generating over two years of data.

- Calendar life testing was conducted on cells stored at three different states of charge and three temperatures, through up to one year for cells at 23°C, resulting in an A123 projection of 8 years life at 35°C and 10 years life at 30°C.
- A 10 cell HEV module was developed and delivered to the National Labs for testing. Module volume and weight were significantly under program goals.
- A 6Ah HEV prismatic cell was designed and developed to greatly improve on cost, energy, and power targets. Cells were delivered as prototypes to the National Labs for testing.

Gap Analysis for HEV 32113 Cells

Characteristics	Units	USABC	Dec-06	Dec-07	Dec-08	Dec-09	Dec-10
Cell Type			26650	32113 G1	32113 G1	32113 G2 B0.1	32113 G2 B1
Pulse Discharge Power (10S)	kW	25	32.6	25	26	32	343
Pulse Regen Power (10s)	kW	20	26.1	20	20	27.6	289
Available Energy	kW	0.3	0.9	0.35	0.33	0.45	0.6
Round Trip Energy Efficiency	%	>90	95	95	95	95	95
Cycle Life, 25 Wh Cycles	Cycles	300k			>450	>400	(see B0.1)
Cold Crank Power at -30°C	kW	5	4.5	3.1	3.2	3.2	
Calendar Life @ 35°C 30°C	Years	15					8 15.5
Max System Weight	Kg	40	14.3	27	26	24	24
Max System Volume	L	32	12.8	19	18	18	18
Max Operating Voltage	V	<400	775	229	215	215	215
Min Operating Voltage	V	55% nax	428	126	118	118	118
Max Allowable Self Discharge Rate	Wh/day	50	0.07	<3.3	<3.3	0.8	0.8
Equipment Operation Temperature Range	°C	-30 to 52	-30 to 52	-30 to 52	-30 to 52	-30 to 52	-30 to 52
Equipment Survival Temperature Range	°C	-46 to 66	-46 to 66	-46 to 66	-46 to 66	-46 to 66	-46 to 66
BSF	Cells		204	62	58	58	58
Production Price @ 100K Vehicles/year	\$	500	2040	1268	1218	1250	1250

Gap Analysis for HEV 6Ah Prismatic Cells

Characteristics	Units	USABC	Jun-08	Jun-10
Pulse Discharge Power (10S)	kW	25	30	31
Pulse Regen Power (10s)	kW	20	26.1	34
Available Energy	kW	0.3	0.34	0.4
Round Trip Energy Efficiency	%	>90		>90
Cycle Life, 25 Wh Cycles	Cycles	300k		
Cold Crank Power at -30°C	kW	5		5
Calendar Life @ 35°C 30°C	Years	15		
Max System Weight	Kg	40	21	<21
Max System Volume	L	32	12	<12
Max Operating Voltage	V	<400	163	144
Min Operating Voltage	V	55% nax	90	79
Max Allowable Self Discharge Rate	Wh/day	50	<3.3	0.8
Equipment Operation Temperature Range	°C	-30 to 52	-30 to 52	-30 to 52
Equipment Survival Temperature Range	°C	-46 to 66	-46 to 66	-46 to 66
BSF	Cells		44	38
Production Price @ 100K Vehicles/year	\$	500		900

Conclusions:

Program spending was achieved on budget, with two no-cost extensions to cover long term cycle life and calendar life testing. Approximately 50% of the funding was allocated to 32113 cell design, cathode development, and cell builds and testing.

At the outset of this program, A123 was a start-up battery manufacturer, with a single product, the 26650 power cell. During the course of this four year program, a new 32113 cell was developed from concept through production and commercialization as a direct result of funding from the USABC and the U.S. DOE.

4.2.1 Computer Modeling Work

– None reported.

4.3 Deliverables/Products Developed

- 32113 Gen2 B0.1 and B1 cells were provided to the National Labs for testing between March and August, 2009.

- A paper pack study was delivered in January, 2010, with a design constructed around the 32113 Griffin Modules. The paper pack design included a thermal management system, electronics and controls, and estimated costs. Ten of the 32113 Griffin modules were delivered to the National Labs for testing in April, 2010, with a module operating instructions/interface control document.
- Finally, 30 of the 6Ah prismatic cells were delivered in April, with test fixtures for each of the test facilities.

4.4 Technology Transfer Activities**4.4.1 Proprietary Reporting**

– Final Report submitted to USABC.

4.4.2 Non-Proprietary Publications and Proceedings

– None reported.

5. Lithium-Ion Cell and System Development

Performing Organization: Johnson Control – Saft Advanced Power Solutions

Project Duration: 5/22/2006 – 2/22/2009

5.1 Executive Summary

An eight-task research effort was conducted by JCS for Lithium-ion cell and system development, followed by testing of VL6P high power cells at the National Laboratories. A complete program was pursued addressing cell design, materials selection and validation, small-scale process development, module design and performance modeling, cell and module abuse testing, advanced manufacturing and recycling study.

5.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

The following tasks were conducted to execute the project:

Task 1– Cell Design Materials Validation & Integration

The goal of this task was to improve the design of the cell to be able to meet the USABC HEV requirements and especially the low temperature and cold cranking goals. In order to achieve these requirements several components of the cell have been studied for improvement. The general idea was to reduce the cell resistance whatever the temperature or to specifically reduce the low temperature (e.g. $<-10^{\circ}\text{C}$) resistance.

The conclusion of this study is that even if increasing surface area of the carbon is good for the cell power, it contributes to increase the consumption of lithium during storage at elevated temperature, which has a negative impact on the calendar life of the battery. It was decided to go back to the lower surface-area

reference graphite. At the same time the negative electrode material was changed, the design of the electrodes was improved and the thickness was reduced. This had a beneficial impact on the power without the detrimental effect observed with the high surface area carbon. Reducing electrode thickness allowed for an increase electrode area in the cell, thus reducing the resistance, also reducing the resistance per surface of electrode. The new design was applied to the last deliverable of cells for the program. The mechanical design of the cells was also improved to optimize the power/energy ratio of the cell, the vent and insulation for abuse tolerance and the internal connection for the power.

Task 2 – Small Scale Process Development and Validation

Scope: Review and improve processes related to prototype fabrication of cells and modules for consistent products and valid results. The goal was to reproduce Saft NCA baseline cell technology in Milwaukee through all steps of cell construction and formation, as well as to build and deliver full VL-prototype cells to USABC, constructed in Milwaukee and optimized for low temperature performance consistent with 40kW power-assist battery criteria.

The accomplishments included the following:

This was used to improve the prototype process in Bordeaux and Milwaukee, including:

- Incoming inspection
 - Validation of electrode process and mechanical assembly
 - Electrolyte filling cover closing
 - Formation and storage management
 - Documentation of the process.
1. Technology transfer from Saft-Bordeaux to JCI-Milwaukee development lab.

2. Implement processing of Saft cell building technology in Milwaukee.
3. Qualify Milwaukee lab cell building capability with cells having electrical performance comparable to Bordeaux-built cells.
4. Deliver Milwaukee-built low temperature cells to USABC for electrical testing.

Conclusions and Lessons Learned on Task 2:

1. Milwaukee is fully qualified to produce VL6P Li-ion cells based on Saft technology.
2. Li-ion cell fabrication can be successfully accomplished using varied processing equipment for mixing, coating, calendaring, slitting, winding, welding, filling, and formation with minor accommodations for machine differences.
3. Cell impedance at 67.5% DOD is primarily a cathode attribute, which is sensitive to mix and coat processing conditions, including proportions, order of addition, and mix, application, and drying rates.
4. Laser welding part fitment and laser operating conditions have a direct impact on the consistency, strength, and hermetic seal quality of welds achieved.

Task 3 – Module, Battery and Battery Protection and Communications (BPC) Optimization

System Design – Three concepts were presented:

- Interlocking sleeves with axial cooling.
- Radial clamped trays with transverse cooling.
- Axially clamped plates with transverse cooling.

A thermal management study was undertaken to quantitatively estimate the thermal propagation from a cell that has experienced a thermal runaway. This conservative analysis indicates that thermal propagation between cells may not be an issue. Short circuit and overcharge tests of single cells in systems also indicate that thermal runaway may not be an issue. More investigation into details such as heat conduction through bus bars is needed. Also needed is quantitative definition of potential internal short circuits.

A system cost model was also developed and delivered to USABC in September 2008 together with a Cost Roadmap:

- VL6P low temperature cell with all identified cost optimization to design and manufacturing processes
- Developed system design
 - Quoted system components
 - Improved labor and assembly estimates
- Volume updated to 100K systems
 - Increased component prices from lower volume
 - Significantly lower factory utilization and efficiency
 - Recycling not included
- \$1,770 (at an annual system volume of 175K \$1,570).

Task 4 – Cell and Module Performance Testing

The final cell design was evaluated using USABC test manual procedure. The purpose of that task was to demonstrate that the cells were able to meet both the cycle life (300,000 cycles at 30°C) and calendar life (15 years at 30°C). To summarize this task, it has been demonstrated that the new cell design perform well in both cycle life and calendar life. To be able to meet all of the performance requirements of the USABC Hybrid Electric Vehicle Applications

program, a minimum of 72 VL6P cells is recommended.

Task 5 – Cell and Module Abuse Tolerance Testing

The purpose of this task was to demonstrate acceptable cell and system abuse tolerance that would enable Li-ion technology to become a viable HEV battery system solution. The cell design used in the full system delivered at the end of this program was adapted from the baseline, VL7P high-power cell, which had demonstrated acceptable abuse tolerance in testing done during Saft's LION HEART program completed in 2006.

VL6P (B-sample design) cells were delivered to the USABC in February 2007. Abuse tests were run by Sandia National Laboratory in May 2007, and results were reported to JCS in June of 2007. VL6P (C-sample design) cells were delivered to USABC in July 2007. Abuse tests were run by Sandia in October 2007, and reported to JCS in December 2007.

In conclusion, JCS has demonstrated that the VL6P high power cell design demonstrates acceptable abuse tolerance for the JCS full system design. This is accomplished at the cell level by the release gas early and efficiently when the cell is abused (e.g. overcharge, overheat). The gas generation is used to activate the vent and open the current circuit in order to retard or stop further reactions before the cathode decomposes.

Task 6 – Advanced Manufacturing Task

This task will investigate and demonstrate the feasibility specific process improvements of cell manufacturing, with the goal of adding efficiency and thereby reducing cost for serial production. The three significant areas under investigation are: dual-sided electrode coating; high speed calendaring with laser slitting and cell filling through centrifugation. Additional process improvements were investigated under this program.

Task 7 – Recycling Summary

The scope of this task addressed the following as they relate to Li-ion HEV batteries and cells:

- Investigate opportunity for cost reduction on battery production.
- Identify the hazardous nature of associated materials.
- Determine regulatory compliance in U.S. and EU.
- Demonstrate the state of recycling infrastructure.

The baseline of Saft's recycling development investigation for the USABC 42V HEV program used the NCA cell technology.

The JCS gap analysis versus USABC targets is provided in the table below.

Power Assist	EOL Target	Average of 3 cells (Cycle life test at 30°C)				
		BOL	Previous Results	Cur Qtr results	Estimated EOL	Actual EOL
Discharge Pulse Power (kW)	40	54.5	47.8	49.0	46.0	
Regenerative Pulse Power (kW)	35	47.7	41.8	42.9	42	
Available Energy (kWh)	0.5	0.83	0.74	0.69	0.64	
Efficiency (%)	>90	95			95	
Cycle Life (50Wh profile)	300k		180k	240k	300k extrapolated	
Cold Cranking Power @ -30C (kW) (@ 30%SOC)	7	6.8	6.8		6.0	
Calendar Life (Yrs)	15				12 *	
Maximum Operating Voltage (Vdc)	440	270.6	270.6	270.6	270.6	
Minimum Operating Voltage (Vdc)	0.55 x V _{max}	165	165	165	165	
Self Discharge (Wh/day)	50	7.3			N/A	N/A
Thermal Performance @-30°C	10%	7%			N/A	N/A
Thermal Performance @-10°C	30%	23%			N/A	N/A
Thermal Performance @ 0°C	50%	36%			N/A	N/A
Thermal Performance @ 50°C	> 100%	197%			N/A	N/A
Ampere Hour Capacity (Ah)	6.5	6.80	6.42	6.46	6.34	
Battery Size Factor (BSF)	66	66	66	66	66	66
System Cost (est. for BSF=66)	\$1,200	\$1,287	\$1,570	\$1,570	\$1,570	\$1,570
System Volume (L - est. for BSF=66)	45	43	43	43	43	43
System Weight (kg - est. for BSF=66)	46	55	55	55	55	55
* Note: Estimated that a BSF of 72 will result in 15 years of Calendar Life.						

5.2.1 Computer Modeling Work

– None reported.

5.3 Deliverables/Products Developed

As described above in Tasks 1-7.

5.4 Technology Transfer Activities

5.4.1 Proprietary Reporting

– Final Report to USABC dated 2/18/2009.

5.4.2 Non-Proprietary Publications and Proceedings

– None reported.

6. USABC/DOE Phase 1 and 2 Projects

Performing Organization: EnerDel

Project Duration:

5/22/2006 – 7/1/2007 Phase 1

8/22/2007 – 7/31/2009 Phase 2

6.1 Executive Summary

This final report covers the work completed for Phase 1 and 2 of the USABC/DOE program. EnerDel proposed a long term goal of developing a lithium nano-titanate anode ($\text{Li}_4\text{Ti}_5\text{O}_{12}$ or LTO)/manganese-spinel cathode (LiMn_2O_4 , $\text{Li}_{1+x}\text{Mn}_{2-x}\text{O}_4$, $\text{Li}_{1+x}\text{Mn}_{2-x-y}(\text{Metal})_y\text{O}_4$, or LMO) battery technology into mass production for the automotive market. The primary goal of this 38-month program was to demonstrate that the $\text{Li}_4\text{Ti}_5\text{O}_{12}$ /Mn-spinel system could meet the performance, life and cost goals of the USABC program. A secondary goal of the program was to identify failure modes of the cell system through experimentation of active materials and cell packaging designs. More specifically, EnerDel was tasked to determine the root cause of cell power fade at elevated temperatures, and to develop measures that would address the elevated temperature fade problem of the cells of preceding program, Phase 1 deliverables. The program involved three major tasks for cell development, life & safety development, and a module design study. Five generations of HEV cells were developed in the course of the EnerDel programs which are highlighted in terms of their key characteristics and configurations. EnerDel has successfully scaled up the cell capacity from a 2.0Ah CD sized cell to a 4.5Ah A5 sized cell while still maintaining the same safety characteristics.

6.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

Program Objectives and Accomplishments:

The primary goal of this program was to demonstrate that the $\text{Li}_4\text{Ti}_5\text{O}_{12}$ /Mn-spinel system can meet the performance, life and cost goals of the USABC program. A secondary goal of the program was to identify failure modes of the cell system through experimentation of active materials and cell packaging designs. More specifically, EnerDel was tasked to determine the root cause of cell power fade at elevated temperatures, and to develop measures that would address the elevated temperature fade problem of the cells of preceding program, Phase 1 deliverables.

In Phase 1 of the HEV program, EnerDel has been able to demonstrate that the LMO/LTO cell chemistry is capable of achieving 13 years of life at room temperature. This was tested and evaluated by INL in the Gen1 cells. During the testing, the cells demonstrated excellent low temperature performance in addition to delivering high power across a wide usable energy window.

In Phase 2, EnerDel completed a root-cause analysis of the power fade observed in the LMO/LTO cell technology. A solution has also been implemented and demonstrated in the R&D cell configuration which has shown an improvement of the power retention compared to the Gen2.1 cells. This solution has been scaled up and implemented in the Gen4 cell deliverables.

EnerDel has also successfully scaled up the cell capacity from a 2.0Ah CD sized cell to a 4.5Ah A5 sized cell while still maintaining the same safety characteristics.

Task 1 – 4-6Ah Cell Development

During the scale-up process development for the A5 cell, EnerDel needed to optimize the process in order to obtain performance similar to what was achieved in the previous CD size cell format.

Task 2 – Life and Safety Improvement

This task involved life evaluation that included a one-year permeation test, and a longevity study to improve the life of the LTO/LMO chemistry, which showed that the gas generation at elevated temperature is the main cause of the power fade. It was observed in the Gen1 testing conducted by Idaho National Laboratory that significant power fade occurs at 60°C. Less power fade was observed at 45°C compared to 60°C. However, in spite of large power fade at elevated temperatures, Idaho using an Arrhenius plot still projected a remarkable 13-year life at 30°C.

From the key findings described above, EnerDel concluded that the mechanism of the gas generation is through the decomposition of the electrolyte on the surface of the LTO electrode, serving as a catalyst. The active materials have no source of hydrogen and the XRD analysis does not point to any change in their crystalline structure. The only material with a source for hydrogen is the electrolyte. EnerDel conducted a number of tests which showed the gas generation is strongly correlated with the power fade.

Task 3 – Pre-Module and Cost Model Study

EnerDel built a total of 14 pre-modules utilizing 12 Gen3 cells each. The pre-modules were shipped to the various National Laboratories per USABC assignment. The pre-module consists of 12 cells connected in series.

The pre-modules also include a Battery Management System (BMS). The BMS has a modular architecture in that the master controller can interface to one or more sub-packs internally. The BMS maintains a high-speed automotive grade serial communication link (CAN) to each sub-pack.

EnerDel conducted a cost model study to evaluate the cost of an HEV system based on varying volumes of production.

Conclusion:

EnerDel completed Phase 2 activity of the HEV program. This program was divided into three tasks: Task 1) Cell development, Task 2) Life/safety improvement, and Task 3) Pre-module development and Cost model study. EnerDel successfully scaled up the capacity of the 1.8Ah LMO/LTO CD size cell to a 4.5Ah Gen3 cell. This cell demonstrated power equivalent to the CD sized cell while still maintaining the same safety characteristics. The majority of the activity in this program was focused on the life improvement of the LMO/LTO system. In Phase 1 of this program, it was observed that the power fade of the Gen2.1 cells was quite rapid at 60°C. Power fade was also observed at 45°C, but less than that observed at 60°C. In Task 2 activity, the power fade was traced back to the generation of gas caused by the interaction of electrolyte and LTO. EnerDel investigated a variety of methods for a solution and determined that the most efficient and cost effective approach was the use of an additive. The additive demonstrated a significant reduction of gas generation and correspondingly less power fade in the cells stored at 60°C. Gen3 cells were made without this additive for comparison with the Gen2.1 cells. Gen4 cells incorporated this additive and are comparable to the Gen3 cells in performance. The pre-modules were assembled using Gen3 cells. One pre-module consisted of 12 Gen3 cells connected in series. Task 3 also included a cost model study which EnerDel conducted and provided an estimate for the cost of the LMO/LTO HEV system dependent upon the production volumes.

EnerDel has demonstrated in this program that the LMO/LTO system can meet the USABC goals for power and energy, at a competitive cost and unparalleled safety performance.

A central objective of this program to identify root-cause of the elevated temperature power fade was successfully met and demonstrated through a variety of tests and experiments, leading to the development and build of the final EnerDel Gen4 of the LMO/LTO system. The packaging study will continue to be conducted and updated as new data is collected. The table below shows the gap analysis for the cell builds of Phase 1 and Phase 2.

6.2.1 Computer Modeling Work

– None reported.

6.3 Deliverables/Products Developed

A total of 27 Gen3 cells were delivered for evaluation and a total of 29 Gen4 cells were delivered for evaluation. In the final task of this program, Task 3, 14 pre-modules were built and delivered for evaluation.

Five generations of HEV cells were developed in the course of the two phases of EnerDel programs. The Gen1, Gen2 and Gen2.1 cells were a part of the Phase 1 of the HEV program. Gen1 was the first deliverable cell that utilized commercial LTO. The Gen2 deliverable differed

from the Gen1 deliverable since the anode used LTO produced by Argonne National Laboratory. The Gen2 cells were succeeded by Gen2.1 cells with improved tab sealing. In the current Phase 2 program, EnerDel built Gen3 and Gen4 cells for delivery. These cells are larger in capacity compared to the previous generation cells. The 4.5Ah Gen3 cell requires a larger form factor which is termed the A5 size. The Gen3 cell uses commercially available LTO and a standard conventional electrolyte. The final deliverable of this program was the Gen4 cell. The Gen4 cell is a 4Ah sized cell made in the A5 format. The major differences between Gen3 and Gen4 are in the electrolyte composition and in the process improvements made to address the elevated temperature power fade problem.

6.4 Technology Transfer Activities

6.4.1 Proprietary Reporting

– Final Report to USABC dated August 2009.

6.4.2 Non-Proprietary Publications and Proceedings

– None reported.

Gap Chart Comparing EnerDel's Cell Performance of Gen1, Gen2.1, Gen3 and Gen4 with USABC Targets

			HEV Phase I		HEV Phase II		
Characteristics at EOL		USABC	Gen1 BOL	Gen2.1 BOL	Gen3 BOL	Gen4 BOL	
10s Discharge Pulse Power (kW)	kW	25	33.5	28.4	32.81	32.26	Battery Design Studio calculation
10s Regenerative Pulse Power (kW)	kW	20	26.8	22.72	26.25	25.81	Battery Design Studio calculation
Available Energy (kWh)	kWh	0.3	0.46	0.43	0.49	0.55	Battery Design Studio calculation
Efficiency	%	>90	96.6	--	--	--	USABC test manual
Cycle Life (25Wh profile)	Cycles	300,000	--	--	--	--	
Cold cranking power at -30°C	kW	5	5.1	--	--	--	USABC test manual
Calendar Life	year	15	--	--	--	--	
Maximum System Weight	kg	40	0.11 / 15.4	.11 / 13.8	.228 / 12.31	.233 / 15.15	
Maximum System Volume	Liter	32	0.08 / 11.2	.078 / 9.8	.1254 / 6.77	.1457 / 9.47	
Selling Price	\$/system @ 100k/yr	300	--	--	991	991	EnerDel Cost model study
Maximum Operating Voltage	V dc	400	2.9 / 406	2.9 / 406	2.9 / 185.6	2.9 / 237.8	Battery Design Studio calculation
Minimum Operating Voltage	V dc	>0.55 x Vmax	1.6 / 224	1.6 / 224	102.1	130.8	Battery Design Studio calculation
Self Discharge	Wh / day	50	1.8	--	--	--	USABC test manual
Thermal Performance @-30°C	%	10	8.3%	--	--	--	not in USABC manual
Thermal Performance @-10°C	%	30	26.1%	--	--	--	not in USABC manual
Thermal Performance @0°C	%	50	44.9%	--	--	--	not in USABC manual
Thermal Performance @50°C	%	>100	136%	--	--	--	not in USABC manual
Operating & Charging Temperature Range	°C	-30 to +52	-30 to 52	--	--	--	Gen3 is red carried over from Gen2
Survival Temperature Range	°C	-46 to +66	--	--	--	--	
Battery Size Factor			140	125	54	65	

Section C – Plug-In Hybrid Electric Vehicle

Section C – Plug-In Hybrid Electric Vehicles (PHEV) Final Reports

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1. A High Performance PHEV Battery Pack

Performing Organization: Compact Power, Inc.

Project Duration: 12/19/2007 – 3/10/2010

1.1 Executive Summary

The goal of this program was to develop a battery for use in PHEV-10 electric vehicles using a spinel-based mixed cathode, a proprietary mechanically robust separator and laminated packaging. The principal objective of this program was to demonstrate that this system was capable of meeting or exceeding the USABC target of 5,000 cycles and 15-year calendar life. An additional key focus of the program was to develop a battery pack that was mechanically, electrically and thermally robust and abuse tolerant for use in PHEVs.

A total of three generations of cells were developed and tested to establish the above targets. While the 1st generation cell delivered good performance and cyclability, it suffered from poor calendar life. The 2nd generation cell yielded good calendar life but had the drawback of poor cycle life. Drawing from the lessons learned from these two generations of cell development, CPI were able to develop the 3rd generation of cell which is on track to meet the 5,000 cycles target. Although only preliminary, the calendar life data also appear quite promising and needed to be validated in ongoing tests. While considerable improvements were brought about in the cell performance, significant efforts were devoted to developing a novel thermal management system based on the concept of refrigerant-to-air cooling. Several generations of this system were built in the course of the program and CPI was able to deliver six fully functional battery packs to the USABC for testing and validation. This cooling system once fully optimized will be highly attractive for PHEV and BEV applications.

1.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

Objectives and Approach:

The main objective was to develop a Li-ion cell that will meet the energy, power and life requirements of the USABC PHEV 10-mile program. A 15-yr calendar life and 5,000 cycles were the targets for this cell. While addressing these key issues, focus was also directed at evaluating the abuse tolerance and low temperature performance of these cells.

The above cell work was supplemented by studies related to modules leading to the development, testing and delivery of packs to the USABC. These studies were directed at finding a design solution that maximizes the effectiveness of the enclosed cells in terms of performance, life and abuse tolerance, while minimizing system weight, volume, and cost. The emphasis of the pack development work, leveraging the lessons learned from the HEV program with the USABC, was on the analysis, design and test of a novel thermal management system that is reliable, efficient and amenable to facile scale-up, manufacturing and validation. An important objective of this program was to meet or approach the long-term USABC cost-target of \$1,700/unit for the battery pack.

The following major tasks were the focus of the program:

1. Demonstrate 15-year calendar life and 5,000 cycle life.
2. Improve low temperature performance.
3. Evaluate abuse tolerance.
4. Develop a battery pack that is mechanically and electrically robust and most importantly thermally very efficient and reliable.

Task 1 – Demonstrate 15-Year Calendar Life and 5,000 Cycle Life

Four generations of cells were tested in course of this program. These were:

- G4.3 – This was the baseline cell carried over from the development program with the USABC on HEV batteries
- PLG0 – Initial PHEV cell, designed to have high specific energy
- PLG1 – 1st iteration of the PLG0 cells
- PLG2 – 2nd iteration of PLG1 and final cell deliverable.

The PLG2 were found from testing to be the most likely to meet the 5,000 cycle target of the USABC program.

The calendar life studies were conducted by storing the cells at multiple temperatures and certain SOC's while subjecting them to a daily HPPC pulse. The resulting data show improved calendar life for the PLG2 cells.

Task 2 – Improve Low Temperature Performance

The PLG2 cells show cold cranking power very similar to that of the PLG1 cell.

Task 3 – Evaluate Abuse Tolerance

The abuse tolerance of CPI's cells was evaluated by Sandia National Labs and a stand-alone report by SNL is available. In summary, the data show attractive features for the cell. For example, the cell behaved quite well for short-circuit as well as under thermal ramp at 50% SOC. During overcharge, the cell did not undergo runaway conditions till 160% SOC. These tests were followed up by abuse tolerance testing on modules and packs at Sandia.

Task 4 – Develop a Robust, Efficient and Reliable Battery Pack

The PHEV performance requirements and cell characteristics introduce unique challenges, particularly in the area of thermal management and abuse tolerance, related to the higher energy

densities of the cells and their expected operation in charge depleting and plug-in recharge modes over large SOC windows. Using the variable cell module design CPI had developed earlier in the HEV program, a significant and substantial amount of new work was carried out to define, design, build and test PHEV packs having a potentially reliable and efficient thermal management system.

An initial pool of 32 pack cooling concepts were down-selected to 4 thermal management systems using the Thermal Concept Matrix. These were then evaluated further through modeling and construction of proof-of-concept designs and prototype builds.

Conclusions:

CPI developed three generations of a PHEV cell which are believed to be capable of meeting the cycle and calendar life targets of USABC.

Varying the anode and most importantly the electrolyte compositions, CPI has been able to significantly improve the cycle life and also the calendar life of these cells. CPI expected to have these data validated in ongoing tests as well as in tests currently underway in the National Labs.

CPI has also been successful in developing a novel thermal management system which it believed will be more efficient, reliable and cost-effective for use in PHEV and BEV applications. While CPI gave best efforts to optimize this new refrigerant-to-air cooling system, significant opportunities still remain to advance this technology further and they hope to be able to pursue those ideas in a future program.

No gap analysis matrix was submitted by CPI.

1.2.1 Computer Modeling Work

– None reported.

1.3 Deliverables/Products Developed

PLG2 – 2nd iteration of PLG1 and final cell delivered to SNL for testing.

1.4 Technology Transfer Activities

1.4.1 Proprietary Reporting

- Final Report as submitted to USABC on April 16, 2010.

1.4.2 Non-Proprietary Publications and Proceedings

- None reported.

2. Lithium-Ion Cell and System Development for Plug-In Hybrid Electric Vehicles

Performing Organization: Johnson Controls-Saft

Project Duration: 6/16/2008 – 10/31/2011

2.1 Executive Summary

The scope of the USABC project was changed from design optimization of an existing NCA cylindrical cell to a new NMC prismatic cell design. The scope change approval included a 10-month program extension and additional funding. A key goal in the JCS PHEV program was to deliver battery systems/designs that combined Saft/JCS cell technology with JCI automotive system expertise to meet USABC goals of life, cost and high energy density. The redefined program moved from Saft developed NCA-graphite cylindrical cells to new JCS-designed NMC-graphite rigid prismatic cells. This required fundamental development on all technical fronts: electrochemistry, cell mechanical design and system design. In a few short months, JCS generated a proprietary cell and system design, and initial cell hardware builds to support early baseline performance characterization.

The 2010 builds included hundreds of prismatic cells used for baseline deliverables, characterization, life testing, as well as module and system builds for internal evaluation and design verification. Builds included wound and stacked electrodes for direct comparison of the two formats. Cylindrical and prismatic cells using identical NMC electrodes were also fabricated, enabling a unique comparison of the decoupled impact of form factor on degradation rate. The projected system-level energy density improvement was achieved. The final pre-scope-change gap analysis for the 10-mile AER system predicted an end-of-program volume of 84 L. The current end-of program projection for the production-intent prismatic system with

twice the range (20-mile AER) is 71 L. Abuse tests of prismatic cells have also yielded promising results, showing tangible improvement over Phase I counterparts. JCS has demonstrated rapid engineering response in pursuit of what will be a fundamentally new and compelling product to add to their portfolio of offerings for the automotive sector. JCS plans to commercialize the technology developed within this program in 2013.

The new prismatic cells were subsequently utilized in the design of a 20-mile AER PHEV system. Two bench test systems were delivered to the National Labs for validation testing of cells and systems. In parallel, JCS developed commercial-intent designs for 20-mile and 40-mile AER PHEV systems. JCS delivered baseline prismatic cells in November, 2010 and improved prismatic cells in April, 2011 to the National Labs for validation.

2.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

The initial Statement of Work for this development program funded JCS for a duration of two years. In June, 2009, the scope of the project was changed from design optimization of an existing NCA cylindrical cell to a new NMC prismatic cell design. The scope change approval included a 10-month program extension and additional funding. A central goal in the JCS FreedomCAR PHEV program was to deliver battery systems/designs that combined Saft/JCS cell technology with JCI automotive system expertise to meet USABC goals of life, cost and high energy density.

Approach:

The development tasks were executed in seven main areas to achieve the project goals:

Task 1 – Cell Electrochemistry Development and Testing

The first part of the program focused on LiNi_{0.8}Co_{0.1}Al_{0.1}O₂ (NCA) –graphite optimization work and a comprehensive abuse tolerance evaluation, including characterization of cell response to the new blunt nail test. A new LiFePO₄ (LFP) chemistry was developed. At mid-program it was decided, in agreement with USABC, to change the scope of the program to further the development of a prismatic LiNi_{0.8}Mn_{0.1}Co_{0.1}O₂ (NMC) cell, which was better suited for the prismatic format and energy density target. The development of the NMC electrochemistry began by comparing the behavior of NMC material to NCA when used in the identical cylindrical cell design. As soon as the behavior of the chemistry was understood in the cylindrical format, it was then evaluated in the prismatic cell. Throughout the program, several cell formats and chemistries were also tested for abuse tolerance.

Task 2 – Cell Mechanical Development

The program aimed to develop a robust mechanical design for a prismatic cell suitable for high volume production. In addition to cell design, interconnection at the cell and module levels, and possible Current Interrupt Device (CID) concepts were evaluated. JCS' initial prismatic design served as an electrochemistry test vehicle. The design progressed through several design iterations supported by prototype builds, culminating in a cell design intended for 2013 production. JCS developed three main iterations of the cell design with stacked and wound electrodes. JCS also built several wound cells using the existing cylindrical cell winder. The 2013 Production Cell is an improvement from the A-sample that begins to optimize the design for cost, manufacturing, and energy density. Lessons learned from the A-sample

build will be applied to this cell with a target of high volume manufacturing.

Consistent with the USABC/DOE funding program, JCS has significantly advanced the development and design of its NMC prismatic cell. JCS is on target with the roadmap to close the gap in energy density between the stacked and wound cells, having already achieved over a 20% increase of wound cell energy density from the proof-of-concept design. JCS is meeting the abuse tolerance target to a EUCAR 4 or better level in testing. Several concepts were evaluated to improve the robustness of the cell interconnections and reduce cost, compared to screw terminals and bus bars.

Task 3 – PHEV Battery System Development

Several iterations of the PHEV battery systems were developed and evaluated: bench-test system, commercial-intent system, and scaled commercial-intent system.

Task 4 – Thermal Management Development and Testing

The thermal management design objectives set out at the onset of the program were achieved, namely:

- a) Designed the thermal interfaces to minimize temperature gradient within the cells in the module and battery pack. The maximum temperature gradient predicted and test values were better than predicted results for straight flow design concept.
- b) Packaged cell modules in a housing that separates the thermal management system from prismatic cells in the modules. This approach ensured that when a cell in the pack vents, the vent gases are contained in the housing and separated from thermal management coolants (air or liquid). This objective was achieved because the battery pack was built and tested with good thermal test results showing that the concept can

should be considered for future development programs.

- c) Evaluated the thermal design of a prismatic cell module to investigate whether thermal propagation would occur if any individual cell went into thermal runaway. The results obtained from thermal propagation testing validated the concept of isolating the cells in a sealed containment housing. The thermal mass of cells adjacent to a cell undergoing thermal runaway reduces risk of propagation.
- d) Designed the thermal management system in such a way that the battery pack may be easily interchangeable between liquid or air cooling.

Task 5 – System and Module Performance Testing

The deliverable systems for ANL and NREL were designed to be bench test only systems which utilize electrical components, electronics, and software from other JCS programs. The use of proven components and software was intended to provide a reliable platform on which to evaluate the performance of prismatic NMC cells and a new thermal management concept.

Results:

- 72 cell PL25M NMC prismatic bench test system built.
- System utilized software, electronics, and electrical hardware from previous cylindrical NCA based JCS programs, which were successfully adapted to a prismatic NMC system.
- Thermal management concept models validated by system level tests.

The cycle life testing performed yielded no significant decrease in system capacity. While an increase in system resistance and reduction in pulse power capability was observed, the system still exceeded the BSF (battery size factor) scaled USABC goals by a significant margin.

More important, trends suggest that, due to the expected impedance growth stabilization and sizeable power margin, life testing will not be prematurely terminated as a result of this issue.

The 72 cell PL25M NMC prismatic bench test system, utilizing many existing JCS system components and proprietary software, provided a sound platform on which to evaluate its PL25M cell in a system configuration as well as assessing a new thermal management concept. The system completed functional, power capability, cycle life, and thermal performance testing at JCS as anticipated, giving confidence that the deliverable systems for ANL and NREL were ready for outside testing. This system's energy, power, and life capabilities will continue to be evaluated at ANL and JCS and its thermal management concept will be further evaluated at NREL.

Task 6 – Cell Manufacturing Process Development and Facilities Expansion

Throughout the program, electrode and assembly development activities were pursued to produce early prismatic prototypes for cell and system evaluations. Utilizing the existing processing equipment in the Milwaukee Development Line combined with process improvements, cell capacity and reliability were significantly improved to provide reliable cells for evaluations. Facility expansion in Milwaukee is in the final stages of completion to allow for additional development tools to support ongoing cell development. Pilot line construction is under way with operation expected by early 2012 that will allow for significantly larger quantities of cells to support future product launches.

Task 7 – Cost Model

JCS has made significant achievements in cost reductions for both systems but are still not able to reach the stretch objective of cost targets identified by USABC. The 20-mile system target unit was \$2,200 (\$260/kWh) and the 40-mile system target unit cost was \$3,400 (\$200/kWh). Meeting the USABC objectives

will require breakthroughs in some fundamental manufacturing processes that are still being addressed by the internal teams and raw materials to provide lower pricing for the most costly part of the system.

Conclusions:

The JCS USABC PHEV program delivered successes on numerous fronts. In a relatively short 2-year period of time, JCS has developed a completely new prismatic cell format, using a new NMC electrochemical technology and developed an associated new system design concept. In the process, JCS gained the capability to internally manufacture prismatic cells, in larger quantities with consistent quality. In short, this program has allowed JCS to feed the genesis of a new product portfolio, which will significantly improve their competitive position.

Sixty-three cells were delivered to USABC for National Laboratory validation testing, including: cylindrical NCA cells in 2008, cylindrical NMC and prismatic NMC baseline cells in November 2010 and prismatic NMC cells in April 2011. All cells used wound electrodes and hard-shell casing. The early direction was to ultimately pursue a stacked-electrode design, which would have yielded a higher cell capacity and therefore, a lower BSF. In December 2011, a decision was made to prioritize the wound-electrode design, most

specifically due to equipment constraints and also in consideration of future high volume manufacturability.

It was agreed with USABC to produce a bench-test system for an end-of-program deliverable, instead of an optimized commercial-intent design. The system was designed for a BSF of 72, based on early calculations (and capacity assumptions consistent with the original stacked format). Once the decision was made to utilize wound-electrodes in the cell, the optimized design for 20 and 40-mile range system was scaled to an achievable BSF of 84. Systems delivered to USABC for National Laboratory validation testing included: a baseline system using 88 cylindrical NCA cells in December 2008 and two final bench-test systems using 72 prismatic NMC cells.

There were several technical barriers to overcome in the development process, the greatest of which were an entirely new form factor and an entirely new electrochemistry for JCS. After a lengthy first cell build at partner-Saft's facility, JCS has in one year developed design resolutions to those barriers to the extent that the material components of the prismatic cell are near the final definition. Of course, material and design refinement opportunities remain and will be aggressively pursued in the short term to deliver a robust, production-ready cell technology.

Gap Analysis with USABC Goals (shown for 20-mile PHEV and 40-mile PHEV)

Gap Analysis - JCS (20 Mile PHEV)			10-Mile	20-Mile						
Characteristics	Unit	USABC Goal	Pre-Scope Change VL22M NCA	Baseline VL22M NMC Gen0 (BOL)	Current VL22M NMC Gen0 (at 1500 cycles)	PL25M NMC Gen1 (Coated Foil) (BOL)	PL25M NMC Gen1 (Coated Foil) Current at 600 cycles	PL25M NMC Gen1 (BOL)	PL25M NMC Gen1 (Projected EOL)	Commercial Intent Prismatic (Projected EOL) *
2s Discharge Pulse Power	kW	45	122	96	79	89	88	105	88	80
10s Discharge Pulse Power	kW	37	99	91	61	82	82	101	80	74
10s Regen Pulse Power	kW	25	80	35	31	39	32	43	30	30
Available Energy for CD Mode, 10 kW	kWh	5.8	3.4	6	5.9	6.3	6.3	6.7	>5.8	5.9
Available Energy for CS Mode	kWh	0.3	0.5	3.4	3.4	3.4	3.4	3.4	3.4	>0.3
Min Round Trip Energy Efficiency	%	> 90	90	90	90	90	90	90	90	90
Cold-Cranking Power at -30 deg C	kW	7	7	9	9	9	9	9	>7	>7
Charge Depleting Cycle-life	Cycles	5,000	<5,000		<4000		600		5000	TBD
Charge Sustaining Cycle Life, 50 Wh Profile	300k		>300000		>300000		>300000		>300000	>300000
Calendar-life (At 35 deg C)	Years	15	TBD		<15		** 12 - 15		** 12 - 15	** 12 - 15
Maximum System Weight	kg	70	105							107
Maximum System Volume	Liter	47	88							71
Selling Price/System @ 100k/yr	\$	2200	3,080 - 3370							3477
Maximum Operating Voltage	Vdc	≤ 400	361	437	437	470	470	470	470	353
Minimum Operating Voltage	Vdc	≥ 0.55 V _{max}	220	260	260	280	280	280	280	210
Self-discharge	Wh/day	50	4.2	<50	<51	<51	<51	<52	<50	<50
System Recharge Rate at 30 deg C	kW	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4
Operating Temperature Range	°C	-30 to 52	-30 to 52	-30 to 52	-30 to 52	-30 to 52	-30 to 52	-30 to 52	-30 to 52	-30 to 52
Survival Temperature Range	°C	-46 to 66	-46 to 66	-46 to 66	-46 to 66	-46 to 66	-46 to 66	-46 to 66	-46 to 66	-46 to 66
Battery Size factor (BSF)			88	104	104	112	112	112	112	84

** Based on 6 months of calendar-life data using a similar cell (coated vs. uncoated foil)

Gap Analysis - JCS (40 Mile PHEV)			10-mile:series	20-mile: 2 parallel-1 series						
Characteristics	Unit	USABC Goal	Pre-Scope Change VL41M NCA	Baseline VL22M NMC-Gen0 (BOL)	VL22M NMC-Gen0 Current Results	PL25M NMC Gen1 (Coated Foil) (BOL)	PL25M NMC Gen1 (Coated Foil) Current at 600 cycles	PL25M NMC-Gen1 (BOL)	PL25M NMC-Gen1 (Projected EOL)	Commercial Intent Prismatic (Projected EOL) *
2s Discharge Pulse Power	kW	46	46	108	97	103	103	113	105	92
10s Discharge Pulse Power	kW	38	82	105	89	100	100	111	101	89
10s Regen Pulse Power	kW	25	25	71	63	78	64	86	60	60
Available Energy for CD Mode, 10 kW	kWh	11.6	11.2	12	11.8	12.7	12.7	13.4	>11.6	11.8
Available Energy for CS Mode	kWh	0.3	0.3	6.8	6.8	6.8	6.8	6.8	6.8	> 5
Min Round Trip Energy Efficiency	%	> 90	90	90	90	90	90	90	90	90
Cold-Cranking Power at -30 deg C	kW	7	7	9	9	9	9	9	>7	>7
Charge Depleting Cycle-life	Cycles	5,000	<5,000		<4000		600		5000	TBD
Charge Sustaining Cycle Life, 50 Wh Profile	300k		>300000		>300000		>300000		>300000	>300000
Calendar-life (At 35 deg C)	Years	15	TBD		<15		** 12 - 15		** 12 - 15	** 12 - 15
Maximum System Weight	kg	120	153							218
Maximum System Volume	Liter	80	165							128
Selling Price/System @ 100k/yr	\$	3400	5170							6368
Maximum Operating Voltage	Vdc	≤ 400	369	470	470	470	470	470	470	470
Minimum Operating Voltage	Vdc	≥ 0.55 V _{max}	225	280	280	280	280	280	280	280
Self-discharge	Wh/day	50	4.2	<51	<51	<51	<51	<51	<51	<51
System Recharge Rate at 30 deg C	kW	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4
Operating Temperature Range	°C	-30 to 52	-30 to 52	-30 to 52	-30 to 52	-30 to 52	-30 to 52	-30 to 52	-30 to 52	-30 to 52
Survival Temperature Range	°C	-46 to 66	-46 to 66	-46 to 66	-46 to 66	-46 to 66	-46 to 66	-46 to 66	-46 to 66	-46 to 66
Battery Size factor (BSF)			96	208	208	224	224	224	224	168

** Based on 6 months of calendar-life data using a similar cell (coated vs. uncoated foil)

2.2.1 Computer Modeling Work

- Computer modeling and predictive analysis were used mainly in Task 5 to perform thermal management studies of prototype cells.

2.3 Deliverables/Products Developed

- JCS deliverables to USABC are listed in the table below.

JCS Deliverables to USABC Based on Original and Amended SOW

	USABC Program Deliverables	Status	Due	Actual	Comment
1	Deliver (1) Baseline PHEV hardware system based on VL22M (BSF=88).	CLOSED	10/31/08	12/1/08	Received at ANL; testing underway
2	Submit a report of baseline VL22M nail penetration characterization test summary.	CLOSED	11/26/08	12/2/08	Review at Quarterly Meeting
3	Present a summary of VL22M power fade characterization testing.	CLOSED	12/23/08	1/28/09	Review at Quarterly Meeting
4	Present a performance summary of 10-mile baseline system	CLOSED	1/30/09	4/15/09	Review at Quarterly Meeting
5	Present a comparison of NCA and LiFePO ₄ chemistry summary.	CLOSED	7/31/09	7/15/09	Review at Quarterly Meeting
6	Present a Packaging and Thermal Management Study of Prismatic vs. Cylindrical Systems.	CLOSED	10/15/09	10/15/09	Review at Quarterly Meeting
7	Deliver Cylindrical VL22M NMC Cells (with re-tuned vent) for National Labs	CLOSED	11/1/10	11/12/10	Build same electrodes as first prismatic. 2 months of testing.
8	Deliver a report of a 20-Mile system thermal design review summary.	CLOSED	10/15/10	4/14/11	Presented at each Quarterly Review. Final on 14-Apr.
9	Deliver baseline prismatic cells for 20-mile PHEV application.	CLOSED	11/1/10	11/15/10	Build in Aug-2010. 2 month of testing.
10	Present Abuse Tolerance Summary.	CLOSED	1/14/11	4/13/11	Presented at each Quarterly Review. Final on 13-Apr.
11	Deliver a report of a 40-Mile system thermal design review summary	CLOSED	4/11/11	4/14/11	Presented at several Quarterly Review. Final on 14-Apr.
12	Delivery of thermal response modeling case output.	CLOSED	4/11/11	4/14/11	Presented at several Quarterly Review. Final on 14-Apr.
13	Present performance comparison summary of NMC and NCA.	CLOSED	1/15/11	4/13/11	Presented at each Quarterly Review. Summary on 13-Apr.
14	Present a summary of Prismatic cell power fade characterization testing.	CLOSED	4/11/11	4/13/11	Moved to accommodate more months of data
15	Deliver improved prismatic cells for 20-mile PHEV application.	CLOSED	4/29/11	4/29/11	Build in progress. 1 month of testing.
16	Deliver a 40-mile system paper design proposal.	CLOSED	4/4/11	4/14/11	Present final design at April Review, based on 20-mile cell
17	Deliver (2) 20-mile PHEV systems: 1 system each to ANL and NREL.	CLOSED	4/18/11	5/3/11	Build in progress. Discuss cell RPT data at Review.
Hardware Deliverables					
Due	Description	ANL	Sandia	NREL	Comment
Dec-08	1 Baseline PHEV (VL22M) System (delivered)	1	0	0	Delivered
Nov-10	20 VL22M-NMC Cells (delivered)	10	0	10	Common NMC electrode
Nov-10	20 Baseline Prismatic Cells (delivered)	10	0	10	Common NMC electrode
Apr-11	45 Improved Prismatic Cells (delivered)	15	15	15	uncoated cathode
Apr-11	2 PHEV 20-mile Systems (delivered)	1	0	1	coated cathode

- Sixty-three cells were delivered to USABC for National Laboratory validation testing, including: cylindrical NCA cells in 2008, cylindrical NMC and prismatic NMC baseline cells in November 2010 and prismatic NMC cells in April 2011. All cells used wound electrodes and hard-shell casing.
- Systems delivered to USABC for National Laboratory validation testing included: a baseline system using 88 cylindrical NCA cells in December 2008 and two final bench-test systems using 72 prismatic NMC cells.

2.4 Technology Transfer Activities

2.4.1 Proprietary Reporting

- Final Report submitted to USABC dated June 10, 2011.

2.4.2 Non-Proprietary Publications and Proceedings

- None reported.

3. PHEV Battery System

Performing Organization: EnerDel

Project Duration: 2/18/2008 – 12/31/2009

3.1 Executive Summary

EnerDel has developed a lithium-ion cell that employs a 5V spinel cathode material with the spinel LTO and demonstrated the chemistry's capability in a PHEV application, that can meet the 10-mile PHEV requirements while maintaining long life, excellent safety and low cost. The chemistry for the system is an $\text{Li}_4\text{Ti}_5\text{O}_{12}$ anode coupled with a high voltage cathode, $\text{LiMn}_{1.5}\text{Ni}_{0.5}\text{O}_4$. The combination of a high voltage cathode material enables an increase in the overall operational voltage of a cell utilizing the LTO anode material. This enables an increase in the energy density of the system thus enabling an LTO based lithium-ion cell for higher energy applications. ANL has demonstrated that the high voltage LNMO cathode material can be synthesized in batches that are larger than laboratory scale. ANL has delivered to EnerDel batches that have been more than 10kg in size. This quantity of material was necessary in order to utilize EnerDel's prototype coating machines. The material was also treated to have a surface coating which improved the stability of the material and reduced the possibility of oxidation of the electrolyte when operating at potentials greater than 4V.

3.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

Objectives and Goals:

EnerDel's objective under the USABC award was to develop a new battery system based on a novel chemistry, that can meet the 10-mile PHEV requirements while maintaining long life, excellent safety and low cost.

These objectives were met by work performed under the following four tasks.

Task 1 – Cathode Material Scale-Up

EnerDel received a total of eleven material deliverables for the cathode material $\text{LiMn}_{1.5}\text{Ni}_{0.5}\text{O}_4$ from ANL. Overall, all the batches show consistent physical properties. The material scale-up was successful.

Two LNMO materials from outside vendors, LNMO-1 and LNMO-2 have been tested and compared to ANL LNMO. ANL's surface coated material has demonstrated its superior performance to the commercially available LNMO materials. While ANL LNMO shows the best overall electrochemical performance, LNMO-1 does show some good potential, especially its high reversible capacity. If surface modification is properly applied on LNMO-1, some of the drawbacks of LNMO-1 material, e.g. limited cycling performance, can possibly be eliminated. However, LNMO-2 does not show any appealing electrochemical performances. From the physical and electrochemical data of LNMO-2, its synthesis process may need further optimization. EnerDel has therefore continued to use the ANL LNMO material for its cell deliverables. This material was used in the final deliverables of the 40 cells.

Task 2 – Electrolyte Development

In order to fully realize the potential of the LNMO/LTO system, EnerDel evaluated electrolyte systems that were capable of withstanding the high operating potential of the LNMO cathode material. ANL focused on developing an electrolyte with a sulfolane solvent based system. EnerDel evaluated ionic liquids, fluorinated solvent systems, higher purity electrolytes, and additives. The evaluation of these electrolyte systems was first characterized via Linear Sweep Voltammetry (LSV) and then later assembled into LNMO/LTO cells in order to determine the

stability upon cycling. EnerDel assembled and prepared the final deliverables utilizing a conventional electrolyte system after determining that the new electrolyte systems could not provide enough stability at this time. With a negative capacity limited design, EnerDel was able to utilize conventional electrolytes to provide stable cycling for the LNMO/LTO system. However, EnerDel continues to evaluate solvent systems in order to more fully enable and realize the full potential of the LNMO/LTO chemistry.

During this program, many electrolytes designed for high voltage cathode were evaluated. From linear sweep voltammetry study only two electrolytes were stable enough to be used as possible candidates for the LNMO electrode: high purity electrolyte and fluoro solvent 1 electrolyte. When cycled in LNMO/LTO full cells, the two selected electrolytes did not perform well. Only 50% capacity retention was obtained after 50 cycles. A possible explanation is that the LNMO electrode plays a catalytic role and reduces the stability in oxidation for the two electrolytes. Also, the low purity of the fluoro-alkylcarbonates solvent can contribute to the poor performance of the fluoro solvent 1 electrolyte.

Task 3 – Cell Development

Once EnerDel understood the effects and requirements for processing the LNMO material, EnerDel delivered 20 CD cells in December and 20 CD cells in March to complete the deliverables for the USABC PHEV program. EnerDel will continue development of this system by improving the electrolyte and material capacity.

Task 4 – Cell Testing

EnerDel conducted testing the final deliverable cells for static capacity and rate capability, HPPC at 30°C, charge sustaining and charge depleting cycle, and cold cranking at -30°C. EnerDel tested the final deliverable cells and

static capacity shows that the energy has to be improved. Rate capability has been improved due to better electrode quality. HPPC results at 30°C have shown good power capability for both first and final deliverable cells. Ten thousand (10,000) charge sustaining cycles at 30% SOC have been completed and no capacity loss observed. Charge depleting cycle is still ongoing with 0.49MWh having been transferred to date. The tested cells have exceeded the USABC requirement of 7kW cold cranking power. The actual cold cranking power obtained was 19.8kW.

Conclusions:

EnerDel has developed a lithium-ion cell that employs a 5V spinel cathode material with the spinel LTO and demonstrated the chemistry's capability in a PHEV application, that can meet the 10-mile PHEV requirements while maintaining long life, excellent safety and low cost. EnerDel evaluated electrolyte systems that included ionic liquids, sulfolane solvent systems, fluorinated solvent system, and additives. Evaluation through linear sweep voltammetry and cycling of the LNMO/LTO system demonstrated that conventional electrolyte systems can be used in the LNMO/LTO cell if side reactions are reduced by controlling the cathode voltage and reducing side reactions by creating a surface coating on the cathode. However, further testing and development of electrolyte systems can improve the energy density of the LNMO/LTO system by enabling a design that is not so anode capacity limited.

EnerDel's approach for an optimum cell design for the initial development of the LNMO/LTO cell was created using a negative capacity limited design which is only possible with LTO since its potential is above the potential of lithium dendrite formation. This type of design is only possible with the LTO anode material since the LTO's potential is not in the range where lithium dendrites can occur. Therefore, the LNMO high voltage cathode material is well

suited to be paired with the LTO anode material. Continuous optimization for electrode production has also resulted in improved LNMO/LTO cell performance. The homogenous electrodes obtained from the production scale coaters demonstrate improved rate over the R&D coater produced electrodes. These optimizations have allowed EnerDel to deliver 20 CD cells in December and 20 CD cells in March to complete the 40 CD cell deliverables for this program.

EnerDel has begun USABC characterization testing as well as charge sustaining and charge depleting cycling on the CD cells produced. The power capability of these cells shows results that can meet the USABC PHEV goals with a BSF of 600. EnerDel has also conducted cold cranking tests that demonstrate that the LNMO/LTO system can meet the cold cranking goals.

Future Work Planned:

EnerDel will continue the development of the LNMO/LTO system by improving the energy density of the system. Evaluation of more methods for stabilizing the active material at higher potentials is necessary. The use of surface coatings will be investigated further to reduce the side reactions that can occur with the electrolyte at the high potentials. More electrolyte systems must be evaluated in order to capitalize on the higher capacity obtained at higher voltages with the LNMO. This will enable the system to move away from a less negative, LTO limited design and therefore utilize the full capacity of the LNMO material. In addition, cycle life stability and operation at elevated temperatures will continue to be evaluated in order to enable the system for use in a PHEV battery application.

Gap Analysis of Cells Produced by EnerDel Compared with USABC PHEV Requirement

Characteristics at EOL	Unit	High Power/Energy Ratio Battery			
		USABC	EnerDel Gen0 BOL	EnerDel Gen1 BOL	EnerDel GenX BOL
Reference Equivalent Electric Range	miles	10	10	10	10
Peak Pulse Discharge Power - 2sec / 10sec	kW	50 / 45	100, 10sec	180, 10sec @50% SOC	65 / 58.5 with 3.4 kWh
Peak Regen Power (10sec)	kW	30	65, 10sec	60, 10sec @50% SOC	39 with 3.4 kWh
Available Energy for CD mode, 10kW rate	kWh	3.4	4.4	4.4	4.42 with 50 / 45 kW
Available Energy for CS mode	kWh	0.5	0.5	0.5	0.65 with 50 / 45 kW
Minimum Round-Trip Energy Efficiency (USABC HEV Cycle)	%	90	-	>97%	>97
Cold cranking power at -30°C, 2sec - 3pulse	kW	7	-	19.8	7, min V: 1.5
CD Life / Discharge Throughput	Cycles/MWh	5,000 / 17	-	on-going, 167/ 0.49	5,000 / 17
CS HEV Cycle Life, 50Wh Profile	Cycles	300,000	-	on-going, 10,000	300,000 (TBD)
Calendar Life, 35°C	year	15	-	-	15 (TBD)
Maximum System Weight	kg	60	360	87, cells only	60
Maximum System Volume	Liter	40	240	50, cells only	40
Maximum Operating Voltage	V dc	400	360	360	360
Minimum Operating Voltage	V dc	>0.55 x Vmax	198	198	198
Maximum Self-discharge	Wh / day	50	-	-	<50
Thermal Performance @-30°C			-	-	10%
Thermal Performance @-10°C			-	-	30%
Thermal Performance @0°C			-	-	50%
Thermal Performance @50°C			-	-	>100%
System Recharge Rate at 30°C	kW	1.4 (120V/15A)	-	-	1.4 (120V/15A)
Unassisted Operating & Charging Temperature Range	°C	-30 to +52	-30 to +52	-30 to +52	-30 to +52
Survival Temperature Range	°C	-46 to +66	-46 to +66	-46 to +66	-46 to +66
Maximum System Production Price @ 100k units / yr	\$	1700	2544	2544	2544
Battery Size Factor			80,000	600	100

3.2.1 Computer Modeling Work

– None reported.

3.3 Deliverables/Products Developed

As described in Section 3.2.

3.4.2 Non-Proprietary Publications and Proceedings

– None reported.

3.4 Technology Transfer Activities

3.4.1 Proprietary Reporting

– Final Report to USABC was received in April 2010.

4. Nanophosphate for 10-Mile and 40-Mile Plug-In Hybrid Electric Vehicle Applications: A Multi-Generational Approach

Performing Organization: A123 Systems, Inc.

Project Duration: 3/6/2008 – 12/31/2011

4.1 Executive Summary

In March 2008 A123 Systems, Inc. initiated a three-year program to develop cylindrical and prismatic cells, packs and modules for 10-mile and 40-mile plug-in hybrid electric vehicle batteries; high voltage materials to support “smart” batteries were also targeted for development. A123 System’s novel nanophosphate-based lithium-ion battery technology was leveraged in order to achieve program goals.

During the course of the program A123 Systems developed, characterized and life tested a new 19.6Ah prismatic cell that is now in production at company facilities in Michigan. This cell is sold to a wide range of OEMs for use in passenger and commercial vehicle applications worldwide. This 19.6Ah cell and the associated modules and packs achieved all performance, abuse tolerance, and life goals set by the program except for cold crank in the 10-mile PHEV system. However, system weight, volume and cost targets were not met but strategies to close the gaps in future systems were identified. Research was also completed in new high voltage cathode, separator and electrolyte materials that would support these strategies.

Cells, modules and packs developed during this program were delivered to Sandia National Laboratory, Argonne National Laboratory, and the National Renewable Energy Laboratory for validation testing; these tests were still ongoing at the time of the project conclusion in December 2011.

4.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

This program leveraged A123’s novel nanophosphate-based lithium-ion battery technology to produce cells, modules and packs targeted at achieving USABC’s 10-mile and 40-mile goals. A123’s technology has already been applied to a variety of commercial and automotive applications including power-assisted hybrid electric vehicles for passenger and full electric vehicles in commercial transportation applications. The key advantages of A123’s nanophosphate technology include:

- Increased available power – A123’s flat power vs. SOC curve allows the battery to be discharged to a lower SOC set-point.
- Inherent safety – A123’s chemistry provides superior abuse tolerance which allows batteries to be charged to a higher SOC.
- Longer life – A123’s excellent deep-discharge cycle life allows higher SOC swings during operation.

These advantages ultimately result in higher available energy for PHEV applications. In order to realize the technology for PHEV applications, a series of technical barriers for performance and cost needed to be addressed: price, performance, cell life, system weight and volume.

The strategy for reducing system price was two-fold: a) reduce cell count by optimizing the power and capacity of the cell while still achieving required abuse tolerance; and b) optimizing the pack configuration to minimize hardware and electronics contributions. Performance targets would be achieved by two short term and a third longer term approach:

a) increase the energy density of the cell; b) leverage the wide state of charge swing possible with nanophosphate; and c) study and develop high voltage materials (cathode, electrolyte, separator) future lower cost and higher performance cells. Cell life would be extended by optimizing cell materials, especially cathode and electrolyte. Finally, system contributions (weight, volume, cost) would be lowered to reduce impact on total vehicle capacity and energy use.

Initial work in the program focused on the development of a larger capacity 32157 cylindrical cell for 10-mile PHEV system applications. These cells leveraged A123's manufacturing experience with 32113 HEV thin electrode cells; the 32157 cells used A123's code name M1 cathode materials with anodes and separators designed to improve abuse tolerance. The first three quarters of development and testing of these cells demonstrated that the energy and power requirements of the USABC 10-mile application could be achieved. However, abuse tolerance targets were not met.

During this same time progress was proceeding rapidly with the large prismatic cell design for the 40-mile PHEV system; this design showed strong potential to produce an efficient, lower cost solution for the 10-mile PHEV application as well. With USABC approval in 2009, the program direction and deliverables were changed to utilize prismatic cell technology to cover both 10 and 40-mile range PHEV applications. Deliverables were also extended to include module and pack designs and hardware to demonstrate the cell technology within the context of a battery system.

At the end of the program, the 19.6Ah prismatic cell design was demonstrated to overcome all technical challenges except system price, weight and volume. Future strategies to further reduce system cost to achieve these targets include: a) decreasing cell count by adopting higher voltage

materials and higher cell loadings; b) reducing cell cost through lower cost materials and assembly processes; c) simplifying pack hardware and electronics through a 1P and possibly air-cooled design.

Objectives and Goals:

The objectives of the program were to design, build and test cells, modules and packs to meet USABC and DOE goals for 10-mile and 40-mile PHEV applications, specifically in order to achieve high volume commercial production. The program would also investigate novel high voltage materials for future "smart" battery applications.

Approach and Main Tasks:

Development effort began in March 2008 with three tracks:

1. Development of a low cost 32157 cylindrical cell, and associated module and pack, optimized for 10-mile PHEV application.
2. Design and development of a large capacity prismatic cell and a paper-only design of module and pack optimized for 40-mile PHEV application.
3. Research into new "smart" materials including cathode, separator and electrolyte for higher energy batteries.

Technical challenges proposed in the program objectives were focused on how to achieve cell cycle and calendar life goals while also meeting the aggressive system weight, volume and cost targets.

Evaluation of the initial 32157 cells demonstrated that the target power and energy for the 10-mile PHEV application could be achieved. However, the cell could not meet EUCAR 4 targets on USABC abuse tolerance testing whereas the larger format prismatic cell development indicated surprisingly robust abuse tolerance. A123 proposed, and was granted,

permission to change the scope of development work to a single, large prismatic cell for both 10-mile and 40-mile PHEV applications. Program deliverables were renegotiated to include modules for 10-mile and 40-mile PHEV systems, with a 10-mile system pack design and prototype to be provided for testing. The program was refocused in March 2009 in this direction.

Fundamental prismatic cell design was completed in 2009, with a final product design completed in 2010; 19.6Ah prismatic cells and packs were delivered in 2010 and early 2011, respectively. Two no-cost extensions allowed for completion of performance and life testing through December 2011. All power and energy targets except cold crank were achieved for the 10-mile applications at end of life (EOL) Ten-mile cycle life achieved approximately 90% of goal before EOL was reached; analysis predicts that a small increase in battery size factor would have allowed this goal to be achieved.

The 40-mile application cycle life tests are still in progress but based on cell behavior, all performance and cycle life tests are projected to pass. Calendar life for the 19.6Ah cell is projected to exceed goals for both 80% and 100% state of charge, projecting to more than 21 and 16 years, respectively. Initial production of the 19.6Ah cell was performed at A123's Enerland facility in Incheon, Korea. During the timeframe of the program A123 commissioned new manufacturing facilities in Romulus and Livonia, Michigan for electrode coating and cell assembly, respectively. Today, A123 produces 19.6Ah cells developed under this program in its Michigan manufacturing facilities and ships these cells to global OEMs for use in a wide variety of EV and PHEV light and heavy duty vehicles on the road today.

Key Accomplishments:

During the course of this USABC program, A123 launched the AMP20M1HD PHEV cell with manufacturing capability in both Korea and the United States. This cell is now in use in vehicles on the road in the United States and around the world, in both passenger and commercial vehicles. USABC/DOE 10-mile and 40-mile pack goals are projected to be achieved for all performance indicators except system weight, volume and price.

Specific program accomplishments:

- By end of program, reduced 40-mile PHEV system price from \$15,360 (2008 estimate with existing technology, 345 x 16Ah cells) to \$8,290 (end of program technology, 255 x 19.6Ah cells), an improvement of 46%. This was achieved as a direct result of cell level cost reduction efforts and improved cell power, leading to lower BSF. System weight and volume were improved by 11% and 18%, respectively.
- Designed, built and characterized a new 32157 cylindrical cell for the 10-mile PHEV pack applications. Evaluation of these cells showed that power and energy performance goals could be achieved but that abuse tolerance did not achieve EUCAR 4. In response, an alternative program plan was developed that leveraged the parallel work to develop a large capacity prismatic PHEV cell for the 40-mile system application.
- Developed two design generations (Gen1 and Gen1.5) for 19.6Ah prismatic cells; Gen1.5 cells meet all 10-mile and 40-mile system power and energy PHEV targets except 10-mile system cold crank. Abuse tolerance characterization of EUCAR 4 or better was achieved for all tests, at both the cell and pack levels.
- Performed cycle life testing under 10-mile and 40-mile PHEV charge depleting (CD) conditions using USABC test methods. Ten-mile cells achieved more than 87 but less than

95% of target cycle life (pass at 4,347 cycles, fail at 4,850 cycles vs. 5,000 cycle goal) and are projected to achieve the USABC target with a slight BSF increase. Cells are still being tested under the 40-mile PHEV CD cycle, and are projected to achieve the 5,000 cycle goal.

- Performed calendar life testing on Gen1 and Gen1.5 19.6Ah prismatic cells using USABC test methods; testing continues beyond program termination at the end of 2011. Current projections indicate that Gen1.5 cells have a storage life of greater than 15 years at 100% SOC and 19 years at 80% SOC at 30°C storage temperature.
- Designed new 10-mile and 40-mile PHEV modules and a full 10-mile PHEV pack. These were the first modules designed and tested by A123 and were preceded by extensive thermal and performance modeling. New battery management and control systems were designed and manufactured during this program.
- Developed a new manufacturing process for A123's nanophosphate material, M1, that reduced material costs by increase production throughput by 100%. This new cathode material was used in the cells delivered as part of this program.
- Completed work on new high energy materials for future PHEV cells. These materials were not used in the cells delivered for this program as per the Statement of Work

their development continued beyond the cell deliverable timeframe.

- Developed higher energy Nanophosphate cathode material, M1x, which has demonstrated targeted capacity of greater than 150mAh/g at C/5 and provides greater than 20% higher voltage than the current lithium iron phosphate chemistry. Assembled and tested 18650 cells with M1x to demonstrate cell level results.
- Researched and scaled up a next generation, nanocomposite separator (NCS) coating and evaluated performance in small format and full size, 19.6Ah prismatic cells.
- Formulated, screened and cell tested new electrolytes and additives to support the new higher energy cathode material, M1x.
- Delivered 19.6Ah Gen1.5 cells to Sandia National Laboratory for abuse tolerance testing, the National Renewable Energy Laboratory for thermal analysis and performance testing, and Argonne National Laboratory for performance and life testing.
- Delivered 10-mile modules, 40-mile modules and a full 10-mile PHEV pack to NREL for thermal analysis and performance testing.

Future Work Planned:

Focus on power-limit estimation.

Gap Analysis vs. USABC Goals

A123 Packs vs. FreedomCAR Energy Storage System End-of-Life Performance Goals 10-Mile PHEV System

Characteristics	Units	USABC Goals	A123 BOL Gen 1.5	A123 EOP/EOL Gen 1.5	A123 EOP/EOL Gen 1.5
				Last Pass Point RPT 8	BSF Adjusted
Peak Pulse Discharge Power, 2 second	kW	50	144	73	82
Peak Pulse Discharge Power, 10 second	kW	45	105	50	47
Peak Regen Pulse Power, 10 second	kW	30	70	34	32
Available Energy for CD Mode	kWh	3.4	4.6	3.4	3.4
Available Energy for CS Mode	kWh	0.5	1.7	0.5	0.5
Minimum Round Trip Energy Efficiency	%	>90		93	93
Cold Crank Power at -30°C	kW	7	7.3		2.2
Charge Depleting Cycle Life	cycles	5000		4347	5000
Charge Sustaining Cycle Life	cycles	300,000			
Calendar Life, 30°C and 100% SOC	years	15		16	16
Maximum System Weight	kg	60	60	60	60
Maximum System Volume	liter	40	48	48	51
Maximum Operating Voltage	V	≤ 400	300	300	319
Minimum Operating Voltage	V	≥ 0.55 V	165	165	176
Maximum Self Discharge	Wh/day	50	<20	<20	<20
System Recharge Rate at 30°C	kW	1.4	1.4	1.4	1.4
Unassisted Operating & Charging Temp Range	°C	-30 to 52	-30 to 52	-30 to 52	-30 to 52
30°C - 52°C % Energy % Power Retained	%	100	100 100		
0°C % Energy % Power Retained	%	50	85 58		
-10°C % Energy % Power Retained	%	30	70 48		
-20°C % Energy % Power Retained	%	15	60 26		
-30°C % Energy % Power Retained	%	10	45 11		
Survival Temperature Range	°C	-46 to 66	-46 to 66	-46 to 66	-46 to 66
System Selling Price at minimum 100k units/year		\$1,700	\$2,956	\$2,956	\$3,143
Battery Size Factor (BSF)			79	79	84

A123 Packs vs. USABC Energy Storage System End-of-Life Performance Goals 40-Mile PHEV System

Characteristics	Units	USABC Goals	A123 BOL Gen 1.5	A123 Q1-2012 Gen 1.5	A123 Q1-2012 Gen 1.5
				As Tested	300A Limit
Peak Pulse Discharge Power, 2 second	kW	46	475	360	209
Peak Pulse Discharge Power, 10 second	kW	38	336	268	196
Peak Regen Pulse Power, 10 second	kW	25	221	176	129
Available Energy for CD Mode	kWh	11.6	16	14.3	14.3
Available Energy for CS Mode	kWh	0.3	4.7	3.0	3.0
Minimum Round Trip Energy Efficiency	%	90	>90	93	93
Cold Crank Power at -30°C	kW	7	11.4		
Charge Depleting Cycle Life Throughput	cycles MW	5000 58		2480 to date	2480 to date
Charge Sustaining Cycle Life	cycles	300,000		300,000	300,000
Calendar Life, 30°C and 100% SOC	years	15		16	16
Maximum System Weight	kg	120	174	174	174
Maximum System Volume	liter	80	95	95	95
Maximum Operating Voltage	V	400	323	323	323
Minimum Operating Voltage	V	>0.55*Vmax	178	178	178
Maximum Self Discharge	Wh/day	50	<20	<20	<20
System Recharge Rate at 30°C	kW	1.4	1.4	1.4	1.4
Unassisted Operating & Charging Temp Range	°C	30°C - 52°C	-30 to 52	-30 to 52	-30 to 52
30°C - 52°C % Energy % Power Retained	%	100	100 100		
0°C % Energy % Power Retained	%	50	85 58		
-10°C % Energy % Power Retained	%	30	70 48		
-20°C % Energy % Power Retained	%	15	60 26		
-30°C % Energy % Power Retained	%	10	45 11		
Survival Temperature Range	°C	-46 to 66	-46 to 66	-46 to 66	-46 to 66
System Selling Price at 100k units/year	\$	\$3,400	\$8,290	\$8,290	\$8,290
Battery Size Factor (BSF)			255	255	255

4.2.1 Computer Modeling Work

- None reported.

4.3 Deliverables/Products Developed

As described in Section 4.2, A123 delivered the following products for evaluation:

- Delivered 19.6Ah Gen1.5 cells to Sandia National Laboratory for abuse tolerance testing, the National Renewable Energy Laboratory for thermal analysis and performance testing, and Argonne National Laboratory for performance and life testing.

- Delivered 10-mile modules, 40-mile modules and a full 10-mile PHEV pack to NREL for thermal analysis and performance testing.

4.4 Technology Transfer Activities

4.4.1 Proprietary Reporting

- Final Report submitted to USABC dated June 8, 2012.

4.4.2 Non-Proprietary Publications and Proceedings

- None reported.

5. Advanced Mixed Metal Nanophosphate-Based Batteries for Plug-In Hybrid Electric Vehicle Applications

Performing Organization: A123 Systems, Inc.

Project Duration: 5/21/2012 – 8/14/2012

5.1 Executive Summary

In May 2012 A123 Systems, Inc. initiated a three-year program sponsored by United States Advanced Battery Consortium (USABC) to develop high energy cells, packs and modules for 40-mile plug-in hybrid electric vehicle batteries using hybrid cathode material that leverages the exceptional power and safety of lithium iron phosphate based materials and the energy benefits of nickel based materials. The program was funded by the United States Department of Energy through USABC and was targeted achieve the performance, life and economic goals established by these groups. Progress was made in cell design, electrode and electrolyte formulation and advanced separator development during the short tenure of the program. No new products were developed and no full cell test data was obtained.

Due to the sale of A123 to a foreign company, the program was terminated by USABC on August 24, 2012 and all work was ended at that time.

5.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

A123 submitted a response in 2011 to a USABC Phase 2 Request for Proposal Information (RFPI) for the development of advanced high performance batteries for plug-in hybrid electric vehicle (PHEV) applications. The objectives of the program were to design, build and test cells, modules and packs to meet USABC and DOE goals for high energy/power ratio, 40-mile PHEV applications, specifically in order to

achieve high volume commercial production. The proposal leveraged the work completed in A123's successful USABC PHEV program "Nanophosphate for 10-Mile and 40-Mile Plug-In Hybrid Electric Vehicle Applications: A Multi-Generational Approach" (PHEV I) that ended in December 2011 and the ongoing low energy storage system (LEESS) program "High Power, Low Cost Nanophosphate Batteries for Power-Assist Hybrid Electric Vehicle Applications" to develop a new high capacity but low cost battery system. The 19.6Ah PHEV cell that was a direct result of PHEV 1 program and whose performance would be the baseline for all follow-on program work.

Objectives and Goals:

The objectives of the program were to design, build and test cells, modules and packs to meet USABC and DOE goals for high energy/power ratio, 40-mile PHEV applications, specifically in order to achieve high volume commercial production. Technical challenges proposed in the program objectives were focused on how to achieve cell cycle and calendar life goals while also meeting the aggressive system weight, volume and cost targets and employing new materials in nearly every aspect of the final cell.

Task Areas:

Development effort on this new three-year program, "Advanced Mixed Metal Nanophosphate-Based Batteries for Plug-in Hybrid Electric Vehicle Applications" (also known as High Capacity PHEV Program) began in May 2012 with five focus areas: 1) new high energy hybrid cathode material, M1xide; 2) new high energy aqueous graphite anode; 3) temperature resistant layers; 4) reformulated electrolyte for long life; and 5) new cell assembly technology. These activities were designed to close the gap between cell performance and cost at the end of the initial

PHEV I development program and the USABC 40-mile PHEV application goals.

Key Accomplishments:

During the three months tenure of this USABC program the baseline cell was designed, cathode and anode materials development was initiated, new separator materials were evaluated for improved abuse tolerance, and electrolyte additives were designed to improve calendar and cycle life.

Specific program accomplishments were:

- Design was completed for initial 20Ah reference cell that employed A123's proprietary M1x cathode powder without high energy materials.
- Analysis and design was completed for 65Ah program deliverable cell to achieve USABC targets. This cell was designed to use M1xide, a combination of M1x powder and a high energy nickel based material.
- Information on non-cathode cell materials and regions or origin was compiled and provided to USABC.

- Experiments to identify appropriate high energy cathode material to blend with A123's proprietary M1x powder were started.
- Experiments to develop a water-based anode formulation with natural graphite were started and coating adhesion results were obtained.
- First experiments were completed demonstrating the capability of temperature resistant layers for improved abuse tolerance in high capacity and high energy cells.
- First experiments to identify additives to extend storage life were performed.

Due to the short tenure of this program no measureable progress was made on closing the gaps between initial technology capability and the final program goals.

Gap Analysis vs. USABC Goals:

The most recent technology gap analysis, presented to USABC at the Q2-2012 program review, is shown below.

A123 Packs vs. USABC Energy Storage System End-of-Life Performance Goals 40-Mile PHEV System

Characteristics	Units	USABC Goals	EOP Q2-2012 *	EOP / EOL Projection
			Gen1.5 19.6Ah Stacked Prismatic	65Ah Wound Prismatic
Peak Pulse Discharge Power, 2 second	kW	46	203	66
Peak Pulse Discharge Power, 10 second	kW	38	193	49
Peak Regen Pulse Power, 10 second	kW	25	127	66
Maximum Current, 10 second	A	300	300	300
Available Energy for CD Mode	kWh	11.6	14.3	11.7
Available Energy for CS Mode	kWh	0.3	3.0	0.7
Minimum Round Trip Energy Efficiency	%	90	93	>90
Cold Crank Power at -30°C	kW	7		9.2
Charge Depleting Cycle Life Throughput	cycles MW	5000 58	3480	5,000 58
Charge Sustaining Cycle Life, 50Wh Profile	cycles	300,000		300,000
Calendar Life, 30°C	year	15	16	15
Maximum System Weight	kg	120	174	127
Maximum System Volume	liter	80	95	72
Maximum Operating Voltage	V	400	323	319
Minimum Operating Voltage	V	>0.55* Vmax	178	190
Maximum Self Discharge	Wh/day	50	<20	<20
System Recharge Rate at 30°C	kW	1.4	1.4	1.4
Unassisted Operating & Charging Temperature Range	°C	30 - 52	30 - 52	30 - 52
30°C - 52°C % Energy	%	100		100
0°C % Energy	%	50		70 - 80
-10°C % Energy	%	30		60 - 70
-20°C % Energy	%	15		50 - 60
-30°C % Energy	%	10		30 - 40
Survival Temperature Range	°C	-46 to 66	-46 to 66	-46 to 66
System Selling Price at minimum 100k units/year	\$	\$3,400	\$8,290	\$4,529
Battery Size Factor (BSF)			255	76

* updated with RPT8 Data

5.2.1 Computer Modeling Work

– None reported.

5.3 Deliverables/Products Developed

None. Program was terminated in three months after USABC award.

5.4 Technology Transfer Activities

5.4.1 Proprietary Reporting

– Final Report of partial work completed and submitted to USABC dated October 26, 2012.

5.4.2 Non-Proprietary Publications and Proceedings

– None reported.

6. Advanced Cathode Materials for PHEV Applications

Performing Organization: 3M Company

Project Duration: 4/2/2009 – 4/30/2011

6.1 Executive Summary

The Lithium-ion Battery (LIB) has become one of the most promising power sources for replacing the traditional combustion engine in vehicles. LIB-powered vehicle types like the Electric Vehicle (EV), the Hybrid Electric Vehicle (HEV), and the Plug-in Hybrid Electric Vehicle (PHEV), are being developed aggressively by the major auto makers. PHEV requires that the LIB have high energy density, high power capability, and high safety, which provide stringent requirements to the cell chemistry, especially for the cathode materials within. Only a few cathode materials currently available are suitable for PHEV.

The objective of this program was to leverage 3M's strong R&D capability and cathode manufacturing "know-how" to develop and scale-up an advanced cathode material for PHEV applications. The project utilized 3M's BC-618 (NMC 111 composition) as a baseline. The relevancy and appropriateness of this baseline is borne out by the utilization of this composition in currently mass produced PHEV vehicles. The specific targets for the material to be developed during the course of this program included ~ 10% higher capacity, 10% lower raw materials costs while maintaining cycle life and thermal stability. 3M's primary focus was on materials development, designing and implementing an 18650 test cell as well as scaling materials to pilot scale levels in order to enable the fabrication of 18650 cells. A final 3M deliverable of this program in conjunction with submitting a final report was to submit sample 18650 cells to the respective DOE laboratories for abuse and electrochemical performance/cycling verification.

6.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

PHEV requires that the LIB have high energy density, high power capability, and high safety, which provide stringent requirements to the cell chemistry, especially for the cathode materials within. Only a few cathode materials currently available are suitable for PHEV. LiFePO_4 has excellent thermal stability and high power capability but low energy density. LiMn_2O_4 (spinel) has excellent thermal stability and power capability, but the cycle life of LIB made with LiMn_2O_4 at high temperatures needs to be improved. LiNiO_2 -based cathode materials have excellent energy density, but the inferior thermal stability of LiNiO_2 prevents its broad application, especially in the large format cells used in PHEV. Recently, $\text{Li}[\text{Mn-Ni-Co}]\text{O}_2$ (MNC) materials has been regarded as a promising cathode candidate for PHEV applications due to good thermal stability, excellent cycle life, high energy density, and high power capability. One such material is $\text{Li}[\text{Mn}_{0.33}\text{Ni}_{0.33}\text{Co}_{0.33}]\text{O}_2$, commonly referred to as MNC 111 or NMC 111. Due to the positive properties of this 111 material and its fit with automotive application requirements, it has become one of the few battery cathodes now found in mass-produced commercial PHEV vehicles.

Objectives and Goals:

The objective of this program was to leverage 3M's strong R&D capability and cathode manufacturing "know-how" to develop and scale-up an advanced cathode material for PHEV applications. The project utilized 3M's BC-618 (NMC 111 composition) as a baseline. The specific targets for the material to be developed during the course of this program included ~ 10% higher capacity, 10% lower raw materials costs while maintaining cycle life and

thermal stability. Delivering a material with these properties would be consistent with the FreedomCar objectives of enabling higher performance, lower cost PHEV vehicles.

Approach:

3M approached the project by conducting a systematic mixture design around the ternary compositional map of nickel, manganese and cobalt in the layered oxide structure. Capacity, cost and thermal stability data of these compositions was statistically analyzed and modeled to determine the best composition to meet the programs goals. Two potential compositions which met the project goals were identified, one optimized for cost and the other optimized for thermal stability. These two optimum compositions were both carried through process optimization, including systematic process conditions, production at pilot scale and verification of process robustness. Comparative data was collected on both prospective USABC compositions, down-selection to a final composition was based on superior capacity retention upon storage at elevated temperatures and maximum cost reduction. An 18650 sized test vehicle was designed and coatings of the USABC prospective material as well as coatings of the baseline BC618 (NMC 111) were utilized to produce comparative 18650 cells. The 18650 cells were evaluated by the standard USABC protocols for electrochemical evaluation agreed to by Argonne National Lab and for thermal and abuse stability utilizing agreed to protocols with Sandia National Lab.

Tasks and Accomplishments:

The following technical tasks were carried out according to the project plans and SOW.

1. *Compositional Exploration and Identification* – 3M explored 12 compositions in the ternary diagram $\text{LiCoO}_2 \bullet \text{LiMn}_{1/2}\text{Ni}_{1/2}\text{O}_2 \bullet \text{LiNiO}_2$, in order to meet the objectives of the proposal: increasing capacity, reducing cost and maintaining thermal stability over baseline NMC 111. Two target NMC compositions were selected for scale-up and large scale evaluations. Although the goal of the USABC program and SOW required only one material to be down-selected, two materials were carried forward based on slightly different results from optimizing on lowest cost or highest thermal stability.
2. *New Materials Process Scale Optimization* – Typical progression in scaling up a new material is performed according to the following course:
 - 500ml Bench Scale – composition identification
 - 2 to 10L Bench Scale – composition and morphology study – preliminary process to develop an understanding of key process parameters on particle morphology and to produce quantities of materials to allow systematic optimization of sintering conditions, such as time and temperature profiles as well as Lithiation levels. The key process conditions were identified during the work in the 2-10L reactor.
 - 300L Pilot Scale – pre-manufacturing process verifying composition and morphology. The pilot scale reactor utilized is based on similar design principles to the 2-10L bench scale reactors; however, it is comprised of multiple 300L reactions tank and is directly related to mass production manufacturing process. More than 10 reaction conditions were evaluated to identify best process conditions.
 - Mass Production - The scope of this proposal covers the activities in Step 1 to 3, bench to pilot scale.

3. *Cell Design –*

3M was required to prepare 18650 cells for head-to-head materials evaluations in a cell design that more closely related to automotive cells than traditional coin cells. During the course of the work a number of deficiencies in 3M's 18650 hardware and preparation procedures were identified which prevented accurate assessment of the materials. These deficiencies were identified and successfully addressed in order to complete the project. Two methods were considered for comparing the baseline material with the higher capacity advanced materials of this project. Method 1 is introducing new cathode material by maintaining the same loading (mg/cm²) of the composite cathode. Method 2 is introducing new cathode material by maintaining the same capacity (mAh/cm²) of the composite cathode. A decision was made by 3M and the USABC working group to utilize Method 2, maintaining a constant capacity for the composite cathode, to build comparative cells with the baseline BC618 (NMC 111) and the Advanced, C1P2 and C2P2 materials. Utilizing this method it would be anticipated that for cells containing C1P2 and C2P2 approximately 10% less cathode material would be utilized relative to same capacity cells containing the baseline BC618 and there would be no significant improvement in the BSF.

4. *Electrode Coating –*

Electrode coating is a key aspect to cell and material performance. Optimized electrode coatings are required to prepare 18650 sized cells for final USABC testing protocols. The focus of this program is developing a new cathode material and therefore significant effort was conducted to optimize electrode coatings for fabrication into 18650 cells. In summary, electrode fabrication studies confirmed that sufficient composite electrodes could be prepared with the Advanced Compositions at 90% active

levels, 5% Super P and 5% PVDF. The electrodes had sufficient durability to be fabricated in 18650 cells and that materials prepared by the "P2" process were more resistant to particle fracture during the calendaring process. Based on battery design covered in separate section the following electrodes were coated to fabricate 18650 cells for internal performance testing and external verification at Sandia and Argonne National Laboratories.

5. *18650 Fabrication Hardware*

Troubleshooting –

- Hardware troubleshooting
- Formation QC protocol
- 18650 fabrication
- Sample shipment

6. *New Material and Baseline Material Evaluations in Test Vehicle –*

- Abuse evaluations
- Electrochemical evaluations

7. *Data Package Summary of New Materials vs. Baseline Material –*

Summary of All Abuse Testing

Requirement	BC618 Benchmark	USABC Comp 2
Nail Penetration Description	Venting/ Smoke and Combustion	Venting/ Electrolyte Boiling
Max Temperature (°C)	425	183
Hot Block Description	Venting/ No smoke	Venting/ No smoke
Max Temperature (°C)	163	166
Thermal Ramp Thermal Runaway Temperature (°C)	227	229
DSC Temperature of Peak Exotherm (°C)	315	315

In summary, Composition 2 has been shown to meet the programs objectives by having

comparable stability performance relative to the Benchmark BC618 (NMC 111) material as determined by evaluation both at the material level and at the 18650 cell level.

Conclusions:

During the course of this project, a new MNC cathode composition was identified and scaled to the pilot level. Evaluation in 18650 cells relative to cells containing baseline BC618 NMC 111 material demonstrated the following performance of the new MNC material:

- Improved capacity by 8% meeting project goal of 5-10%.
- Reduced raw materials cost by 28% exceeding project goal of 15%.
- Improved cold crank power meeting USABC requirement of >7kW.
- Comparable self discharge rate meeting USABC requirement of <50Wh/day.
- Comparable thermal and abuse stability as measured by thermal ramp, hot block and nail penetration.
- Comparable cycle life as measured by the USABC charge depletion and reference performance testing methods demonstrating >750 cycles and meeting project goal of >500 cycles.

Based on these above results, 3M delivered 40, 18650 cells to the relevant National Laboratories for abuse and electrochemical performance/cycling verification.

Future Work Planned:

The material developed in this program demonstrates performance and cost benefits over MNC 111 material which is currently being utilized in mass produced vehicles. These benefits have been demonstrated in 18650 cell format with cycling up to 750 cycles. Additional testing in large vehicle ready cells (10-20 Ah) for multiple 1,000 cycles would be the next appropriate step towards implementing this material in vehicle applications.

6.2.1 Computer Modeling Work

- None reported.

6.3 Deliverables/Products Developed

40 units, 18650 cells to the relevant National Laboratories for verification purposes such as:

- Energy density
- Power capability (HPPC tests)
- Thermal stability test
- Cycling life test
- Abuse tolerance (oven, nail, etc.)

6.4 Technology Transfer Activities

6.4.1 Proprietary Reporting

- Final Report to USABC at the conclusion of the project, “Advanced Cathode Materials for PHEV Applications Program.”

6.4.2 Non-Proprietary Publications and Proceedings

- None reported.

7. A High Performance PHEV Battery Pack

Performing Organization: LG Chem/Compact Power Inc.

Project Duration: 4/4/2011 – 12/31/2013

7.1 Executive Summary

The goal of this 32-month USABC Program was to develop and evaluate a battery for use in PHEV-40 electric vehicles using a Manganese-rich cathode (MRC), a proprietary mechanically robust separator and laminated packaging. The main objective was to demonstrate that this system is capable of meeting or exceeding the USABC target of 5,000 cycles and 15-year calendar life. An additional key focus of the program was to develop a battery pack that is mechanically, electrically and thermally robust and abuse-tolerant for use in PHEVs.

Comprehensive studies were carried out to utilize the MRC-based cathode to develop a cell that is capable of meeting the PHEV 40-mile targets. By first studying its behavior under various test conditions and examining its failure modes, efforts were made to improve upon the materials properties. A total of two generations of cells were developed and tested to demonstrate these results, as well as submitted to USABC for further testing and verification. The 1st generation cells consisted of MRC/NMC blends but did not exhibit adequate life characteristics especially at elevated temperatures. The 2nd generation used an Atomic Layer Deposition (ALD) -coated MRC cathode and shows improved performance but still less than adequate life characteristics are anticipated.

LG Chem/CPI also devoted significant efforts to develop a novel thermal management system based on the concept of indirect cooling using a refrigerant, cold-plate and solid fin. Two generations of packs were built and delivered to USABC for further testing and verification.

7.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

This USABC award was originally a 24-month program aimed at developing and demonstrating Li-ion cell and pack technologies which will meet the performance, life and cost targets of the USABC 40-Mile PHEV program. An objective was to develop the cell using the next-generation, Mn-rich layered-layered composite cathode material, which shows an attractive potential for delivering high specific capacity and, thus, significant cost reduction.

The above cell work was supplemented by pack studies with the goal of developing an automotive-grade, self-contained battery pack using a refrigerant-based cooling system. The objective was to significantly increase the efficiency of the thermal management system to increase life, lower BSF and, thus, and more importantly, lower pack cost. This was achieved via an indirect cooled the refrigerant-to-solid fin thermal management system.

The following major tasks were the focus of the program:

- Demonstrate 5,000 cycle life and 15-year calendar life
- Evaluate abuse tolerance
- Develop a battery pack that is mechanically and electrically robust and most importantly thermally very efficient and reliable.

Objectives and Goals:

The overall objective of this USABC program was to develop a battery for use in PHEV-40 electric vehicles using a manganese-rich cathode (MRC), a proprietary mechanically robust separator and laminated packaging. The principal objective of this program was to

demonstrate that this system is capable of meeting or exceeding the USABC target of 5,000 cycles and 15-year calendar life. An additional key focus of the Program was to develop a battery pack that is mechanically, electrically and thermally robust and abuse-tolerant for use in PHEVs.

Approach and Tasks:

Cell Studies: The following major tasks were the focus of the cell development studies using the Mn-rich cathode:

- Evaluate the performance characteristics (e.g. capacity, HPPC, self-discharge)
- Evaluate life (cycle and calendar)
- Evaluate low temperature performance
- Carry out abuse tolerance tests
- Cost-modeling studies
- Support of National Lab performance and abuse tolerance studies.

Pack Development:

One key objective of the pack development activities was to develop a volumetrically more efficient pack than the one developed in CPI's earlier program using the refrigerant-to-air cooling concept. Major tasks for this present program thus, included the development and optimization of an indirect cooling system using a refrigerant-cooled cold plate and solid fin especially with respect to volume, weight and cost.

The specific tasks included:

- Development of operational scenarios and thermal models
- Development and optimization of compressor/condenser/evaporator assembly
- Optimization of the pack housing and integration

- Optimization of the electrical and BMS systems
- Pack validation, testing and delivery to National Labs.

Since the program cell did not have sufficient maturity in the beginning, all of CPI's initial pack development studies were carried out using PLG2 cells developed in the earlier program. It had the capacity of 15Ah, in contrast to the 60Ah proposed for the program but the footprint was the same as the cell CPI used later for the final program cell and pack builds and deliverables.

Accomplishments:

Comprehensive studies were carried out to utilize the MRC-based cathode to develop a cell that is capable of meeting the PHEV 40-mile targets. By first studying its behavior under various test conditions and examining its failure modes, efforts were made to improve upon the materials properties. CPI studied, for example, the impact of various formation voltages and voltage limits on capacity and cyclability. Based on these results, studies were carried out to identify effective solutions such as multi-stage formation and degassing protocols, doping and coating of cathode powders, use of electrolyte additives and cathode blends to improve the durability of this cathode system.

A total of two generations of cells were developed and tested to demonstrate these results. The 1st generation cells consisted of MRC/NMC blends and did not exhibit adequate life characteristics especially at elevated temperatures. The 2nd generation used an ALD-coated MRC cathode and shows improved performance but still less than adequate life characteristics. The cycle life of the cells was critically dependent on the charge voltage limit. When charged beyond 4.4V limit to increase the available capacity, there was a significant decay in cycle life. The failure modes, though, for both the cells appear to be similar. Significant gassing, Mn dissolution and consequent anode

passivation were the key failure modes. Coating the cathode particles with a conformal coating of ALD considerably enhanced the cycle-life; however, it did not mitigate the voltage fade issue. The cells, though, showed quite good abuse characteristics. Both of these generations of cells were submitted to USABC for further testing and verification.

CPI also devoted significant efforts to develop a novel thermal management system based on the concept of indirect cooling using a refrigerant, cold plate and solid fin. It is a self-contained pack, housing not only the electrical components but also the thermal management system.

Work was carried out to optimally package cells into modules mechanically and electrically, optimize the compressor size, attach fins to the cold plate, etc. to develop a pack that is volumetrically and gravimetrically efficient. Two generations of packs were built and delivered to USABC for further testing and verification. CPI believes that this cooling system, once fully optimized, will be attractive for PHEV and BEV applications.

Conclusion:

Considerable insight into the material properties and ways to improve upon them for the MRC cathodes have been obtained in this program that will be highly valuable to the development of a high energy, long life and low cost battery for PHEV battery. Similarly, the development of a stand-alone, self-contained battery pack provides a good alternative to packs built using conventional cooling methods such as liquid and air

Gap Analysis vs. USABC Goals:

A gap analysis was not submitted by LG Chem/CPI for this project.

7.2.1 Computer Modeling Work

– None reported.

7.3 Deliverables/Products Developed

As described above, two generations of cells were developed and tested to demonstrate these results:

- The 1st generation cells consisted of MRC/NMC blends and did not exhibit adequate life characteristics especially at elevated temperatures. These first generation of large cells (PLG3a) were delivered to the National Labs. These cells had a capacity of about 24Ah yielding a specific energy of about 190Wh/kg at the 0.1C rate.
- 60 units of 2nd generation of PLG3b cells used an Atomic Layer Deposition (ALD) - coated MRC cathode and show improved performance but still less than adequate life characteristics.
- Two generations of packs (12V and 24V packs, based on the PLG2 cells) were built and delivered to USABC for further testing and verification.

7.4 Technology Transfer Activities

7.4.1 Proprietary Reporting

– Final Report submitted to USABC dated May 29, 2014.

7.4.2 Non-Proprietary Publications and Proceedings

– None reported.

8. PHEV Advanced High Performance Cell Program

Performing Organization: Johnson Controls, Inc.

Project Duration: 2/12/2012 – 3/30/2014

8.1 Executive Summary

The PHEV Advanced High Performance Cell program funded by the Department of Energy (DOE) and guided by the United States Advanced Battery Consortium (USABC) was kicked off by Johnson Controls, Inc. (JCI) in early April, 2012. The purpose was to extend the results of the prior USABC-JCI PHEV program ending in May 2011 and focus specifically on three main dimensions related to a prismatic energy cell. Those dimensions were increasing energy density, improving abuse tolerance, and to reduce cost, all with the 20-mile AER gap chart targets in mind. Within a few months of the program start, the USABC Management Committee requested, and JCI proposed aligned additional stretch goals with no change to the overall program cost or duration. At approximately the half way point of the program, JCI requested a reduction in scope and cost to accommodate the demands on resources within JCI's technical team. The scope change did not alter the program end date, and stretch goals were retained.

Deliverables included baseline prismatic cells at Month 4 for evaluation at Argonne National Labs. Final cells were delivered to Argonne as well as National Renewable Energy Lab and Sandia National Lab at the end of the program for life, thermal, and abuse testing respectively. Regular Quarterly Reviews were conducted at alternating locations to update USABC regarding program status and plans. The program was concluded in March 2014 with all hardware deliverables shipped.

8.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

This follow-on program was proposed to the USABC organization to build on the success of the previous program which saw the development of JCI's prismatic NMC-graphite technology. Starting conditions for this program were based on the PL27M cell that was derived from that prior program which was being prepared for mass production by JCI. This PHEV2 prismatic cell had a 27Ah capacity, and a specific energy density of 275Wh/L.

This program was created to conduct research in five major areas in parallel, with results converging at final cell delivery all in support of the three main dimensions mentioned above. They included: 1) High Energy Density Materials, 2) Electrode Processing and Design Optimization, 3) Increased Upper Voltage, 4) Mechanical Design and Manufacturing Improvements, and 5) Abuse Tolerance Improvements.

Over the course of the two year program, results were extracted from pouch and prismatic cell builds resulting in the expected convergence of decisions and eventually the finally cells with 36Ah rating delivered to the National Laboratories.

Objectives and Goals:

The central goal of this PHEV program focused on cell-only research and design while also considering the impact to a potential battery system as described in the USABC 20-mile AER Gap Chart targets. The JCI PL27M prismatic energy cell was the baseline cell to initiate this program.

Two scope changes were made to accommodate changing JCI resources/capabilities and

additional stretch goals requested by the USABC. At the July 2012 Quarterly Review, the cell deliverable count was modified to account for limited test capacity at the National Labs. The change was 45 baseline cells to 9, 60 mid-program cells to 18, and 45 final cells to 38. In August 2012 the USABC management committee requested the existing program include aggressive stretch goals to investigate riskier avenues of research and stay within the existing funding agreement. JCI proposed several ideas which resulted in three approved additional stretch goals. They were a) Higher Energy Chemistry with targeted 375Wh/L, b) High Energy Cathode specifically Li rich layered-layered structure with targeted 450Wh/L, and c) Mechanical Component Opportunity focusing on a plastic cell enclosure for a true neutral design with lower part and tooling costs.

Tasks:

The project was executed in five main tasks as summarized below:

Task 1 – Higher Energy Density Materials

Investigating new cathode materials was an important part of the program. Evaluation of new and improved NMC materials constituted one of the main activities. Higher Ni content materials offer one pathway to increase cell energy density. However, increased Ni content is usually associated with lower thermal stability and life. Suppliers have recently made notable improvements by stabilizing the structure of these materials through doping or by improving the purity of the structure. Another way to stabilize the cathode active material and reduce the reactivity with the electrolyte is to apply a surface treatment. Materials incorporating these stabilization techniques were evaluated over the course of the program.

The other challenge to a higher energy result was found in the primary anode material, graphite, where increased loading and density

without adversely affecting life and abuse tolerance was found to have no advantage over the baseline formulation. Initial approaches were focused on high density and high compressibility materials, and blends of graphite materials. After preliminary investigation of alternatives to the current material, a decision was made to focus on optimizing the electrode design using baseline graphite. Improving energy density to the next level requires using new material like alloys and Si-based anode materials that are not in the scope of this program.

Initially, the program focus was on high nickel content NMC materials as a means to enhance energy density at the cell level. However, high nickel chemistry showed unacceptable performance in calendar life and cycle testing at high temperature. Initial testing concluded that the baseline NMC and the other NMC materials closer to the baseline with regard to structure and metal composition have more potential to improve the energy density of JCI prismatic cell. Thus, two candidate materials along with JCI baseline NMC (1/1/1) were assembled into PL27M cells for high voltage cycling. Increasing the voltage stability and upper voltage limit is another path to increased energy density. Finally, it was demonstrated that the baseline NMC (1/1/1) is still the best overall cathode material for a high energy JCI prismatic cell within the intended operating window.

Task 2 – Electrode Processing Optimization/Design Optimization

The sub-tasks performed included: High Solids Mixing for Cathode (to reduce the cost of electrode manufacturing by using a reduced amount of solvent), Ultra High Molecular Weight Binders (various suppliers and grades of high MW PVDF's were researched), Organic Solvent Elimination from Positive Electrode, Electrode Design Optimization (balancing higher loading and higher density electrode designs along with an optimized electrode

formulation), and Development of Prismatic Cells with High Density Anode and Cathode.

The program has demonstrated that higher electrode loading is not an efficient means of increasing the cell energy density as it has a detrimental impact on cell life, particularly cycle life. It will also bring more risk on the life performance. Higher cathode and anode densities were studied individually to quantify the impact on cell energy density, power capability, and life performance. The final deliverable cells have an electrode design outlined below:

- Cathode: the same loading as the baseline cell; paste mixing process; 7% density increase with optimized formulation.
- Anode: the same loading as the baseline; 10% density increase.

Task 3 – Increased Operating Voltage

Through the use of rating and testing the cell at a higher charge voltage, an increase in the available energy can be achieved. This is one of the most efficient method to increase energy density and reduce \$/kWh. Key questions to be addressed were the impact of elevated voltages on life and abuse tolerance. Accordingly, long term storage and characterization of JCI prismatic cells was conducted at higher voltages, with the goal of evaluating performance at upper voltage limits above 4.1V. In addition to directly increasing the cell upper voltage limit, another approach studied was to widen the usable state of charge (SOC) window, also known as the depth of discharge range. This would maximize cell energy utilization in real applications and also reduce \$/kWh metric at a system level. Three sub-tasks were identified to address this goal: 1) increase upper cell voltage limit; 2) increase SOC (state of charge) usage window; and 3) improved electrolyte solvents or additives.

Over a two year period of the program, numerous electrolyte compositions and additives have been studied in both pouch cells

and PL27M cells. Based on the extensive matrix of test results, the electrolyte selected for the final design was the current baseline composition with the addition of an overcharge additive from manufacturer Elec_2.

Task 4 – Mechanical Design & Advanced Manufacturing

The program aimed at improving energy density by minimizing the void volume in the cell and reducing cost through design and assembly process optimization. The targets of WBS 4.0 were to increase the active material, by 3-5% inside the cell by minimizing the void volume, while maintaining the external dimensions, and reduce the overall cost of the mechanical components by 10-15%. Novel design concepts and design optimization were evaluated to improve cell energy density and drive down cell cost. Different manufacturing processes were investigated to improve yield, throughput and robustness, and reduce capital cost. In addition, new features were assessed to improve the abuse tolerance of cells with significantly increased energy density.

The present design uses a rigid aluminum can as the enclosure for prismatic cells. Reducing the can sidewall thickness enables cost reduction and energy density improvement. Experimental and analytical studies on the cells with different wall thicknesses and geometry were conducted to evaluate the mechanical strength and thermal performance.

Computer simulations were performed to understand the impact of using different aluminum alloys and reducing cell can wall thickness on strength and thermal performance. From those results, the can sidewall thickness was reduced to about 80% of baseline through use of an aluminum alloy with higher strength. This increased cell capacity by about 2% and saved approximately 7% in cost of can. No performance degradation was found in the cells built with the thin wall cans, in characterization and abuse tests.

To improve the functionalities of the can, a special design, which potentially could improve the cell heat dissipation, was explored. Thermal performance evaluation was conducted using the cells built with the special cans.

Throughout the program, opportunities of improvement of energy density and reduction of cost in mechanical design and manufacturing process were pursued. Optimized component designs (e.g. size reduction in can wall thickness and current collectors and mandrel removal) were included in the final design, which has an increase of cell capacity, by 8% (exceeded the target of 3-5% capacity improvement), and a reduction of cell cost, by 15% (met the target of 10-15% cost reduction). Completely new designs (e.g. plastic cell canister, cell internal and external coating) were investigated and explored in the program to pave a way for further optimization and cost reduction.

Task 5 – Abuse Tolerance Improvements

Increasing energy density of the cell results in lower thermal stability either because of less stable materials or decreased heat dissipation. Therefore, the evaluation of separators for high thermal stability initiated during the previous program was continued. The objective was to delay the temperature at which an internal short circuit is created in the cell as well as limit heat propagation between the two electrodes. This improves the safety margin. JCI has also worked closely with ENTEK on their new experimental ceramic-filled separators to conduct testing to establish whether the ceramic filled separator could significantly enhance the abuse tolerance of the prismatic energy cell.

A few issues were found during the cell building process:

- Strong static and weak web strength; difficult to handle and wind in cell assembly.

- High moisture: moisture level at a few thousands ppm after vacuum drying.
- Low breakdown voltage in hipot test (as low as 50V): difficult to use hi-pot to detect other defects during cell assembly.

Nevertheless, SiO₂-filled separators also demonstrated their potential in cell performance. All cells with SiO₂-filled separator had very low cell impedance. The high moisture level was a big concern initially, but after a few trials, it was found that the high moisture did not affect the cell performance. The most remarkable result is the lack of resistance growth in the calendar life tests. After one year and storage at 60°C, no resistance increase was observed in these cells with SiO₂-filled separators.

8.2.1 Computer Modeling Work

– None reported.

8.3 Deliverables/Products Developed

Deliverables included baseline prismatic cells at Month 4 for evaluation at Argonne National Labs. Final cells were delivered to Argonne as well as National Renewable Energy Lab and Sandia National Lab at the end of the program for life, thermal, and abuse testing respectively.

8.4 Technology Transfer Activities

8.4.1 Proprietary Reporting

– Final Report was submitted to USABC dated May 6, 2014.

8.4.2 Non-Proprietary Publications and Proceedings

– None reported.

Section D – Low-Energy Energy Storage Systems

Section D – Low-Energy, Energy Storage Systems (LEESS) Final Reports:

1. A123 – High Power, Low Cost Nanophosphate Batteries for Power-Assist Hybrid Electric Vehicle Applications D-1
2. Maxwell – USABC LEESS Program D-5

1. High Power, Low Cost Nanophosphate Batteries for Power-Assist Hybrid Electric Vehicle Applications

Performing Organization: A123 Systems

Project Duration: 2/23/2011 – 6/1/2013

1.1 Executive Summary

A123 Systems was awarded funding for the development of an HEV LEESS Energy Storage System in February, 2011, in a two year program. The objectives of the program were to design, build, and test cells and modules for HEV Low Energy, Energy Storage systems which would achieve DOE/USABC performance targets and significantly close the gap on system price targets. However, this program was terminated by USABC prior to the conclusion due to concerns about A123 Systems financial status and potential ownership by a foreign entity; as a result, program objectives were not fully met. This report covers the accomplishments and developments of A123 Systems development teams from March 1, 2011 through August 24, 2012.

This program involved the development of a 3.8Ah wound flat wrap (WFW) prismatic cell, which was approached in three phases: 1) materials and chemistry proof-of-concept in 0.34Ah stacked prismatic cells, 2) electrode winding capability proof-of-concept in 1.3Ah wound, then flattened, cells using an adapted cylindrical cell winder, and 3) full cell prototypes in 3.8Ah WFW design, using targeted wound prismatic equipment and processes. This program also funded the evaluation of improved cathode powder for increased pulse power capability, development of an improved anode formulation to reduce cost and improve power capability, development of an improved electrolyte formulation for low impedance at low temperature, and development of a low cost module/pack design.

1.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

Project Objectives and Approach:

The objectives of the program were to design, build, and test cells and modules for HEV Low Energy, Energy Storage systems which would achieve DOE/USABC performance targets and significantly close the gap on system price targets. The final program deliverable was for a system which would include 71 3.8Ah WFW cells in a single module/pack which would meet all performance targets, for a price of \$831.

Development effort was initiated in March, 2011, on an HEV LEESS system based on a modified version of A123's 6Ah prismatic cell. Preliminary evaluation of systems which would be directly based on a 6Ah prismatic cell indicated that these cells provided more energy than required and would far exceed target price. Similarly, systems based off of a 26650 cell design would be high cost/price due to the number of cells required to meet power targets. Therefore, the decision was made to develop a 3.8Ah cell which would be based on the 6Ah prismatic cell chemistry and electrode design, but modified to increase power, decrease materials costs, and enable a wound, flat cell design with lower cost production than the existing stacked prismatic assembly process.

Cathode powder studies and formulation for flexible electrodes, anode formulation and low impedance electrolyte development were completed and demonstrated in 0.34Ah stacked, prismatic lab prototype cells. WFW processes were developed and new equipment purchased five and installed (at A123's expense) to demonstrate performance in full format prismatic cells. A new, minimalist module design with lower cost electronics, cost reduced

cell pressure management and thermal controls was developed. Components were designed and procured in anticipation of assembling module prototypes, conducting module level testing, and meeting committed program deliverables.

Key technologies evaluated within the scope of this program included cell level development (lower cost and potentially higher power cathode powder, new lower cost natural graphite based formulations, lower impedance electrolyte, and WFW cell design) and system Level 6 development (71 cell module with low cost electronics, air cooling, compression pad-free design).

Initial evaluation of A123's technology led to the identification of four technical challenges, which this program was designed to address:

- Increased power density, particularly at low temperatures, to reduce unneeded energy and decrease BSF.
- Increased cycle life for WFW cells.
- Improved calendar life for low cost natural graphite anodes.
- Reduced system cost/price.

Increased power density was addressed by a combination of lower electrode loading, evaluation of higher power lithium iron phosphate (LFP) cathode powders, and development of a low impedance electrolyte. An additional, ultra low impedance separator was to be included in subsequent cell builds.

Increased cycle life in WFW cell format was to be achieved through modifications of electrode formulations and loading, and increased process development for cell assembly. This effort was in process at the end of the program.

Improved calendar life was to be achieved through optimization of the low impedance electrolyte, customizing the formulation to be compatible with the selected anode formulation.

Reduced system cost was addressed by improving power density to reduce BSF, reducing materials cost, introducing the lower cost WFW cell design and assembly process, and by simplification of the module electronics and hardware.

All of the above technical challenges were anticipated to be successfully addressed by June, 2013, which would have required a four month no-cost program extension. Due to the sale and merger of A123 to a foreign entity, the program was terminated in 18 months by USABC in August, 2012, while on track for completion of program objectives by June, 2013.

Task 1 – Cathode Development

Cathode efforts for the HEV LEES program included both an electrode optimization study to select current collector and conductive additives, and a modeling study to determine if an improvement in cathode powder composition could improve low temperature power.

Task 2 – Anode Development

Anode development efforts were focused on achieving high power goals with a low cost anode, preferably natural graphite with an aqueous binder. Seven blends were selected for scale up evaluation in 0.34Ah prismatic and 1.3Ah flattened, wound cells. Blend 5 was selected to use in the 1.3Ah wound prismatic cells to be provided as deliverables to the National Labs.

Task 3 – Electrolyte Development

The objective for electrolyte development in this program was to decrease cell impedance, especially at low temperatures, without impacting calendar life. A series of mixture experiments were conducted to optimize the carbonate solvent composition, salt mixture, and electrolyte additives. Preliminary screening of promising formulations was conducted in coin and small form factor prismatic cells, to narrow the window of compositional ranges and

additive types which were effective in achieving power objectives.

Task 4 – Cell Design

The targeted HEV LEESS cell design was for a 3.8Ah WFW prismatic in a sealed pouch. This design was new to A123, and the decision to employ WFW technology was driven by the need to reduce cost at the cell level. Despite significant reductions in cell materials costs and lower cost processes, there was still a significant gap between predicted and target system cost. A123 was able to leverage the prior experience of team members to accelerate development, and by the time of program termination, the initial prototypes had been assembled and testing initiated.

Task 5 – Module Design

A 71S1P module concept was designed to house the HEV LEESS cells, to provide a low cost alternative to PHEV module/pack design. Module development objectives were to design, test, and deliver fully functional module prototypes which included significantly reduced electronics, air cooling, simplified assembly process, and elimination of compliant pads between cells. This effort was intended to result in a “module as pack” which would not exceed 47% of total system cost (which includes anticipated cost reduction at the cell level). During the course of this program, the module hardware was developed and FEA evaluation conducted, BMS as developed, new electronics boards were developed and received for testing, air cooling system was designed and CFD models completed, and prototype assembly initiated.

Key Accomplishments:

The following lists accomplishments during the 18 active months of this program. USABC price goals were not anticipated to be fully achieved, however, a significant reduction in the gap was accomplished.

- Materials selection, electrode design, cell design, hardware and process development

were conducted on three prototype phases, a 0.34Ah stacked cell, a 1.3Ah wound, then flattened cell, and a 3.8Ah true WFW prismatic cell.

- Accelerated cycle life testing and high temperature storage testing were conducted to benchmark materials selection and wound, flattened 1.3Ah cell designs. Development of assembly capability for 3.8Ah cells was completed just as program was terminated. Cells were assembled however testing was limited due to program termination.
- A 71 cell HEV module was developed in support of cost reduction options. The module was in the process of being assembled at program end. Modeling of critical module characteristics was conducted, including FEA to assess impact of the new pressure plates and CFD to determine air flow and assess thermal impact with the air cooling system.
- Estimated system price was reduced by 53% based on cell and module achievements and projected further achievements through program end.
- Fifteen 6Ah cells were delivered to the National Labs for preliminary benchmark testing.
- Twenty 1.3 wound, flattened prismatic cells and ten test fixtures were provided to the National Labs for interim testing, to assess progress on WFW technology.

Conclusion:

The HEV LEESS Cell and Module Development program provided A123 Systems the resources to develop high power electrodes, a novel, low cost cell design, and an efficient module design. All developments were on track to meet the proposed system performance and pricing. Although the program was not able to run its full course, many key technologies associated with this funding are deployable across other A123 product lines, therefore significant value was achieved.

The final Gap Analysis table for the program is provided below. Life testing conducted during the course of this program used a high rate charge and discharge regime to accelerate the product development cycle time. Since these

test results do not conform to USABC standard protocol, they are not represented in the Gap Analysis, but are shown in the Program Technology Development sections of the detailed final report.

**A123 Packs vs. USABC Energy Storage System End-of-Life Performance Goals
HEV LEASS System**

Characteristics	Units	USABC PA Low Energy EOL Goals	A123 BOL Q3-2012	A123 EOP / EOL Projection
			3.8Ah Wound Prismatic Concept Interim Cell	3.8Ah Wound Prismatic Deliverable Cell
Discharge Pulse Power, 2 second	kW	55	66	56
Discharge Pulse Power, 10 second	kW	20	24	45
Regen Pulse Power, 2 second	kW	40	72	41
Regen Pulse Power, 10 second	kW	30	36	36
Maximum Current	A	300	120	300
Discharge Requirement Energy	Wh	56	591	56
Regen Requirement Energy	Wh	83	405	83
Energy Over Which Both Requirements are Met	Wh	26	405	284
Energy Window for Vehicle Use	Wh	165	541	395
Energy Efficiency	%	95	96	95
Cycle Life	cycles	300,000		300,000
Cold Crank Power at -30°C	kW	5		4.2
Calendar Life	years	15		15
Maximum System Weight	kg	20		20
Maximum System Volume	liter	16		16
Maximum Operating Voltage	Vdc	≤ 400	334	270
Minimum Operating Voltage	Vdc	≥ 0.55 V	167	185
Unassisted Operating Temperature Range	°C	-30 to 52		-30 to 52
30°C - 52°C % Energy	%	100		100
0°C % Energy	%	50		50
-10°C % Energy	%	30		30
-20°C % Energy	%	15		15
-30°C % Energy	%	10		10
Survival Temperature Range	°C	-46 to 66		-46 to 66
System Selling Price at minimum 100k units/year	\$	\$400		\$831
Battery Size Factor (BSF)			88	71

1.2.1 Computer Modeling Work

– None reported.

1.3 Deliverables/Products Developed

Fifteen 6Ah cells were delivered to the National Labs for preliminary benchmark testing.

1.4 Technology Transfer Activities

1.4.1 Proprietary Reporting

– Final Report was submitted to USABC on October 26, 2012.

1.4.2 Non-Proprietary Publications and Proceedings

– None reported.

2. USABC LEESS Program

Performing Organization: Maxwell Technologies, Inc.

Project Duration: 11/11/2010 – 3/31/2014

2.1 Executive Summary

The Maxwell-USABC Low Energy, Energy Storage System (LEESS) program was initiated to demonstrate the possible application of asymmetric capacitors (LiC) in the Power Assisted Hybrid Electric Vehicle (PA-HEV) market. PA-HEV applications are well suited to the combined power and energy density afforded by the hybrid LiC cell. Over the course of 38 months, the research built upon the company's existing asymmetric capacitor technology and significantly improved the performance of such devices in areas of operating voltage, low temperature performance, and cost effective manufacturing.

In order to increase the stable operating voltage from today's 3.6V towards a 4.2V cell, Maxwell advanced state-of-the-art of electrode and electrolyte technology. Low temperature performance was targeted through the development of advanced electrolyte solvent systems. Advanced manufacturing processes have been developed to ensure a cost effective approach for electrodes, asymmetric capacitors, and systems.

The program successfully produced the following:

- A 2200F Rated 1.1Ah LiC Pouch cell that, in the final configuration based on improvements completed beyond the program, indicates that it can be the basis for a pack that meets all USABC-PA-HEV Gap Chart requirements except system volume, weight and cost.
- A pack module that enabled the testing of the LiC cells in the full system

configuration, demonstrated a sophisticated module design, and provided a platform for automotive pack design experience.

- Lab-scale production equipment and processes to manufacture the cells and packs in limited quantity and validate key production metrics.
- A fully developed cost model for the Gen3 cell and FS Module (pack) based on 100K system annual demand.

2.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

Over the course of the USABC collaboration with Maxwell, the research built upon the company's existing asymmetric capacitor technology and significantly improved the performance of such devices in areas of operating voltage, low temperature performance, and cost effective manufacturing.

Objectives and Goals:

This project led by Maxwell was comprised of multiple participants and research facilities, and involved the pursuit of the following main objectives and goals.

Building on existing asymmetric capacitor technology, improve the performance of such devices in the following areas:

- Increase the stable operating voltage from currently 3.6V to 4.0V and up to 4.2V by advancing the electrode and electrolyte.
- Widen the power performance temperature window of existing hybrid technology to perform well at low temperature by advancing the electrolyte solvent system.

- Create a high volume cost effective manufacturing process for electrodes of asymmetric capacitor cells.

Develop a cost effective system solution that approaches the LEES targets:

- Develop a low cost separator technology and method of use that reduces cost from current levels.
- Perfect module architecture which eliminates excess weight, volume and cost from a high voltage ultra capacitor system.
- Advance electronics solutions for ultra capacitor system management beyond today's state of the art for low cost structure.
- Demonstrate program advancement by producing prototype units that features all the technical advancements and validate performance in a comprehensive testing regime.

Tasks:

The following is a brief summary of the tasks and sub-tasks performed during the program.

Task 1 – Electrode and Cell Development

This task was originally separated into Task 1 – Electrode Development and Task 4 – Electrode Design but the two were effectively merged in late 2011 into the current Task 1 – Electrode and Cell Development as the activities started to overlap and combine. Activities performed under Task 1 included the following:

- Cathode Development: Ultimately, standard Maxwell UCAP carbon, which has been optimized for performance and cost over a number of years, were incorporated as the cathode in cell builds.
- Anode Development: More than 12 carbons were evaluated for the anode and the selection was completed in 2012. Ongoing work outside the

program on the anode film, conducted since the cell design freeze for the production run, successfully reduced the anode film thickness to 60µm in 2013 on the existing equipment. Work to further reduce the thickness towards 50µm will likely require major equipment modifications.

- Anode Current Collector Process Development: The initial concept for copper anode perforation was mechanical pin penetration which proved a simple process but the resulting hole pattern was irregular and inconsistent and the holes had raised burrs which caused shorts and debris blocking the holes thus limiting the effectiveness of the perforation.
- Lithium Pre-Doping: Three methods of lithium pre-doping were investigated including Stabilized Lithium Metal Powder (SLMP), Li vapor deposition and electrochemical Li deposition.
- Electrode Pilot Line Scale Up: The electrode pilot line scale up began in 2011. Anode pilot line process development started in 2012 and work proceeded achieving 80µm anode film and functional electrodes in December 2012.
- Cell Development and Fabrication: The lab cell development was completed and Gen1 cells were delivered for National Lab testing in June 2011. Gen2 cell development started and the Gen2 cell build was delivered in 2012. Gen3 cell development started in 2012 and was completed in 2013. Gen3 cell fabrication started in 2013 and proceeded for most of the full year until 2013. Gen3 cells intended for final National Lab testing and

inclusion in the production LEES Pack Build were produced on the pilot-scale electrode and R&D cell build lines.

- The new anode formulation has been scaled up to pilot line production and the film thickness has been reduced to 60 um resulting in a 10% ESR reduction in the Gen3 cell format. Further process improvement activity has resulted in a 30% increase in process speed.

Task 2 – Electrolyte Development

The objective of the task was to expand the operating temperature window to -30°C to 52°C and to enable stable 4.2V operation. The electrolyte development started in Q1 2011 and still continues post-program. The initial work included a full literature search and the compilation of candidate electrolyte was identified in 2011. Next, an electrolyte purification process was developed by Maxwell which enabled higher voltage operation for the LiC cells. Sixteen electrolyte formulations were evaluated and two were identified for further testing.

Of those, one (E08) showed a 15% ESR improvement and stability at -30°C and was selected for cell fabrication in 2012. All Gen3 cells produced for the program include the control electrolyte.

Task 3 – Separator Development

The objective of the task was to identify or develop a separator that both lowered the cell material cost and improved cell performance (ESR). Separator development commenced in 2011 and proceeded until 2012. The task had two parallel tracks. The first was with Porous Power who worked to develop a variant of their proprietary separator to suit the LiC requirements. The second was an internal review of commercially available separators. More than 15 commercial separators were identified as possible candidates and evaluated.

Two separators showed slightly better ESR than the control separator but both were not yet in mass production and supply could not be assured. Because of the inability to develop or locate a separator with better cost and performance characteristics, the Gen3 LiC cells produced continued to use the control separator.

Task 4 – System Design

The design of the system pack (FS module) went through several design revisions dependent on the emerging optimal configuration of the 2200F Rated LiC pouch cell and the Gap Chart requirements for weight, size and cost.

Task 5 – Production Build

In the first part of the task, the GEN3 cell formulation and design configuration was frozen in order to begin production of quantities required for the production FS modules and PS modules. Over 600 GEN3 2200F Rated pouch cells were produced on Maxwell's electrode pilot line and cell fabrication pilot line. In the second part of this task, production LEES Pack Build initially resulted in a large number of scrapped cells. However, the requirement number of FS and PS modules were successfully assembled and packed for shipment to the National Laboratories. Because of the limited number of FS modules (packs) and PS modules available, a revised test plan was agreed upon with USABC. Prior to shipping the packs to the National Labs (INL, SNL and ANL), pre-screen tests were conducted to reveal performance issues, system operation issues or other quality related failure modes.

Conclusion:

The Maxwell USABC LEES program was comprised of multiple objectives and has been a challenging effort touching all areas of the R&D department and incorporating the assistance of several supportive sub-contractors. Specific technical issues emerged in subjects spanning core cell chemistry to electronic component selection. Those issues were successfully navigated such that one of the fundamental

objective – the technical proof-of-concept of LiC cells in the PA-HEV automotive application – has been demonstrated. Work completed outside the program has since improved both parameters and the Projected Gap Chart shows that, with the newer cell configuration, all performance criteria will be met and only system weight, volume and cost remain. Those issues are not unimportant and reflect key parameters that will determine the technology's viability in the PA-HEV or other EV market. Maxwell believes that the technical

success of the program is the first step in the development of the fully commercial product platform and the volume, weight and cost of the cell and associated packs are now the focus of subsequent ongoing work. Throughout the program, Maxwell maintained, updated and analyzed a cost model formatted in a custom MS Excel workbook for USABC.

Gap Analysis vs. USABC Goals:

The Gap Analysis at the end of the program is provided below.

USABC LEES PAHEV		USABC Required EOL		Statement of Work EOL		End of Program EOL		Forward plans to achieve targets:
End of Life Characteristics	Unit	PA (Lower Energy)		PA (Lower Energy)		PA (Lower Energy)		
2s / 10s Discharge Pulse Power	kW	55	20	55	20	55	20	reduce film thickness to reduce ESR
2s / 10s Regen Pulse Power	kW	40	30	40	30	40	30	
Maximum current	A	300		300		250		
Energy over which both requirements are met	Wh	26		26				reduce film thickness to reduce ESR
Energy Efficiency	%	95		95		96.1		
Cycle-life	Cycles	300,000 (HEV)		300,000 (HEV)		300,000 (HEV)		
Cold-Cranking Power at -30°C	kW	5		5		3		-30oC capable electrolyte
Calendar Life	Years	15		15		15		85% Cap, 150% ESR
Maximum System Weight	kg	20		22		35		
Maximum System Volume	Liter	16		25		33		
Maximum Operating Voltage	V _{dc}	<=400		<=400		320		
Minimum Operating Voltage	V _{dc}	>=0.55 V _{max}		>=0.55 V _{max}		0.56		
Unassisted Operating Temperature Range	°C	-30° - 52°		-30° - 52°		-20° - 52°		-30oC electrolyte, film thickness
30° - 52°	%	100		100		67		-30oC electrolyte, film thickness
0°	%	50		50		37		-30oC electrolyte, film thickness
-10°	%	30		30		21		-30oC electrolyte, film thickness
-20°	%	15		15		9		-30oC electrolyte, film thickness
-30°	%	10		10		6		-30oC electrolyte, film thickness
Survival Temperature Range	°C	-46 to +66		-46 to +66		-46 to +66		
Selling Price/System @ 100k/yr	\$	\$400		\$920		\$917		
Hardware Level		System		System		System		
Capacity	Wh					310		
Battery Size Factor (BSF)						80		

2.2.1 Computer Modeling Work

– None reported.

2.3 Deliverables/Products Developed

- Over 600 Gen3 cells were successfully produced with most being assembled into system packs.
- USABC formatted Cost Model submitted.
- All packs, PS modules and GEN3 cells except Pack #3 have been shipped or are being stored as agreed upon at the Q1 2014 Quarterly Program Meeting. Pack #3 is being held for shipment until the system board issue uncovered by the Pack #1

Element test is resolved (estimated May 30, 2014).

2.4 Technology Transfer Activities

2.4.1 Proprietary Reporting

- Final Report was submitted to USABC in April 2014.

2.4.2 Non-Proprietary Publications and Proceedings

- None reported.

Section E – Technology Assessment Program (TAP)

Section E – Technology Assessment Program Final Reports:

1. K2 Energy – Technology Assessment of Cells and Batteries	E-1
2. ActaCell – Technology Assessment of Soft Pouch Cells Based on Lithium Manganese Spinel.....	E-5
3. SKI – EV Technology Assessment	E-9
4. Leyden – Technology Assessment of 10Ah Lithium-Ion Pouch Cells for EV Applications.....	E-13
5. Farasis – Evaluation of High Capacity Cells for EV Applications	E-17
6. SKI – EV Technology Assessment Project.....	E-21

1. Technology Assessment of Cells and Batteries

Performing Organization: K2 Energy Solutions, Inc.

Project Duration: 8/3/2010 – 10/31/2011

1.1 Executive Summary

K2 Energy Solutions and USABC participated in a mutual technology assessment of K2's battery technology in a two-phase program where K2 produced, at the program outset, 54 units of their LFP165HES module (3.2V, 51Ah), that is currently a K2 commercial product used extensively by their partners and customers as a battery module for EV applications. Per the agreed upon Statement of Work, 20 of these modules were shipped to Idaho National Laboratory (INL) for performance testing, 14 were shipped to Sandia National Laboratory (SNL) and the National Renewable Energy Laboratory (NREL) for abuse and thermal testing, and 20 were retained at K2 for in-house performance test at K2's Henderson facility. In the second phase of the program, K2 fabricated 54 units of their 3.2V, 45 Ah "flat-pack" cell that has been in production since mid-2010. These 54 cells were allocated for testing as described above for the LFP165HES module. The goal of this testing was to evaluate K2's present battery technology against USABC targets and identify areas for improvement and additional development.

1.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

Project Objectives and Approach:

The goal of this TAP award for testing was to evaluate K2's present battery technology against USABC targets and identify areas for improvement and additional development.

All the cells evaluated under this program utilize K2's lithium iron phosphate against graphite chemistry. The 165HES module consists of 16 of K2's LFP26650EV cells connected in parallel. The assembly of this module is the subject of U.S. and PCT patent applications (U.S. #12/794,054 & PCT/US2010/037451). The LFP45 cell is currently being manufactured by K2's European partner, European Battery, at their Varkaus, Finland manufacturing facility.

Phase I – In Phase I of this effort, K2 was to fabricate 50 units of the LFP165HES battery modules and ship 18 of these to the National Laboratory designated by USABC. Fourteen additional units were to be shipped to SNL for abuse testing (two of these may be shipped to NREL for thermal testing prior to being forwarded to SNL). K2 was to retain the remaining 18 units for parallel testing at the Henderson facility. K2's test plan would utilize standard USABC tests and would be reviewed with USABC personnel prior to commencement.

Phase II – In Phase II of this effort, K2 was to ship eighteen 45Ah prismatic cells to the National Laboratory designated by USABC. Fourteen additional units would be shipped to SNL for abuse testing (two of these may be shipped to NREL for thermal testing prior to being forwarded to SNL). An additional 18 units were to be tested by K2 at its Henderson facility. K2's test plan was to utilize standard USABC tests and would be reviewed with USABC personnel prior to commencement.

The following tests were performed during the USABC award, with the summary of outcomes:

Test 1: Static Capacity Test – The static capacity test was run on eighteen 165HES cells and twenty LFP45 cells. Each pack was placed in a 30°C incubator and allowed to soak for a period of two hours. Afterwards, the pack was

charged at C/3 and discharged at C/3 three times, with an hour rest between each cycle. Finally, the pack was charged at C/3 and discharged at 1C three times. All discharge capacities were within 2%, as specified in the test plan.

Test 2: Hybrid Pulse Power Characterization Test (Low Current) – This test was performed with a calculated profile that was scaled for each type of cell. For the 165HES, it was a 30s discharge at 63.75A, 40s rest, 10s charge at 47.85A, and 360s discharge at 51A. For the LFP45, it was a 30s discharge at 52.27A, 40s rest, 10s charge at 39.23A, and a 360s discharge at 42A.

Test 3: Self-Discharge Test – This test was performed by discharging at a C/3 rate for a C/3 static capacity, then charging it to 50% SOC and placing the packs in a 30°C incubator for 7 days. After discussing the results with INL, it seems likely that 165HES packs 31 and 32 developed leaks as a result of the prior thermal performance test.

Test 4: Thermal Performance Test – A C/3 discharge and low current HPPC test was performed on four 165HES cells at -30°C, -10°C, 0°C, and 50°C. The low current HPPC test parameters used were a 30 second discharge at 63.75A, 40 second rest, 10 second charge at 47.85A, and 360 second discharge at 51A. This was then repeated nine times.

Test 5: DST Cycle Life Test – A DST load profile was applied to two 165HES cells and two LFP45 cells at 30°C. A reference performance test (RPT) was performed monthly. The 165HES modules were out to RPT4 at the end of the project and the LFP45 cells were out to RPT2 at project end due to their later starting date.

Test 6: Calendar Life Test – Sixteen cells of each type were divided among four temperatures: 30°C, 40°C, 50°C, and 60°C. The pulse per day load profile specified in the test plan was applied daily. An RPT test was performed monthly.

Gap Analysis vs. USABC Goals

EV Targets	BOL Target	165HES (module)	165HES (system)	LFP45 (cell)	LFP45 (system)
Power Density(W/L)	460	393	328	NA	NA
Specific Power – Discharge, 80% DOD/30 sec(W/kg)	300	284	247	NA	NA
Specific Power -Regen, 20% DOD/10 sec (W/kg)	150	320	278	NA	NA
Energy Density -C/3 Discharge Rate(Wh/L)	230	123	103	281	234
Specific Energy - C/3 Discharge Rate(Wh/kg)	150	89	77	176	153
Specific Power/Specific Energy Ratio	1:2	3.2	3.2	NA	NA
Total Pack Size(kWh)	40	40	40	40	40
Calendar Life(Years)	10	NA	NA	NA	NA
Cycle Life - 80% DOD (Cycles)	1,000	NA	NA	NA	NA
Selling Price - 25,000 units @ 40 kWh(\$/kWh)	<150	550	660	615	738

1.2.1 Computer Modeling Work

- None reported.

1.3 Deliverables/Products Developed

54 units of K2's LFP165HES battery modules were produced in total and used as follows:

- 20 modules were shipped to Idaho National Laboratory for performance testing.
- 14 modules were shipped to Sandia National Laboratory and the National Renewable Energy Laboratory for abuse and thermal testing.

- 20 modules were retained at K2 for in-house performance testing.

1.4 Technology Transfer Activities

1.4.1 Proprietary Reporting

- Final Report was submitted to USABC on September 30, 2011.

1.4.2 Non-Proprietary Publications and Proceedings

- None reported.

2. Technology Assessment of Soft Pouch Cells Based on Lithium Manganese Spinel

Performing Organization: ActaCell, Inc.

Project Duration: 8/5/2010 – 10/31/2012

2.1 Executive Summary

ActaCell participated in USABC's Technology Assessment of ActaCell's soft pouch cells based on Lithium Manganese Spinel. The cells were designed to meet the requirements set forth in the guidelines for low energy-energy storage systems (LEESS) for power-assist hybrid electric vehicle (PA-HEV) applications. The cells were distributed in the following manner:

- 6 cells to be tested at ActaCell for performance testing.
- 18 cells to be tested at ANL (USABC) for performance testing.
- 12 cells to be tested at Sandia (USABC) for safety/abuse testing.
- 3 cells to be tested at NREL (USABC) for thermal analysis.
- Performance testing includes calendar life tests, hybrid pulse cycling, cold crank, characterization and reference performance tests (RPT).

2.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

ActaCell Energy Systems is a materials development company focused on rechargeable batteries. Current work focuses on high powered cathode materials based on manganese spinel, as well as high energy anode nano-composite materials based on antimony and silicon. Located in Austin, Texas, ActaCell develops materials and cell designs in a facility that includes a dry room, materials laboratory, electrode coater, calendar, and stacker

machines, electrode material production equipment, thermal test chambers, and over a hundred channels of cell and module cyclers. ActaCell became a wholly owned subsidiary of Contour Energy Systems in July 2012.

Objectives and Goals:

The goal of this TAP award for testing was to evaluate ActaCell's present Lithium Manganese Spinel battery technology against USABC targets and identify areas for improvement and additional development.

Approach:

ActaCell was in development of an 8Ah cell targeting the medium and heavy duty HEV market at the time of the start of the program. Although the amount of energy was double what the LEESS program required, it was assumed that the cell's performance could be scaled after the program was complete. ActaCell does not have pilot scale manufacturing capability, so ActaCell contracted the assembly of the cells to Eagle Picher Technologies (EPT) of Joplin, Missouri. Although EPT is an experienced lithium-ion cell manufacturer for the aviation and aerospace markets, they had not yet assembled a soft pouch cell. As part of the contract build agreement, ActaCell assisted EPT with the purchase of the soft pouch sealing equipment needed to complete the cells. The ordering, delivery, and installation of this machinery dictated that the program be put on hold for a few months. After a few delays, the cells were finally built. In retrospect, multiple runs should have been made to get familiar with the equipment, dial in various parameters, and prove consistency in production.

However, due to time constraints, ActaCell accepted the first 75 cells to come off the line. These 8Ah cells were cycled and delivered to USABC for analysis. It soon became apparent

that many of the cells experienced inconsistent capacity cycling, changing their value depending on time spent between cycles. It is believed that due to limited wetting procedures and experience, the 8Ah cells did not get adequate electrolyte wetting for full electrode coverage. This caused difficulty in gauging the true cell capacity and led ActaCell and USABC managers to conclude that ActaCell should remake the cells by hand in Austin. This allowed ActaCell to redesign the cell to the more appropriate size 4Ah cell (for LEES goals).

Although making the cells by hand allowed for more control over construction, it was laborious and time consuming. Yields were less than what would come from a fully automated facility, but 18 cells were able to be sent to ANL for performance testing.

Sandia National Lab received cells from the EPT 8Ah build, while NREL received cells from both the 8Ah and 4Ah builds.

Tests Performed:

During this TAP program, the following tests were performed:

- Calendar/Storage at 30, 40, 50, 60°C (#1520, #1542, #1554, #1555)
- Hybrid Cycle Life (#1527)
- Cold Crank (#1525)

Testing Results:

To meet the requirements of the LEES program, it was determined that a 4Ah cell should be built using a battery scale factor (BSF) of 74. Since a limited number of cells were available for test purposes, ActaCell was only able to use one cell in each of the desired tests, rather than the preferred amount of three.

Gap Analysis vs. USABC Goals

LEES EOL Goals			4 Ah Cell BOL		4 Ah Cell 150 days	
End of Life Characteristics	Unit	PA (Lower Energy)	ActaCell 74S Pack		ActaCell 74S Pack	
2s / 10s Discharge Pulse Power	kW	55 20	27	28	28	28
2s / 10s Regen Pulse Power	kW	40 30	38	36	36	36
Discharge Requirement Energy	Wh	56	74	78	78	78
Regen Requirement Energy	Wh	83	106	100	100	100
Maximum current	A	300	300	300	300	300
Energy over which both requirements are met	Wh	26	349	249	249	249
Energy window for vehicle use	Wh	165	529	427	427	427
Energy Efficiency	%	95	96	96	96	96
Cycle-life	Cycles	300,000 (HEV)			currently at 120k	currently at 120k
Cold-Cranking Power at -30°C	kW	5			at 60% DOD	at 60% DOD
Calendar Life	Years	15				
Maximum System Weight	kg	20	20	20	20	20
Maximum System Volume	Liter	16	10.8	10.8	10.8	10.8
Maximum Operating Voltage	Vdc	≤400	310.8	310.8	310.8	310.8
Minimum Operating Voltage	Vdc	≥0.55 V _{max}	183	183	183	183
Unassisted Operating Temperature Range	°C	-30 to +52	-30 to +52	-30 to +52	-30 to +52	-30 to +52
30° -52°	%	100	100	100	100	100
0°	%	50				
-10°	%	30				
-20°	%	15				
-30°	%	10				
Survival Temperature Range	°C	-46 to +66	-46 to +66			
Selling Price/System @ 100k/yr	\$	400	\$ 1,365.00	\$ 1,365.00	\$ 1,365.00	\$ 1,365.00

2.2.1 Computer Modeling Work

– None reported.

2.3 Deliverables/Products Developed

Sandia National Lab received cells from the EPT 8Ah build, while NREL received cells from both the 8Ah and 4Ah builds.

2.4 Technology Transfer Activities

2.4.1 Proprietary Reporting

– Final Report submitted to USABC in December 2012.

2.4.2 Non-Proprietary Publications and Proceedings

– None reported.

3. EV Technology Assessment

Performing Organization: SK Innovation Co., Ltd.

Project Duration: 11/8/2010 – 12/31/2011

3.1 Executive Summary

SK introduced their 25Ah cell called E250 for USABC EV technology assessment. The E250 has high energy density of 148Wh/kg maintaining high power and reliable life performance. SK has focused on improving both performance and safety of the cell. The technology for E250 enables electric vehicles to be more safe and reliable. SK sources materials which meet requirements for robustness and safety, and the cell design is also focused on life reliability and safety. Cost is the key factor SK expects to achieve as well as performance. SK has reduced the cost by integrating materials and decreasing the number of parts in the cell and pack. SK believes that mass production in Seosan could be helpful to achieving large cost reductions. To make the EV with long-range driving distance, SK aims to effectively design future cells in terms of weight and volume.

3.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

The SK E250 cell is a pouch type design, which can be used for various applications and has high specific energy density. It has higher safety characteristics than the steel container type Li battery. The pouch-type cell design has additional advantages such as effective cooling, vibration resistance, and higher cost performance with less number of parts. The cell's high power and energy characteristics

mainly originate from blended Mn-spinel cathode/surface modified graphite chemistry and unique separator technology, which is a result of high-rate electrode coating technology which enables a thinner and more uniform electrode. SK cells have excellent safety level based on proprietary ceramic-coated separator technology, Mn-spinel cathode and pouch-type cell design. Both ceramic coating layer and heat resistant base film separator impart high thermal stability to SK batteries. Additionally, Mn-spinel is known to be one of the safest cathodes.

Objectives and Approach:

The TAP project led by SK proposed the 25Ah cell for this program, and established the goal of confirming high power characteristics, and reliable cycle life performance through this program.

The project's approach was to evaluate SK's GEN2.0 cells versus USABC goals through the following three tests:

1. High energy density EV LIPB development with maintaining high power.
2. EV LIPB for long cycle life and calendar life performance.
3. EV LIPB development with high abuse tolerance.

All tests were performed according to the USABC test procedures. The life characteristics were evaluated through both cycle tests and high temperature storage tests. Abuse tests were also performed by SK and SNL in the program. Test cells that SK sent to ANL, SNL and NREL are listed in table below.

Test Items			Sample	In-house 35 cells	ANL 21 cells	SNL 12 cells	NREL 3 cells
Cycle Life Tests			6 cells	○	○		
Accelerated Calendar tests	SOC 100%	25°C	3 cells	○	○		
		35°C	3 cells	○	○		
		45°C	3 cells	○	○		
		55°C	3 cells	○	○		
		55°C (Re-test)	3 cells	○			
		55°C (w/pressure)	2 cells	○			
	SOC 40%	55°C	3 cells	○	○		
Abuse tests			12 cells	○		○	○

Test Results:

Cycle Life Tests – SK achieved the E250 cycle life target, which is capacity retention higher than 80% after 1,000 cycles. It is expected that E250 could achieve 1,800 cycles following the current trend.

Accelerated Calendar Life Tests – A total of 17 cells were used for the calendar life tests. From the trends, It is clear that calendar life is dependent on temperature and SOC, and there was a critical temperature.

Life Estimation – Based on calendar life test results, SK estimated the life of E250 by assuming that capacity loss is proportional to square root of storage time. E250 is guaranteed for 3.1 years.

Improvements of Performance at High Temperature – After several electrolyte optimization experiments to improve the E250 cell performance, SK decided not to change the chemistry. The new life estimate was calculated of improved cells. Through the same estimation method with E250, it might be guaranteed 16.8 years at 30°C in case of 60% SOC.

The company has also succeeded in commercializing Li-Ion Battery Separators through proprietary technology development, which will play a crucial role in providing competitive advantage to its LiPB business

Conclusions:

SK has performed the USABC technical assessment program with E250 which represents standard EV cell of SK. It shows high power, long cycle life and safe characteristics which are resulted from the materials and cell design. Program test items were cycle life tests, accelerated calendar life tests and abuse tests. The final report contains one year test results of the program.

Cycle life tests at 30°C have proceeded for 1,050 cycles. The capacity retention and the power capability after 1,050 cycles remain 86.7% and 93% each. 750 more cycles for the cycle life tests could go on until EOL. Therefore, E250 could exceed the USABC's minimum cycle life goal.

The calendar life test results for 44 weeks were 90.3%, 86.4% and 75.3% at each temperature of 25°C, 35°C and 45°C. Power retentions were 98.2%, 95.6% and 7.4%. The value of 7.4% was obtained because the capacity dropped below 20% of initial capacity by peak power method. In case of 55°C storage, capacity dropped below 80% and DC-IR increased sharply in 16 weeks. The main reason for degradation is Mn-dissolution of LMO. It caused electrolyte decomposition and cell swelling. Through the previous work, SK found solutions to improve high temperature characteristics. SK optimized electrolyte and electrolyte wetting property. The

electrode interface reaction has been also enhanced. In addition of these results, they have been trying to change chemistry reducing blending ratio of Mn-dissolution source, and have improved high temperature performance with changing chemistry. Therefore, the product can achieve excellent high temperature performance as well as cycle life.

SK also can confirm the high level of safety of E250. Over discharge and blunt rod test results were L2 of EUCAR hazard level. Short circuit and overcharge test results were L3 and thermal stability test results got L4. Therefore, E250 cells showed no fire or no explosion at all abuse tests. In conclusion, SK's cell showed excellent characteristics, exceeding USABC's minimum goals for long term commercialization.

Future Work Planned:

SK proposed a 40Ah cell with high energy density of 200Wh/kg for follow up program. They are highly confident that their cell will ultimately meet USABC's long term goal.

3.2.1 Computer Modeling Work

– None reported.

3.3 Deliverables/Products Developed

21 cells were delivered for testing at ANL, 12 cells for SNL and 3 cells for NREL.

3.4 Technology Transfer Activities

3.4.1 Proprietary Reporting

– Final Report was submitted to USABC dated February 10, 2012.

3.4.2 Non-Proprietary Publications and Proceedings

– None reported.

4. Technology Assessment of 10Ah Lithium-Ion Pouch Cells for EV Applications

Performing Organization: Leyden Energy

Project Duration: 8/13/2010 – 11/7/2011

4.1 Executive Summary

The objective of the USABC – Leyden Energy Technology Assessment Program was to undertake a series of tests to measure the performance, cycle life, accelerated calendar life and abuse tolerances of 10Ah electric vehicle (EV) prismatic pouch cells made by Leyden Energy and thus assess their potential for use in EV batteries by USABC members. Testing was performed on 68 units at Leyden Energy, Idaho National Laboratory (INL), and Sandia National Laboratory (SNL). Specifically, the USABC – Leyden Energy TAP project evaluated Leyden Energy's 10Ah rechargeable lithium-ion pouch cell for their applicability for use in EVs. The cell chemistry was proprietary Leyden Energy technology which utilized lithium imide electrolyte salts and a graphite foil cathode current collector in place of the aluminum current collector normally used in standard cell construction. Prior tests by Leyden Energy with 18650 cells had indicated that this new cell architecture results in improved performance, life, and abuse tolerance at above ambient temperatures. The 10Ah lithium-ion pouch cell achieved some of the program objectives, demonstrating excellent energy density and safety, but also generated inconclusive data on high temperature calendar life. Through this program, Leyden Energy and the USABC intended to determine whether the same improvements can be realized in larger capacity prismatic pouch cells that are more appropriate for use in future EVs.

4.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

The scope of this program incorporated two distinct cell builds and delivery of cells from both builds to INL and SNL for testing. Specifically, Leyden Energy supplied 68 10Ah flexible pouch cells for testing. The cells were delivered according to the following schedule:

- 2 months after start of program 10 cells from Build 1
- 5 months after start of program 58 cells from Build 2.

These cells consisted of a nickel-cobalt-manganese (NCM) cathode and graphitic carbon anode, utilizing a 45 micron thick graphite foil current collector on the cathode side and a 10 micron thick copper current collector on the anode side. Cell electrolyte consisted of a lithium imide salt dissolved in a mixed carbonate solvent based solution with several SEI forming additives.

Concurrent with testing at INL and SNL, Leyden Energy conducted parallel internal evaluations following test plans from INL and SNL.

Objectives and Goals:

The key objective of the USABC – Leyden Energy Technology Assessment Program was to undertake a series of tests to measure the performance, cycle life, accelerated calendar life and abuse tolerances of 10Ah EV prismatic pouch cells made by Leyden Energy and thus assess their potential for use in EV batteries by USABC members, as well as to set improvement goals for future battery components.

Tests Performed:

Energy Density – Using the USABC Static Capacity test protocol, Leyden Li-ion pouch cells were able to achieve approximately 10.8Ah versus the initial design target of 10Ah. With these results, Leyden was able to achieve the following values for the energy density and specific energy, which were greater than 150Wh/kg and nearly 300Wh/l at the cell level for the 10Ah prismatic Li-ion cells.

Power Density – The Leyden Energy Li-ion pouch cells can sustain an approximate 3C discharge and achieved 378W/kg at the cell level. Leyden met all of the targets listed by INL.

Self-Discharge Test – The cells exhibited low self-discharge during the seven day storage test. This short term test was listed in the INL test plan as a test drawn from USABC Electric Vehicle Battery Test Procedures Manual, Rev 2, DOE/ID-10479, January 1996.

Cycle Life – Cycle life was measured using a 0.5C constant current charge to 4.0V, allowed by a constant voltage mode at 4.0V until the rate dropped to 0.05C. Discharges were done at 1C rate. Between each charge and discharge the cells were rested for 30 minutes. Every 25 cycles a 0.2C discharge was used. Linear results were achieved to approximately 850 cycles at 20°C and the cell continues to cycle. At 40°C 750 cycles were achieved prior to the cell hitting a knee and fading.

Calendar Life – Calendar life testing was done on cells at 100% SOC (4.0V) at 30°C, 40°C, 50°C and 60°C. During the life testing a once-per-day calendar life pulse was used. This was modified from the manual to be the same as the EVPC profile, with an additional clamp charge step:

- Step 1: 30s discharge at 10A
- Step 2: 40s rest
- Step 3: 10s charge at 7.5A

- Step 4: taper charge at C1/3 rate to target voltage for 900s.

Reference Performance Tests (RPTs) were not done on these cells at Leyden, but were done at INL. Anomalies were observed in the behavior of 10Ah pouch cells during calendar life testing at elevated temperatures. The fact that the results for 50°C were inferior to that of 60°C indicated that these results did not reflect actual chemistry issues, as the cell behavior should have been reversed if the degradation was temperature related. Leyden Energy believes that these results were due to cell construction issues.

As a consequence of the inconsistent results from the 10Ah pouch cell builds, Leyden repeated the calendar life testing using the high volume production INR-18650-CE cells. Only small differences were seen in the end-of-discharge and end-of-charge voltages during the pulses over the time period of the calendar testing for these INR-18650-CE cells. The results for the multiple cells at each temperature and for each SOC were very consistent. There were no discrepancies between different temperatures as seen in the pouch cell builds, agreeing with the diagnosis of issues with the large cell construction. A tremendous improvement in calendar life was seen over the large pouch cells.

Reference Performance Tests – INL ran a set of cycle life and calendar testing using 16 pouch cells. Cycle life was measured at 30°C using the DST discharge profile. Calendar tests were done at 30°C, 40°C, 50°C and 60°C. RPTs on the pouch cells were performed monthly. Discharge capacity and energy was measured. INL's test data is available in a separate report from INL. Similar to the INL pouch cell testing, Leyden performed RPTs on the INR-18650-CE.

Abuse Tolerance Tests – These consisted of overcharge tests and heating tests in various test configurations.

4.2.1 Computer Modeling Work

– None reported.

4.3 Deliverables/Products Developed

Leyden Energy supplied 68 10Ah flexible pouch cells for testing.

4.4 Technology Transfer Activities

4.4.1 Proprietary Reporting

– Final Report was submitted to USABC in late 2011.

4.4.2 Non-Proprietary Publications and Proceedings

– None reported.

5. Evaluation of High Capacity Cells for EV Applications

Performing Organization: Farasis Energy, Inc.

Project Duration: 6/11/2012 – 12/31/2013

5.1 Executive Summary

The goal of this 16-month Technology Assessment project was to demonstrate the potential to achieve world-leading performance and capacity in a large prismatic pouch cell suitable for EV applications utilizing Farasis Energy technology based on high capacity layered/layered NCM cathode material originally developed at Argonne National Laboratory (HE-NCM). The base cathode material for the project was supplied by BASF, a licensed material manufacturer that is developing this new material for the Li-ion industry. The project would provide USABC and DOE the first direct assessment of the Farasis approach to enabling the use of this new, high capacity cathode material and the first assessment of a material supplied by a licensed manufacturer.

Farasis were able to achieve some of the objectives of the project but fell short of others. The key accomplishments at the conclusion of the project were Farasis post-processing two different generations of BASF material, building cells, testing and delivering to the National Labs. During the USABC project various material development, cell development and cost modeling activities took place, with the final cells meeting the energy, power and temperature targets. With respect to DST cycle life and calendar life, the cells continue testing and more data is required for a robust model and prediction to be made. With respect to the USABC cell cost targets, more development is required to meet the gaps.

5.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

For this USABC Technical Assessment project Farasis Energy, Inc proposed to build and provide prototype Li-ion pouch cells based on Farasis Energy technology designed around high capacity, layered-layered NCM cathode material supplied by BASF, a fully licensed cathode material supplier.

Objectives and Goals:

The overall goal of the USABC Technology Assessment project was to demonstrate the potential to achieve high performance and capacity in a large prismatic pouch cell suitable for EV applications utilizing Farasis Energy technology based on high capacity layered/layered NCM cathode material originally developed at Argonne National Laboratory (HE-NCM). The cathode material for the project was supplied by BASF, a licensed material manufacturer that is developing this new material for the Li-ion industry. The cells were designed around the BASF cathode material which underwent a secondary, proprietary coating/surface stabilization process at Farasis. The cells were built at Farasis' manufacturing facility in China.

Approach and Key Tasks:

Farasis proposed to build 52 each, approximately 30Ah, prismatic Li-ion pouch cells, 34 of which were to be provided to USABC for performance and safety testing and calorimetric characterization. In addition, Farasis agreed to provide 18 each small prototype pouch cells used for internal development with a capacity of ~ 2.0Ah for additional testing by USABC. The duration of the program was proposed to be approximately 16 months, including ~ 8 months of

performance testing at Farasis and the National Laboratories assigned to test the cells.

The two main tasks for this project included:

Task 1 – Cell Manufacturing

Manufacture and supply at least 52 each 30Ah or higher capacity prismatic pouch cells and at least 18 each 2.0Ah prismatic pouch cells.

Task 2 – Cell Testing

Test the cells according to agreed upon protocols with USABC in parallel to independent testing occurring at the National Laboratories.

A summary of the status of the objectives and key findings at the end of the project is given below:

- Farasis successfully built large pouch cells using both the FEI-2 and FEI-3 Cathode material. 75 large pouch cells were built and 42 were delivered to the various National Laboratories for evaluation. The initial energy density of the cells at C/3 discharge rate at 30°C is approximately 210Wh/kg, significantly lower than the original target of 250Wh/kg. A major reason for missing the target was based on the decision to limit the charging voltage to 4.5V instead of 4.55V to increase cycle life and to not use a constant voltage “taper” charge at the top of charge for the same reason.
- The testing of the large deliverable cells was to begin in early 2014 and Farasis intends to provide detailed benchmarking against the other aspects of the USABC EV battery system performance, safety and thermal goals.
- The technology still has a number of weaknesses that likely prevent its commercial use at this stage. In particular, the impedance, impedance growth and capacity loss, particularly if the cells are held at high states of charge, result in major limitations on the theoretical potential of these high capacity cathode materials.

- Farasis was able to show that its secondary processing technology for HE-NCM type cathode materials does improve the performance of the HE-NCM materials. However, it was also found that the base material can have a major impact on the cell performance and process optimization is required for any change in cathode material morphology or composition.

Gap Analysis Relative to Key Program Accomplishments:

Farasis built ~70 each 30Ah Li-ion cells using the FEI-3 version of the BASF HE-NMC cathode material. Of these cells 21 were delivered to Idaho National Laboratory for performance testing, 16 were delivered to Sandia National Laboratory for safety testing and six were delivered to NREL for thermal characterization. The table below shows the gap analysis for the Farasis deliverable FEI-3 30Ah Li-ion pouch cells showing the beginning of life status based on initial testing and the current status of cells cycling at Farasis. During this project Farasis was successful in demonstrating the capability to stabilize the HE-NCM BASF cathode material for cycling at elevated voltages and capacities, achieving >600 cycles in cells with minimal excessive impedance growth and energy loss. The original energy density goal of 250Wh/kg, based on initial cell designs and materials was not met in the final deliverable cells.

The barriers to achieving that energy density included a decision related to enhancing the cycle life achievable by these system including lowering the upper voltage cutoff and not including a constant voltage taper charge as part of the charge profile. In addition, the final FEI-3 cathode electrodes exhibit slightly greater impedance reflected in a lower average voltage than the FEI-2 cathode electrodes used to make prototype large cells. The remaining deficiencies of the HE-NCM material related to impedance

and voltage fade are being addressed by Farasis through ongoing materials development efforts in collaboration with Argonne National Laboratory.

Future Work Planned:

Farasis will continue its work on these high capacity high voltage Li-ion battery systems to address the remaining issues and work to fully enable the potential of this new class of cathode material. BASF has suspended pilot scale work on the HE-NCM material but is continuing lab scale efforts. Based on this, Farasis plans to continue its work by sourcing precursor material from another supplier so that it can synthesize

and control morphology and composition through out the development process and not depend on a secondary partner that makes these decisions independent of Farasis.

Under new project awards, Farasis are directly sourcing precursor material and synthesizing the final cathode materials. While the focus of those projects will be to demonstrate the technology in smaller pouch cells, Farasis plans to build on the large pouch cell design developed under this USABC project to make cells using the same “30Ah” pouch cell form factor incorporating the improved technology. These cells will be available to any entities that are interested in evaluating them near the end of those projects.

Gap Chart for 30 Ah FEI-3 Li-ion Pouch Cells

Parameters (Units) of Fully Burdened System	Minimum Goals for Long Term for Commercialization	Long Term Goals	Farasis System Goal (est. for 30 Ah Cells)	Farasis Deliverables FEI-3 30 Ah Cells (BOL)	Farasis-3 30 Ah Cells (Current Status)
Power Density (W/L), 80% DOD/30 sec	460	600	740	860	
Specific Power - Discharge, 80% DOD/30 sec (W/kg)	300	400	400	490	
Specific Power - Regen, 20% DOD/10 sec (W/kg)	150	200	200		
Energy Density - C/3 Discharge (Wh/L)	230	300	380	350	330
Specific Energy - C/3 Discharge Rate (Wh/kg)	150	200	200	205	190
Specific Power/Specific Energy Ratio	2 : 1	2 : 1	2 : 1	2.3 : 1	
Total Pack/Cell Size (kWh)	40	40	40		
Life (Years)	10	10	8		
Cycle Life - 80% DOD (Cycles)	1000	1000	400	0	78
Power and Capacity Degradation (% of rated spec)	20	20	20	0	4.8
Selling Price - 25,000 units @ 40 kWh (\$/kWh)	<150	100	<180		
Operating Environment (°C)	"-40 to +85, 20% Performance Loss, (10% Desired)	-40 to +85	-33 to +55		
Normal Recharge Time (hr)	6 hours (4 hours desired)	3 to 6 hours	6 hours	5 hours	
High Rate Charge	20-70% SOC in <30 min @ 150 W/kg (<20 min @ 270 W/kg desired)	40-80% SOC in 15 min	40-80% SOC in 30 min		
Continuous discharge in 1 hr - No Failure (% of rated energy capacity)	75	75	75		

5.2.1 Computer Modeling Work

– None reported.

5.3 Deliverables/Products Developed

65 Large FEI-3 Pouch Cells (42 Distributed to the National Laboratories as outlined below):

- 21 cells to INL for Performance Testing
- 16 cells to Sandia National for Safety Testing
- Cells to NREL for Thermal Evaluation.

Small pouch cells were built and delivered to INL for evaluation but were recalled after issues were found with their performance in testing at Farasis.

Testing will be ongoing at Farasis and the National Laboratories.

Approximately 70 each FEI-3 30Ah deliverable cells were built at the factory and completed at the end of June. 65 cells were delivered to Farasis in Hayward. Of these cells, 21 were shipped to INL, 16 cells were shipped to Sandia, and 5 cells were shipped to NREL at the end of June.

5.4 Technology Transfer Activities

5.4.1 Proprietary Reporting

– Final Report was submitted to USABC in 2014.

5.4.2 Non-Proprietary Publications and Proceedings

– None reported.

6. EV Technology Assessment Project

Performing Organization: SK Innovation, Ltd.

Project Duration: 9/28/2012 – 1/31/2014

6.1 Executive Summary

SKI introduced the E400 cell for a new USABC EV Technology Assessment Program (TAP) as a follow-on to a previous evaluation of the Balanced E250 battery which concluded in 2011. SKI set a goal of higher energy density and improved calendar life maintaining good cycle life and stability. First, SKI increased the ratio of NCM whose energy density is higher and calendar life performance is better than those of Mn-spinel. By applying shell-core NCM, structural instability of Ni²⁺ is also improved and this means an improved cycle life can be expected. Secondly, SKI optimized cell design by modification of electrode formula and electrolyte additives, and were able to get higher power output both at room temperature and at low temperature than the previously developed E400. SKI could also expect comparatively more improved life performance. Lastly, to achieve even higher life performance at high temperature, Manganese-Spinel (LMO) was completely removed, and an optimized electrolyte introduced. With these developments, the new LMO-free E400 battery is expected to show significantly enhanced performance. A remaining concern of the LMO-free 400 battery is safety, which is likely to be addressed using a stable ceramic-coated separator.

6.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

In the middle of the ongoing USABC TAP program, SKI introduced LMO-free E400 cell in an attempt to obtain improved calendar life. Therefore, previous versions of E400s were withdrawn from long-term performance tests.

The USABC assessment program 2012 was originally planned to be completed in September 2013 but SKI applied for a year's extension to evaluate and report life performance trends longer than 10th RPT of LMO-free E400. This report includes all results of testing conducted on LMO-free E400.

Objectives and Goals:

The overall goal of this TAP award was to evaluate and improve SKI's E400 system for higher energy performance and calendar life, with improved safety attributes.

TAP Tests:

During this program, the following types of tests were performed on LMO-free E400 cells.

Core Tests – Core tests have been performed with 18 cells of LMO-free E400; 6 for cycle life and 12 for accelerated calendar life tests. SKI followed USABC test manual and specific test conditions.

Cycle Life Tests – After successfully completing the core tests, cycle life test have been conducted with 6 cells. RPTs have been conducted at every 100 cycles (~1/month).

Accelerated Calendar Life Tests – 12 cells were tested for accelerated calendar life; 3 cells with SOC 100% are stored at each temperature of 25, 35, 45, and 55°C. RPTs have been conducted at four week intervals.

Life Estimation – SKI developed their own life modeling method. In regards with calendar life estimation, they have developed life model referring to USABC Battery Life Estimator manual. Cycle life and calendar life estimation were carried out using more than 10 months of life data. Initial results indicate that when LMO-free E400 takes a purely drive mode, it can run more than 248,000 miles with 343kWh of energy throughput (per unit cell; 91.0MWh per

pack system) based on 80% of capacity retention.

Calendar life estimation was also conducted using a semi-empirical model based on Arrhenius behavior and applying test data. On 80% retention basis, LMO-free E400 can maintain 3.1 years, 3.6 years, 4.6 years, and 9.8 years in 30°C, Phoenix, Honolulu, and Minneapolis, respectively. This is the result from SOC100% storage, and when SOC conditions responding real life are applied, calendar estimated life will be much longer than this. From accumulated NCM cell experiences of SKI, calendar life of SOC 50% is approximately five times longer than that of SOC 100%, and calendar life of lower end SOC is approximately 20 times longer than that of SOC 100%. Thus, it is considered to last more than 10years when real life SOC conditions are applied to LMO-free E400.

Abuse Test Results of LMO-Free E400 – All abuse test items passed with no fire, no explosion except thermal ramp and overcharge. SKI conducted overcharge tests according to test method from SNL, and for obtaining more information, 5V overcharging was additionally included in the tests. It was found the LMO-free E400 is very safe up to 5V with 36°C of maximum temperature. However, the cell could not hold up to 6V overcharging and it is considered that structural instability of Ni raised partial pressure of oxygen and this brought other chemical reaction with large amount of gases to be vented out at 6V.

Conclusions:

In conclusion, SKI has developed LMO-free E400, and this battery cell has shown several achievements and can produce high energy

density, long life (cycle and calendar performance) and stable safety. Firstly, LMO-free E400 satisfies the USABC goal for total pack energy of 40kWh and system specific energy of 150Wh/kg. Secondly, based on capacity retention of 80%, cycle life is expected to exceed 2,000 cycles and calendar life is at least 100 weeks at 35°C and SOC 100% condition, which is far superior life performance and also exceeds the USABC life goal. Thirdly, abuse tests were carried out and LMO-free E400 showed safe and stable behavior in thermal stability, penetration, short circuit and overdischarge tests. In thermal stability, the cell lasted up to 200°C with 30 minutes of hold time at each temperature step and maximum temperatures in other tests were 52°C, 104°C and 56°C in penetration, short circuit and overdischarge, respectively. Overcharge tests were also conducted and LMO-free E400 showed very stable behavior up to 5V. In 6V overcharge tests, it generated large amount of gases and venting occurred. In order to improve overcharge, cell design modification will have to be followed such as adjustment of electrolyte additives for suppressing over-potential chemical reaction, enhancing of cathode materials and application of heat stability reinforced CCS.

Future Work Planned:

SKI would like to continuously develop and modify cell design including electrolyte optimization and adjustment of cathode formulation in order to improve life performance further.

Gap Analysis vs. USABC Goals:

The table below shows the gap analysis between USABC goals and SKI's LMO-free E400 cells.

Parameter(Units) of fully burdened system	USABC minimum goals	Beginning of Development	SK Proposal	Results of LMO free E400
Power density (W/L)	460	436	485	617
Specific power Dis. 80%DOD/30s (W/kg)	300	287	304	462
Specific power Regen. 20%DOD/10s (W/kg)	150	300	365	483
Energy density C/3 Discharge rate (Wh/L)	230	206	230	230
Specific energy C/3 Discharge rate (Wh/kg)	150	115	150	150
Specific power/Specific energy ratio	2 : 1	2.7 : 1	2.5 : 1	3.1:1
Total pack size (kWh)	40	40	40 (BOL)	40(BOL)
Life (Years)	10	10	10	10
Cycle life - 80% DOD (Cycles)	1,000	900	1,000	1,000
Power & Capacity degradation (% of rated spec)	20	20%	20%	20%
Selling price – 25,000units @ 40kWh (\$/kWh)	<150	<500 (by 2015)	500	<500 (by 2015)
Operating environment (°C)	-40 to +50 20% performance loss (10% desired)	-30 to +50 for cell -40 to +50 for BMS	-30 to +50 for cell -40 to +70 for BMS	-30 to +50 for cell -40 to +50 for BMS
Normal recharge time	6 hours (4 hours desired)	4-6 hours	4 hours	4-6 hours
High rate charge	20-70% SOC < 30minutes @150 W/kg (<20min @ 270W/kg Desired)	20-70% SOC < 30 minutes @150 W/kg	20-70% SOC < 30minutes @150 W/kg	20-70% SOC < 30minutes @150 W/kg
Continuous discharge in 1 hour – No failure (% of rated energy capacity)	75	75	75	75

6.2.1 Computer Modeling Work

– None reported.

6.3 Deliverables/Products Developed

SKI delivered the following cells during the USABC TAP Program:

- High Energy E400 cells (Reference)
- Low IR E400 cell (Improved)
- LMO-free E400 cells

6.4 Technology Transfer Activities

6.4.1 Proprietary Reporting

– Final Report to USABC was submitted on December 3, 2014.

6.4.2 Non-Proprietary Publications and Proceedings

– None reported.

Section F – Ultracapacitors

Section F – Ultracapacitors Final Reports:

1. Maxwell – USABC 42V Ultracapacitor Module Development Program..... F-1
2. NESSCAP – Development of Ultracapacitor Technologies for Automotive Applications..... F-7

1. USABC 42V Ultracapacitor Module Development Program

Performing Organization: Maxwell Technologies

Project Duration: 2/25/2005 – 1/31/2007

1.1 Executive Summary

This program was based upon a 24-month cost shared development activity between Maxwell and the USABC, with the focus on developing a module with a revolutionary low cost architecture while enhancing technical performance of the device. Throughout the 24-month technical product development period, deliverables occurred as defined in the SOW. Those deliverables were comprised of hardware of various configurations and progress reporting on a regular basis. Significant knowledge with data point validations occurred during the execution of this program. The progression was down a path of highly advanced 42V ultracapacitor module technology development for energy storage and power delivery needs, along with moving dramatically toward system cost goals for the 42V FSS, as defined within the FreedomCAR program. As demonstrated in the USABC program award, Maxwell, will continue the significant progress and technical developments toward commercialization of useful ultracapacitor systems applicable to a variety of automotive needs.

1.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

Project Objectives and Approach:

The program tasks were organized under three phases: Cell Developments, Module Developments, and Testing.

Cell Developments:

The baseline cell delivered in 4Q04 was Gen3 (2600F, 2.5V). This cell configuration evolved

to Gen8 (3850F, 2.85V). A four-pronged technical approach was pursued to achieve targeted improvements to Maxwell's ultracapacitor cell product:

Increasing Capacitance – During this same time frame other possible methods continued to be evaluated, with more tests:

- Improve specific capacitance with new Asian carbon.
- Increasing the electrolyte absorption by blending carbons.

By 4Q05, and as part of the 2.85V development, Maxwell R&D continued to evaluate the feasibility of new carbons. New carbon samples were received from the following vendors: CHC40 & KH15. The CHC40 coin cell data showed 5% higher capacitance and 30% higher ESR, than the control carbon. The KH15 coin cell data showed similar capacitance and 30% higher ESR, than the control carbon. Other samples were being pursued, along with requesting cleaner versions of carbon sample batches.

Increasing Cell Voltage to Make a 2.85

Ultracapacitor – The 3Q05 Quarter focused on determining the mechanisms of gas generation inside cells, so as to minimize gas generation at high application voltages. Current testing at this time used asymmetric electrode thickness Thick-thin or TT cell design (although this was later dropped at the end of 2005). Some of the key findings as a result of a prior 5-month gas generation study were as follows:

- Understood that gassing is a thermally assisted, electrochemical reaction.
- Understood that activated carbon is the major contributor to gas generation in the cell.
- Understood that reducing surface functional groups on the carbon is a key to

reducing gas generation (therefore new version of activated carbon was developed).

- Identified a cleaner carbon source and process that is suitable for 2.85V ultracapacitors.

Continuing R&D research demonstrated that a Stabilizer in the Electrolyte improved the gas generation situation. It was shown that:

- Gas generation and therefore pressure rise is reduced by stabilized electrolyte.
- Drying temperature is a factor in controlling internal gas pressure.

These results are repeatable by electrolyte suppliers' different manufacturing sites. Still, a stabilizer does not improve cycle life significantly.

Reduce ESR – By 3Q05, it was generally recognized that reducing the resistance of activated carbon would reduce the bulk resistance of the electrode (without addition of conductive carbon), which is known to affect the life performance of the cells. This quarter, an experimental activated carbon C#41 supplied by commercial vendor and tested in coin cells, reduced ESR by 13%.

Increasing Film Density – By 3Q05, Maxwell R&D team realized that increased film density was possible by varying carbon particle size distribution through smaller particle size and by the blending of different particle size distributions. Increasing the electrode film density improves the energy density of the electrode. Test results in coin cells indicated that 0.66 g/cc is achievable, but large cell evaluation remained at that time, although was being pursued.

Using laboratory film and electrodes at the end of 2005, electrodes were made by increasing lamination passes from 1 to 20. The results indicated that the electrode density and the

capacitance increased as the lamination passes increased. After (10) passes, there was no further improvement in density or capacitance. It was noted that after (5) passes, ESR also increases and an investigation was conducted to determine if this was the result of possible foil damage. Electrical DC life testing was in progress at that time.

Module Developments:

By 2Q05, the module consisted of a aluminum enclosure and contained all module-to-module and cell balancing required internally. It represented a good first effort in packaging and performance. The thermal performance of the module when related to the higher voltage counterpart, was not sufficient to meet the needs of the USABC and therefore design refinements were required. This refinement took place both at the cell and at the module level. The cell resistance needed to be reduced and the efficiency of the module thermal transfer needed improvement.

By 3Q05, engineering was addressing the energy gap by increasing energy density through module and cell (packaging) design. Then, by selecting appropriate cell form factors that give resistance and energy required given:

- Electrode active width drives cell height.
- Electrode capacitance (active width and carbon capacitance) drives cell diameter.
- Electrode capacitance and packaging drives cell weight.

As a result, engineering optimized module material selection and form factor based on cell packaging and form factor.

By now (3Q05), Maxwell had a cell design that would meet start-of-life energy required to provide end-of-life electrical performance, and could manufacture this cell for the final deliverable in 1Q06. The module designed around this cell met the program goals for lifetime, energy and weight. Two open technical

considerations remained: self-discharge and module volume requirements.

Module thermal performance was enhanced as a result of the lower resistance. Module designed around larger (75mm) diameter cell and end-of-life energy requirements:

- Reduced resistance due to aspect ratio changes (diameter/height).
- Reduced resistance due to thinner electrode.

As a result, higher power was available as a result of lower cell/module resistance.

Maxwell procured new software to aid in module thermal analysis. With this new analytical tool, thermal data from Maxwell's existing 48.6V module were confirmed. The plan of record then called for a plastic enclosure on the sides with aluminum top plates. Module showed very good continuous current thermal characteristics.

By the end of 2005, Engineering was designing, analyzing and prototyping parts for the module to reduce volume and weight. In addition, engineering had developed the test plan for modules scheduled to be delivered to USABC in March 2006.

Testing Results:

In 3Q06 the test result updates were available for the 2.85V (Gen8) cell, from the 2Q06 tests. Module testing at that time was being impacted by Maxwell Production requirements. The following best summarizes the delivered 3850 Farad (Gen8), 1.24Ah cell:

- Exceeds power and energy goals at the beginning of testing.
- Still exceeds energy and power goals after 750K UC10 cycles at approximately 36Wh.
- Exceeds energy goals after 168-days of calendar life with 34Wh energy available.

- Self-discharge <8% after 725K cycles.
- Self-discharge <5% after 102-days calendar life.
- Meets cold crank goal at ~80% DOD (1.92V/cell).
- Meets cold crank goal after 1-month open circuit voltage (2.13V/cell).

Overall, excellent performance has been achieved from the Gen8 cells developed for this program largely exceeding all targets for electrical performance. It is likely then that the Gen8 cell is over designed and the cell required to meet the electrical performance targets can realize a cost advantage over the cell cost shown at the close of the program. This represents an easy way to take more cost out of the product through optimization of the energy and power in the cell.

Product Cost Status:

As this program progressed, considerable improvements to cost extrapolation through actual vendor material costs provided better projection estimates. The progression to final hardware configuration and accurate bill of materials (BOM's), provided the proper incremental cost reduction focus required. It must be noted that the current product cost is a considerable way from the ultimate USABC FSS target of \$80. This target was too far from current technology and manufacturing state-of-the-art to achieve in the timeframe of the program. However further work is proceeding which will significantly close the gap between the ending program cost and ultimate FSS target.

Gap Analysis with USABC Targets:

The following chart depicts the gap analysis as the program progressed from 4Q04 through 3Q06 to the program (interim) goal, and the FSS target. It can be seen in the gap chart that with the exception of cost and self discharge, the module developed conforms excellently to the

program goals established. It should be mentioned that at the close of the program, the topic of self discharge remains a highly visible

topic. It is anticipated that the specification will be revised for the coming programs.

Parameter (Units)	Gen3	Gen5	Gen6	Gen8				Status 3Q06	Program (Interim) Goal	FSS Target
	Status Beginning of Program	Status 1Q05	Status 2Q05	Status 3Q05	Status 4Q05	Status 1Q06	Status 2Q06			
Selling Price (\$) @100K units/yr	760	346	265	342	332	332	298	298	217	80
Operating Voltage Max (V _{OC}) of Module	45	40	43.2	45.6	45.6	45.6	45.6	45.6	45	48
Operating Voltage Max (V _{OC}) of Cell	2.5	2.5	2.7	2.85	2.85	2.85	2.85	2.85	2.85	3
Operating Voltage Min (V _{OC}) of System	27	27	27	27	27	27	27	27	27	27
Available Energy (Wh) CP@1kW	20 BOL	20 BOL	22.9 BOL	30 EOL	30 EOL	30 EOL	30 EOL	36 @ 750K Cycles	30 EOL	30 EOL
Self Discharge (%) 72 hrs from V _{max} , RT	N/A	N/A	16%	TBD	TBD	TBD	TBD	BOL – 45% EOL 5-7%	<4	<4
Cycle Life (cycles) UC10 Profile [≈150K miles]	750K	750K	245K+ UC30	750K	750K	750K	750K	750K	750K	750K
Calendar Life (years)	10	10	TBD	15	15	15	15	TBD (Accelerated Test in Progress, @168 days)	15	15
Pulse Discharge Power (kW) @2s	N/A	N/A	N/A	6	6	6	6	6	6	6
Cold Cranking Pulse @ -30°C (kW)	13.6	11.8	17.30	31.60	31.60	31.60	31.60	18kW@50% DOD	8	8/21 V _{min}
Temperature Range, Operating (°C)	-40 to 65	-40 to 65	-40 to 65	-40 to 65	-40 to 65	-40 to 65	-40 to 65	-30 to 65	-40 to 65	-30 to 52
Temperature Range, Survival (°C)	-40 to 70	-46 to 66	-46 to 66	-46 to 66	-46 to 66	-46 to 66	-46 to 66	-46 to 66	-46 to 66	-46 to 66
System Weight, Max (kg)	16	15	11	12.4	13.8	14.3	13.7	13.7	14	10
System Volume, Max (liters)	22	15	12	13.2	11.6	10.2	9.8	9.8	10	8

1.2.1 Computer Modeling Work

– None reported.

1.3 Deliverables/Products Developed

Generally, all deliverables throughout the program were made on time met the expectation and intent of the performance objective of the deliverable.

- With prior approval, the first deliverable was in 4Q04 for (22) baseline 2600F, 2.5V (Gen3) cells and (5) pre-existing modules that were current Maxwell state of technology at the start of the program.
- During 2Q05, a quantity of (24) 2600F 2.5V (Gen5) cells and (6) 2600F 2.7V (Gen5) cells were shipped to Idaho National Laboratory (INL).

- In July 2005, the deliverable of (24) 2600F 2.7V (Gen6) cells was met. On 8/1/05, additional (20) cells of the current Gen6 variety was shipped to Sandi National Laboratory (SNL) for the start of abuse testing.
- The final deliverables shipped in early April 2006, and consisted of (13) 3850F 2.85V (Gen8) cells to INL and (16) 42V modules to various USABC locations.

1.4 Technology Transfer Activities

1.4.1 Proprietary Reporting

– Final Report submitted to USABC on October 21, 2006.

1.4.2 Non-Proprietary Publications and Proceedings

– None reported.

2. Development of Ultracapacitor Technologies for Automotive Applications

Performing Organization: NESSCAP Co., Ltd.

Project Duration: 2/1/2006 – 11/18/2008

2.1 Executive Summary

The NESSCAP Co., Ltd. achieved important improvements in various aspects of its ultracapacitor technology in an effort to develop a 42V Start-Stop (42V FSS) ultracapacitor module. Tasks were categorized into three areas: electrode formulation, process verification, and module design. Various electrode formulations were tested for energy and power. After several evolutions, a formulation codenamed USABC 5.0 was obtained. NESSCAP verified the process of incorporating USABC 5.0 into large cylindrical cells at its manufacturing line. Several 42V FSS modules were designed based on USABC 5.0 cells.

Both propylene carbonate (PC) and acetonitrile (ACN) electrolytes were investigated. Significant improvements were made with PC, particularly in terms of capacity and cold cranking, but it was determined during a go/no-go evaluation that meeting USABC requirements with PC-based cells would be difficult. The project focused solely on ACN after the go/no-go evaluation.

Major accomplishments during this project include the following: (1) 40% increase in beginning-of-life (BOL) energy density for ACN-based USABC 5.0 cells; (2) Improvements in low-temperature performance of PC-based cells; and (3) Estimated cost reduction from \$543 to \$198 for ACN-based USABC 5.0 modules. It has been recently reported by Idaho National Laboratory (INL) that a USABC 5.0 cell is exceeding end-of-life (EOL) energy and power requirements after 750k cycles. However, weight, volume,

calendar life and selling price still remain challenging.

2.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

This work by NESSCAP was conducted under a two-year USABC contract between September 2005 and August 2007. Two main challenges for ultracapacitors were identified at the beginning of the project:

- EOL energy requirement
- Selling price.

The project consisted of three parts: electrode formulation, process verification, and module design. For the part of materials formulation, essential materials such as carbon, electrolyte, and binder were studied. Several electrode formulations were attempted for higher energy density and lower cost. Once a viable formulation was obtained, it was put through the process verification at NESSCAP's manufacturing line. The process verification spanned from electrode coating to large cylindrical cell packaging. Module design accompanied these processes and resulted in four different designs.

Key Tasks:

Electrode Formulation –

Carbon: Commercially available carbons were investigated for its capacitance, life characteristics, and cost. Chemically activated phenolic carbon shows best overall characteristics. However, because of its more costly precursor and activation process, it is two to three times more expensive than an alternative such as physically activated coconut shell carbon.

Partially graphitized carbon was investigated. 1,840 cells were fabricated with the partially graphitized carbon. Electrolyte used was ACN with 1 M of TEABF₄. The cells were electrically activated at 2.7V for 12 hrs. However, cells failed due to severed electrodes. Based on this result, it was concluded that obtaining an electrode made entirely of partially graphitized carbon would be difficult. However, given its higher specific capacitance and the need for higher energy, it was decided to continue exploring potential methods to benefit from partially graphitized carbon. It was concluded that 2-3% increase in capacitance was possible by mixing large and small carbon particles.

Electrolyte: This project started with PC as the main electrolyte because of its relative safety compared to ACN. However, PC has lower conductivity than ACN and also exhibits severe conductivity degradation at low temperature. Moreover, meeting the cold cranking requirement of USABC was also regarded as an area of challenge.

Various electrolyte systems of better conductivity were tested. In order to improve the conductivity, smaller salt ions, higher concentration of salts, higher conductivity solvents, additives, and ionic liquids were tried. More improvements were necessary to meet the cold cranking requirement and it was realized that the electrolyte alone was not going to resolve the issue.

The next focus was placed on carbon. A carbon (Carbon 7) with larger pore volume was incorporated to see its effect at low temperature. It was thought that larger pores should lead to less restriction on salt movements at low temperature. As the content of Carbon 7 is increased, a better defined power pulse is seen. During a go/no-go decision event, it was decided that PC-based ultracapacitors will be no longer pursued.

Electrode Formulation: One of the most effective ways to increase available energy is by incorporating more activated carbon into an electrode. This can be accomplished by coating a thicker active layer and/or replacing materials in an electrode that do not contribute to energy storage with activated carbon. Less of these materials will enable more activated carbon content. However, if not done correctly, these reductions will lead to a physically unstable electrode with high electrical resistance.

The final electrode formulation of this project codenamed USABC 5.0 is shown in the following table.

USABC 5.0 Formulation Obtained After Reducing Weight % of Conducting Carbon to 5% from 17%

Material	wt %
AC	90.0
CC	5.0
C1	1.6
BR2	3.0
PTFE	None
SR1	0.4

High Operating Voltage: Higher operating voltage provides various advantages, and achieving a higher operating voltage than 2.7V has been an active topic in the ultracapacitor industry. It has, however, turned out to be a very challenging task.

Several useful conclusions can be drawn from the series of experiments by NESSCAP: In order to achieve an operating voltage beyond 2.7V, a full cell, not just the electrolyte, should be the subject of analyses; TEABF₄/ACN by itself is not a limiting factor; and cells fabricated with TEABF₄/ACN has a voltage window larger than the current operating voltage of 2.7V.

Process Verification – The process of manufacturing USABC 5.0 large cylindrical cells was defined and verified at NESSCAP’s manufacturing facility. The process is categorized by three sub processes: electrode coating, cell assembly, and electrolyte impregnation.

Module Design – Four modules of increasingly complex ultracapacitor designs were proposed based on USABC 5.0 large cylindrical cells. Each module contained 18 units of USABC 5.0 large cylindrical cells connected in series. Cells were connected via laser-welded buss bars and balanced with active balancing circuitry.

The main focus of Design 1 was on achieving the low cost. It does not have water proofing or thermal management feature. It was the simplest design being proposed where 18 of USABC 5.0 large cylindrical cells are connected in series with active balancing circuitry per cell. Design 2 incorporated waterproofing and thermal management in addition to the features of Design 1. Design 3 incorporated CAN bus communication in addition to the features of Design 2. Design 4 was the pre-program design, and it contained the most number of parts and required most labor in manufacturing and assembly. It was a proof-of-concept design and served as the starting point of the module design at the beginning of the project.

Conclusions:

In order to narrow the gap between USABC requirements and the NESSCAP technology as

of 2005, various challenges had to be overcome. A new electrode formulation had to be developed in order to meet the energy density requirements. A large cylindrical cell that was far more efficient in terms of weight and volume than the pre-program prismatic cell had to be developed. Both the new electrode and the cylindrical packaging had to be verified at the manufacturing line. Then a much more cost effective module design had to be accomplished.

The development of a large cylindrical cell with USABC 5.0 formulation was completed. Its manufacturability was verified at NESSCAP’s manufacturing line. This accomplishment was accompanied by an effective module design that reduced the number of parts by about 60%.

Future Work Planned:

Much work remains. Gravimetric and volumetric energy density requirements are not yet completely satisfied. The selling price of \$80/module is still far away from the current estimate of \$198/module. Calendar life needs to be verified. However, NESSCAP firmly believes that continuing to work with the USABC for next several years will close the gap between the USABC requirement and NESSCAP technology even further and that NESSCAP will be able to eventually satisfy the USABC requirements.

**Gap Analysis Based on USABC 5.0 Cells (it is the BOL characteristics of USABC 5.0 cells –
* results reported by INL)**

System Attributes	USABC Goal for 42 V Start-Stop (FSS)	Phase 1 (USABC 5.0)	Phase 1 (USABC 5.0)
		BOL	EOL
Discharge Pulse (kW)	6 / 2s	10.2*	6.8*
Regenerative Pulse (kW)	N / A		
Cold Cranking Pulse @ -30 °C	8 / 21V _{Min.}		
Available Energy (CP @ 1 kW)	30	34.9*	31.1*
Cycle Life / Equiv. Road Miles (cycles / miles)	750 k / 150,000	750 k	750 k
Calendar Life (Yrs)	15		
Energy Efficiency on Load Profile (%)	95 / UC10		
Self Discharge (% , 72hr from Max. V)	< 4		
Maximum Operating Voltage (Vdc)	48	48.06	48.06
Minimum Operating Voltage (Vdc)	27	27	27
Operating Temperature Range (°C)	-30 to +52		
Survival Temperature Range (°C)	-46 to + 66		
Maximum System Weight (kg)	10	12.9 (packaged)	12.9 (packaged)
Maximum System Volume (Liters)	8	14.9 (packaged)	14.9 (packaged)
Selling Price (\$/system @100k/yr)	80	198	198
Currently meet/exceed , or low risk to achieve USABC target			
Medium risk to meet/exceed USABC target			
High risk to achieve USABC target			

2.2.1 Computer Modeling Work

– None reported.

2.3 Deliverables/Products Developed

As described in Section 2.2, NESSCAP delivered large cylindrical cells with USABC 5.0 formulation for testing at INL.

2.4 Technology Transfer Activities

2.4.1 Proprietary Reporting

– Final Report to USABC dated August 24, 2008..

2.4.2 Non-Proprietary Publications and Proceedings

– None reported.

Section G – 12 Volt Start-Stop

Section G – 12V Start-Stop Final Reports:

1. Leyden – Development of an Advanced Lithium-Ion 12V Start-Stop Battery..... G-1
2. Saft – 12V Start-Stop Battery Development G-7

1. Development of an Advanced Lithium-Ion 12V Start-Stop Battery

Performing Organization: Leyden Energy, Inc.

Project Duration: 2/8/2013 – 3/30/2014

1.1 Executive Summary

Under a 16-month USABC award, Leyden Energy Inc., of Fremont California, has developed an affordable, advanced lithium-ion 12V Start-Stop battery system to meet the challenging performance, life and cost targets set by USABC. In addition to its innovative cell technology, Leyden engaged with a high volume cell manufacturer (Dow Kokam) and a capable pack manufacturer (Flextronics) to leverage their respective expertise and resources to overcome technical challenges and advance low cost, domestic manufacturing. The project concluded with delivery of generation “A” sample prototypes to the designated National Labs, which will enable production “C” samples in 30 months. The key elements of the program include:

- Use of a LTO/LMO couple with Leyden’s Li-imide electrolyte to enable a system that meets target capacity, cold cranking power, cycle life, calendar life and price targets.
- Advancement of Leyden’s technology platform and leveraging of Dow Kokam’s, the targeted high volume manufacturer, investment in large volume, domestic lithium-ion manufacturing footprint.
- Utilization of an innovative cell and battery design to result in a lower weight and lower volume system, approximately 7.2Kg and 6L.
- A system whose voltage profile is an excellent match for the start-stop application.

1.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

Project Objectives and Approach:

The main program objective was to design, develop, and assemble 12V Start-Stop cell and battery “A” sample prototypes that meet and exceed the USABC targets using an LTO-LMO system and Leyden Li-imide electrolyte.

During the course of this USABC program Leyden Energy has developed a technology and prototype cell design capable of meeting stringent USABC requirements for an in-cabin 12V Start-Stop battery. Of all of the electrification designs in automobiles, 12V Start-Stop batteries are the most economical and will increase fuel economy and lower CO₂ emissions, providing a strong benefit to the environment.

Technical tasks performed by Leyden and its team are listed below with brief status.

Task 1 – Improvement of Low Temperature Rate Capability –

Task 1 is complete, although electrolyte solvent base optimization will continue beyond the timeline of this program. Cold cranking was significantly improved by press density optimization. Sub-tasks included anode and cathode press-density optimization, optimization of electrolyte formulation, and anode and cathode formulation optimization.

Task 2 – Improvement of High Temperature Performance –

In Leyden’s prototype cells, good cycle life (100% DOD) was observed at 20°C and 40°C but noticeable performance degradation occurs at 60°C after approximately 400 cycles. The sub-tasks included: improvement of high temperature performance through electrolyte

optimization, and improvement of the high temperature cycle life through surface treatments.

Task 3 – Cost Reduction –

To achieve the USABC cost targets, the team tested and evaluated new materials and worked in partnership with the supply base to bring the material costs down. The task of cost reductions proceeded in parallel to the procurement of materials. In addition to a material cost advantage, this project also leveraged Dow Kokam's high volume manufacturing facility to optimize cell yield and reduce cell manufacturing costs. Dow Kokam, now XALT Energy, was the intended high volume cell manufacturer at the start of the program.

Task 4 – Cell Testing –

Screening of electrolyte and surface treatments is best performed in prototype cells approximately 0.8Ah (2.7mm x 62mm x 90mm) cells. Leyden's pilot line is presently capable of sustainably making hundreds of these cells per week. To test the large number of cells needed for the design of experiments in Tasks 1, 2 and 3, Leyden has 1500 - 5V channels with current capability up to 3.75A per channel or 30A with 8 channels in parallel; in addition to 48 high power channels with capability of 160A with 8 channels in parallel. Cell characterization included discharge as function of C rate and temperature, cold cranking, thermal characterization and/or heat capacity, HPPC and abuse testing (overcharge, over discharge, short circuit, nail penetration and thermal stability). Preliminary data on life at room temperature and extreme temperatures was also collected.

Task 5 – Scale Up –

The final cell size was targeted to be 20Ah pouch 7.5mm thick with the x-y dimensions of 224mm by 225mm. After the initial testing in 0.8Ah prototype pouch cells, Leyden scaled up to 3Ah cells to finalize some of the cell assembly and electrolyte filling parameters, which facilitated scale up to the final 20Ah cell size at the end of the first year. Dow Kokam,

were instrumental, as the final design was based on a pouch cell currently in production at the Midland, MI factory. Over 350 of the 20Ah dry cells were assembled at Dow Kokam/XALT and completed final assembly (electrolyte filling, formation, final degas/seal) at Leyden's facility. Cells were characterized and sorted. Several battery modules/packs have been built for internal and external testing. Leyden documentation was released for BOM, Material Purchasing Specifications, Incoming Quality Inspection (IQC), In-Process Quality Control (IPQC), Standard Operating Procedures (SOPs).

Task 6 – Pack Design and Electronics –

Flextronics were the lead in the design of the 12V 40Ah battery pack, including a voltage balancing circuit board. They incorporated pack design features and functionality from packs and modules previously developed. This expertise was used in designing this 12V battery pack including provisions to make connection for external venting of the battery case. The "A" sample pack was designed for bench testing to demonstrate the general performance of the pack. This was intended to demonstrate the cell capabilities at the battery pack level.

Task 7 – Deliverables and Testing –

The team provided both, cell and battery deliverables during the program culminating in three fully operational "A" Sample 12V 40Ah packs for the USABC designated National Laboratories. Fifty (50) individual cells were delivered at Month 10 and another 50 Cells and 3 notational "A" sample packs at conclusion of the program (Month 16). In addition, parallel testing and evaluations were conducted at Leyden and the large volume cell and pack manufacturers facilities, to demonstrate this system meets all of the targets outlined in the gap analysis.

Task 8 – Secure Contracts with Targeted high Volume Manufacturers –

Leyden brought Flextronics on as the battery assembly partner at the beginning of the program and both Flextronics and Dow Kokam,

who assembled the cells, agreed to participate on a no-cost basis.

Conclusions and Lessons Learned:

The resulting technology was demonstrated in 2.2Ah cells shipped by Leyden to USABC in October 2013 and January 2014 as well as in larger 20Ah cells assembled by XALT/Dow Kokam and processed by Leyden in Q1 of 2014. The 20Ah cells were shipped to USABC as final deliverables, and a sub-group of cells has been assembled into 12V packs by Flextronics for delivery to USABC by the end of April. During this program, Flextronics has designed a 12V demo battery pack and voltage balancing circuitry.

LTO-LMO technology developed under this program has met the most critical performance targets: cold cranking at -30°C and an extrapolated cycle life of >450k start-stop cycles at 30°C. In addition, They demonstrated >200k cycles and good calendar life at 50°C. The cost of the pack is highly sensitive to the manufacturing costs, but found it is possible to meet the target cost under high volume production scenarios.

The following parameters were found to be critical during cell development for meeting the technical targets:

- Electrolyte Formulation
- LTO Surface Treatment
- A/C Ratio
- Formation Procedure
- Electrode Processing and Storage Guidelines
- Electrode Formulation
- Press Density.

The changes led to significant improvement in high temperature performance with >200k USABC cycles projected at 50°C.

In parallel to the development work, Leyden has made significant strides in the scale up of its technology. Anode and cathode coatings were performed on production-scale high speed slot die coating equipment, and the resulting electrodes were assembled into more than 300 - 20Ah cells at the Dow Kokam facility in Midland, Michigan (now XALT Energy).

Three main challenges were encountered and are being mitigated by the development team as listed below:

1. Cells built by XALT/Dow Kokam had some defects and performance deficiencies when compared to identical 2.2Ah cells built at Leyden; this led to a delay in getting cells ready for pack assembly.
2. Pack deliverables were delayed by four weeks and will be delivered by the end of April 2014.
3. Leyden lacks sufficient calendar life data; additional builds were made to generate this data.

The overall program accomplishments include the following:

- Met critical USABC performance targets.
- Demonstrated the possibility of meeting cost targets under a scenario of 1M packs a year with reduced manufacturing costs.
- Delivered required cell samples to USABC and National Labs for evaluation and testing.
- Established excellent traction with major U.S. cell/battery manufacturing partners – a prerequisite for robust supply-chain for high-volume manufacture.

Gap Analysis with USABC Requirements:

Table 1 shows USABC requirements evaluated against the projected performance of the proposed 12V LTO-LMO battery with a 5S2P (5 series x 2 parallel) configuration delivering 40Ah at the beginning-of-life and 30Ah at end-of-life.

The gap analysis shows that the Leyden LTO-LMO system meets most of the USABC targets. The system has excellent voltage match, very high power, high charge/discharge efficiency, low self-discharge, high regen capability, and wide operating temperature range. Parameters highlighted green are clear passes; blue either pass at BOL and have some uncertainty at EOL

or more information is needed for certainty; yellow are at risk and do not meet the requirement at EOL at this time.

Over the final quarter of the project, further improvements were made in cold crank and thermal performance. Additional efforts were directed towards improving the 66°C survival test results but fell slightly short of meeting the USABC target of <5% degradation in power. It's important to note that the thermal performance and the cold crank numbers are for the BOL battery and the ongoing testing will determine the EOL values. The volume and weight of the pack was found to be higher than anticipated at the beginning of the program.

Table 1. 12V Start-Stop Gap Analysis Leyden EOP Targets Present Status (EOL)

End of Life Characteristics	Units	Target		Leyden EOP Targets	Present Status (EOL)	Comment
		Under hood	Not under	Not under hood	Not under hood	Not under hood
Discharge Pulse, 1s	kW	6		10	15.75	21kW BOL, derated by 25% for EOL
Max current, 0.5s	A	900		900	1200	Based on 40C rate capability and derated by 25%
Engine-off accessory load	W	750		2000	1700	BOL, based on high current to low current HPPC ratio
Cold cranking power at -30 °C (three 4.5-s pulses, 10s rests between pulses at lower SOC)	kW	6 kW for 0.5s followed by 4 kW for 4s		6 kW, 4 kW	100% SOC: 5.9kW	Data is for BOL
					50% SOC: 4.8kW	
					30% SOC: 4.0kW	
Extended Stand Test (30 days at 30°C followed by cold crank test)	kW	6 kW for 0.5s followed by 4 kW for 4s		6 kW, 4 kW	4.3kW	At the end of third pulse power capability is higher than 4kW
Min voltage under cold crank	Vdc	8		8	8	Based on 1.6V cell min voltage
Available energy (750W)	Wh	360		480	383	BOL for 40Ah pack is 512Wh at 100% DOD (75% of 510Wh is 383Wh), however, it seems that USABC specifies it at 90% SOC.
Peak Recharge Rate, 10s	kW	2.2		2.2	3.68	At 10% DOD value (so it is very conservative value)
Sustained Recharge Rate	W	750		1350	1700	Based on high current HPPC to low current HPPC ratio
Cycle life, every 10% life RPT with cold crank at min SOC	Engine starts/miles	450k/150k		450k/150k	>450k at 30°C >200k at 50°C	Approximated from 25-50k shallow cycles
Calendar Life at 30°C, 45°C if under hood	Years	15 at 45°C	15 at 30°C	15 at 30°C	15 at 30°C	Based on approximation from 50°C storage
Minimum round trip energy efficiency	%	95		96	99.9	Shallow cycling, 2.2Ah prototypes
Maximum allowable self-discharge rate	Wh/day	10		10	2	Based on extended stand test
Peak Operating Voltage, 10s	Vdc	15		15	14.5	Based on 2.9V as Max voltage
Sustained Max. Operating Voltage	Vdc	14.6		14	13.5	Based on 2.7V recommended charge voltage
Minimum Operating Voltage under load	Vdc	10.5		10.5	10.5	Based on 2.1V as Min Voltage
Operating Temperature Range (available energy to allow 6 kW (1s) pulse)	°C	-30 to +75	-30 to +52	-30 to +52	-30 to +52	Please note: numbers are for the BOL cells
30 °C ~ 52 °C	%	100 (to 75°C)	100	100	100	
0 °C	%	50		50	88	
-10 °C	%	30		30	84	
-20 °C	%	15		15	73	Data for BOL
-30 °C	%	10		10	14	
Survival Temperature Range (24 hours)	°C	-46 to +100	-46 to +66	-46 to +66	-46 to +66	Almost meet 66°C survival: power loss is >5% but <10%
Maximum System Weight	kg	10		7.2	12	8.3kg for the cells; 14.5kg for demo pack; 12kg for optimized pack
Maximum System Volume	L	7		4.9	9	5L cell volume, 17.5L demo pack, 9L optimized pack
Maximum System Selling Price (@100k units/year)	\$	\$220	\$180	\$180-250		Several cost scenarios presented
Cell Capacity	Ah			20	20	
Battery Size Factor, Cells				10 (2P5S)	10(2P5S)	

1.2.1 Computer Modeling Work

- None reported.

1.3 Deliverables/Products Developed

- 20Ah cell deliverables made by XALT/ Dow Kokam and processed at Leyden.
- 21 cells to ANL shipped on 3/31/2014.
- 4 cells to NREL shipped on 3/31/2014 then shipped to Sandia after test completion.
- 8 cells to Sandia shipped on 3/31/2014.
- 3 packs assembled by Flextronics to be shipped to ANL on 4/24/2014.

- 10 of the 2.2Ah cells made from the same electrodes as XALT/ Dow Kokam cells to ANL – ready to be shipped once the agreement is reached on test matrix.

1.4 Technology Transfer Activities

1.4.1 Proprietary Reporting

- Final Report submitted to USABC on April 18, 2014.

1.4.2 Non-Proprietary Publications and Proceedings

- None reported.

2. 12V Start-Stop Battery Development

Performing Organization: Saft

Project Duration: 3/1/2013 – 3/31/2014

2.1 Executive Summary

Saft proposed to develop an advanced, high-performance battery for 12V Start-Stop (12VSS) vehicle applications based on their proprietary NMC-LTO lithium-ion battery technology. Saft's NMC chemistry is the current high-quality industry standard Li-ion cathode active material. The high temperature stable NMC cathode is paired with an LTO anode for long cycle and calendar life, and low system cost. Saft illustrated in the gap analysis, included in the SOW, that the NMC-LTO technology was projected to meet or exceed the USABC requirements for this application.

The development program scaled up the Saft LTO technology from the development test vehicles, 0.4 Ah, as well as, 0.8 and 1.2Ah pouches to a 10-15Ah prismatic cell to be manufactured in the Saft hard can PHEV-2 VDA size cell as a demonstration of the technology's ability to meet the cost and size requirements. Saft also proposed to study the concept of polymer battery housing and propose suitable materials for such a Li-ion battery.

Saft made several major accomplishments as follows: Designed and built three iterations of the proposed LTO-NMC cells and delivered several cells to National Labs for verification testing; Extensive testing and design of experiments were performed on the deliverable cells to measure the cells' performance against the gap chart; Saft also generated several monoblock design concepts throughout the program and conducted experiments to vet those concepts. Additionally, Saft worked with Virginia Commonwealth University (VCU) to conduct a paper study of potential polymer material candidates for the monoblock housing;

Saft also worked with Wildcat Technologies to conduct electrolyte optimization studies using high throughput screening methods; and Saft conducted an investigation into the cost of the full monoblock battery during this program.

2.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

Program Approach:

For several years now, Saft has embarked on developing a multi-chemistry line of Li-ion products for various markets. Every chemistry seems to be particularly better suited for use in one application or another. For the USABC 12V Start-Stop requirement noted in the June 1st 2012 RFQ, Saft proposed a high temperature stable NMC chemistry with an LTO anode for long cycle and calendar life and low system cost.

Saft had successfully supplied NMC based Li-ion cells for high power, high temperature automotive application. The result includes a product that won the 2012 Innovation Award from the customer and allows operation for multiple races at temperatures above 100°C. The LTO anode is well-known to offer excellent cycle, calendar life, and safety due to the anode potential in a region of electrolyte stability, but is also known to have significant life issues at elevated temperature. Saft has successfully resolved this through a combination of electrode processing and electrolyte formulation developments to stabilize the electrolyte interface even at temperature extremes.

Saft is producing the NMC line of products in two large formats, cylindrical and prismatic. The large format prismatic cell has been designed to be produced in Saft's Jacksonville, FL factory. The first of these cells to be manufactured is the LP28M. For the USABC

12V start-stop application, Saft proposed a new cell design, PL46P, for the best fit to the requirements. The Saft proposed PL46P electrode design provides greater energy margin than is needed but is being challenged on the cold cranking requirement. A higher power/energy ratio, as well as an optimized electrolyte would better fit the application requirement. Improvements would also be made in low temperature power, high temperature tolerance, and electrode cost.

In the final stop-start battery design Saft proposed to use a single 1p5s configuration in a 46Ah electrode design. However, to reduce the need for mechanical cell development, the optimized electrode for this design would be placed in a Saft existing VDA-sized prismatic cell hardware (PL20P) and would be tested against the scaled program requirement to show objectives have been met.

The development plan for this 12VSS program included program management, cell development, polymer material studies, process development for novel assembly, cell testing, and deliverable and gate tasks. The development plan did not include battery management or thermal management activities. For the evaluation of the NMC, LTO, electrolyte, and separator candidates, the emphasis was to be placed on optimization of LTO and electrolytes for -30°C performance, while maintaining calendar life and cycle life up to 45°C.

Tasks:

The following highlights the main tasks pursued to achieve the program objectives.

1. *Electrochemistry Development* – The electrochemistry portion of this program focused on the development and demonstration of an LTO-NMC cell that would fulfill all USABC gap chart requirements. The development was carried out during three deliverable cell builds.

2. *Deliverable Cell Testing* – Shown in Table 1.
3. *Electrolyte Studies* – The focus of the electrolyte studies work is to a select set of electrolyte formulations for optimal low temperature performance while maintaining stable high temperature impedance growth. The studies were conducted using Wildcat Discovery Technology's rapid prototyping and testing. Due to delayed receipt of electrolyte/additives from our supplier and an issue at Wildcat (accidental contamination of the initial round of cells), results are not yet available for this report. Approximately 100 cells are currently on test at Wildcat with results available by the end of May 2014. A brief addendum to this report will be submitted when the final results are available
4. *Polymer Trade Summary Report* – In this task, different polymer materials were studied for their inherent hermeticity, ability to be formed/manufactured at low cost, mechanical strength, their ability to be hermetically-joined, and their ability to withstand the stack pressure exerted onto the battery walls. With Li-ion cells being sensitive to moisture, they must be housed such that a hermetic sealing from the atmosphere can be guaranteed throughout the battery life time. Furthermore, the housing must withstand the volatile organic solvents from the electrolyte. Additional polymer material studies tasks within this program have been conducted at VCU. Due to the short duration of this project, activities related to polymer materials selection were paper studies only. No physical testing was conducted, but the project aims generally at delivering knowledge of the polymer housing hermeticity, the

system design for confining cell stacks, and an estimate of the system costs for the different solutions.

5. *Nanoindentation Using Atomic Force Microscopy (AFM)* – The goal of this nanoindentation study was to establish whether swelling of the near surface would affect mechanical properties. That is, this study explores whether plasticization of the near surface might be different from the bulk due to an almost “pure” polymer domain at the surface. This part of the VCU study can be considered “pioneering” in the sense that nanoindentation has not been done on materials immersed in non-aqueous solvents in published literature as of yet. The relevance is clear: plasticization near-surface plasticization could be a forerunner of more extensive long term solvent/polymer interactions
6. *Novel Cell Assembly* – To meet the goals/SOW for this program, the desired monoblock configuration, and goals for high volume manufacturing and reduced cost, requires development of novel assembly methods. This task involved studies of the internal bussing of electrode stacks, their feed through from within the battery to the circuit board, and eventually to the terminals on the outside of the start-stop battery. Possible high volume assembly processes connected to the bussing and feed through were reviewed, as well as, the processes of electrolyte filling and final closing of the battery. It should be noted that due to the reduced program scope the processes were only briefly studied on a more conceptual basis.
7. *Cost-Reducing Solutions Study* – Per the SOW for this program, a dramatically

different approach to the way Li-ion cells are fabricated and assembled into a module and/or a pack is needed for a significant reduction in the hardware cost. This is because the hardware in a Li-ion battery pack is typically responsible for a combined 80% of the cost add-on before indirect costs. Saft’s goal for this portion of the program was to investigate the effectiveness of the use of injection molded thermoplastics for the battery housing instead of traditional metallic housings. Saft also investigated ways to reduce the cost of the electrochemistry. Saft has clearly shown a path to reaching the USABC price target of \$220/each in the year 2020 at an annual production volume of 100k.

Task Accomplishments and Gap Analysis vs. USABC Requirements:

Table 1 shows a summary of the system level and end of project cell deliverable performance metrics gap analysis at the end of the USABC project. The metrics shown in green as passing. Yellow indicates that the measured value is within 10% of the target. Red is shown where the measured results do not meet the USABC target. A blank field in the gap chart indicates that the testing for that item is not completed.

As can be seen in the table above, the LTO-NMC cells delivered during this program and the calculated monoblock battery meet most of the performance requirements listed in the gap chart. The cells are able to pass all thermal performance tests and the cell impedance is better than Saft’s original proposal. The monoblock battery is calculated to have sufficient available energy and the peak recharge rate is nearly double the requirement. There is also no excessive gas generation observed in cells fitted with pressure transducers.

Table 1. End of Program Saft LTO-NMC Gap Chart Analysis

End of Life Characteristics	Units	USABC Target	Saft LP10P scaled Target	Saft's 1st Deliverable Cell Actual	Saft's 2nd Deliverable Cell Actual	Saft's 3rd Deliverable Cell Actual	Monoblock Calculated from 2nd Deliverable Cell Actual
		Under-hood	Under-hood	1s (LP10P)	1s (LP10P)	1s (LP10P)	5s1P, 46Ah, 11.25V
Cold cranking power at -30 °C (3- 4.5s pulses w/10s rests @ lower SOC) , 0.5s followed by 4s	kW, 0.5 sec	6	1.2	0.0	0.15	0.11	3.5
	kW, 4 sec	4	0.9	0.0	0.06	0.05	1.3
Available energy (750W)	Wh	360	15.7	23.2	24.2	21.0	557
Peak Recharge Rate, 10s	kW	2.2	0.1	0.1	0.2	0.2	4.3
Cycle life, every 10% life RPT with cold crank at min SOC	Engine starts/miles	450k/150k		6.2k	In progress, 18.2k		In progress, 18.2k
Calendar Life 30°C / 45°C under hood	Years	15 at 45°C			No change @ 64d		No change @ 64d
Minimum round trip energy efficiency	%	95%		> 99%	> 99%		> 99%
Maximum self-discharge rate	Wh/day	10	0.43	0.1	0.1	0.2	2.3
Peak Operating Voltage, 10s	Vdc	15	3.0	3.0	3.0	3.0	15.0
Sustained Max. Operating Voltage	Vdc	14.6	2.9	2.9	2.9	2.9	14.6
Minimum Operating Voltage under Autostart	Vdc	10.5	2.1	2.1	2.1	2.1	10.5
Minimum Operating Voltage Under Load (below -30°C)	Vdc	8	1.6	1.6	1.6	1.6	8.0
Operating Temperature Range (available energy to allow 6 kW-1s pulse)	°C	-30 to + 75					
75 °C	Wh	360	15.7		18		414
45 °C	Wh	360	15.7		21	20	478
30 °C	Wh	360	15.7	17	18		414
0 °C	Wh	180	7.8		21		480
-10 °C	Wh	108	4.7		18		420
-20 °C	Wh	54	2.3		9		215
-30 °C	Wh	36	1.6		7		161
Survival Temperature Range (24 hours)	°C	-46 to +100					
Maximum System Weight	kg	10	N/A	N/A	N/A	N/A	9.765
Maximum System Volume (Displacement)	L	7	N/A	N/A	N/A	N/A	6.99
Maximum System Selling Price (@100k units/year)	\$	\$220	N/A	N/A	N/A	N/A	\$219.46
Battery Scaling Factor (BSF)				5.00			
Cell Scaling Factor (CSF)				4.60			

However, although the DCR decreased with each build and there was ample available energy, the LTO-NMC cells were not able to pass the cold crank test after discharging 360Wh equivalent per the test manual. The cells are only able to pass cold crank above 60% SOC. They believe that NMC positive is not an appropriate material for the cold crank requirement due to the NMC/LTO open circuit potential which is too low at minimum SOC. LMO/LTO or a blend of NMC and LMO paired with LTO are more appropriate couples. Further investigation of LMO for use in the cathode is needed.

The calendar life and cycle life of the LTO-NMC cells built during this program cannot yet

be determined. After 45 days in storage, the cells increased slightly in 750W discharge energy and DCR did not change. Additional time in storage is needed to begin to see available energy degradation. For cycling, only two data points are currently available. Saft observed a 6% energy fade after 7k cycles however more data points are needed since fade rate is not linear. Continued cycling of the cells is needed to accurately predict the cycle life.

A robust monoblock design concept was generated during this program and a significant amount of analysis was conducted which showed that the mass and volume requirements can be achieved. An extensive paper study of polymer materials candidates for the monoblock

was conducted which resulted in a short list of potential materials. Those candidates were tested for electrolyte and water compatibility and a single polymer, polyphenylene sulfide, was identified as a highly likely housing material. However, additional longer term permeation testing is needed to completely validate this polymer candidate.

An in-depth analysis of the cost of the monoblock has resulted in a projection that the USABC required sell price of \$220/unit at an annual production volume of 100k units/year in 2020 can be met. The monoblock design concept is a large enabler for meeting this critical requirement. Inclusion of LMO in the cathode also helps meet the price target while also potentially improving cold crank performance.

An extensive electrolyte study was also conducted during this program. However, it did not result in identification of a single electrolyte formulation that was statistically significantly better than any other electrolyte formulation. Saft believes that this may be related to premature failure of the seals in the coin cells used for this testing. Additional electrolyte studies are needed utilizing a more robust test vehicle.

Conclusion and Future Work Planned:

Saft has performed the program tasks as outlined in the SOW to develop a 12V start-stop battery. VCU has concluded their polymer study which resulted in a recommendation for monoblock housing material. Wildcat Technologies has provided data from electrolyte testing which has allowed Saft to select an optimized electrolyte for the final deliverable cells. The mechanical team has completed the conceptual design of the monoblock battery. The chemistry team has conducted several experiments to design a 3rd deliverable cell

which best meets the requirements in the gap chart. Saft has completed the design and build of the 3rd deliverable cells which are a culmination of the knowledge gained throughout this program. Additional electrolyte studies are needed utilizing a more robust test vehicle.

2.2.1 Computer Modeling Work

– None reported.

2.3 Deliverables/Products Developed

- Progress reports of cell performance
- Report of gas generation data
- Polymer-electrolyte compatibility
- Cell testing progress reports
- Report of cost-reducing solutions
- Program cell build and test summary report
- Polymer trade summary report
- Novel cell assembly findings report
- Prototype cell test report
- Hardware deliverable – (5) LP10P Ah cells
- Hardware deliverable – (15) LP10Ah cells
- Hardware deliverable – (20) LP10Ah cells.

2.4 Technology Transfer Activities

2.4.1 Proprietary Reporting

– Final Report submitted to USABC dated May 16, 2014.

2.4.2 Non-Proprietary Publications and Proceedings

– None reported.

Section H – Separators

Section H –Separator Final Reports:

1. UMT – Dry-Stretch Low-Cost Separators for EV/HEV Lithium Batteries H-1
2. AMS – The Development of a Low-Cost, 100°C Shutdown Separator with 200°C
Melt Integrity for Lithium-Ion Batteries H-5
3. Celgard – High Temperature Melt Integrity Lithium-Ion Battery Separators..... H-9
4. ENTEK – Multifunctional, Inorganic-Filled Separators for Large Format, Li-Ion
Batteries (Phases I, II, & III)..... H-15

1. Dry-Stretch Low-Cost Separators for EV/HEV Lithium Batteries

Performing Organization: Ultimate Membrane Technology, LLC

Project Duration: 12/16/2002 – 8/31/2006

1.1 Executive Summary

This program was funded by USABC to develop low-cost separators for rechargeable lithium-ion batteries for applications in electric vehicle (EV)/hybrid electric vehicle (HEV). It included three distinct phases: (I) low-cost separators, (II) low-cost shutdown separators and (III) low-cost low temperature shutdown separators. The focus of this summary report is on the results of Phase III which was executed during the term of the current DOE Cooperative Agreement.

The achievement in Phase I was to develop an all-continuous dry-stretch process for bringing down the cost of polypropylene (PP) separators. The cost of PP separators was set to be \$1/M2 and successfully achieved based on the process model. The all continuous process was achieved by consequently connecting a bi-layer PP/PP co-extrusion process with annealing process and then with stretching process successfully. The challenge of connecting all the processes consequently and successfully was to decrease the extrusion line speed to 35 ft/min for quality precursor so that the stretched membrane will not be damaged during the following high-temperature stretching in heated oven. The low extrusion line speed for quality precursor was achieved by a 300mm circular die equipped with a 200-mil die gap, and the properties of the stretched membrane in the lab oven met the targeted requirements.

The achievement in Phase II was in developing the core-skin separator from directly blending PP and PE resin during extrusion for simplifying the manufacturing process, in which the PP skin is formed to maintain melt integrity

while the PE core is shutdown. The core/skin structure was developed by forcing the extrudate passing through a section of narrow slit within the die. The process was greatly simplified for lowering the cost of manufacturing. Two analytical techniques (FTIR-ATR; SPM (scanning-probe microscopy)) show that the skin region contains PP richer component while the core region contains PE richer component while not 100% PP skin was observed. However, the obtained separator showed the shutdown at 135°C and maintained the melt integrity at least up to 172°C under the hot air environment.

The achievement in Phase III was in developing a successful PP/PE-PB/PP tri-layer low temperature shutdown separator with good mechanical strength via a unique co-extrusion and co-stretching process for the objective of low cost manufacturing. The middle layer was based on the immiscible PE-PB blend with a co-continuous PB line structure as immiscible PP-PE blend discovered in Phase II. The PE-PB blend middle layer provided the shutdown and pre-shutdown function while the PP outer layers provided the strength. The grade of PP resin was selected to stretch a temperature compatible to the melting points of PE and PB resins so that we could obtain reasonably high gas permeability and reasonably low shrinkage.

1.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

The high-energy density of rechargeable lithium batteries necessitates a shutdown feature built into separator so that the battery containing it can be safely used in our daily life. The prior arts of the shutdown separators have been seen shutdown often at 135°C, which is the melting point of high density polyethylene. Technically, the lower the shutdown temperature is, the safer the battery is for its gain of longer time for

shutdown. However, there is little success in the separators having a shutdown temperature lower than 135°C reported in the prior arts. The possible reasons are the following: All the shutdown function is based on the pore collapse during fusion of polymeric crystal of the separators when they reach the melting point. The lower shutdown temperature means the lower melting point of the polymeric crystal, which further implies the weaker strength of the crystals and then the separators. The separator with reasonably good strength is needed in the field of battery manufacturing. So, there is a technical dilemma.

In addition, the lower crystal strength is difficult to be processed into microporous membrane separators. The micropore formation is primarily relying on the fast crystallization to set the pores during stretching either in dry stretching technology platform or in wet-stretching technology platform. The wet-stretching process may be slightly easier than the dry-stretching process. Both have more difficulty.

The typical polymeric materials suitable for this purpose are low-melting-point polyethylene and polybutylene-1. The low-melting-point polyethylene is mostly linear low-density polyethylene for its uniform crystal distribution. The polybutylene-1 has a melting point of 110~125°C with few choices of grades. In this project, Ultimate Membrane Technology (UMT) made attempts to break the technical dilemma to obtain a lower-temperature shutdown temperature separator with good strength.

Project Objectives and Approach:

This program was to develop low-cost separators for rechargeable lithium-ion batteries for applications in electric vehicle (EV)/hybrid electric vehicle (HEV). It included three phases: (I) low-cost separators, (II) low-cost shutdown separators and (III) low-cost, low temperature shutdown separators. The specific objective of

Phase III was to develop a low-cost separator having a shutdown temperature lower than 135°C (preferably 110~125°C) for rechargeable lithium batteries.

The approach in this Phase III was to develop an acceptable PP/PE-PB/PP tri-layer low temperature shutdown separator with good mechanical strength via a unique co-extrusion and co-stretching process for the objective of low-cost manufacturing. The middle layer was based on the immiscible PE-PB blend with a co-continuous PB line structure like the immiscible PP-PE blend discovered in Phase II. The PE-PB blend middle layer provided the shutdown and pre-shutdown function while the PP outer layers provided the strength. The grade of PP resin was selected for being stretched at temperature compatible to the melting points of PE and PB resins so that UMT could obtain reasonably high gas permeability and reasonably low shrinkage. These outcomes were achieved with work performed in four task areas as summarized below.

Task 1 – Low-Cost Low Temperature Shutdown Separators

Dry-stretching technology was continually adopted as a processing platform for low-cost separators. Before the major shutdown of PE component was initiated, an immiscible component having a lower melting point was incorporated into the membrane to pre-shutdown the pores for further enforcing the safety.

Task 2 – Product Concept: Co-Continuous Structure of Blends for Uniform Separators
Polybutylene-1 (PB) having a melting point of 110~125°C was blended with PP or PE to form a low temperature shutdown separators. With the die design described in Phase II, PB phase formed a stable co-continuous structure with the other phase (PP or PE) in the blend and then a uniform precursor and membrane. The co-continuous blend precursor can be sandwiched by two outside layers via coextrusion for melt integrity. To have a low temperature shutdown

and reasonably good melt integrity, two blend systems were tried for the membrane separators: (1) PP-PB blend separator and (2) PP/PE-PB blend/PP tri-layer separator.

Task 3 – Separator From PP/PB Blend

In the initial effort, PB was blended with PP and extruded with the designed circular die in Phase II for the precursor. The precursor was further cold-stretched and hot-stretched into membrane. The following describes the resins used and the processing conditions:

Resins: PP/PB (80/20) blends
 PP: 165°C Tm MFI = 1.5 g/10min
 PB: 125°C Tm; MI = 4.0 g/10min
 Processing Conditions: Anneal/Stretch at 120°C

The stretched films turned into almost clear although its elastic annealed precursor should be good for forming a microporous membrane. The major challenge here is to stretch the annealed precursor further below the melting point of PB (110~125°C). The PP component was not able to form pores at low temperature. A grade of PP resin suitable for being stretched in such low temperature range is needed.

Task 4 – PP/PE-PB Blend/PP Tri-Layer Separator

The product concept was based on PE-PB blend as a shutdown layer and on PP as melt integrity layers. In the blend, the PB component was served as pre-shutdown function by its lower melting point (110~125°C), and the PE component plays the role of major shutdown (135°C) after pre-shutdown. The blend and PP were co-extruded at 216°C with the designed circular die described in Phase II. Before getting to PP/PE-PB blend/PP tri-layer separator, membrane separators from pure PE, PE-PB blend, PP/PE-PB blend/PP tri-layer were also studied. Their precursors were analyzed on TEM, and the pure PE precursor shows no line structure at all.

Conclusions and Future Needs:

PB shows the co-continuous structure in the layer of PE-PB blends. The co-continuous structure helps the extrusion stable and generates uniform film precursor. It helps produce the quality separators and raise the yield. The PP/PE-PB blend/PP tri-layer separator yielded a targeted Gurley, and puncture strength although the shrinkage remains high. The PP layers are expected to maintain the melt integrity until its melting points (160°C) and above. The PB component in the layer of PE-PB blend has a melting point of 110~124°C and is expected to perform the pre-shutdown at its melting point before the major shutdown at the PE melting point of 135°C.

More developmental work is needed to bring down the shrinkage unless the battery manufacturers can adjust their process for the separators with the high shrinkage.

1.2.1 Computer Modeling Work

– None reported.

1.3 Deliverables/Products Developed

PP/PE-PB blend/PP tri-layer separators for Li-ion batteries and associated processing technology.

1.4 Technology Transfer Activities

1.4.1 Proprietary Reporting

– Final Report was submitted to USABC on January 15, 2007.

1.4.2 Non-Proprietary Publications and Proceedings

– None reported.

2. The Development of a Low-Cost, 100°C Shutdown Separator with 200°C Melt Integrity for Lithium-Ion Batteries

Performing Organization: Advanced Membrane Systems, Inc.

Project Duration: 11/29/2004 – 3/1/2006

2.1 Executive Summary

A low temperature shutdown separator technology was developed by Advanced Membrane Systems (AMS) as the culmination of Phase I work for the USABC under a previous award. The separator was tested for shutdown by Sandia National Laboratories, and showed promising outcome and as a result of that, the project moved to Phase II. The overall goals of the 18-month Phase II were to continue product and process research and refinement in order to meet all of the USABC specifications of lithium-ion cells including the low cost, low shutdown temperature (preferably 100°C), high melt integrity (preferably 200°C), and develop roll-form samples to meet battery manufacturers cell assembly and testing requirements. Throughout the duration of the Phase II program, AMS ran many trials (extrusion, stretching, extraction, and annealing) and tested thousands of samples, modified equipment and developed a product that met most of the USABC's requirements. As deliverable, AMS also produced samples in roll-form for cell testing.

2.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

During Phase I, AMS performed the development work in order to meet the gateway criteria set forth by the USABC team. More R&D work and additional equipment were needed to further improve the uniformity, shrinkage and production of roll-form product for cell testing. The first goal of Phase II was to

produce a membrane of 25 micron thickness in roll-form that shuts down at approximately 110°C and maintains its melt integrity above 175°C. In order to meet this goal, AMS had to complete the equipment installation, and have it ready for trials that gave AMS the capability to produce thin membranes in roll-form. Work was done to further optimize formulation, process conditions and investigate how far the shutdown temperature could be lowered without compromising other properties of the membrane. In order to expedite the development process, AMS started making small samples in bench scale and utilized the information to produce larger samples in roll-form. The second goal of this phase was to assure having proper and adequate in-house test capabilities so; constructive work could be done on improving and verifying properties of the roll-form samples in order to measure against the Phase II (gap) requirements. The ultimate task of this phase was to deliver sample rolls to Sandia National Labs for cell testing and work with the USABC's battery manufacturers to meet all of their cell requirements and move on to commercialization.

Objectives and Goals:

There were two main objective of their project. The first objective of this phase was to address the entire product and process issues, have proper instruments and tools for accurate bench level testing, and make roll-form material for cell testing. The second objective was to show that the high-volume production costs stay less than \$1 per square meter and successfully pass all of the requirements and cell testing so as to provide compelling reasons to move on to a future Phase III (commercialization). The technical objectives were to meet all of the gateway criteria of Phase II.

Approach:

In order to make micro-porous membranes suitable for this project, there were three technologies to select: Dry technology (example: method used to produce Celgard), wet technology (example: method used by Tonen, Asahi, Entek and AMS to produce micro-porous film, MPF) and finally a very expensive chemical etching method. AMS selected the wet technology for this project due to its flexibility, lower costs, in-house availability of all of the required equipment and could easily be scaled up. Not all wet processes are the same; based on required properties and available machinery, process steps could vary.

During Phase I, AMS used a stretching fixture and an oven to simulate the stretching process. AMS also ran several trials at a subcontractor's site for stretching, however, the trials were not successful. In order to use the subcontractor's machine, the machine had to be modified and this modification was above the scope of this project. In addition, trial costs and scheduling would have been a major issue and set back. In January 2004, immediately following the USABC's project Phase I deadline, AMS determined that for Phase II of the USABC project needed a stretching machine. AMS purchased a used tenter frame and modified it so it could be used for cross machine direction stretching required for this project which gave AMS the capability of making prototype samples in roll-form for Phase II of this project.

During Phase I of this project, due to small sample size and low yield, all of the required testing could not be done. During Phase II, in order to make larger samples for bench level and cell testing, AMS incorporated new machinery and optimized the process parameters for extrusion, calendaring, stretching, extraction and annealing. AMS also added all of the lab equipment needed and used correct test methods for proper testing of the samples. As a result of this project, AMS has full in-house capability of making sample rolls

and performs all of the required testing. AMS made samples that show low shutdown temperature and have high melt integrity.

Tasks:

In the Separator Development task, AMS utilized a wet process method for making a microporous separator to meet the stated requirements and performance criteria. In this process, a special type of ultra high molecular weight polyethylene (UHMWPE) and low molecular weight polyethylene (LMWPE) polymers and a particulate filler (TiO₂) were mixed and extruded using wet film technology, calendaring and stretching. The basic technology used for this particular wet process consisted of eight steps: Mixing, wet extrusion, calendaring, hot stretching, extraction, annealing, testing and slitting/packageing.

Throughout the Phase II program, in order to meet all of the gap criteria, AMS ran over one hundred trials. Each trial consisted of, mixing, extrusion, calendaring, stretching, extraction, annealing and producing small sample rolls for testing. AMS performed bench level tests on over 2,000 samples and compared them against the required criteria (thickness, air permeability, MacMullin #, wet out, shutdown, melt integrity, shrinkage, puncture and tensile strength).

During the Phase II program, AMS also spent substantial amount of time and money to streamline its facility in Billerica and made it ready for pilot production. In order to meet all of the regulatory requirements, AMS hired consultants and incorporated all of the necessary equipment into its pilot line and made it ready so it can produce larger rolls in pilot scale.

Key Accomplishments:

- Because of this project, AMS has also learned to make separators with shutdown behavior in the range of 110 to 150°C
- AMS wrote detailed procedures and is capable of accurately measuring:

- Shutdown temperature: AMS designed and built system with a built-in data acquisition for accurately measuring the shutdown behavior of the separator
 - Melt integrity
 - MacMullin #: AMS made a specially designed cell that could accurately measure the MacMullin #
 - Shrinkage
 - Tensile strength: AMS purchased a motorized tensile tester for more accurate measurement
 - Puncture test: AMS built a special fixture and pin and utilized the motorized tester
- AMS streamlined its pilot facility and is capable of making larger samples in roll-form for battery testing.

Conclusions:

While the Phase II project objectives have been met and this has been a successful project, AMS had to overcome many hurdles, however, it also acquired valuable knowledge and experience throughout this project. AMS was able to make sample rolls that meet most of the gap criteria. Also, as a result of this project, AMS developed proper test methods and will be capable of making large sample rolls for HEV cell testing.

Future Work Planned:

In order to achieve lower shutdown temperatures and make a product with less shrinkage, AMS may need polymers with

narrow molecular weight distributions. Since commercially available polymers do not provide the desired properties, the last, most expensive but viable option is to have a polymer tailor-made for this application. This option definitely has a merit, but it requires cost justifications.

However, making a polymer with a narrow molecular weight distribution is costly, as it needs special process and catalyst requirements that may not be so attractive for polymer producers. It may require the outreach and influence of the USABC to accomplish that possibility.

2.2.1 Computer Modeling Work

- None reported.

2.3 Deliverables/Products Developed

Product ID 5J07 AMS rolls made for evaluation by Sandia National Labs.

2.4 Technology Transfer Activities

2.4.1 Proprietary Reporting

- Final Report for Phase II submitted to USABC in March 2006.

2.4.2 Non-Proprietary Publications and Proceedings

- None reported.

3. High Temperature Melt Integrity Lithium-Ion Battery Separators

Performing Organization: Celgard, LLC

Project Duration: 9/4/2008 – 9/1/2010

3.1 Executive Summary

The thermal abuse tolerance of Li-ion cells depends not only on the stability of the active materials in the anode and cathode but also on the stability of the separator which prevents direct interaction between electrodes. A High Temperature Melt Integrity (HTMI) separator that possesses good mechanical integrity at high temperatures to prevent the electrodes from contacting one another becomes very critical to provide the greater margin of safety needed by lithium-ion cells at higher temperatures. This two-year USABC project award to Celgard was executed in two phases, namely separator development and test standard development for evaluation of separators. In the first phase, Celgard R&D team explored numerous options towards producing an HTMI separator. Hand-fabricated samples were used to carry out initial film tests. Options included HTMI-I (a coating process with single and double sided coatings, different binders, different ceramics), HTMI-II (ceramic blended samples), and HTMI-III (high temperature resins) approaches. Most development work was carried out with an HTMI-I approach and was taken to the next level of making them at a pilot-scale level. In the second phase, Celgard has pursued and documented a systematic approach to develop an HTMI film test protocol for subjecting HTMI separators to a series of film tests that quantify the dimensional stability in X, Y, and Z directions at higher temperatures.

At the project conclusion, Celgard was able to demonstrate an HTMI concept separator with shrinkage as low as 5% at 150°C, negligible hole propagation in response to a 450°C hot spot, and no Z-direction shorting up to 220°C. In addition, the chemical and electrochemical

stability of the HTMI separators were also proven together with the ability to produce master rolls of HTMI separators.

3.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

As new automotive applications for lithium batteries emerge, cell design and performance requirements are constantly evolving and present unique challenges to traditional battery producers. A strong demand for improved safety and increased reliability of these high energy and high power density Li-ion batteries thus become inevitable. The thermal abuse tolerance of Li-ion cells depends not only on the stability of the active materials in the anode and cathode but also on the stability of the separator which prevents direct interaction between electrodes. By entering into the automotive industry, Li-ion cells and its components face stringent requirements on properties under extreme operating conditions. Thus a need for a HTMI separator that possesses good mechanical integrity at high temperatures to prevent the electrodes from contacting one another becomes very critical to provide the greater margin of safety needed by Li-ion cells at higher temperatures.

An HTMI separator is defined as a film material that possesses good mechanical integrity at high temperatures to prevent the electrodes from contacting one another. This capability increases the margin of safety for high temperature operation of Li-ion cells.

Objectives and Goals:

- Develop a standard test protocol for evaluating HTMI property in Li-ion battery separators.

- Design and develop a separator product that demonstrates the HTMI criteria at 220°C.

Project Approach and Scope:

The USABC project award to Celgard included two segments, namely separator development and test standard development. The separator development part involved exploring several options on the laboratory scale. Hand-fabricated samples were used to carry out initial film tests. Options included HTMI-I (a coating process with single and double sided coatings, different binders, different ceramics), HTMI-II (ceramic blended samples), and HTMI-III (high temperature resins) approaches. Most development work was carried out with an HTMI-I approach and was taken to the next level of making them at a pilot-scale level. Some studies were also done with the HTMI-II approach.

The standard test-method development task started with a baseline study where Celgard focused on identifying the critical separator properties that influence high temperature performance of Li-ion cells. A few existing Celgard commercial separators were tested first to create a baseline or reference point for comparison against the new HTMI separator to be developed later. Celgard has both dry and wet process technologies and for this study, products were used from these different technologies to establish the baseline. The study focused upon the properties which were believed to be the characteristics of HTMI behavior in the Li-ion industry. This is also based on the information from other competitors and also the feedback received from customers. The properties included X and Y shrinkage from heat, Z-direction strength, and high temperature stability. Several test methods and test conditions were proposed and were developed to measure the critical HTMI film properties. After completing the film tests for the baseline separators, similar tests were performed on

several variants of HTMI separators that were developed during the course of the project.

The next step was the cell tests which were carried out using both baseline separators and newly developed HTMI separators to look for safety improvement. Li-ion cells were fabricated at Celgard and testing was done focusing on mechanical abuse and high temperature tests to study how the HTMI property of separator influences Li-ion cell safety. Upon validating the proposed HTMI film test protocol and correlating the film tests with the cell abuse tests, HTMI film test standards were finalized and the operating procedures were also documented.

Film Test Methods to Quantify HTMI Property:

The following are the film test methods proposed to measure or quantify the safety characteristics of a HTMI separator:

- Shrinkage measurement in a conventional oven at different temperatures for fixed duration.
- Thermal Mechanical Analysis (TMA) test to measure the strain with temperature in machine direction (MD) and transverse direction (TD).
- Hot spot test that simulates the internal short and a measure of the propagation area.
- Measurements of film resistance as a function of temperature to determine the Z-direction integrity at higher temperatures.

Tasks for HTMI-I (2009) Separator Development:

The main activity was development of ceramic coating using a Celgard base film. The sub-tasks included the following:

1. *Development Activities* – A coated separator with ceramics has been used

recently in the lithium battery market. It has been shown that the ceramic coating provided certain HTMI advantages over regular separators, especially in higher capacity cells. Developing a coated separator requires a two-stage R&D effort. The first stage, is to develop a suitable and thin substrate base film; and, the second, is to apply a coating onto one or both sides of the base film.

2. *Developing a Thin Base Film* – During coating trials with different substrate films which included both PE and PP films and also dry/wet process films, it was found that the wet process based PE film had better coating performance in terms of adhesion. Therefore, the development focus was on the wet process PE film only.
3. *Applying Coating onto the PE Base Film* – Based on preliminary studies, Alumina ceramics had the best performance (battery chemistry compatibility and performance) among the different ceramic particles being tried (Alumina, SiO₂, TiO₂, CaCO₃, etc.). So the Alumina ceramic was our top choice. For polymer binders, a variety of polymers (polyolefin, PVDF, polyimide, and poly-aramide) were studied. Among these binder polymers, poly-aramide stood out as the top choice due to its high temperature properties and its binding and adhesion properties to the ceramics and the base film. In order to achieve the balanced HTMI properties (adhesion as well as good porosity in the coating layer) Celgard chose a formulation of 50:50 between ceramics and polymer binders. Dip coating technology was chosen because this allowed coating of both sides of the base film in one step.

Summary of HTMI (2009) Sample Evaluation:

Based on the film tests and the cell abuse tests conducted internally, HTMI-I v1 separator shows an advantage compared to baseline separators. However, the results were quite inconsistent and not reproducible. HTMI-I v1 separator need to be optimized further to overcome some of the existing limitations that included: void areas as seen in the SEM cross section, excess variation in the Z-direction strength, and variation in dielectric breakdown.

HTMI-II Separator Evaluation:

The HTMI-II concept involved blending of ceramic filler material into polypropylene and making the separator using a dry stretch process. Hand samples were successfully made and the film was evaluated for all the proposed HTMI film tests and hot box tests.

Based on the HTMI film tests conducted, HTMI-II separator showed little benefit on hot-spot test and high temperature shrinkage tests compared to the baseline separators. Resistance retention tests and rupture temperature tests showed no additional benefit for HTMI-II separator. Hot-Box test at 150°C and 160°C showed similar results compared to trilayer and mono-PP separator. Hand-fabricated samples were successfully made for the HTMI-II approach and the film was evaluated for all the proposed HTMI film tests and hot box tests. Further attempts were made to produce the film in pilot scale, and several processing issues were identified with the sample. Since the sample was weaker than the base film, it had slitting issues, and as a result, slit rolls were not able to be prepared. Further sample evaluation (Nail test with 18650 cells) was not carried out. Several trials were attempted to improve upon the strength, but with a ceramic loading of ~50 wt%, the trials were unsuccessful and no further trials for HTMI-II concept were planned.

Conclusions:

Overall, the project objectives have been achieved successfully. The deliverables included a standard test protocol for evaluating HTMI film properties in Li-ion battery separators and designing and developing an example separator product that meets the HTMI criteria of 220°C.

- HTMI-Standard Test Development:** A systematic approach has been taken to develop an HTMI film test protocol. To be qualified as HTMI, a separator may be subjected to a series of film tests that quantify the dimensional stability in X, Y, and Z-directions at higher temperatures. The tests include TMA in MD and TD, Hot electrical resistance test in Z-direction, and the Hot-Spot test. TMA involves measuring X-Y dimensional stability at higher temperatures. Shrinkage, elongation, and rupture can all be measured in machine and transverse directions. The Hot-ER test involves measuring film resistance as a function of temperature to determine the Z-direction integrity at higher temperatures. The Hot Spot test is a simulation test that can be used evaluate the role/effect of a separator in during internal short circuits in Li-ion cells. This test is a measure of hole propagation in response to a hot spot of 450°C.
- Correlation Study:** The test protocol has been validated with several HTMI separators made as a result of product development efforts, and the film test data were correlated with the cell abuse tests conducted internally at Celgard. Based upon the validation of the HTMI test protocol and the data gathered internally, qualified HTMI separators shall need to have: very low shrinkage (<15%) in MD and TD at higher temperature (~140°C); higher melt integrity in Z-direction (>160°C); and, reduced propagation in

response to a hot spot (~35% reduction compared to baseline separators). Separators exhibiting these superior high temperature properties are expected to perform marginally better than conventional separators, at high temperatures and for internal short conditions.

- HTMI Film Development:** During the course of this USABC project, the Celgard R&D team explored numerous options towards producing an HTMI separator. The approaches included: HTMI-I (single and double sided coatings, different binders, different ceramics), HTMI-II (ceramic blended samples), and HTMI-III (high temperature resins). With these successful development efforts, Celgard was able to demonstrate an HTMI concept separator with shrinkage as low as 5% at 150°C, negligible hole propagation in response to a 450°C hot spot, and no Z-direction shorting up to 220°C. In addition, the chemical and electrochemical stability of the HTMI separators were also been proven. Finally, the ability to produce master rolls of HTMI separators was demonstrated.

Future Work Planned:

High-volume manufacturing processes and quality control specification for automated production of a durable separator product that meets the HTMI criteria of 220°C.

3.2.1 Computer Modeling Work

– None reported.

3.3 Deliverables/Products Developed

- A standard test protocol for evaluating HTMI film properties in Li-ion battery separators.

- Development of an example separator product that meets the HTMI criteria of 220°C.

3.4 Technology Transfer Activities

3.4.1 Proprietary Reporting

- Final Report to USABC dated October 2010 with the deliverable HTMI Film Standard Test Protocol.

3.4.2 Non-Proprietary Publications and Proceedings

- None reported.

4. Multifunctional, Inorganic-Filled Separators for Large Format, Li-Ion Batteries (Phases I, II, & III)

Performing Organization: ENTEK Membrane, LLC

Project Duration: 10/31/2008 – 12/31/2013

Phase I: 10/13/2008 – 12/31/2009

Phase II: 2/8/2010 – 6/30/2011

Phase III: 8/22/2011 – 12/31/2013

4.1 Executive Summary

The Separators are integral to the performance, safety, and cost of lithium-ion batteries. During normal operation, the principal functions of the separator are to prevent electronic conduction (i.e. shorts or direct contact) between the electrodes while allowing ionic flow through the electrolyte. In the case of large format Li-ion cells for hybrid or plug-in hybrid applications (HEV, PHEV), there are opportunities to handle many failure modes at the system level, through the Battery Management System, (BMS), cooling system or mechanical structure of the battery. A separator that does not shutdown will be required to be thermally stable, resist shrinkage, and provide a high barrier to electrode contact at high temperatures.

In Phase I and II USABC programs, ENTEK produced alumina-filled and silica-filled separators at high filler loadings with good dimensional stability at 200°C. The separators were manufactured using ultrahigh molecular weight polyethylene (UHMWPE) gel processing on ENTEK production scale equipment. Sequential biaxial orientation was identified as the preferred process for making thin films. ENTEK performed both roll-to-roll extraction of oil plasticizer and film annealing.

Thermal integrity testing at Sandia National Labs showed that filled separators are thermally stable to 250°C. While an improvement in safety or abuse tolerance could not be

demonstrated, the inorganic filled separators exhibited a number of desirable properties. The cycle life of cells with silica and alumina filled separators was increased by 80% compared to the controls with unfilled separators. Cells with the silica filled separators also showed lower rates of self-discharge and lower capacity fade rate when stored fully charged at 60°C. Cells with the silica filled separator also show improved low temperature (cold cranking) performance.

ENTEK has sampled and supplied silica-filled separators to numerous cell manufacturers. The lack of shutdown and low mechanical strength are the biggest hurdles preventing commercial acceptance of separator with high levels of inorganic filler. It is difficult to pass overcharge and short circuit test for cells made with inorganic filled separators. Winding and assembling cells is also challenging due to the lower tensile modulus compared to unfilled separators.

A capability to manufacture inorganic-filled separators is built into ENTEK's new Teklon line 3, absent two key components: sheet die and, calendar. This equipment will not be added until a market for this material develops. ENTEK will continue to be able to make pilot runs of inorganic-filled separator using the MDO/TDO line at PTI. Lastly, 18650 cells with filled separators were delivered to INL and Sandia for testing.

4.2 Comparison of Actual Accomplishments with Goals and Objectives of Project

Inorganic fillers are commonly used as reinforcing agents in polymer systems (e.g. silica-reinforced tire tread), but not at the loading levels required to achieve a

3-dimensional (3D) inorganic network. In the manufacture of inorganic-filled separators, the thermally-induced phase separation of the polymer and plasticizer (i.e. oil) ensure that the extracted sheet has 3D interconnecting and interpenetrating pore and polymer networks. Such a structure is required to ensure ion flow or transport from one surface of the separator to the opposite face. In a similar fashion, the interconnected polymer network ensures transmission of a load throughout the bulk structure. As inorganic fillers are added to the polymer oil mixture, they remain as isolated aggregates in the extracted separator until a critical concentration is reached. In the case of monodisperse spheres, a percolation threshold of 18 volume % filler would be required to ensure an interconnected inorganic network from one separator surface to the opposite one. *An inorganic network can be formed at lower volume fractions provided that the filler has a higher dimensionality or fractal dimension than a solid sphere.* As a result of the 3D inorganic network, this separator would be expected to exhibit low shrinkage at temperatures above the polymer melting point.

Objectives and Goals:

The overall objective of the three-phase program with USABC was for ENTEK to achieve volume-manufacturable inorganic-filled separators with high temperature stability, low electrical resistance and, to supply test cells incorporating the separators for evaluation of cycle life that were capable of low self-discharge and low capacity fade.

Summary Approaches and Key Tasks in USABC Separator Development:

Phase I	Phase II	Phase III
Separator Model	Separator model	Separator model
Filler Selection	Process Technology	Process Technology
Polymer Matrix Considerations	Optimization of filler to polymer ratio	Filler dispersion in precursor sheet
Process Technologies	Tensile properties of oil laden precursor sheet	Films Stretched at PTI and Extracted on Teklon Production Extractor
Heat Treatment / Annealing	Heat Treatment of Separator (Annealing) and Shrinkage	18650 Cell testing continued from Phase II
Electrochemical Performance	Cell builds by an outside lab	Moisture Management
Separator Model	Cell testing at ENTEK	Abuse Testing, 18650 Cells
	Thermal integrity testing (separator)	Abuse Testing, Pouch Cells
	Thermal ramp testing (cell)	Large Format Cells
	Moisture Management	Densification of Silica Filled Separators
	Shutdown functionality	Deliverables

Phase I Task Outcomes:

In the Phase I USABC project, ENTEK focused on achieving separators with low impedance and excellent high temperature, mechanical and dimensional stability using the following approaches:

- Incorporation of inorganic fillers into a polyolefin separator at high loading levels to form a 3D inorganic network

- Use of silane-grafted polyethylene to crosslink the polymer matrix in highly filled separators
- Heat treatment (annealing) of bi-axially-oriented, highly filled separators above the melting point of the polymer matrix to reduce residual stress while maintaining high porosity

Separators were manufactured using UHMWPE gel processing in combination with high loading levels of precipitated silica or fumed alumina. The resultant oil-filled sheets were bi-axially-oriented, and then solvent extracted and dried to form microporous separators. SEMs of these separators show an interpenetrating network of UHMWPE fibrils, inorganic filler, and pores.

ENTEK successfully demonstrated 20-30 μ m thick, inorganic-filled separators that shrank less than 5% in both the machine- and transverse-directions after heating the separator in an inert atmosphere for 1 hour at 200°C. The separators were produced without compromising other desirable properties such as high porosity

(> 50%), rapid wetting, and extremely low impedance values. The excellent stability of the separator at high temperature is expected to improve abuse tolerance of Li-ion cells (e.g. internal short circuit). Initial coin cell work with conventional Li-ion electrodes shows promise for the electrochemical stability and performance of these new ENTEK separators. In Phase I, ENTEK demonstrated both alumina-filled and silica-filled separators with extremely low impedance and excellent high temperature melt integrity (i.e. <5% MD and <5% TD shrinkage at 200°C). Further work is required to refine the manufacturing process and to optimize the chemical/physical properties of these inorganic-filled separators.

Based upon the success of this program, ENTEK submitted a follow-on proposal to further refine the chemical/physical properties and manufacturing process for inorganic-filled separators.

Table 1 shows the final gap analysis between USABC goals and the inorganic-filled separators that ENTEK was able to achieve.

Table 1. Gap Analysis Between U.S. ABC Goals and Inorganic-Filled Separators Shown in Phase I

Parameter	Units	USABC Goal	Process A, 67% Al ₂ O ₃	Process B, 69% Silica,
Selling price	\$/m ²	1.00		
Thickness	micron	<25	22	24
MacMullin#	#	<11	<6	<3
Gurley	s/10cc	< 35	14	3.5
Wettability		Wet out in electrolytes	Complies	Complies
Chemical Stability		Stable in battery for 10 years	Not tested	Not tested
Pore Size	micron	<1	<1	<1
Puncture Strength*	gf/25.4 μ m	>300	110	245
Thermal Stability at 200°C		<5% shrinkage	2.8% MD, 3.3% XMD	2.8% MD, 3.3% XMD
Tensile Strength		<2% offset at 1000 psi	1,200	6,900
Skew	mm/m	<2 mm/meter	< 2	< 2
Pin Removal		Easy removal from all winding machines	-	Cells Built
Shutdown	°C	As required	No Shutdown	No Shutdown

Phase II Task Outcomes:

In the Phase II USABC program, ENTEK demonstrated significantly improved cycle life for 18650 cells built with gel process, precipitated silica-filled separators, compared to control cells built with an unfilled polyethylene separator. The cycle life of cells with silica filled separators increased by 80% compared to the controls. Cells with the silica filled separator also showed lower rate of self discharge and lower capacity fade rate when stored at 60°C. Cells made with precipitated silica, fumed silica and fumed alumina filled separators gave no indication of negative impact on cell chemistry. At this point precipitated silica is the preferred filler in terms of cost and cell performance.

Sequential biaxial orientation was indentified as the preferred film making process. Preferred tensile properties for biaxial precursor sheet

have been indentified. Precursor sheet has been made routinely on production scale equipment. Roll-to-roll annealing processes for ensuring high temperature dimensional stability have been identified. These roll-to-roll processes can be adapted to a continuous inline separator production process.

Thermal integrity testing at Sandia National Labs showed that filled separators are thermally stable to 250°C. Thermal ramp testing of 18650 cells at Mobile Power solutions shows that an inorganic filled separator alone will not prevent thermal runaway; if the cell can be discharge by an alternate path, the separator remains intact even at 250°C.

The final gap analysis at the conclusion of Phase II is shown in Table 2.

Table 2. Gap Analysis Between USABC Goals and the Deliverable Separators in Phase II

Parameter	Units	USABC Goal	Program Goal	Silica DY110420.002
Thickness	micron	<25	<25	19
MacMullin #	#	<11	<8	< 4.2
Gurley	s/10cc	< 35	< 20	7.5
Wettability		Wet out in electrolytes	Wet out in electrolytes	Complies
Chemical Stability		Stable in battery for 10 years	Stable in battery for 10 years	Not tested
Pore Size	micron	<1	<1	< 1
Puncture Strength, JIS 1019*	gf	>300 gf/25.4 µm	>300 gf/25.4 µm	285
Thermal Stability at 200°C		<5% shrinkage	<3% shrinkage	4.7% MD
				2.7% XMD
Tensile Strength		<2% offset at 1000 psi	<2% offset at 1000 psi	1390
Skew	mm/m	<2 mm/meter	<2 mm/meter	-
Pin Removal		Easy removal from all winding machines	Easy removal from all winding machines	-
Shutdown	°C	As required	As required	No Shutdown
Selling price	\$/m ²	1.00	1.00	1.2

Phase III Task Outcomes:

In Phase III of this program ENTEK has demonstrated:

- Oil filled bi-axially-oriented film can be extracted on production scale equipment (previously it was demonstrated that precursor sheet can be made on production scale equipment).
- The previously identified performance advantages for 18650 cells built with precipitated silica filled separators are repeatable
 - 80% longer cycle life
 - Lower self-discharge rate
 - Better capacity retention when stored at 60°C
 - Better low temperature performance.
- Cells made with precipitated silica filled separators demonstrate lower self discharge rate better capacity retention than unfilled controls when stored at 70°C and 4.2V with very little resistance growth.
- High levels of moisture in the inorganic-filled separators do not appear to cause problems in the cell designs and chemistries investigated in this project.
- Mechanical properties of the inorganic-filled separators can be improved with higher polymer content, but at a cost of higher shrinkage at high temperature.
- Mechanical properties can be further improved with densification of the film by calendaring.

- ENTEK has sampled silica-filled separators to numerous cell manufacturers. To date, only one, JCI, has demonstrated significant interest. Lack of shutdown and low mechanical strength are the biggest hurdles preventing commercial acceptance of separator with high levels of inorganic filler. It is difficult to pass overcharge and short circuit test for cells made with inorganic filled separators. Winding and assembling cells is also challenging due to the lower tensile modulus compared to unfilled separators.

Gap Analysis:

ENTEK was not able to produce a separator that met all of the USABC goals. This gap analysis in Table 3 presents a comparison of silica-filled separators with three different formulations. Separators with higher silica content have low shrinkage, but also low puncture strength. Increasing the PE content results in higher puncture strength that meets the USABC goal but also higher shrinkage. ENTEK concluded that the final formulation of inorganic-filled separators will be application specific.

Conclusions:

The three consecutive USABC awards allowed ENTEK to achieve inorganic-filled separators with excellent high temperature stability, very low electrical resistance and, cells with excellent cycle life and low self-discharge and low capacity fade at 70°C and 4.2V.

Table 3. Gap Analysis Between USABC Goals and Deliverable Separators in Phase III

Parameter	Units	USABC Goal	Phase II Base Line 2.3:1 S/P	Current Value Roll PR 545-1 1.65:1 S/P	Current Value Roll PR 553-1 1.25:1 S/P
Thickness	micron	<25	19	20.7	20.1
MacMullin #	#	<11	≈2	1.92	2.74
Gurley	s/10cc	< 35	7.5	8.7	12.0
Wettability		Wet out in electrolytes	Complies (fast-wetting)	Complies (fast-wetting)	Complies (fast-wetting)
Chemical Stability		Stable in battery for 10 years	Not tested	Not tested	Not tested
Pore Size	micron	<1	< 1	< 1	< 1
Puncture Strength, JIS 1019*	gf	>300 gf/25.4 μm	285	331	421
Thermal Stability at 200°C		<5% shrinkage	4.7% MD	10.8 %MD	15.7% MD
			2.7% XMD	6.5%XMD	9.4% XMD
Thermal Stability at 150°C				9.9 % MD	13.1% MD
				5.3 % XMD	7.3 % XMD
Tensile Strength		<2% offset at 1000 psi, MD	1390	3438	3357
Skew	mm/m	<2 mm/meter	Not measured	Not measured	Not measured
Pin Removal		Easy removal from all winding machines	Not measured	Not measured	Not measured
Shutdown	°C	As required	No Shutdown	No Shutdown	No Shutdown

4.2.1 Computer Modeling Work

– None reported.

4.3 Deliverables/Products Developed

An initial production of 18650 cells was made by ALEC for delivery to INL and Sandia for testing: 35 cells with Teklon control separator and 35 cells with silica-filled separator. Of these 48 cells were shipped to INL and 22 cells were shipped to Sandia in December of 2012. (During preliminary characterization testing at

INL, it was discovered that several of the cells with silica-filled separators exhibited signs of internal shorting during charging. An investigation of the cells determined that a change in cell design by the manufacturer was responsible for the observed behavior. Consequently, all of the cells were returned to ENTEK and a new set of deliverables was ordered from a different manufacturer, Farasis Energy Inc.)

September 19, 2013 Farasis Energy Inc. delivered the following 18650 cells to ENTEK:

- 39 with control separator (Teklon)
- 35 with silica-filled separator
- 2.0 AH design: NMC/graphite, no PTC

The cells were screened at ENTEK:

- 1 capacity cycle (1C)
- 168 hour open circuit stand and discharge (1C)
- 1 final capacity cycle (1C)

On the basis of screening the following cells were selected:

- Controls: 4 cells with the lowest OCV after 168 hour were removed
 - 35 cells to ship
- SFS: 4 cells with greater than 10% capacity loss on OCV stand and 1 cell that was accidentally reversed while setting SOC for shipment
 - 30 cells to ship
- The total number of cells ready to ship was less than the 70 planned.

Final Disposition of cells (cells were shipped on 10/31/2013) is shown in the table:

Destination	Control Cells	SFS Cells
INL	23	20
Sandia	12	10
Total	35	30

From the cells retained by ENTEK, three each of the controls and SFS cells were placed on cycle test. Initial cycle performance suggests that the cells with the silica-filled separator started with lower capacity than the Teklon controls but have a lower rate of capacity fade

4.4 Technology Transfer Activities

4.4.1 Proprietary Reporting

Three Final Reports to USABC were submitted as follows:

- Phase I dated December 31, 2009 titled “Highly Filled and/or Crosslinked Lithium-Ion Battery Separators for HEV/PHEV Applications.”
- Phase II dated August 28, 2011 titled “Multifunctional, Inorganic-Filled Separators for Large Format, Li-ion Batteries.”
- Phase III dated January 17, 2014 titled “Multifunctional, Inorganic-Filled Separators for Large Format, Li-ion Batteries (Phase III Development Program).”

4.4.2 Non-Proprietary Publications and Proceedings

At the end of Phase I, a provisional patent entitled “Highly Filled Lithium-Ion Battery Separators and Methods of Making the Same” was filed on March 19, 2009.

At the end of Phase II, a poster titled “Development of Separators with Inorganic Fillers for Advanced Lithium Ion Batteries” was presented at the Battery Safety & Lithium Mobile Power 2010 conference in Boston MA on November 3, 2010 by Robert Waterhouse.

Presentations derived from Phase III included:

- [1] R. Waterhouse, Y. Patil, J. Emanuel, J. Frenzel, D. Lee, D. Spitz, and R. Pekala, Highly Filled Lithium-Ion Battery Separators for HEV/PHEV/EV Applications, 220th ECS Meeting & Electrochemical Energy Summit Boston, Massachusetts (October 9-14, 2011).

- [2] R. Waterhouse, Y. Patil, J. Emanuel, S. Peddini, and R. Pekala, Dimensionally Stable, Highly Porous Separators for Large Format Lithium-Ion Batteries, Advanced Automotive Battery Conference, Orlando FL (February 6-10, 2012).
- [3] R. W. Pekala, R. Waterhouse, Y. Patil, S. Peddini, J. Emanuel, J. Frenzel, D. Lee, D. Spitz, and G. Fraser-Bell, Multifunctional, Inorganic-Filled Separators for Large Format, Li-ion Batteries, DOE Annual Merit Review, May 16, 2012.

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