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IRON-AIR BATTERY DEVELOPMENT PROGRAM

Final Report for 1979

By
E. S. Buzzelli
C. T. Liu
W. A. Bryant

May 1980

Work Performed Under Contract No. AC02-76ET13390

Westinghouse R&D Center
Pittsburgh, Pennsylvania

U. S. DEPARTMENT OF ENERGY

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C. T. Liu
W. A. Bryant

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ABSTRACT

This report describes the progress and status of the research and development program on the iron-air advanced technology battery system at the Westinghouse Electric Corporation during the period June 1978-December 1979. This advanced battery system is being developed for electric vehicle propulsion applications. Testing and evaluation of 100 cm² size cells was undertaken while individual iron and air electrode programs continued. Progress is reported in a number of these study areas. Results of the improvements made in the utilization of the iron electrode active material coupled with manufacturing and processing studies related to improved air electrodes continue to indicate that a fully developed iron-air battery system will be capable of fulfilling the performance requirements for commuter electric vehicles.

I. SUMMARY

1.0 Summary

The iron-air battery development program has, during this work period, concentrated on the fabrication and testing of both 100 cm² size cells and individual iron and air electrodes. Significant progress was made towards meeting our near term goals of a specific energy content of 110 wh/kg and a specific power capability of 110 w/kg through the development of a space efficient cell casing structure and a catalyst-coated, electrolytically porous current-collecting grid. Progress in demonstrating extended stable cyclic life in full cells has been slower than anticipated due to air electrode structural limitations.

Prototype cell-size (400 cm²) iron electrodes were prepared and evaluated which demonstrated capacities equal to their research test size (10 cm²) counterparts. Considerable progress was made in the fabricating of iron electrodes with the preferred structure with increased utilization along with reproducible performance characteristics.

Near the end of the reporting period, program emphasis was shifted almost entirely towards bifunctional air electrode development. Progress in the fabrication and construction of reproducible electrodes was made along with improved performance and life. Studies concerning the effects of cycling conditions, active carbon layer electronic resistivity and surface active agent additions to the electrolyte were also initiated. Further fundamental materials processing studies of the air electrode and its components have led to a better understanding of the air electrode while simultaneously resulting in improved cyclic performance.

II. INTRODUCTION

2.0 Introduction

In the selection of candidate battery systems for electric vehicle propulsion applications, several criteria need to be met. First and foremost among these are the necessary electrochemical performance characteristics of stored energy content and specific power capability. A successful system must also incorporate these characteristics in a durable system with long cycle life capability. In addition, the candidate battery should ideally be composed of low cost, readily available materials. Finally, the requisite peripheral systems for normal operation in the electric vehicle environment should be kept to a minimum.

The iron-air cell, consisting of a porous sintered iron electrode coupled to a fully bifunctional carbon-based air electrode is capable of satisfying the required performance characteristics for electric vehicles. The ultimate goals for the iron-air cell are a specific energy content of over 140 wh/kg and a specific power delivery capability of more than 130 w/kg, required for vehicle acceleration. Projected cycle life is to exceed 1000 charge-discharge duty cycles. The primary materials utilized in the iron and air electrode fabrication procedures are carbon and iron powders, both of which are low cost and virtually inexhaustable in supply. Moreover, since the cell is designed to operate at near ambient temperature (or slightly above 45°C), complex and expensive auxiliary subsystems normally associated with the higher operating temperature electrochemical systems are rendered unnecessary.

Attainment of performance and lifetime goals have been technically limited by difficulties in the achievement of extended stable cycle life in 100 cm² size cells. This report describes the progress

and results to date in these areas as well as advancements in electrode performance studies completed during the work period.

III. AIR ELECTRODE STUDIES

3.1 Introduction

Previous testing has demonstrated that promising electrical performance can be met in a bifunctional air electrode structure. More specifically, it has been determined that the creation of a proper wettable surface within the air electrode is of paramount importance with regard to the discharge performance of the bifunctional air electrode. However, the development of specific fabrication techniques to accomplish this has been more difficult than originally anticipated. The results of studies to improve air electrode performance through improved methods of fabrication are discussed in section 3.3.2 (LU Series).

More fundamental studies of air electrode operation and structure have been deemed to be necessary to achieve improved performance and life. The results of this series of experiments are described in section 3.3.3 (B Series).

Both series of electrodes were designed to test specific concepts of air electrode operation. Such concepts are presented in Appendix 8.2.

3.2 Air Electrode Fabrication

3.2.1 Carbon Powder Preparation

3.2.1.1 Carbon Silverizing

Silverized carbon is prepared by precipitating Ag on carbon black (in presence of NH_2NH_2) via the addition of AgNO_3 to a wet carbon slurry. Excess water is vacuum filtered from the slurry and the resulting paste is air dried for ~16 hrs. (For specific carbon/ AgNO_3 weight ratios, see the Appendix.)

3.2.1.2 Active Material Preparation

Dry silverized carbon powder is mixed with Teflon 30B dispersion (and/or other polymeric binders) and various metal catalyst powders (see Appendix for specific metals) in a wet slurry. Material is then air or oven dried (100°C) and blended to form a fine flowing powder.

3.2.1.3 Hydrophobic Material Preparation

Carbon powder is wet slurry mixed with Teflon 30B dispersion and dried as in section 3.2.1.2. The powder is then oven baked (@ 300°C, 2 hrs) to thermally decompose the binder dispersion agent.

3.2.2 Electrode Formation and Processing

The above dry powders are then mechanically pressed into sintered Ni fiber current* collecting grids (see Figures A-8, A-9 of Appendix) to form the green electrode structure. Final processing involves hot flat bed pressing of the above (conditions listed in Appendix).

The finished electrode is tabbed (via spot welding) and mounted into a 1/8" ABS frame (40 cm² air 'window' exposed), as further described in Reference (1).

3.3 Experimental Results and Discussion

3.3.1 Test Conditions

All electrodes are fully cycled in a 25% KOH aqueous solution at 25°C against a Ni sheet counter electrode. Cells are run using unscrubbed air, and electrolyte level within the cell casing is maintained via a siphon tube arrangement. Potentials are measured relative to a Hg/HgO reference electrode. During the discharge (O₂ reduction) mode, electrodes are operated at a current density of 25 mA/cm² for four hours.

*Or other material (see Appendix).

Charging is accomplished at a current density of 12.5 mA/cm^2 , also for four hours. Once a week, electrodes are polarized in the discharge mode over a current density range of $6.25\text{--}125 \text{ mA/cm}^2$.

3.3.2 LU Series Electrodes

Tests were continued on air electrodes LU 046 to 056, whose preparation and composition is described in the 1978 Final Report (#C00/2949-1). These electrodes, utilizing identical active material compositions, were fabricated using both wet pasting and preform molding techniques (see 8.3 in Appendix) and 2-3 layers of nickel or steel fiber current collectors. All were hot pressed at temperatures in excess of 370°C for times of at least 10 min. The performance of these electrodes in the discharge mode (Table III-1) indicated that the wettable region within the electrodes had been limited by the high pressing temperatures. Those electrodes incorporating sponge iron powder in the active material exhibited lower oxygen evolution potentials (by $\sim 50 \text{ mV}$) than those not containing this additive (Table III-2). This effect was independent of electrode preparation method. The cycle life of all but the wet pasted electrodes was less than 100.

Electrodes LU 057 through 064 were cold pressed and then oven baked in lieu of hot pressing as a final processing stage. Due to the prolonged, high temperature baking treatment, discharge performance was poor (Table III-1). It is thought that the use of an FEP coated current collector in these electrodes was a contributing factor towards this performance. This was done in an attempt to ensure active layer-current collector bonding. Electrodes 059 through 061, containing sponge iron powder in the active material, exhibited the aforementioned effect in the charge mode.

In electrode LU 065, the charging and discharging layers were prepared separately to test a new concept. The charging layer contained a mixture of 1 gram of charging active material (see below) and .5 gram of sponge iron powder, pressed in between two low density ($.10 \text{ gm/cm}^2$)

TABLE III-1

Discharge Potentials of Air Electrodes (mV vs. Hg/HgO)
at Current Density of 25 mA/cm²

Electrode	<u>Cycles</u>						<u>Off Test @ Cycles</u>
	<u>3</u>	<u>27</u>	<u>51</u>	<u>75</u>	<u>99</u>	<u>123</u>	
LU-046	-218	-176	-167	-141	-169	--	114
-047	-204	-166	-157	-148	-161	--	114
-048	-222	-180	-285	--	--	--	63
-049	-203	-177	--	--	--	--	27
-050	-177	-159	-252	-230	--	--	93
-051	-200	-278	--	--	--	--	27
-052	-215	-178	-196	-303	--	--	75
-053	-211	-189	-243	--	--	--	75
-054	-209	-163	-138	-184	--	--	72
-055	-164	-287	--	--	--	--	39
-056	-202	-213	--	--	--	--	30
-057	-245	-218	--	--	--	--	48
-058	-278	-206	--	--	--	--	48
-059	-237	-197	--	--	--	--	36
-060	-204	-176	--	--	--	--	36
-061	-275	--	--	--	--	--	24
-062	-139	-181	--	--	--	--	36
-063	-306	-443	--	--	--	--	36
-064	-262	--	--	--	--	--	24
-065	-259	-407	--	--	--	--	33

TABLE III-1 (continued)

Discharge Potentials of Air Electrodes (mV vs. Hg/HgO)
at Current Density of 25 mA/cm²

Electrode	Cycles										Off Test @ Cycles	
	<u>3</u>	<u>27</u>	<u>51</u>	<u>75</u>	<u>99</u>	<u>123</u>	<u>147</u>	<u>171</u>	<u>195</u>	<u>219</u>		
LU-066	-192	-178	-192	-198	-178	-166	-196	-190	-216	-269		
-067	-173	-162	-171	-171	-144	-176	-173	-169	-183	-224		
-068	-163	-166	-200	-177	-168	-147	-172	-176	-215	-285		
-069	-119	-133*	(* @ 18th cycle, this was removed and tested against Fe electrode)									
-070	-121	-137	-157	-142	-159	-203	-297	-350	-311	--		
-071	-130	-139	-154	-136	-147	-182	-223	-253	-267	-327		
-072	-145	-158	-165	-267	-224	-246	-301	-247	-366	-258		
-073	-122	-172	-320	-325	-334	-373	-469	-511	-556	-467		
-074	-192	-167	-158	-155	-155	-151	-156	-162	-169	-240		
-075	-174	-143	-134	-135	-136	-106	-138	-145	-162	--		
-076	-175	-136	-129	-125	- 91	- 95	-177	-141	-435	-385		
	Cycles											
	<u>243</u>	<u>267</u>	<u>291</u>	<u>315</u>	<u>339</u>	<u>363</u>	<u>387</u>	<u>411</u>	<u>435</u>	<u>459</u>	<u>483</u>	
LU-066	-310	-312	-261	-370	-520	-260	-312	-310	-342	--	-- 456	
-067	-205	-204	-246	-211	-199	-318	-389	-367	--	--	-- 426	
-068	-313	-332	-362	-415	-403	-136	-148	-215	-488	--	-- 459	
-069	--	--	--	--	--	--	--	--	--	--	-- 18	
-070	--	--	--	--	--	--	--	--	--	--	-- 213	
-071	-340	-270	-313	-374	-311	-372	-674	-725	-518	--	-- 449	
-072	-592	-531	-196	-550	-460	-576	--	--	--	--	-- 381	
-073	-428	-508	--	--	--	--	--	--	--	--	-- 276	
-074	-275	-360	--	--	--	--	--	--	--	--	-- 289	
-075	--	--	--	--	--	--	--	--	--	--	-- 219	
-076	--	--	--	--	--	--	--	--	--	--	-- 237	

TABLE III-1 (continued)

Discharge Potentials of Air Electrodes (mV vs. Hg/HgO)
at Current Density of 25 mA/cm²

	<u>3</u>	<u>27</u>	<u>51</u>	<u>75</u>	<u>99</u>	<u>123</u>	<u>147</u>	<u>171</u>	<u>195</u>	<u>219</u>	<u>246</u>	<u>273</u>	<u>300</u>	Off Test @ Cycle
LU-077	-.142	--	--	--	--	--	--	--	--	--	--	--	--	26
-078	-.148	--	--	--	--	--	--	--	--	--	--	--	--	26
-079	-.118	--	--	--	--	--	--	--	--	--	--	--	--	26
-080	-.139	--	--	--	--	--	--	--	--	--	--	--	--	24
-081	-.166	-185	-231	-184	-212	-225	-241	-240	-226	-231	-253	-307	-289	324
-082	-.203	-183	-184	-189	-185	-179	-199	-218	-224	--	--	--	--	216
-083	-.331	-160	-151	-150	-150	-185	-219	-234	-271	-308	-324	--	--	267
-084	-.235	-188	-176	-179	-188	-221	-252	-268	-305	-330	-347	--	--	267
-085	-.237	-181	-161	-165	-162	-168	-188	-202	-252	--	--	--	--	207
-086	-.143	-206	-208	-227	-218	-235	-283	-321	-406	--	--	--	--	207
-087	-.136	-184	-185	-179	-232	-269	-324	-370	-495	-633	--	--	--	222
-088	-.262	-195	-182	-181	-194	-215	-242	-258	-288	-300	-338	--	--	267
-089	-.250	-187	-173	-178	-177	-179	-206	-213	-288	--	--	--	--	207
-090	-.241	-186	-179	-179	-191	-205	-236	-263	-292	-264	-266	--	--	264
-091	-.127	-160	-165	-177	-212	-246	-306	-336	-410	-427	-464	--	--	255
-092	-.135	-165	-169	-186	-198	-225	-281	-352	-461	--	--	--	--	207
-093	-.142	-165	-158	-169	-181	-207	-249	-274	-365	-441	-485	--	--	267
-094	-.150	-178	-176	-192	-193	-205	-233	-265	--	--	--	--	--	186
-095	-.160	-173	-171	-167	-194	-232	-273	-292	-350	-373	-482	--	--	267
-096	-.157	-177	-173	-185	-189	-209	-246	-269	--	--	--	--	--	186
-097	-.167	-188	-156	-206	-199	-206	-234	-256	-325	--	--	--	--	207

TABLE III-1 (continued)

Discharge Potentials of Air Electrodes (mV vs. Hg/HgO)
at Current Density of 25 mA/cm² Cycles

	<u>3</u>	<u>27</u>	<u>51</u>	<u>75</u>	Off Test <u>@ Cycle</u>
LU-098	-.158	-.182	-.191	--	54 (Leakage)
099	-.233	-.282	--	--	42 (Poor performance, leakage)
100	-.255	-.213	-.207	--	66 (Leakage)
101	-.270	-.210	-.241	--	54 (Poor performance, leakage)
102	-.270	-.237	--	--	36 (Leakage)
103	-.209	--	--	--	15 (Leakage)
104	-.207	--	--	--	15 (Leakage)
105	-.204	-.202	-.168	--	60 (Leakage)
106	-.144	-.240	--	--	45 (Poor performance, leakage)
107	-.175	Test Still in Progress			
108	-.149				
109	-.193				
110	Not in test				
111					

TABLE III-2

Charge Potentials of Air Electrodes (mV vs. Hg/HgO)
at Current Density of 12.5 mA/cm²

<u>Electrode</u>	<u>Cycles</u>					
	<u>3</u>	<u>27</u>	<u>51</u>	<u>75</u>	<u>99</u>	<u>123</u>
LU-046	+559	+553	+565	+595	+861	--
-047	+605	+634	+679	+791	+913	--
-048	+542	+546	+583	--	--	--
-049	+604	+615	--	--	--	--
-050	+538	+547	+563	+623	--	--
-051	+536	+548	--	--	--	--
-052	+573	+621	+686	+819	--	--
-053	+542	+546	+563	--	--	--
-054	+563	+591	+622	+914	--	--
-055	+516	+548	--	--	--	--
-056	+570	+578	--	--	--	--
-057	+694	+811	--	--	--	--
-058	+713	+767	--	--	--	--
-059	+555	+576	--	--	--	--
-060	+537	+555	--	--	--	--
-061	+540	--	--	--	--	--
-062	+626	+850	--	--	--	--
-063	+638	+690	--	--	--	--
-064	+685	--	--	--	--	--
-065	+616	+1047	--	--	--	--

TABLE III-2 (continued)

Charge Potentials of Air Electrodes (mV vs. Hg/HgO)
at Current Density of 12.5 mA/cm²

<u>Electrode</u>	<u>Cycles</u>										
	<u>3</u>	<u>27</u>	<u>51</u>	<u>75</u>	<u>99</u>	<u>123</u>	<u>147</u>	<u>171</u>	<u>195</u>	<u>219</u>	
LU-066	+539	+527	+521	+528	+575	+520	+527	+524	+529	+547	
-067	+525	+524	+535	+557	+790	+552	+565	+579	+601	+731	
-068	+534	+534	+505	+528	+582	+520	+519	+520	+524	+554	
-069	+530	+512*	(* @ 18th cycle, this was removed and tested against Fe electrode)								
-070	+522	+520	+575	+857	+524	+564	+522	+543	+504	--	
-071	+525	+520	+578	+841	+517	+508	+521	+522	+512	+530	
-072	+536	+523	+557	+505	+506	+501	+497	+523	+535	+624	
-073	+540	+529	+512	+519	+516	+517	+529	+548	+545	+548	
-074	+558	+561	+523	+523	+518	+513	+509	+517	+508	+535	
-075	+648	+604	+668	+600	+588	+603	+611	+573	+603	--	
-076	+544	+530	+529	+522	+515	+515	+537	+516	+526	+609	
	<u>Cycles</u>										
	<u>243</u>	<u>267</u>	<u>291</u>	<u>315</u>	<u>339</u>	<u>363</u>	<u>387</u>	<u>411</u>	<u>435</u>	<u>459</u>	<u>483</u>
LU-066	+521	+515	+523	+526	+531	+772	+623	+572	+562	--	--
-067	+591	+567	+585	+600	+605	+761	+675	+693	--	--	--
-068	+516	+506	+513	+514	+521	+644	+724	+627	+541	+826	--
-069											
-070	--	--	--	--	--	--	--	--	--	--	--
-071	+522	+527	+520	+580	+525	+683	+543	+529	+950	--	--
-072	+693	+520	+685	+543	+542	+554	--	--	--	--	--
-073	+525	+803	--	--	--	--	--	--	--	--	--
-074	+506	+530	--	--	--	--	--	--	--	--	--
-075	--	--	--	--	--	--	--	--	--	--	--
-076	--	--	--	--	--	--	--	--	--	--	--

TABLE III-2 (continued)

Charge Potentials of Air Electrodes (mV vs. Hg/HgO)
at Current Density of 12.5 mA/cm²

	<u>3</u>	<u>27</u>	<u>51</u>	<u>75</u>	<u>99</u>	<u>123</u>	<u>147</u>	<u>171</u>	<u>195</u>	<u>219</u>	<u>246</u>	<u>273</u>	<u>300</u>
LU-077	+ .513	+ .485	--	--	--	--	--	--	--	--	--	--	--
-078	+ .514	+ .483	--	--	--	--	--	--	--	--	--	--	--
-079	+ .516	+ .486	--	--	--	--	--	--	--	--	--	--	--
-080	+ .509	--	--	--	--	--	--	--	--	--	--	--	--
-081	+ .537	+ .529	+590	+526	+543	+548	+553	+566	+636	+611	+520	+540	+622
-082	+ .621	+ .541	+555	+565	+577	+601	+557	+554	+534	--	--	--	--
-083	+ .545	+ .541	+541	+550	+550	+534	+530	+516	+515	+526	+555	--	--
-084	+ .526	+ .518	+598	+521	+537	+519	+514	+503	+525	+511	+612	--	--
-085	+ .553	+ .527	+582	+529	+536	+595	+503	+504	+514	--	--	--	--
-086	+ .557	+ .557	+559	+559	+569	+555	+532	+536	+551	--	--	--	--
-087	+ .546	+ .513	+519	+609	+536	+539	+543	+542	+548	+712	--	--	--
-088	+ .530	+ .528	+529	+542	+544	+527	+520	+504	+508	+513	+551	--	--
-089	+ .565	+ .546	+550	+541	+545	+529	+510	+510	+528	--	--	--	--
-090	+ .544	+ .540	+546	+548	+531	+535	+533	+521	+512	+546	+516	--	--
-091	+ .569	+ .525	+526	+546	+539	+548	+552	+545	+538	+591	+556	--	--
-092	+ .552	+ .537	+533	+541	+529	+515	+535	+541	--	--	--	--	--
-093	+ .545	+ .540	+543	+551	+556	+549	+531	+519	+533	+534	+558	--	--
-094	+ .553	+ .538	+542	+541	+543	+531	+509	+508	--	--	--	--	--
-095	+ .524	+ .521	+524	+534	+535	+525	+520	+510	+509	+515	+535	--	--
-096	+ .560	+ .542	+543	+541	+553	+536	+519	+523	--	--	--	--	--
-097	+ .568	+ .531	+544	+541	+557	+539	+526	+527	+545	--	--	--	--

TABLE III-2 (continued)

Charge Potentials of Air Electrodes (mV vs. Hg/HgO)
at Current Density of 12.5 mA/cm² Cycles

	<u>3</u>	<u>27</u>	<u>51</u>	<u>75</u>
LU-098	+.605	+.609	+.624	--
099	+.611	+.654	--	--
100	+.651	+.627	+.624	--
101	+.635	+.597	+.561	--
102	+.704	+.671	--	--
103	+.640	--	--	--
104	+.614	--	--	--
105	+.644	+.605	+.620	--
106	+.697	+.639	--	--
107	+.587		Test in progress	
108	+.590		Test in progress	
109	+.580		Test in progress	
110	Not in test			
111				

nickel fibers. The discharging active material (see below) was wet pasted in between two low density ($.10 \text{ gm/cm}^2$) nickel fibers.

<u>LU 065 Charging Active Material</u>		<u>LU 065 Discharging Active Material</u>	
Shawnigan Black	10 gm	Darco GX	1 gm
WC-12% Co	8 gm	MnO ₂	2 gm
FeWO ₄	3 gm	AgNO ₃	.075 gm
NiS	3 gm	Teflon 30B	16 drops
FEP	3 gm	H ₂ O	8 ml
Teflon 30B	4 gm		
Triton X-100	1 drop		

A thin hydrophobic layer (.15 gm Shawnigan Black, 2.5 drops Teflon 30B and 1.25 ml H₂O) was then pasted onto the air side of the discharge layer. The composite was then air dried, after which .6 gm of H-3 material (see Appendix, Table A-1A) was pressed onto the air side. Both charging and discharging layers were cold pressed separately and then baked at 350°C for 20 min (Table A-2A). The later operation, as well as the high teflon content are thought to have been responsible for the less-than-anticipated electrical performance of this electrode.

Electrodes LU 066 through 076 were designed to investigate both the effects of Ketjenblack ($1000 \text{ m}^2/\text{gm}$ carbon) on discharge potentials and of teflon content on cycle life. The active layers of electrodes LU 070, 071, and 074 were perforated to enhance electrolyte penetration throughout the entire active layer. All electrodes in this series performed well with respect to charging potentials and cyclic life and all but electrodes LU 066 and LU 067 (higher pressing temperatures) performed well on discharge. A small addition of Triton X-100 to the active material (series J, K of Table A-1A) was shown to enhance O₂ reduction potentials.

Electrodes LU 077 through 079 were fabricated in an identical manner, except that LU 079 contained .3 gm of cobalt powder on the electrolyte side of the electrode. Electrodes LU 077 and 079 were sprayed (electrolyte side) with 2 ml of a dilute Triton X-100 solution,

while LU 078 was sprayed with a 2 ml solution of .1 N AgNO_3 solution. Both were instituted to promote rapid electrolyte penetration of the active layer. All performed well initially, but deteriorated in performance beyond cycle 15, with full electrode leakage occurring at about 26 cycles. The above attest to the detrimental effects of indiscriminate wetting of the electrode structure.

In electrodes LU 080, 081, and 082, PFA powder was added as a binder in conjunction with teflon 30B in an attempt to extend cycle life. Little improvement in achieving this end was attained, as no significant lifetime enhancement over teflon 30B-only bonded electrodes LU 083 through LU 097. There were also the first series of electrodes which were pressed at temperatures in excess of 343°C . Good charging and discharging potentials, as well as a long (over 200) cycle life characterized these samples.

Electrodes LU 098 to LU 111 were prepared to test the effect of active material pore formers on electrode performance. Addition of urea and nickel sponge powders, formation of expanded metal-Ni fiber overlap current collectors and combinations of the above have been employed in these electrodes. These electrodes were, however, prone to premature electrolyte leakage, further illustrating the effects of indiscriminate electrolyte penetration mentioned previously.

3.3.3 B Series Electrodes

The B series of half cells was designed to investigate more fundamental areas than the LU series. As such, few of these electrodes were optimized from a performance standpoint. In this series, numerous active material mixtures, current collecting grid systems, and cell cycling conditions were studied. Test results provided insight into air electrode charge and discharge operational modes and suggested procedures for enhancing performance characteristics.

In electrodes B-1 to B-9 (Tables III-3 and III-4), a neoprene type of contact cement (diluted with a methyl-ethyl ketone in xylene

solution), which was capable of penetrating the air electrode surface was utilized. It was felt that this substance would largely eliminate electrode-cell casing bond failures. In B-1, an expanded metal current collecting grid, having a large open-area structure was chosen to facilitate increased carbon-carbon aggregate contact between adjoining layers. This electrode displayed poor O_2 reduction and evolution potentials due to high cell internal impedance. This is believed to be the result of the electrochemical reaction sites lying farther, on the average, from the current collecting grid structure. B-2, made with steel fiber current collectors, exhibited encouraging discharge potentials, but suffered from current collector oxidation. B-3 and B-4 used the customary Ni fiber current collectors, were of interest performance wise, but failed via hydrophobic layer blistering (see Fig. 3-2). Electrode B-5 contained neither WC, NiS nor $FeWO_4$ and was designed to determine their necessity within the active carbon mixture. Such materials have been found to promote lower O_2 evolution potentials in past electrodes. As the data from Table III-4 shows, however, the addition of these catalysts does not appear to be required to achieve relatively low charge potentials. Half cell B-7 was fabricated using a polyethylene binder. This polymer powder has the advantages of low cost and a low melting point, and is available in free-flowing form. Thus it was thought to be a viable alternative to the relatively expensive TFE dispersion. In practice, however, carbon/polyethylene mixtures proved to be both largely impermeable to O_2 and hydrophillic with respect to the electrolyte. B-8, utilizing a previously successful active carbon layer (see Appendix, Tables A-2D, A-1F) performed well. B-8, as well as B-2, 3, 4, failed via the previously mentioned hydrophobic layer "blistering" (Figures 3-1 and 3-2). This phenomenon, a form of localized delamination, results from several sources, including multilayer construction, and the inclusion of a thick carbon layer on the electrolyte boundary of the electrode. The former leads to excessive KOH penetration of the electrode, while the latter impedes electrolyte exiting via the hydrophobic layer during the charge mode. These factors

Table III-3

Discharge Potentials of Air Electrodes (mV vs Hg-HgO)
 $j = 25 \text{ mA/cm}^2$

Electrode	3	27	51	75	99	123	147	171	195	219	246	273	300	Off Test Cycle
B-1	-.234	-.224	-.331	-.461	-.419									99
B-2	-.177	-.169	-.153	-.155	-.151									123
B-3	-.195	-.155	-.140	-.151	-.161									123
B-4	-.242	-.182	-.093	-.158	-.168	-.188								141
B-5	-.186	-.188	-.211	-.222										78
B-6	Structurally Unsound													
B-7	-.418													1
B-8	-.158	-.154												45
B-9	-.306	-.164	-.262	-.223	-.290	-.286	-.260	-.273	-.271	-.279	-.270			269
B-10	-.114	-.218	-.205	-.220	-.251	-.301	-.398	-.512	-.647	-.507	-.882			249
B-11	-.121	-.184	-.181	-.189	-.185	-.218	-.260	-.386	-.579					193
B-12	Structurally Unsound													
B-13	Continucus Discharge for 5280 hrs $\bar{V} = -.149$ $V_{\max} = -.241$ $V_{\min} = -.098$													
B-14	Intermittent Discharge for 4896 hrs $\bar{V} = -.213$ $V_{\max} = -.277$ $V_{\min} = -.119$													
B-15	Continuous Discharge for 4512 hrs $\bar{V} = -.181$ $V_{\max} = -.226$ $V_{\min} = -.127$													
B-16	-.149	-.192	-.191	-.197	-.246	-.242	-.321	-.309	-.328	-.247	-.271	-.299	-.394	343
B-17	Intermittent Discharge for 4784 hrs $\bar{V} = -.284$ $V_{\max} = -.532$ $V_{\min} = -.163$													598
B-18	-.163	-.180	-.180	-.186	-.242	-.234	-.319	-.285	-.280	-.283	-.326	-.561	-.622	343
B-19	-.159	-.181	-.186	-.182	-.211	-.170	-.313							136
B-20	-.138	-.187	-.180	-.190	-.236	-.203	-.233	-.276	-.442					213

Note: $\bar{V}$ refers to average voltage

$V_{\max}$ and $V_{\min}$ refer to maximum and minimum voltage

(All electrodes removed from test when leakage occurred.)

Except where otherwise noted.

TABLE III-3 (continued)

Discharge Potentials of Air Electrodes (mV vs. Hg-HgO)
 $j = 25 \text{ mA/cm}^2$

Electrode	3	27	51	75	99	123	147	171	195	219	246	273	300	Off. Test Cycle
B-21	-.147	-.195	-.197	-.216	-.259	-.238	-.270	-.294	-.546					213
B-22	-.141	-.210	-.212											57
B-23	-.134	-.183	-.183	-.185	-.224	-.236	-.278	-.375						183
B-24	-.267	-.287	-.288	-.462										93
B-25	-.270	-.283	-.201	-.284	-.426	-.594								138
B-26	-.214													18
B-27	-.161	-.192	-.236	-.262	-.312									102
B-28	-.145	-.196	-.231	-.187	-.306									102
B-29	-.126	-.184	-.221	-.276	-.406	-.520	-.608	-.413						183
B-30	-.214	-.201	-.202	-.222	-.276	-.326	-.334	-.435						177
B-31	-.290	-.240	-.224	-.224	-.225	-.228	-.249	-.274	-.338	-.440	-.522			201
B-32	-.161	-.184	-.257	-.287	-.346	-.490								147
B-33	-.267	-.250	-.253	-.261	-.261	-.275	-.316	-.362	-.579					219
B-34	-.221	-.274	-.302	-.368	-.417	-.498								147
B-35	-.274	-.235	-.236	-.279	-.335	-.398	-.381	-.538						177
B-36	-.314	-.303	-.286	-.325	-.338	-.362	-.389	-.426						
B-37	-.244	-.221	-.204	-.209	-.199	-.206	-.213	-.334						
B-38	-.286	-.204	-.193	-.193	-.214	-.217	-.222	-.241						
B-39	-.212	-.172	-.164	-.169	-.179	-.194	-.204	-.219						
B-40	-.222	-.190	-.169	-.199	-.227	-.218	-.222	-.245						

TABLE III-3 (continued)

Discharge Potentials of Air Electrodes (mV vs. Hg-HgO)
 $j = 25 \text{ mA/cm}^2$

Electrode	3	27	51	75	99	123	147	171	195	219	246	273	300	Off Test Cycle
B-41	-.213	-.191	-.148	-.171	-.186	-.174	-.186	-.205						
B-42	-.226	-.190	-.169	-.199	-.227	-.218	-.222	-.245						
B-43	-.269	-.238	-.242	-.229	-.241	-.235	-.229	-.252						
B-44	-.265	-.220	-.210	-.206	-.226	-.223	-.233							165
B-45	-.238	-.176	-.164	-.152	-.169	-.162	-.172							165
B-46	-.275	-.230	-.235	-.256	-.276									
B-47	-.260	-.236	-.268	-.248	-.283									
B-48	-.243	-.194	-.188	-.181	-.191									

TABLE III-4

Charge Potentials of Air Electrodes (mV vs. Hg-HgO)
 $j = 12.5 \text{ mA/cm}^2$

Electrode	3	27	51	75	99	123	147	171	195	219	246	273	300	Off Test Cycle
B-1	+ .687	+ .709	+ .730	+ .745	+ .880									99
B-2	+ .546	+ .547	+ .540	+ .569	+ .528	+ .506								141
B-3	+ .572	+ .555	+ .554	+ .574	+ .537	+ .486								141
B-4	+ .551	+ .533	+ .513	+ .620	+ .551	+ .501								141
B-5	+ .561	+ .541	+ .573	+ .609										78
B-6	Structurally Unsound													
B-7	Never Charged													1
B-8	+ .569	+ .517												45
B-9	+1.007	+ .800	+ .745	+ .705	+ .757	+ .748	+ .661	+ .658	+ .648	+ .640	+ .615			269
B-10	+ .574	+ .531	+ .553	+ .557	+ .554	+ .555	+ .550	+ .550	+ .540	+ .567	+ .621			249
B-11	+ .614	+ .619	+ .614	+ .612	+ .578	+ .571	+ .577	+ .593	+ .660					193
B-12	Structurally Unsound													
B-13	Never Charged													
B-14	Never Charged													
B-15	Never Charged													
B-16	+ .674	+ .605	+ .600	+ .576	+ .582	+ .559	+ .634	+ .651	+ .560	+ .634	+ .653	+ .614	+ .559	343
B-17	Never Charged													
B-18	+ .609	+ .610	+ .599	+ .574	+ .581	+ .563	+ .585	+ .561	+ .607	+ .569	+ .585	+ .558	+ .579	343
B-19	+ .604	+ .606	+ .536	+ .559	+ .586	+ .510	+ .579							156
B-20	+ .596	+ .591	+ .560	+ .552	+ .555	+ .547	+ .547	+ .555	+ .544					213

TABLE III-4 (continued)

Charge Potentials of Air Electrodes (mV vs. Hg-HgO)
 $j = 12.5 \text{ mA/cm}^2$

Electrode	<u>3</u>	<u>27</u>	<u>51</u>	<u>75</u>	<u>99</u>	<u>123</u>	<u>147</u>	<u>171</u>	<u>195</u>	<u>219</u>	<u>246</u>	<u>273</u>	<u>300</u>	Off Test Cycle
B-21	+ .588	+ .588	+ .581	+ .570	+ .573	+ .568	+ .562	+ .571	+ .561					213
B-22	+ .573	+ .593	+ .603											57
B-23	+ .587	+ .592	+ .573	+ .559	+ .561	+ .552	+ .562	+ .562						183
B-24	+ .741	+ .746	+ .746	+ .812										93
B-25	+ .752	+ .764	+ .761	+ .757	+ .764	+ .739								138
B-26	+ .669													18
B-27	+ .651	+ .648	+ .630	+ .642										102
B-28	+ .609	+ .640	+ .633	+ .650										102
B-29	+ .578	+ .583	+ .569	+ .563	+ .571	+ .520	+ .559	+ .556						183
B-30	+ .651	+ .631	+ .637	+ .607	+ .792	+ .593	+ .588	+ .616						177
B-31	+ .653	+ .617	+ .634	+ .616	+ .624	+ .594	+ .561	+ .652	+ .614	+ .593	+ .605			261
B-32	+ .620	+ .585	+ .583	+ .544	+ .614	+ .576								147
B-33	+ .675	+ .705	+ .646	+ .618	+ .614	+ .596	+ .594	+ .616	+ .609					219
B-34	+ .657	+ .633	+ .625	+ .595	+ .604	+ .596								147
B-35	+ .687	+ .669	+ .631	+ .599	+ .605	+ .592	+ .570	+ .614						177
B-36	+ .860	+ .740	+ .715	+ .749	+ .716	+ .730	+ .706	+ .702						
B-37	+ .714	+ .656	+ .632	+ .640	+ .620	+ .656	+ .656	+ .736						
B-38	+ .875	+ .668	+ .669	+ .699	+ .683	+ .683	+ .687	+ .689						
B-39	+ .746	+ .624	+ .621	+ .675	+ .654	+ .636	+ .629	+ .631						
B-40	+ .713	+ .629	+ .605	+ .699	+ .645	+ .646	+ .632	+ .624						

TABLE III-4 (continued)

Charge Potentials of Air Electrodes (mV vs. Hg-HgO)
 $j = 12.5 \text{ mA/cm}^2$

Electrode	3	27	51	75	99	123	147	171	195	219	246	273	300	Off Test Cycle
B-41	+ .896	+ .639	+ .626	+ .658	+ .652	+ .660	+ .636	+ .661						
B-42	+ .712	+ .639	+ .649	+ .666	+ .664	+ .687	+ .658	+ .659						
B-43	+ .716	+ .651	+ .664	+ .688	+ .675	+ .691	+ .652	+ .655						
B-44	+ .715	+ .654	+ .656	+ .674	+ .668	+ .688	+ .658							165
B-45	+ .676	+ .627	+ .727	+ .648	+ .642	+ .643	+ .624							165
B-46	+ .823	+ .705	+ .690	+ .683	+ .663									
B-47	+ .866	+ .721	+ .742	+ .705	+ .689									
B-48	+ .699	+ .652	+ .640	+ .667	+ .662

Figure 3-1 Air Electrode Blistering
(1 unit = 1 cm)

Figure 3-2 Air Electrode Blistering
(1 unit = 1 cm)

presumably lead to O_2 pressure buildup that effectively forces KOH exiting through the electrode hydrophobic layer. The blistering phenomenon is peculiar only to those electrodes fabricated to include both of the aforementioned structural features. B-9, made with Ag-free active carbon, displayed poor performance in both charge and discharge operational modes, illustrating the necessity of a peroxide decomposition catalyst. B-10, employing a single Ni plated steel fiber current collector, gave substandard results due to current current collector corrosion.

In B-11 and all subsequent electrodes, pressing conditions (370°C, 18 T, 10 min) and active material layer loading (Table A-2D) were standardized. In addition, a bilayer Ni fiber current collecting grid system was employed. Each electrode was built to test one particular facet of study. Half cells B-11 through B-18, identical in composition and construction, were cycled under various conditions. The following data summarize the results.

TABLE III-5
Electrode Cycling Conditions Test

<u>Electrode #</u>	<u>Cycling Conditions</u>	<u>Mean Discharge Potential* vs Hg-HgO (@ 25 mA/cm²)</u>
B-13	Continuous discharge	-.128
B-15	Continuous discharge	-.158
B-14	4 hr discharge; 4 hr open circuit	-.200
B-17	4 hr discharge; 4 hr open circuit	-.223
B-11	4 hr charge; 4 hr discharge	-.256
B-16	4 hr charge; 4 hr discharge	-.242
B-18	4 hr charge; 4 hr discharge	-.235 ⁽¹⁾

* through 300 cycles

⁽¹⁾ through 270 cycles

Although individual variation is evident, Table III-5 data results attest to the benefit of the continuous discharge (O_2 reduction) mode with respect to electrode performance. It is felt that O_2 evolution during the charging process disrupts crucial electrolyte penetration pathways within the active carbon matrix.

B-19 through B-23 were fabricated to evaluate several experimental hydrophobic layer treatments formed by 3M Commercial Chemicals Division. A listing of electrodes and their respective levels of treatment is contained in Table III-6. As data from Tables III-3 and III-4 show, however, electrodes 19-21 (treated) show no significant improvement in lifetime characteristics over the untreated electrodes 22-23. Both charge and discharge potentials were similar in all electrodes.

TABLE III-6
Electrode Hydrophobic Layer Treatment Study

<u>Electrode #</u>	<u>Treatment</u>
B-19	.15 gm L-2573 ⁽¹⁾ -applied by coarse spray
B-20	.30 gm L-2573 -applied by fine spray
B-21	.18 gm L-2573 -applied by flooding
B-22	No treatment
B-23	No treatment

(1) courtesy 3M-Commercial Chemicals Div.

B-24 and B-25, as well as electrodes B-30 through B-35, were intended to provide insight into the O_2 reduction reaction site location with respect to the current collecting grid. This was done by testing electrodes fabricated to contain active layers from carbons of equal surface areas, but different electronic resistivities. It was thought that the dependence of discharge potential values upon electron conduction pathways within the electrode would manifest itself in the form of

higher potentials for the higher electronic resistivity carbon electrodes. Results from this series would appear to point to the reverse relationship, at least among the higher surface area carbons, as the following data illustrate. Also, as shown, the lower resistivity active material carbon electrodes appear to have a longer cycle life. Further investigation in this area is required.

TABLE III-7
Carbon Active Layer Resistivity Study

Electrode #	Specific Area (m ² /g)	Resistivity (relative)	Discharge Potential (mV vs Hg-HgO)	Charging Potential (mV vs Hg-HgO)	Cycle Life
B-32	290	High	-.288	.587	147
B-33	220	Low	-.314	.630	219

B-26, constructed entirely of a high surface area carbon (1000 m²/g) material, failed rapidly due to leakage; while B-36, utilizing a graphitic carbon active layer, displayed poor potentials in both modes. Electrode B-29, made with a porous, metal catalyst coated active layer current collector (Table A-2E), displayed good charging potentials at the expense of the discharge mode. Thus, highly conductive (relative to carbon) metal powders on the current collecting surface of this electrode effectively eliminate the three dimensional current collecting network vital to oxygen reduction.

Electrodes B-27 and B-28 were built incorporating wire probes on the active layer current collector in an attempt to experimentally map the potential distribution on an operating bifunctional air electrode. Results point to a large tab-electrode current collector grid ohmic potential drop and to an inverse current magnitude distribution with distance from this tab.

Electrodes B-37 through B-43 and B-46 through B-48 make up a study of the effects of the addition of various surface active agents to the electrolyte. Table III-8 gives the specifics. As shown, both the Triton X-100 and the FC-430 additives result in improved discharge performance with little cycle life deterioration.

TABLE III 8
Surface Active Agent Addition Study

Electrode #	Surface Active Agent Addition (0.1 g/cc KOH)	Mean Discharge Potential (-mV vs Hg/HgO) (1)	Cycle Life (2)
B-37	Hercules 5779-32 (1)	256.28	256
B-38	FC-95 (3) (7)	229.31	252
B-39	FC-171 (3)	204.05	252
B-40	FC-430 (3)	195.13	256
B-41	Triton X-100 (4)	189.25	316
B-42	Lauryl Trimethyl Amine Chloride (5)	225.30	252*
B-43	0	250.71	332+
B-46	FC-129 (3)	272.54	246
B-47	FC-170C (3)	293.21	256+
B-48	FC-98 (3)	231.89 (6)	159

(1) Through 240 cycles

(2) Until gross electrolyte leakage

(3) Courtesy Hercules, Inc.

(4) Courtesy Rohm & Haas Co.

(5) Courtesy Armour Industrial Co.

(6) Through cycle 159

(7) .001 g/cc KOH

(*) Internal short circuit

(+) Removed prior to leakage point

In summary, the aforementioned half-cell air electrode test program continues to clarify our understanding of the bifunctional air electrode, while simultaneously providing an avenue for the application of novel approaches to the problem of developing long life, reproducible, and electrochemically satisfactory structures.

IV Iron-Electrode Studies

4.1 Procedure

Iron electrodes were prepared by the pressing and sintering of a mixture of sponge iron powder and urea pore former between perforated nickel current collectors. For many studies, the electrodes had a major face area of 10 cm^2 (1.25 in. x 1.25 in.), however, larger cell-size electrodes were also fabricated and studied. Hydrogen sintering was accomplished in a moving belt furnace and employed various combinations of temperature and time at temperature to achieve the desired densities. Following sintering, determinations were made of weight loss, shrinkage and density change. Electrodes to be half-cell tested were provided with nickel current collector tabs.

Half-cell testing of the electrodes utilized a sheet nickel counter electrode and a Hg/HgO reference probe. Temperature stability at 44-46°C was normally provided by immersion of the acrylic half-cell cases in a stirred water bath. [Heated ovens were used to test the largest electrodes (400 cm^2 , or 8 in. x 8 in.).] Charge and discharge current densities, upper plateau cutoff voltage (0.750 volts vs. Hg/HgO) and cycle times were controlled automatically. A large excess of electrolyte was used in the half-cell testing to minimize the need for water makeup to the electrolyte (which, unless stated to be otherwise, was 25 weight percent KOH containing 15 gm/l LiOH).

More specific aspects of the experimental procedure are contained, where necessary, in the appropriate sub-sections on Experimental Results.

4.2 Experimental Results

4.2.1 Fe Powder Studies

Work was concentrated on an evaluation of powder prepared by 700°C hydrogen reduction of lot 19 Fe_2O_3 , a lot shown previously to possess superior properties. Iron powders were prepared by three processes, designated R, RS and RST. The process steps, developed in the previous report period, are as follows:

- (R) Reduce Fe_2O_3 at 700°C in flowing hydrogen. [The Fe_2O_3 bed depth is limited to about 1/2 inch while the time at temperature is 30 minutes.]
- (S) Break up the loosely agglomerated iron powder by forcing it through a screen of between 60 and 100 mesh. The iron particles are then screened and separated according to size with only the smallest particles (less than 38 μm in size) retained for further processing.
- (T) A measured weight of iron particles is mixed overnight in a rotating ceramic jar with about 10 parts by weight of deionized water and 2.4 weight percent (based on iron weight) of $\text{CdSO}_4 \cdot 8 \text{H}_2\text{O}$. After mixing, most of the water is removed by decanting. Further drying to a caked mass is accomplished by heating in air at between 65°C and 80°C. The dried cake is crushed to powder by forcing it through a 60 mesh screen.

B.E.T. nitrogen adsorption surface areas were determined and correlated with sinter shrinkage (Table IV-1).

In addition, lot 19 Fe_2O_3 was hydrogen reduced at 650°C (rather than at the established temperature of 700°C) to determine if greater iron powder activation and, hence, greater electrode capacity

TABLE IV-1

Surface Area and Sinter Shrinkage of
Lot 19 Fe Powders

<u>Preparation Process</u>	<u>Surface Area (m²/gm)</u>	<u>Sinter Shrinkage (%)</u>
R	0.98	13.7
RS	1.05	12.5
RST	7.31	22.2

could be realized. Reduction for 30 minutes produced a pyrophoric iron powder. Two electrodes were prepared from powder prepared by 60 minute reduction. However, their discharge capacities were inferior to those electrodes prepared from powder made by 700°C reduction.

Further attempts at increased powder activation consisted of lowering the temperature of air drying (one of the requirements of process T) below the normal temperature range of 65-80°C. A temperature of 30-40°C could not be maintained since the powder oxidation which accompanies drying was sufficient to raise the temperature to about 70°C. Drying at ambient temperature produced a very active powder (16.2 m²/gm). However electrodes produced from this powder possessed capacities which were inferior to those prepared from powder which had been dried at 65-80°C.

4.2.2 Optimization of Fe Electrode Density

Lot 19 iron powder prepared by the RST process was dry blended with 10 volume percent (8.2 weight percent) urea of a nominal particle size of 115 μ m (-100, +170 mesh). Small (10 cm²) electrodes were prepared by pressing at about 3000 psi and sintering at various combinations

of time and temperature. Each electrode contained a perforated nickel current collector on its two major faces and was nominally 0.095 inch thick.

Discharge capacity in 25 weight percent KOH plus LiOH was measured as a function of electrode density (Figure 4-1). It is readily apparent that optimum discharge capacity is associated with a density of about 22% TD_{Fe} . This result is in excellent agreement with that previously found for RST electrodes prepared from lot 4 Fe_2O_3 powder.⁽¹⁾ The maximum discharge capacity for a lot 19 RST electrode of 0.44 A·hr/gm was accomplished by sintering for 20 minutes at 800°C. In lithiated 35 weight percent KOH the maximum capacity was 0.53 A·hr/gm (Figure 4-2).

Since preliminary results (Section 4.2.4) had shown that a less active powder may be needed for the fabrication of cell-size electrodes to prevent the formation of shrinkage cracks, capacities were also determined as a function of density for lot 19 R and RS electrodes. Maximum capacity was rated at 22 to 24% TD_{Fe} and had the value, for both electrode types, of 0.36·A hr/gm for lithiated 25 weight percent KOH.

Formulation and proof of a sintering model for a porous iron electrode was finalized. The detailed results are contained in Appendix A.

4.2.3 Current Collector Improvement

In the previous work period it was determined that the discharge capacity of iron electrodes, when computed on a total weight basis (iron plus current collector), could be greatly enhanced by combined elimination of most of the external current collector and incorporation of a small amount of matted nickel fiber to the electrodes interior. In the present work period this study was extended to lot 19, 10 cm² electrodes. Figure 4-3 presents the results of some of this work, based on electrodes of very nearly optimum density (22% TD_{Fe}).

Curve 714331-A

Figure 4-1. Discharge Capacities of Lot 19 RST Electrodes as a Function of Density

Figure 4-2. Discharge Capacities of Lot 19 RST Electrodes as a Function of Density. [KOH Concentration Varied Between 30 and 35 Weight Percent.]

Figure 4-3. Discharge Capacity of Lot 19, Type RST Iron Electrodes Containing Various Current Collectors

For electrodes containing perforated nickel current collector on both major faces but without internal current collector (electrodes -8L and -9L) discharge capacities ranged between 0.32 and 0.35 A·hr/gm of total electrode for the first 15 cycles of operation for which the electrolyte concentration did not exceed 30 weight percent KOH. With the elimination of all but 0.15 gram of external current collector and incorporation of 0.10 gram of nickel fiber, either in matted form (electrode -6L) or shredded form (electrode -30L), discharge capacities in the range 0.39 to 0.44 A·hr/gm of total electrode were realized over the first 15 cycles.

When current collector elimination was carried even further (no internal collector, electrode -45L) a discharge capacity as great as 0.50 A·hr/gm of total electrode was achieved. From Figure 4-3, it is apparent that the discharge capacity of this particular electrode is very sensitive to the changes in electrolyte concentration which accompany continued cycling with only intermittent makeup of water to the electrolyte.

It is apparent from these results that iron electrodes of 10 cm² size operated very well with practically no current collector. However, it remained to be demonstrated that similar results could be obtained with larger, cell-size electrodes.

4.2.4 Characteristics of Process R, Lot 19 Iron Electrodes

This type of electrode was studied in detail since it has been used exclusively in the iron-air cells built to date. The results are presented and discussed in the following sub-sections.

Electrode Density

Discharge capacity was found to be very sensitive to the density of the electrode (Figure 4-4). Over the range of densities studied (19.5% to 26.5% TD_{Fe}), first cycle capacity decreased from about

Curve 715182-A

Figure 4-4. Discharge Capacity of Process R, Lot 19 Electrodes as a Function of Their Density. Test Conditions Included 100 mA/cm² Charge and 50 ma/cm² Discharge.

0.6 Ahr/gm to about 0.2 Ahr/gm. Later cycle capacities, typified by the data for the eighth cycle, were at a maximum for the intermediate range of density between about 21.5% and 23.5% TD_{Fe} . A discussion of the physical factors which determine the relationship between electrode density and capacity is presented in Appendix A. Further test results were obtained on electrodes from each of three densities, namely low, intermediate and high density represented in turn by percent theoretical density of iron values of 19.6, 23.1 and 26.5. These results are described in the following sub-section.

Half Cell Performance

The results of Figure 4-5 demonstrate that the discharge capacities of iron electrodes were favored by low charge current density. The employed discharge current density of 50 mA/cm^2 corresponds to the expected cruise condition of an electric vehicle powered by an iron-air battery. The preferred charge current density of 25 mA/cm^2 would correspond to an overnight charge of the battery.

For all three electrode densities, high capacity was also favored by relatively low discharge current density (Figure 4-6). The influence of discharge current density on capacity was extensive for electrodes of intermediate and high density while a more moderate influence was noted for the low density electrode. Superior capacity was observed at all levels of discharge current density when the high capacity (23.1% TD_{Fe}) electrode was charged at 25 mA/cm^2 rather than 100 mA/cm^2 .

Initial portions of several charge curves are shown in Figure 4-7. The high capacity electrode had a charge voltage of -1.06 volts (versus Hg/HgO). Its discharge voltage (about -0.91 volts measured against Hg/HgO) also compared favorably to that of electrodes of both higher and lower density (Figure 4-8).

Curve 715179-A

Figure 4-5. Discharge Capacity of Process R, Lot 19 Electrodes as a Function of Charge Current Density. Discharge was at 50 mA/cm².

Curve 715183-A

Figure 4-6. Discharge Capacity of Process R, Lot 19 Electrodes as a Function of Discharge Current Density

Curve 715181-A

Figure 4-7. Charge Potential of Process R, Lot 19 Electrodes
Using a Current Density of 25 mA/cm²

Figure 4-8. Discharge Potential for Process R, Lot 19 Electrodes at 50 mA/cm²

The results of polarizations determined on the three electrodes are given in Figure 4-9. The degree of polarization was greatest for the electrode of highest density.

Further tests were conducted only on electrodes of intermediate density (maximum capacity). For convenience in testing, charging was conducted at 100 mA/cm^2 instead of 25 mA/cm^2 , the rate associated with superior discharge capacity. Increased KOH concentration significantly enhanced discharge capacity, as evidenced by the data of Figure 4-10.

Obviously the physical structure (as determined by density) of an iron electrode influences many features of its performance. For an electrode of relatively low density (e.g., 19.6% TD_{Fe}), capacity was found to be much less affected by discharge current density than were electrodes of greater density (Figure 4-6). Both the charge potential and initial (zero time) discharge potential of the low density electrode were found to be appreciably different from the corresponding potentials of the optimally dense electrodes (Figures 4-7 and 4-8, respectively).

LiOH Addition

The effect on discharge capacity of LiOH addition to 25 percent KOH was studied in detail. The results are presented in Figure 4-11. First cycle discharge capacity was found to increase linearly with increasing LiOH addition. For later cycles (typified by cycles 6 and 23) capacity was significantly enhanced for LiOH contents of up to about 30 gm/l, while for greater LiOH contents the further increase in capacity was minimal at cycle 6 and negligible at cycle 23.

Following half-cell testing, a small portion of each electrode was chemically analyzed for lithium. The results of Figure 4-12 (in which Li content is relatively independent of LiOH addition) suggest that other factors besides incorporation of Li^+ into the iron lattice are also responsible for the beneficial effect of LiOH addition to discharge capacity.

Curve 714875-A

Figure 4-9. Discharge Polarization Characteristics of Process R, Lot 19 Electrodes

Figure 4-10. Discharge Capacity of Process R, Lot 19 Electrodes as a Function of KOH Concentration. Electrodes were Charged at 100 mA/cm² and Discharged at 50 mA/cm².

Figure 4-11. Improvement to the Capacity of Lot 19, Type R Electrodes due to LiOH Addition

Curve 714504-A

Figure 4-12. Iron Electrode Lithium Content

4.2.5 Cell-Size Electrodes

Both 100 cm² (4 in. by 4 in.) and 400 cm² (8 in. x 8 in.) electrodes were prepared from type R powder (800°C, 10 minute sintering). Their capacities were determined and compared to those achieved with test size 10 cm² (1 1/4 in. by 1 1/4 in.) electrodes (Figure 4-13). Included are electrodes made with and without nickel current collector since the results of Section 4.2.3 showed that their capacities were comparable when determined on an active weight basis. It is apparent from Figure 4-13 that electrodes of a size much greater than the half-cell test size (10 cm²) can be prepared and made to possess equally large capacities of about 0.34 A·hr/gm. Two 100 cm² electrodes delivered significantly lower capacities (0.25 to 0.28 A·hr/gm). However, both had low density (20.0 to 21.0% TD_{Fe}) compared to the optimum (about 24% TD Fe) for electrodes prepared from this type of powder. Consequently, their appreciable first cycle discharge capacities (0.7 A·hr/gm) were apparently great enough to permanently depress their further capacity performance by means of interparticle neck fractures.

Work was also devoted to the development of 100 cm² electrodes made from higher surface area powders. In the past, 100 cm² RST electrodes had exhibited excessive sinter shrinkage with resultant cracking and bowing when either nickel mesh current collector was placed on the major faces or was entirely eliminated from the electrodes. However with the nickel mesh current collector placed beneath the surface of the electrode, cracking was virtually eliminated and bowing was reduced. Discharge capacities for electrodes sintered 20 minutes at 800°C ranged from 0.29 to 0.38 A·hr/gm. This performance is not, however, up to the level found for 10 cm² electrodes of this type.

Additional 100 cm² electrodes were made from both RT powder and mixtures of type R and RST powders. Electrode densities were held at or very near 22% TD_{Fe}. The aim was to achieve capacity greater than

Figure 4-13. Discharge Capacity of Lot 19, Type R Iron Electrodes as a Function of Their Size

that found for type R powder and to be free from shape change and cracking which occur as the result of excessive sinter shrinkage. Type RT electrodes, when sintered at 800°C for 20 minutes and containing nickel mesh current collector beneath the surface, have produced capacities of 0.39 A·hr/gm and 0.46 A·hr/gm for respective KOH concentrations of 25 and 30-35 weight percent. Electrodes prepared from the mixed R and RST powders had capacities as great as 0.38 A·hr/gm.

RT powder has been judged to provide the best cell-size electrodes from the viewpoint of the optimum combination of capacity and structural integrity.

With the use of more active powders for preparing 100 cm² electrodes, new welding procedures had to be adopted to assure sound bonds between the electrodes and their current collector tabs. The most successful procedure utilized 700°C hydrogen reduction of the electrode followed by immediate welding, either in air or flowing nitrogen. Tab weld problems have also been essentially eliminated by substituting 0.005 in. thick tabs for 0.015 in. thick ones.

4.2.6 Iron Electrode Life Testing

The life testing of a series of 100 cm² iron electrodes was initiated to determine their capacity characteristics during extended periods of cycling. Among the variables to be evaluated are ambient atmosphere (CO₂-scrubbed air versus unscrubbed air) and wetting agent addition to the electrolyte.

4.3 Summary of Iron Electrode Studies

The B.E.T. surface area of lot 19 powder prepared by the RST method was found to be $7.31 \text{ m}^2/\text{gm}$. Efforts to further increase the area of this powder were successful but the electrodes prepared from the more active powder did not possess greater capacity.

Maximum capacity was realized for electrodes made from lot 19, RST powder when the sintered density was about $22\% \text{ TD}_{\text{Fe}}$. Some evidence has been obtained, however, which suggests that this powder may not be appropriate to the fabrication of larger, cell-size electrodes because of the undesirable sinter shrinkage associated with its extensive surface area. For this reason powders of lower surface area were also employed in the fabrication of cell-size electrodes. Initial results were encouraging. For example, high capacity, independent of electrode size up to 400 cm^2 , was realized for a starting powder of $0.98 \text{ m}^2/\text{gm}$ (lot 19, R powder). Other performance characteristics of electrodes made from this relatively low surface area powder were also determined. Promising results were also obtained from cell-size electrodes prepared from other lot 19 powders, including types RT and mixtures of types R and RST.

Other studies yielded the formulation of a working model of the iron electrode and improved the capacity of iron electrodes through development of alternate current collector designs.

V. CELL STUDIES

5.1 Introduction

During the work period, a concerted effort was made to optimize conditions, configurations and procedures for the testing of individual 100 cm^2 iron-air cells. It is expected that knowledge gained from the above can readily be applied to the more difficult testing of full scale (prototype) 400 cm^2 iron-air cells to be incorporated into modules for electric vehicle battery application. In addition to the above, a primary goal, particularly during the first half of this work period, was the demonstration of stable cyclic life in individual cells (with respect to established charge-discharge potentials*) while simultaneously striving to improve the latter. Later, as increased performance was achieved, efforts were directed toward the attainment of specific energy and power goals (140 w-hr/kg , 100 w/kg) - i.e., cell casing weights were reduced, auxiliary systems consolidated, etc. This section describes efforts in these areas as well as summarizing the performance of all 100 cm^2 iron-air cells built and testing during this period.

5.2 Cell Testing Procedures

All full cell systems were comprised of two 100 cm^2 , carbon-based air (oxygen redox) electrodes coupled to a 100 cm^2 sintered Fe electrode. The iron electrodes used in the cells were all of the process R, lot 19 type which are described in section 4.2.4 of this report. Air electrodes were fabricated according to the information in Tables A-3 and A-4 of the Appendix. The air electrodes were encased in $1/8''$ ABS plastic frames, which then comprised the two major faces of the

* Discharge -100 mV vs. Hg/HgO
Charge $+550\text{ mV}$ vs. Hg/HgO

external casing structure. The Fe electrode was held midway between the air electrodes (see Figures 5-1 and 5-2). Cells were discharged at a current density of 25 mA/cm^2 (anticipated electric vehicle cruise condition) with polarizations up to 125 mA/cm^2 , in a 25% KOH electrolyte solution (both with and without 15 g/l LiOH added). The LiOH addition has been shown to be beneficial in extending the capacity of the Fe electrodes.⁽¹⁾ Cells were recharged primarily at a 20 A-hr rate.

Auxiliary Equipment

The following test conditions were maintained throughout full cell testing:

1) CO_2 introduced into a cell had been previously found to lead to potassium carbonate formation with resultant KOH dilution and decreased iron electrode capacity. For this reason, a nitrogen-22% oxygen mixture was substituted for ambient air. This was done primarily to eliminate the need for troublesome CO_2 removal scrubbers and/or special cell test chambers previously used. The gas mixture was introduced into the cell by means of an acrylic chamber fitted over each air electrode and mounted to the ABS frame.

2) The gas mixture was heated to approximately 40°C (anticipated operating temperature in electric vehicle battery application) and moistened with water prior to its introduction into the cell. The water addition served to prevent excessive concentration of OH^- ions in solution, which as discussed shortly, has been demonstrated to have a detrimental effect on cell capacity.

3) Electrolyte was circulated slowly to maintain a constant volume in the cell case.

In other respects, the cell testing configuration closely followed that described in Reference (1).

Figure 5-1. Fe-Air Cell Casing

Figure 5-2. Fe-Air Cell Cross Section

5.3 Cell Testing Results

Two series of cells were tested. Those of the A series were not optimized for size nor weight. The B series cells utilized a space efficient casing which reduced both cell weight and electrolyte volume considerably. Detailed results are contained in Table V-1. A discussion of the results follows.

A Series

The initial cell of this series (A-1) was studied in considerable detail. To a large extent, the information obtained from this study served as a basis upon which subsequent cell design and test procedures rested. The following information was obtained.

1) It was determined that the addition of LiOH (15 gm/l) to KOH electrolyte resulted in increased cell voltage of ~ 0.1 V and cell capacity of $\sim 40\%$. However, the addition of LiOH may be of mixed benefit since lithium carbonate crystal deposition and growth in the air electrode (resulting in electrode expansion with concurrent leakage of electrolyte) may occur.

2) A beneficial effect of increased KOH concentration on cell capacity was established. Above 40% concentration levels, however, cell performance declined due to a concentration polarization effect on the O_2 reduction mechanism.

3) Prolonged interruption of electrolyte heating decreased cell discharge voltage by 50 to 130 mV (with voltages of both electrodes being adversely affected) and discharge capacity by about 25%. However, both cell voltage and capacity were readily regained once the cell temperature was re-established.

4) No adverse, long term effects were noted to occur as the result of repeated full discharges (to essentially zero cell voltage). Thus, at least with respect to the air electrode, the charging process (O_2 evolution) represents the major, long-term cause of structural disruption.

Cells A-2 through A-7 were near duplicates of cell A-1 and, again, were designed primarily for cycle life and as opposed to voltage performance. The majority of these cells were run for approximately 50 charge-discharge cycles before testing was terminated (usually due to leakage). In most, air electrode discharge potentials were from 20-80 mV higher than that shown for half cells of identical composition (Tables A-3, A-4 of Appendix). It is felt that inefficient current collection largely account for this differential. Also, delamination of successive carbon layers presented a larger problem in the 100 cm² structures than it did in 40 cm² half cells, since it is more difficult to produce a uniformly thick electrode in the larger size. Thus upon hot pressing, homogeneous teflon-teflon bonding is less likely, therefore, the electrode loses its structural integrity. The iron electrodes in these cells generally performed closer to their demonstrated half cell results. In several instances, however, problems were encountered with poor tab-current collector weld integrity which resulted in abnormally low discharge potentials for these cells (A-4, A-6, A-7, A-8).

Cell A-4 casing incorporated a smaller electrolyte volume and displayed somewhat improved cell capacity due to the beneficial effect on iron electrode capacity of increased OH⁻ ion concentration (electrolyte concentration effect). As in cell A-1, the addition of LiOH to electrolyte resulted in ~10% cell capacity increase.

Typical polarization curves for the above cells are shown in Figure 5-3. As shown, for the region of interest (current densities 25-125 mA/cm²), cell internal resistance is in the range of 20 mΩ.

Cells A-8 through A-11 were run using non-lithiated electrolyte, and, as Figure 5-4 shows, exhibited similar polarization characteristics to cells discharged in lithiated electrolyte. Capacities of the former cells were lower (Table V-1), however, After cell A-11, lithiated electrolyte was again used. Cell capacity improvement can be seen in Table V-1.

Curve 717677-A

Figure 5-3. Cell Polarization Profiles
(Cells A-1, A-2, A-4)

Curve 717676-A

Figure 5-4. Cell Polarization Profiles
(Cells A-8, A-13)

Table V-1 - Electrical Characteristics of 100 cm² Iron-Air Cells

Cell	Capacity (A.hr)				Electrode Voltage for Discharge of 5A (V)**		Cell Voltage for Discharge of 5A (V)**	Discharge Polarization** (change in mv for 5A to 20A)		Number Cycles to Termination	Reason For Termination
	25% KOH		25% KOH + 15g/l LiOH		Iron	Air	5A (V)**	Air	Fe		
	Typical	Maximum	Typical	Maximum							
A-1			(10.7)	(14.7)	--	--	0.8	170	50	67	Leak
A-2	(10.5)	(15.5)	***	(21.9)	0.91	0.13	0.78	120	100	50	Leak
A-3	--	--	(17.0)	(25.0)	0.90	0.15	0.75	170	90	12	Short circuit
A-4	(12.1)	(14.5)	***	(18.3)	0.86	0.16	0.70#	170#	50#	47	Unsatisfactory performance.
A-5	(8.2)	(10.4)	(9.0)	(13.0)	0.98	0.24	0.74#	210#	30#	46	Unsatisfactory performance.
A-6	(6.4)	(7.8)	(7.1)	(8.2)	0.95	0.24	0.71	270	70	41	Leak
A-7	(10.6)	(13.8)	--	--	0.87	0.12	0.75#	70#	160#	24	Check cell interior.
A-8	(10.6)	(12.0)	--	--	0.90	0.18	0.72#	170#	110#	57	Check cell interior.
A-9	(9.0)	(11.8)	--	--	0.91	0.35	0.58#	430#	90#	44	Check cell interior.
A-10	(9.2)	(10.6)	--	--	0.80	0.20	0.60#	200#	240#	54	Check cell interior.
A-11	(8.1)	(8.1)	--	--	0.87	0.26	0.61#	350#	100#	33	Leak (air-frame joint.

**in 25% KOH + g/l LiOH unless noted otherwise

***widespread in data

#25% KOH

Table V-1 (Continued) - Electrical Characteristics of 100 cm² Iron-Air Cells

Cell	Capacity (A.hr)				Electrode Voltage for Discharge of 5A (V)		Cell Voltage for Discharge of 5A (V)	Discharge Polarization change in mv for 5A to 20A)		Number Cycles to Termination	Reason For Termination
	25% KOH + 15 g/l Li		25% KOH		Iron	Air	Air	Fe			
	Typical	Maximum	Typical	Maximum							
A-12	12.99	17.90	--	--	.93	.13	.80	240	45	16	Delamination
A-13	13.66	25.0	--	--	.92	.15	.77	188	84	93	Delamination
A-14	14.50	25.5	--	--	.93	.25	.68	188	114	92	Delamination
A-15	11.80	26.1	--	--	.89	.23	.66	--	--	98	Delamination
A-16	11.77	19.5	--	--	.89	.21	.68	--	--	54	Poor performance.
B-1	8.29	14.0	--	--	.92	.24	.68	--	--	6	Delamination
B-2	13.41	29.8	--	--	.90	.18	.72	109	93	40	Leakage
B-3	11.92	21.5	--	--	.89	.28	.61	--	--	34	Electrode bulging
B-4	13.42	22.25	--	--	.87	.24	.63	147	146	22	Poor performance
B-5	NA	NA	--	--	.81	.25	.56	--	--	4	Poor performance
B-6	8.66	18.50	--	--	.92	.19	.73	--	--	43	Poor performance
B-7	12.32	23.00	--	--	.90	.14	.76	147	89	35	Poor performance
B-8	12.95	15.85	--	--	.90	.13	.77	--	--	23	Leakage
B-9	29.95	33.23	--	--	.89	.14	.75	77	112	2	Delamination
B-10	24.53	33.23	--	--	.90	.14	.76	104	91	2	Delamination

Cells A-7, A-8, A-9, and A-10 employed a center-mounted iron electrode current-collecting tab which was expected to lower cell ohmic resistance. However, due to poor tab-current collector welding, results were below expectations.

In the air electrodes of cells A-9 and A-11, a heterogeneous, size graded mixture of active material was used (A-1 through A-10 used a non-size graded mix). Air electrode discharge potentials for this combination were not up to expectations.

The air electrode of cell A-12, fabricated using a large particle size binder* (23 μ avg.) and high pressing forces, failed through delamination after approximately 12 cycles. Cells A-13 and A-14, each of which contained an air electrode which utilized another large particle size binder,* but lower pressing temperatures, lasted considerably longer (93 cycles). Cell A-14 also incorporated an additional steel fiber-Fe exmet charging current collector to promote even charging of the Fe electrode. The improved capacities of both of these cells can be seen from Table V-1.

Starting with cell A-15, a two layer air electrode construction, coupled with 36 Ton, 370°C (previously 330°C) pressing conditions, was instituted. The use of PFA (per fluoro alkoxy) and FEP (fluorinated ethylene propylene) powders as binders, which, because of their previously noted large particle size (20-25 μ) had resulted in inferior carbon layer binding, was discontinued in favor of a TFE dispersion (particle size .05-.5 μ). Care was taken in active material layer fabrication to ensure a uniform thickness. As shown in Table V-1, this cell exhibited enhanced lifetime characteristics at the expense of long-term performance. Cell A-16, constructed using an identical active material mixture, but employing a Ni Fiber-Ni Exmet current collecting grid, suffered from performance limitations and was removed from test for this reason prior to actual air electrode structural failure.

* See Tables A-3A, A-4A of Appendix.

The effects of various charging current-time sequences (constant ampere-hr charge) on mean subsequent discharge capacities were determined in the A series of cells. It was found that a moderate charging current resulted in a 15-40% extension of discharge capacities as the following composite data from cell A-9 illustrates:

<u>Charging Sequence</u>			<u>Mean Subsequent Discharge Capacity</u>
<u>Amps</u>	<u>Hours</u>	<u>Amp-Hr</u>	<u>(Amp-Hr)</u>
5.0	4.0	20.0	13.94
10.0	2.0	20.0	10.58
2.5	8.0	20.0	11.41

Polarization curves from some of the latter A cells are shown in Figure 5-3.

B Series

Starting with cell B-1, a more efficient cell frame, one in which the air electrodes form an integral part of the structure, was employed (Figure 5-1). With this design, cell frame weight was cut by 67% and electrolyte volume by 84%. Cell B-1 performed poorly due to poor bonding within the air electrode between the expanded metal and Ni fiber current collectors. In addition, the use of a high surface area active material mixture resulted in insufficient bonding between successive carbon layers which led to premature cell leakage. It is felt that that teflon binder particles may be absorbed into the carbon, in these cases, resulting in a loss of TFE particle-particle contact.

Cells B-2 and B-3, using in the air electrode both a lower surface area active material and on all expanded metal current collecting grid system,* showed slight improvement with respect to cycle life, but exhibited low cell voltages.

* Expanded metal current collectors provide better electronic conduction, while effectively increasing reaction site-current collector path length.

With cell B-4, we initiated the use of a new type of current collector prepared by sintering various oxygen overpotential reduction metal powders to an expanded metal grid. This electrolytically porous structure enables more precise control of O_2 evolution sites within the air electrode via creation of low overpotential areas on the inner current collector. In addition, it is expected that cell internal impedance will be lowered since the requisite electrochemical reactions take place in the immediate vicinity of the current collecting grid. In B-4, Ni powder was used to coat the active layer current collector, while in cell B-5, a Ni-Fe-WC-Co mixture was employed. In the latter cell, the active carbon layer immediately adjacent to the coated current-collecting grid was hand-pasted to enhance electrolyte penetration. Considerable improvement in initial discharge potentials was realized in this electrode as shown in Table V-2.

Table V-2: Initial Discharge Potentials
of Cell B-5 Air Electrode

Current Density (mA/cm ²)	Discharge Potential (mV vs Hg/HgO)
12.5	110
25.0	134
50.0	180
75.0	190
125.0	222

The results of Table V-2 suggest that previously established cell "break-in" procedures are unnecessary. Corrosion of the Fe powder sintered onto the porous current collector resulted in poorer than anticipated performance in cells B-4 and B-5. This corrosion, with its attendant iron particle displacement apparently reduced the effective electrolytically porous area of the air electrode.

Cell B-6 was fabricated with an air electrode active carbon layer composed of a mixture of hydrophillic and hydrophobic materials to establish a more controlled electrolyte penetration pathway through

the electrode structure. Current collectors (one coated with a porous metal powder mixture) were welded together along both upper and lower surfaces to ensure a better electron conduction pathway to/from the cell external circuit. However, cell performance was below anticipated levels due both to the aforementioned metal catalyst corrosion and to high resistivity of the coated current collector caused by the use of heavier gauge grid material.

Cells B-7 and B-8, which utilized a lighter current collector base material (also coated) displayed a discharge capacity improvement over cell B-6. Cell B-8 which contained an air electrode having a thin active layer, failed via electrolyte leakage.

The air electrodes of cells B-9 and B-10 were designed to test a high surface area ($1000 \text{ m}^2/\text{gm}$) active carbon layer that had been found to be successful in half cell studies. Impressive discharge capacities and low air electrode polarization voltage changes characterized these cells. However, both failed rapidly due to delamination. Thus we learned that processing conditions (pressures, loading) beneficial to half-cell (40 cm^2) air electrodes cannot, in general, be extrapolated to full size (100 cm^2) air electrodes purely by way of an area proportionately.

5.4 Summary of Cell Studies

Due to a number of factors, which have already been discussed, progress in demonstrating extended stable cyclic life (over 100 cycles) coupled with meeting projected cell performance characteristics has been more difficult than originally anticipated. As of the end of this reporting period, testing of prototype-size 400 cm² Fe-air cells for electric vehicle module applications has not been initiated. It is felt, however, that this slower development pace has been more than offset by a gain in fundamental understanding of the Fe-air system (particularly the air electrode). Promising developments such as new cell casing configurations and the air electrode porous current collecting structure were tested. Through further experimental air electrode processing studies, fundamental carbon studies⁽⁷⁾ and cyclic voltammetry studies of air electrode components, further gains will be realized.

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11. W. A. Bryant, "The Structure and Performance of Sintered Iron Electrodes," Electrochimica Acta, 24 (1979)
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VIII. APPENDIX

8.1 Iron Electrode Working Model

Experimental Results

Because of its relative ease of measurement, density has often been adopted as a measure of the physical structure of a pressed and sintered body. To test the usefulness of this measure to correlate the capacity data of the present work, the capacities of electrodes prepared by numerous combinations of sintering time and temperature were determined as a function of electrode density. These results, for lot 4 RST and lot 19 RST* electrodes respectively, are given in Figures A-1 and A-2. In both cases the data are seen to be fairly well correlated.

According to the porosity model proposed by Selanger⁽²⁾, a maximum achievable discharge capacity can be calculated for any value of initial electrode porosity (or density). This model, when applied to an iron electrode, yielded the plot of Figure A-3. [According to this curve the largest maximum capacity (which is potentially realizable only for porosities of about 80 percent and greater) is less than the theoretical capacity of iron (0.962 A·hr/gm) since the electrodes of the model were assumed to contain 6 volume percent current collector, the amount incorporated into the electrodes of the present experimental study.] The molar volume of $\text{Fe}(\text{OH})_2$ upper plateau discharge product ($26.4 \text{ cm}^3/\text{mole}$) is significantly greater than the molar volume of iron ($7.1 \text{ cm}^3/\text{mole}$). Thus, according to the model and for the condition of insufficient initial porosity (density exceeding approximately 20 to 25% TD_{Fe}), the pores would be filled with $\text{Fe}(\text{OH})_2$ before all the iron could be utilized. On the other hand, excessive initial porosity

*Corresponding iron powder surface areas were $4.4 \text{ m}^2/\text{gm}$ and $7.3 \text{ m}^2/\text{gm}$.

Curve 714328-A

Figure A-1. Relation Between Discharge Capacity and Density for Electrodes Prepared from Iron Powder Lot 4 RST

Curve 714331-A

Figure A-2. Relation Between Discharge Capacity and Density for Electrodes Prepared from Iron Powder Lot 19 RST

Curve 714329-A

Figure A-3. Discharge Capacity of an Iron Electrode According to the Porosity Model of Selanger

(density less than about 20% TD_{Fe}) would permit all the iron to react to form $Fe(OH)_2$ but a portion of the void volume would remain following completion of the upper plateau discharge since only a relatively small amount of iron would comprise the electrode. As shown by Figure A-3, capacity measured on the basis of active material weight is not expected to fall below its peak values. However, for this range of densities, capacities determined on a volume of electrode basis would be much lower than the peak value. Only for the ideal amount of initial porosity (density of 20 to 25% TD_{Fe}) would the pore volume be just sufficient to contain the $Fe(OH)_2$ formed by the discharge reaction of all iron in the electrode.

Over the range of density common to both the porosity model and the present experimental study, the discharge capacities predicted by the model are considerably greater. This is to be expected. The model is based on maximum achievable capacity while the experimental work utilized test conditions which were known to be compatible with a particular battery system (iron-air) and were not optimized, therefore, for achieving maximum iron electrode capacity. This is not to say that capacities as great as those predicted by the porosity model are unobtainable. For example with the use of non-battery conditions (usually 35 percent KOH electrolyte containing 50 gram/liter LiOH, 50°C cell temperature and 12 mA/cm² discharge current density), discharge capacities of between 0.72 and 0.76 A·hr/gram have been obtained for 25 percent dense electrodes.

Important differences do exist between the shapes of the experimentally derived curves (Figures A-1 and A-2) and the curve derived from the porosity model (Figure A-3). These differences indicate that factors other than the relative proportions of iron and porosity must also determine obtainable capacity. These factors are discussed in the following section.

Discussion

The limited amount of sintering densification experienced by the experimental electrodes indicated that only first stage sintering had occurred. This initial stage in a sintering process is marked both by considerable neck growth between adjoining particles and by significant decrease in total surface area. Discharge capacity is favored by interparticle neck growth since electrical resistance is thereby lowered. On the other hand, decreased surface area lowers discharge capacity since it is only at the surface that the discharge reaction between iron and hydroxyl ions can proceed.

Based on this background knowledge, it was contended that the regions of positive slope in the experimentally derived curves of Figures A-4 and A-5 were associated with capacity controlled by the extent of neck growth, while the regions of negative slope were indicative of capacity being determined by the amount of surface area remaining after sintering. These two hypotheses were tested in the following manner.

Kuczynski⁽³⁾ has shown that the half-thickness of a neck formed during sintering (X) is related to time of sintering (t) by the relation

$$X = K_1 t^{1/n} \quad (1)$$

For surface diffusion, the predominant mechanism in stage one sintering, n has the value of 7.^(3,4) Therefore, if indeed one determinant of discharge capacity is the degree of neck growth, the positive sloped component of each curve of Figures A-4 and A-5 should have a slope of $1/7$ when replotted on a log-log basis. Figure A-6 shows such a plot. The lines drawn through each set of data have slopes of $1/7$ and are seen to represent the data points fairly well, thus supporting the hypothesis.

Curve 714327-A

Figure A-4. The Effect of Sintering Time and Temperature on the Capacity of Electrodes Prepared from Iron Powder Lot 4 RST.
[Open Symbol - Cycle No. 1; Solid Symbol - Cycle No. 8]

Figure A-5. The Effect of Sintering Time and Temperature on the Capacity of Electrodes Prepared from Iron Powder Lot 19 RST.
 [Open Symbol - Cycle No. 1; Solid Symbol - Cycle No. 8]

Curve 714332-A

Figure A-6. Plots to Determine Value of Exponent in Equation (1).
[Open Symbol - Powder 4 RST Electrodes;
Solid Symbol - Powder 19 RST Electrodes]

According to German and Munir^(5,6) the change (decrease) in surface area (ΔS) which accompanies surface diffusion in the initial stage of sintering is given by the relation

$$\frac{\Delta S}{S_0} = K_2 t^{1/\gamma} \quad (2)$$

In this relation S_0 is the initial surface area ($t = 0$) and γ has a value of 3.5. If, according to the second hypothesis, the second determinant of discharge capacity is indeed the amount of remaining surface area, the negative sloped portion of each curve of Figures A-4 and A-5 should have a slope of minus $1/3.5$ when replotted on a log-log basis. Examination of Figure A-7 demonstrates that the data are well represented by lines having this slope and thus the hypothesis is supported.

A mechanistic description of the capacitance behavior of an iron electrode is now possible. Consider, for example, the capacity of lot 4 RST electrodes sintered at 700°C (right-hand portion of Figure A-4). For the first discharge, the number of interparticle contacts would be expected to be very large based on the small iron particle size employed. Therefore, only for very brief sintering times (20 minutes or less) is first cycle capacity controlled in lot 4 RST electrodes by internal resistance. For greater times of sintering, the amount of retained surface area controls capacity. Evidently this amount was considerable since the discharge capacities are a significant fraction of the theoretical capacity of iron. As first cycle discharge proceeds, however, many interparticle necks would be expected to be broken due to the $\text{Fe}(\text{OH})_2$ being formed having a greater molar volume than that possessed by iron. Only a fraction of the original necks would remain following first cycle discharge and their thickness would determine, for electrodes sustained for short times, the discharge capacity for succeeding cycles. For electrodes given longer sintering times, succeeding cycle discharge

capacity would be limited by the amount of active area reclaimed by charging of the electrode. This amount will generally be somewhat less than the active area available to first cycle discharge, presumably the result of physical factors such as blockage of some of the electrolyte feed channels and local regions of overly thick Fe(OH)_2 .

Curve 714333-A

Figure A-7. Plots to Determine Value of Exponent in Equation (2).
[Open Symbol - Powder 4 RST Electrodes;
Solid symbol - Powder 19 RST Electrodes]

8.2 Concepts on the Bifunctional Air Electrode

O₂ Reduction Mode

Previous testing has demonstrated that discharge performance (O₂ reduction mode) is enhanced via construction of electrodes incorporating a large wettable surface area within the electrode structure. Although conceptually simple, the implementation of the above has proven difficult. Various approaches, including the size grading of active material carbons and the usage of differentially dried carbon components have been attempted. At present, the mixing of size graded hydrophillic and hydrophobic materials within the active material is employed, as it has shown itself to be the most promising both in terms of the performance characteristics it gives the electrode, and of relative processing simplicity.

O₂ Evolution Mode

In general, more success has been achieved in the lowering of the O₂ overpotential than has in the O₂ reduction mode. It has been shown that a charging site distribution on the carbon or in the interface between carbon and oxygen overvoltage reduction catalysts in the vicinity of the current collecting grid results in a better performance - lifetime stability combination than a charging site distribution on the grid itself. Creation of micro-reservoirs of electrolyte solution at the charging sites is deemed essential for ensuring that the requisite electrochemical reactions occur. The degree to which the O₂ overpotential is lowered is dependent on the number of such sites in existence.

Failure Modes

The principal failure modes of bifunctional air electrodes have been found to be delamination of electrode layers and/or excessive electrolyte flooding of the electrode structure. Although both appear to result from structural damage during the O₂ evolution process, such electrochemical phenomena as carbon corrosion (oxidation) or breakdown

of catalytic materials (WC, NIS, FeWO_4) may also be contributing factors. One view proposes that, during the charging mode, carbon in the hydrophobic layer is constantly exposed to water saturated O_2 existing through this layer, and, as a result, becomes susceptible to water condensation. As water vapor condenses on this layer, moisture-laden pathways are established by which electrolyte can penetrate entirely through the structure. As electrolyte floods the region between active material layers, pressure builds, eventually exceeding the inter-layer bonding limit, resulting in electrode delamination.

8.3. Construction of Air Electrodes - Electrode Testing

Notes for Accompanying Tables

Carbon Powder Preparation:

Carbon Silverizing - silver precipitated on carbon (in presence of NH_2NH_2) via AgNO_3 addition to a wet carbon slurry (ratio as in subsequent tables)

Active Material Preparation - components (in active material tables) mixed in a wet slurry, air dried

Hydrophobic Layer Preparation - as above, but oven baked 300°C , 30-60 min

Processing Terminology:

Wet Pasting - refers to hand or roll pasting of carbon, in wet slurry form, into current collecting grid. Usually applies to active carbon layer only.

Preform - mechanical pressing of dry carbon powder in a die structure (as per Figure A-9)

Dry Powder - mechanical pressing of dry carbon powder directly into current collecting grid in a manner similar to the preform process

Construction Terminology:

Concept of electrode active and hydrophobic layers as per Figure A-8.

Additional Notes:

Expanded metal current collector mesh designations

ex. 5Ni7-2/0 trademark Exmet Corp.

5Ni7-189 trademark Delker Corp.

AIR ELECTRODE CROSS SECTION

Figure A-8. Air Electrode Cross Section

1. Load Powder

2. Press Powder

3. Release Die

4. Remove Preform

Figure A-9. Mechanical Preform Process

TABLE A-1A

Air Electrode Active Materials

	H-1	H-2	H-3	H-4	J-1	J-2	J-3	J-4	J-5	J-6	K-1	K-2	K-3
Shawinigan Black*	10	10	10 ¹	3	4.5	5.5	6.5	7.5	8	--	30	30	30 ¹
Ketjenblack*	--	--	--	--	4.5	3.5	2.5	1.5	1	10	--	--	--
WC-12 w/o Co	--	--	--	1	2	2	2	2	2	3	--	4.5	--
FeWO ₄	--	--	--	--	2	2	2	2	2	3	--	4.5	--
NiS	--	--	--	1	2	2	2	2	2	3	--	4.5	--
Teflon 30B	3	3	5	1	3.2	3.2	3.2	3.2	3.2	3	9	12	9
Teflon FEP	4	4	6	1	2.6	3.6	3.6	3.6	3.6	3.5	--	--	--
Fe-powder	23	13	--	--	--	--	--	--	--	--	--	--	--
H ₂ O (ml)	60	60	60	23	45	45	45	45	45	60	160	160	150
Triton X-100 (drop)	--	--	--	2	3	3	3	3	3	4	--	6	--

NOTE: All numbers are in grams, except for water.

*Silverized carbon (contains 0.0248 g of Ag per gram of silverized carbon),

‡ - carbon not silverized

TABLE A-1B Air Electrode Active Materials

	<u>79-01</u>	<u>-02</u>	<u>-03</u>	<u>-04</u>	<u>-05</u>	<u>-06</u>	<u>-07</u>	<u>-08</u>	<u>-09</u>	<u>-10</u>	<u>-11</u>	<u>-12</u>
Shawinigan Black*	--	30	10 [‡]	--	5	7.5	10	10 [‡]	5	7.5	5	7.5
Ketjenblack*	30	--	--	10	5	2.5	--	--	5	2.5	5	2.5
WC-12 w/o Co	9	9	--	3	3	3	--	3	3	3	3	3
FeWO ₄	6	5	--	2	2	2	2	2	2	2	2	2
NiS	6	5	--	2	2	2	2	2	2	2	2	2
Teflon PFA	--	--	--	4	4	4	4	4	3.6	3.6	3.3	--
Teflon FEP	10	10	3	--	--	--	--	--	--	--	--	--
Teflon 30B	10	10	3	6	6	6	6	6	4.5	4.5	3.5	3
H ₂ O (ml)	200	250	85	75	75	75	75	75	75	75	75	70

NOTE: All numbers are in grams, except for water.

*Silverized carbon (contains 0.0248 g of silver per gram of silverized carbon).

‡ - carbon not silverized

TABLE A-1C Air Electrode Active Materials

	<u>79-13</u>	<u>-14</u>	<u>-15</u>	<u>-16</u>	<u>-17</u>	<u>-18</u>	<u>-19</u>	<u>-20</u>	<u>-21</u>
Shawinigan Black*	10	10	10	20‡	20‡	5	5	7	7
Ketjenblack*	--	--	--	--	--	5	5	3	3
WC-12 w/o Co	3	3	3	--	--	3	3	3	3
Fe WO ₄	2	2	2	--	--	2	2	2	2
NiS	2	2	2	--	--	2	2	2	2
Teflon 30B	3	4	5	6	8	5	3	5	3
H ₂ O (ml)	70	70	70	120	120	60	60	60	60

NOTE: All numbers are in grams, except for water.

*Silverized carbon (contains 0.0248 g of silver per gram of silverized carbon).

‡ - carbon not silverized

TABLE A-1D

Air Electrode Active Materials

	79-22	-23	-24	-25	-26	-27	-28	-29	-30	-31	-32	-33
Shawinigan Black*	15	10	20 ¹	7.5	7.5	10	7.5	7.5	5	30	20	20
Ketzenblack*	10	15	--	--	--	--	--	--	--	--	--	--
WC-12 w/o Co	--	9	--	--	--	--	--	--	--	--	--	--
Teflon 30B	30	30	20	12.5	12.5	50	6.25	6.25	5	80	8	4
Teflon 120 FEP	--	--	--	--	--	--	--	--	--	--	6	3
Ni-powder	--	--	--	--	--	--	6.0	--	--	--	--	--
H ₂ O (ml)	120	100	100	50	50	30	50	50	200	230	170	170

NOTE: All numbers are in grams, except for water.

*Silverized carbon (contains 0.0248 g of Ag per gram of silverized carbon).

¹ - carbon not silverized

TABLE A-1E.

Air Electrode Active Materials

	79-34	-35	-36	-37	-38	-39	-40	-41
Shawinigan Black	10	10	--	10	5	5	10	20
Ketjenblack	--	--	1	--	5	5	--	--
WC-12 w/o Co	--	--	--	--	5	5	5	6
Fe WO ₄	--	--	--	--	--	3	--	4
NiS	--	--	--	--	--	3	--	4
Teflon 30B	4	4	1	4	7.5	7.5	5	20
Teflon 120 FEP	3	3	1	3	--	--	--	--
Ni powder	3	--	--	4	--	--	--	--
H ₂ O (ml)	85	85	5	55	65	65	60	130

NOTE: All numbers are in grams, except for water.

*Silverized carbon (contains 0.0248 g of Ag per gram of silverized carbon).

‡ - carbon not silverized

Table A-1F

B Air Electrode Active Materials

	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>	<u>E</u>	<u>F</u>	<u>G</u>	<u>H</u>	<u>I</u>	<u>J</u>	<u>K</u>	<u>L</u>	<u>M</u>	<u>N</u>
Shawinigan Black (Ag) ⁽¹⁾	30	30 ^N	30	30	30 ^N	10	10	10	-	25	-	-	-	-
Ketjenblack (Ag) ⁽²⁾	-	-	-	-	-	-	-	-	10	25	-	-	10 ^N	20
WC - 12% CO	4.5	-	-	4.5	-	-	4.5	-	3	17	-	-	-	-
NiS	4.5	-	-	4.5	-	-	1.5	-	2	-	-	-	-	-
Fe WO ₄	4.5	-	-	4.5	-	-	1.5	-	2	-	-	-	-	-
H ₂ O	175	200	150- 175	150- 175	150- 175	50	50	50	75	200	70	70	70	140
Teflon 30B	15	15	9	12	9	-	-	-	6	20	5	5	10	10
S.D.P.	-	-	-	-	-	20	20	20	-	-	-	-	-	-
Triton X-100	-	-	-	.18	-	.18	.9	.18	-	-	-	-	-	-
Mogol L	-	-	-	-	-	-	-	-	-	-	10	-	-	-
Vulcan XC-72	-	-	-	-	-	-	-	-	-	-	-	10	-	-
F.E.P.	-	-	-	-	-	-	10	-	-	-	-	-	-	-
P.F.A.	-	-	-	-	-	-	-	-	4	-	-	-	-	-

(1) 200g Shawinigan
12g AgNO₃

(2) 300g Ketjenblack
11.4g Ag

S.D.P. - Super Dylan Polyethylene

F.E.P. - Fluorinated Ethylene Propylene

P.F.A. - Perfluoroalkoxy

N - No Ag

TABLE A-1G

B Air Electrode Active Materials

	O	P	Q	R	S	T	U	V
Shawinigan (Ag) ⁽¹⁾	-	-	-	-	-	10 ^N	30	-
Regal 400R	10	-	-	-	-	-	-	-
Regal 330R	-	10	-	-	-	-	-	-
CSX-136A	-	-	10	-	-	-	-	-
Monarch 880	-	-	-	10	-	-	-	-
Regal 660R	-	-	-	-	10	-	-	-
Superior Graphite	-	-	-	-	-	2	-	-
Teflon 30B	5	5	5	5	5	6	15	5
H ₂ O	70	70	70	70	70	70	200	70
Narco P-100	-	-	-	-	-	-	-	10

(1) 200g Shawinigan
12g AgNO₃

N - No Ag

TABLE A-2A
Air Electrode (40 cm²) Construction

Electrode Identi- fication	Current Collector			Active Layer			Hydrophobic Layer			Fabrication Process	Final Process Conditions			Thickness (mils)
	Type	No.	Wt. (g)	No.	Type	Wt. (g)	No.	Type	Wt. (g)		Temp. (°C)	Load (Ton)	Time (min)	
LU-057	Steel Wool	4	4.6	4	H-1, H-2	7	1	H-3	2	Preform	300	6++	30	57
LU-058	Steel Wool	3	3.8	3	H-1, H-2	6	1	H-3	2	Preform	300	6++	30	54
LU-059	Steel Wool	3	3.0	3	G-1, G-2, Fe-sponge	5	1	G-3	0.8	Preform & Paste	300	3++	20	35
LU-060	Ni Fiber*	3	5.3	3	G-1, G-2, Fe-sponge	2.9	1	G-3	0.6	Paste	300	3++	30	37
LU-061	Ni Fiber*	3	4.3	3	G-1, G-2, Fe-sponge	3.6	1	G-3	0.4	Paste	300	5++	30	36
LU-062	Ni Fiber*	3	4.2	2	G-1(plus Triton X-100)	2.2	1	H-3	0.6	Paste	350	12++	20	33
LU-063	Ni Fiber**	3	4.5	2	G-2, H-4, Fe-sponge	4.9	1	H-3	0.6	Powder & Paste	350	12++	20	47
LU-064	Ni Fiber	3	4.2	2	G-1, G-2, H-4	3.6	1	H-3	0.6	Powder & Paste	350	12++	20	45
LU-065	Ni Fiber	4	5.6		charge-discharge sites were prepared separately (see text)						350	12++	20	20, 25
LU-066	Ni Fiber	5	4.7	4	J-1, J-2, J-3, J-4	6.8	1	G-2	1.2	Powder	320	5	10	59
LU-067	Steel Wool	5	4.8	4	J-1, J-2, J-3, J-4	6.8	1	G-2	1.2	Powder	320	5	10	58
LU-068	Ni Fiber	5	4.1	4	J-1, J-2, J-3, J-4	7.4	1	G-2	0.2	Powder	300	5	10	55
LU-069	Ni Fiber	4	3.4	3	J-1, J-6	6.2	1	H-3	0.8	Powder	300	5	10	47
LU-070	Ni Fiber+	5	5.0	4	J-1, J-2, J-6	6.1	1	H-3	0.5	Powder	300	5	10	61
LU-071	Ni Fiber+	5	5.0	4	J-1, J-2, J-6	6.1	1	H-3	0.5	Powder	300	5	10	57
LU-072	Ni Fiber	4	5.0	3	J-1, 79-15	6.0	1	H-3	1.9	Powder	300	5	10	47
LU-073	Ni Fiber	6	5.7	5	J-5, 79-01, Fe-sponge	7.3	1	A-1	1.7	Powder	300	5	10	59
LU-074	Ni Fiber+	4	5.0	3	79-23(less Triton X-100)	4.5	1	A-1	1.7	Powder	300	5	10	39
LU-075	Ni Fiber	4	5.7	3	K-1, K-2	5	1	K-3	1.4	Powder	300	5	10	48
LU-076	Ni Fiber	4	6.0	3	K-1, K-2	5	1	K-3	1.4	Powder	300	5	10	49

* Current collectors were coated with Teflon FEP

** Ni fiber coated with H-4 material

+ Active layer was perforated before final process

++ Cold press load, baked at final process without load

TABLE A-2B

Air Electrode (40 cm²) Construction

Electrode Identi- fication	Current Collector			Active Layer			Hydrophobic Layer			Fabrication Process	Final Process Conditions			Thickness (mils)
	Type	No.	Wt. (g)	No.	Type	Wt. (g)	No.	Type	Wt. (g)		Temp. (°C)	Load (Ton)	Time (min)	
LU-077	Ni Fiber	4	4.6	3	79-01, 79-02	6.7	1	79-03	2	Powder	300	18	15	53
LU-078	Ni Fiber	4	4.7	3	79-01, 79-02	6.5	1	79-03	2	Powder	300	18	15	55
LU-079	Ni Fiber	4	4.7	3	79-01, 79-02, Co	6.8	1	79-03	2	Powder	300	18	15	54
LU-080	Ni Fiber	4	5.5	3	79-04, 79-05, 79-06, 79-07	6.5	1	79-08	2	Powder	330	18	10	44
LU-081	Ni Fiber	4	5.8	3	79-04, 79-05, 79-06, 79-07	6.5	1	79-08	2	Powder	330	18	10	44
LU-082	Ni Fiber	4	5.6				1	79-08	2	Powder	310	5	10	60
LU-083	Ni Fiber	2	2.8	1	79-13	2.2	1	79-16	2	Powder	343	12	10	30
LU-084	Ni Fiber	2	2.8	1	79-14	2.2	1	79-17	2	Powder	343	12	10	30
LU-085	Ni Fiber	2	2.8	1	79-15	2.2	1	A-1	2	Powder	371	12	10	29
LU-086	Ni Fiber	2	3.1	1	79-18	2.2	1	A-1	2	Powder	371	12	10	28
LU-087	Ni Fiber	2	2.9	1	79-19	2.2	1	79-16	2	Powder	343	12	10	30
LU-088	Ni Fiber	2	2.8	1	79-13	2.2	1	79-16	2	Powder	357	12	10	30
LU-089	Ni Fiber	2	2.8	1	79-14	2.2	1	79-17	2	Powder	357	12	10	29
LU-090	Ni Fiber	2	2.8	1	79-15	2.2	1	A-1	2	Powder	357	12	10	29
LU-091	Ni Fiber	2	2.9	1	79-18	2.2	1	A-1	2	Powder	357	12	10	28
LU-092	Ni Fiber	2	3.0	1	79-19	2.2	1	79-16	2	Powder	357	12	10	29
LU-093	Ni Fiber	2	3.0	1	79-20	2.2	1	A-1	2	Powder	343	12	10	30
LU-094	Ni Fiber	2	3.1	1	79-20	2.2	1	A-1	2	Powder	357	12	10	29
LU-095	Ni Fiber	2	2.9	1	29-21	2.2	1	79-16	2	Powder	343	12	10	30
LU-096	Ni Fiber	2	3.0	1	79-21	2.2	1	79-16	2	Powder	357	12	10	30
LU-097	Ni Fiber	2	2.6	1	79-15, 79-18	2.2	1	A-1	2	Powder	357	12	10	46

TABLE A-2C

Air Electrode (40 cm²) Construction

Electrode Identi- fication	Current Collector			Active Layer			Hydrophobic Layer			Fabrication Process	Final Process Conditions			Thickness (mils)
	Type	No.	Wt. (g)	No.	Type	Wt. (g)	No.	Type	Wt. (g)		Temp. (°C)	Load (Ton)	Time (min)	
LU-098	Ni Fiber and Exmet	2	2.5	1	79-22, 79-23	3.2	1	79-24	0.8	Powder	343	18	30	25
LU-099	Ni Fiber and Exmet	2	2.0	1	79-25, 79-26	1.0	1	79-27	2.0	Powder	275	3+	240	-
LU-100	Ni Fiber and Exmet	4	3.9	1	79-28, 79-29, Ni-sponge	1.1	1	79-30	1.5	Powder	371	18	10	16
LU-101	Ni Fiber	2	2.8	1	79-32, Ni-sponge	1.5	1	79-32 (baked)	1.0	Powder	300	18	10	21
LU-102	Ni Fiber	2	2.8	1	79-32, Ni-sponge	1.5	1	79-32 (baked)	1.0	Powder	275	18	10	22
LU-103	Ni Fiber	2	2.9	1	79-33, Ni-sponge	1.5	1	79-33 (baked)	1.0	Powder	300	18	10	-
LU-104	Ni Fiber and Exmet	4	5.5	2	79-34 urea	3	1	79-32	2.0	Powder	300	18	10	-
LU-105	Ni Fiber and Exmet	4	5.5	2	79-34	3.5	1	79-35	2.0	Powder	300	18	10	31
LU-106	Ni Fiber	1	6.6	1	79-36	2.2	1	79-35	2.0	Paste	300	12++	60	-
LU-107	Ni Fiber	4	5.4	3	79-20, 79-32, 79-38	5.5	1	79-32	2.0	Powder	300	10	20	58
LU-108	Ni Fiber	4	5.4	3	79-20, 79-37, 79-38	5.5	1	79-32	2.0	Powder	300	10	20	53
LU-109	Ni Fiber	4	5.3	3	79-20, 79-32, 79-39	5.5	1	79-32	2.0	Powder	300	10	20	57
LU-110	Ni Fiber	4	5.4	3	79-20, 79-32, 79-39, urea	6.0	1	79-32	2.0	Powder	300	25++	60	61
LU-111	Ni Fiber	4	5.3	3	79-37, 79-41	3.4	1	79-35	2.0	Powder	300	10	20	34

TABLE A-2D

B Air Electrode Construction (40 cm²)

Electrode Identi- fication	Current Collector			Active Layer			Hydrophobic Layer			Fabrication Process	Final Process Conditions			Thickness (mils)
	Type	No.	Wt. (g)	No.	Type	Wt. (g)	No.	Type	Wt. (g)		Temp. (°C)	Load (Ton)	Time (min)	
B-1	5 SS70-1/0	4	34.0	3	A	12.0	1	B	5.0	Dry Powder	300	5	10 ⁽¹⁾	
B-2	Steel Fiber	4	5.7	3	A	12.0	1	B	3.0	Dry Powder	300	5	10	
B-3	Ni Fiber	4	5.7	4	C, D	12.5	1	E	3.0	Dry Powder	300	5	10	
B-4	Ni Fiber	4	5.8	4	C, D	12.5	1	E ⁽²⁾	3.0	Dry Powder	300	5	10	
B-5	Ni Fiber	4	5.4	3	C	6.25	1	E	3.0	Dry Powder	300	5	10	
B-6	5 Fe7-2/0 ⁽³⁾	4	6.0	4	C, D	12.5	1	E	3.0	Dry Powder	300	5	10	
B-7	Steel Fiber	4	6.0	4	F, G	2.3	1	H	.75	Dry Powder	300	2	30 ⁽¹⁾	
B-8	Steel Fiber	4	6.25	3	J	7.0	1	J	2.5	Dry Powder	300	5	10 ⁽¹⁾	
B-9	Ni Fiber	4	5.9	4	E	6.25	1	E	1.5	Dry Powder	300	5	10	73.2
B-10	Steel Fiber ⁽⁴⁾	1	3.7	1	I	4.0	1	B	4.0	Dry Powder	330	18	10	67.8
B-11	Ni Fiber	2	2.9	1	N	4.0	1	B	4.0	Dry Powder	370	18	10	70.0
B-12	Steel Fiber ⁽⁴⁾	1	3.7	1	79-01 ⁽⁵⁾	4.0	1	V	4.0	Dry Powder	370	18	10	
B-13	Ni Fiber	2	2.5	1	N	4.0	1	B	4.0	Dry Powder	370	18	10	
B-14	Ni Fiber	2	2.55	1	N	4.0	1	B	4.0	Dry Powder	370	18	10	79.2
B-15	Ni Fiber	2	2.5	1	N	4.0	1	B	4.0	Dry Powder	370	18	10	75.0
B-16	Ni Fiber	2	2.5	1	N	4.0	1	B	4.0	Dry Powder	370	18	10	69.2
B-17	Ni Fiber	2	2.5	1	N	4.0	1	B	4.0	Dry Powder	370	18	10	70.0
B-18	Ni Fiber	2	2.5	1	N	4.0	1	B	4.0	Dry Powder	370	18	10	71.3
B-19	Ni Fiber	2	2.5	1	N	4.0	1	B	4.0	Dry Powder	370	18	10	66.7
B-20	Ni Fiber	2	2.5	1	N	4.0	1	B	4.0	Dry Powder	370	18	10	67.8

(1) Cold rolled 25', 4 dir.

(2) Unbaked

(3) Unannealed

(4) Ni plated

(5) See Table A-1B

TABLE A-2E

B Air Electrode Construction (40 cm²)

Electrode Identi- fication	Current Collector			Active Layer			Hydrophobic Layer			Fabrication Process	Final Process Conditions			Thickness (mils)
	Type	No.	Wt. (g)	No.	Type	Wt. (g)	No.	Type	Wt. (g)		Temp. (°C)	Load (Ton)	Time (min)	
B-21	Ni Fiber	2	2.5	1	N	4.0	1	B	4.0	Dry Powder	370	18	10	64.7
B-22	Ni Fiber	2	2.5	1	N	4.0	1	B	4.0	Dry Powder	370	18	10	74.7
B-23	Ni Fiber	2	2.5	1	N	4.0	1	B	4.0	Dry Powder	370	18	10	73.0
B-24	5 Fe7-189	2	2.9	1	K	4.0	1	B	4.0	Dry Powder	370	18	10	61.2
B-25	5 Fe7-189	2	2.9	1	L		1	B	4.0	Dry Powder	370	18	10	68.5
B-26	5 Fe7-189	2	2.9	1	M ⁽¹⁾		1	M	4.0	Dry Powder	370	18	10	69.3
B-27	5 Ni7-2/0	2	4.45 ⁽²⁾	1	N		1	B	4.0	Dry Powder	370	18	10	76.5
B-28	5 Ni7-2/0	2	4.30 ⁽²⁾	1	N		1	B	4.0	Dry Powder	370	18	10	79.5
B-29	(3)	2	3.1	1	N		1	B	4.0	Dry Powder	370	18	10	78.3
B-30	5 Ni7-2/0	2	2.6	1	O		1	B	4.0	Dry Powder	370	18	10	66.3
B-31	5 Ni7-2/0	2	2.7	1	P		1	B	4.0	Dry Powder	370	18	10	63.8
B-32	5 Ni7-2/0	2	2.6	1	Q		1	B	4.0	Dry Powder	370	18	10	68.8
B-33	5 Ni7-2/0	2	2.6	1	R		1	B	4.0	Dry Powder	370	18	10	71.0
B-34	5 Ni7-2/0	2	2.6	1	K		1	B	4.0	Dry Powder	370	18	10	62.3
B-35	5 Ni7-2/0	2	2.6	1	S		1	B	4.0	Dry Powder	370	18	10	61.2
B-36	5 Ni7-2/0	2	2.7	1	T		1	B	4.0	Dry Powder	370	18	10	56.8
B-37	5 Ni7-2/0	2	2.6	1	C		1	B	4.0	Dry Powder	370	18	10	57.0
B-38	5 Ni7-2/0	2	2.55	1	C		1	B	4.0	Dry Powder	370	18	10	53.0
B-39	5 Ni7-2/0	2	2.6	1	C		1	B	4.0	Dry Powder	370	18	10	53.8
B-40	5 Ni7-2/0	2	2.6	1	C		1	B	4.0	Dry Powder	370	18	10	54.2

(1) Unbaked

(2) Wire probes attached for resistivity measurements

(3) 3 Ni5-2/0 + 7 Ni12-2/0 (coated with Ni-Fe-WC-Co Powder) + 5 Ni7-2/0

TABLE A-2F

B Air Electrode Construction (40 cm²)

Electrode Identi- fication	Current Collector			Active Layer			Hydrophobic Layer			Fabrication Process	Final Process Conditions			Thickness (mils)
	Type	No.	Wt. (g)	No.	Type	Wt. (g)	No.	Type	Wt. (g)		Temp. (°C)	Load (Ton)	Time (min)	
B-41	5 Ni7-2/0	2	2.6	1	C	4.0	1	B	4.0	Dry Powder	370	18	10	52.8
B-42	5 Ni7-2/0	2	2.6	1	C	4.0	1	B	4.0	Dry Powder	370	18	10	51.8
B-43	5 Ni7-2/0	2	2.6	1	C	4.0	1	B	4.0	Dry Powder	370	18	10	53.7
B-44	5 Ni7-2/0	2	2.6	1	C	4.0	1	B	4.0	Dry Powder	370	18	10	47.7
B-45	5 Ni7-2/0	2	2.6	1	C	4.0	1	B	4.0	Dry Powder	370	18	10	48.8
B-46	5 Ni7-2/0	2	2.6	1	C	4.0	1	B	4.0	Dry Powder	370	18	10	47.0
B-47	5 Ni7-2/0	2	2.6	1	C	4.0	1	B	4.0	Dry Powder	370	18	10	51.0
B-48	5 Ni7-2/0	2	2.6	1	C	4.0	1	B	4.0	Dry Powder	370	18	10	48.8

8.4 Construction of Air Electrodes - Cell Testing

TABLE A-3A

Full Cell Air Electrode Active Materials (100 cm²)

	<u>AA</u>	<u>BB</u>	<u>CC</u>	<u>DD</u>	<u>EE</u>	<u>FF</u>	<u>GG</u>	<u>HH</u>	<u>II</u>	<u>JJ</u>	<u>KK</u>	<u>LL</u>	<u>MM</u>	<u>NN</u>
Shawinigan Black(Ag) ⁽¹⁾	30	30 ^N	30	30	30 ^N	--	30	10 ^N	--	5	7.5	10	10 ^N	10
Ketjenblack (Ag) ⁽²⁾	--	--	--	--	--	30	--	--	10	5	2.5	--	--	--
WC - 12% Co	4.5	--	--	4.5	--	9	9	--	3	3	3	3	--	3
NiS	4.5	--	--	4.5	--	6	6	--	2	2	2	2	--	2
Fe WO ₄	4.5	--	--	4.5	--	6	6	--	2	2	2	2	--	2
H ₂ O	175	200	150- 175	150- 175	200	200	200	85	75	75	75	75	75	70
Teflon 30B	15	15	9	12	9	10	10	3	6	6	6	6	6	5
Triton X-100	--	--	--	--	--	--	--	--	--	--	--	--	--	--
FEP	--	--	--	--	--	10	10	3	--	--	--	--	--	--
PFA	--	--	--	--	--	--	--	--	4	4	4	4	4	--

(1) 300 g Shawinigan
12 g AgNO₃
(7.65 g) Ag

(2) 300 g Shawinigan
11.4 g, Ag

TABLE A-3B

Full Cell Air Electrode Active Materials (100 cm²)

	<u>OO</u>	<u>PP</u>	<u>QQ</u>	<u>RR</u>	<u>SS</u>	<u>TT</u>	<u>UU</u>	<u>VV</u>	<u>WW</u>	<u>XX</u>	<u>YY</u>	<u>ZZ</u>	<u>AAA</u>	<u>BBB</u>
Shawiningan Black (Ag) ⁽¹⁾	5	10 ^N	5	5	30	20	10	30 ^N	5	--	20	20 ^N	--	20
Ketjenblack (Ag) ⁽²⁾	5	--	5	--	--	--	--	--	5	20	--	--	20	--
WC - 12% Co	3	--	--	--	--	--	--	--	3	6	6	--	--	--
Ni S	2	--	--	--	--	--	--	--	2	4	4	--	--	--
Fe WO ₄	2	--	--	--	--	--	--	--	2	4	4	--	--	--
H ₂ O	60	70	70	35	240	70	50	120	60	--	--	--	140	200
Teflon 30B	3	17	16	8	48	32	16	48	5	20	20	20	10	10
Triton X-100	--	--	--	--	--	--	--	--	--	--	--	--	--	--
F.E.P.	--	--	--	--	--	--	--	--	--	--	--	--	--	--
PFA	--	--	--	--	--	--	--	--	--	--	--	--	--	--
Ag NO ₃	--	--	--	0.2	--	--	--	--	--	--	--	--	--	--
CO	--	--	--	--	--	20	--	--	--	--	--	--	--	--

(1) 300 g Shawiningan
12 g AgNO₃
(7.65 g) Ag

(2) 300 g Ketjenblack
11.4 g, Ag

TABLE A-4A

Full Cell (100 cm²) Air Electrode Construction

Electrode Identi- fication	Current Collector			Active Layer			Hydrophobic Layer			Fabrication Process	Final Process Conditions			Thickness (mils)
	Type	No.	Wt. (g)	No.	Type	Wt. (g)	No.	Type	Wt. (g)		Temp. (°C)	Load (Ton)	Time (Min)	
A-1	Ni Fiber	4	11.1	3	AA	12.0	1	BB	3.0	Dry Powder	300	5	10 ⁽¹⁾	
A-2	Ni Fiber	4	11.9	3	AA	12.0	1	BB	3.0	Dry Powder	300	5	10	59.3
A-3	Ni Fiber	4	12.1	3	AA	12.0	1	BB	3.0	Dry Powder	300	5	10	59.1
A-4	Ni Fiber	4	12.2	3	AA	12.0	1	BB	3.0	Dry Powder	300	5	10	60.1
A-5	Ni Fiber	4	11.95	3	AA	12.0	1	BB	3.0	Dry Powder	300	5	10	63.2
A-6	Ni Fiber	4	12.1	3	AA	12.0	1	BB	3.0	Dry Powder	300	5	10	61.3
A-7	Ni Fiber	4	12.1	3	AA	12.0	1	BB	3.0	Dry Powder	300	5	10	62.8
A-8	Ni Fiber	4	12.0	3	AA	12.0	1	BB	3.0	Dry Powder	300	5	10	59.55
A-9	Ni Fiber	4	11.75	4	CC, DD	12.5	1	EE	3.0	Dry Powder	300	5	10	76.7
A-10	Ni Fiber	4	12.0	3	AA	12.0	1	BB	3.0	Dry Powder	300	5	10	63.0
A-11	Ni Fiber	4	11.8	4	CC, DD	12.5	1	EE	3.0	Dry Powder	300	5	10	74.8
A-12	Ni Fiber	4	8.45	4	FF, GG	12.1	1	HH	3.6	Dry Powder	300	32	15 ⁽¹⁾	
A-13	Ni Fiber	4	11.1	4	II, JJ, KK, LL	11.7	1	MM	3.6	Dry Powder	330	32	10	45.4
A-14	Ni Fiber ⁽²⁾	5	16.4	4	II, JJ, KK, LL	11.7	1	MM	3.6	Dry Powder	330	32	10	
A-15	Ni Fiber	2	6.1	2	NN, OO	7.8	1	PP	3.0	Dry Powder + Preform	357	36	10	37.4
A-16	Ni Fiber- 5 Ni7-2/0	2	11.4	2	NN, OO	7.8	1	PP	3.0	Dry Powder + Preform	357	36	10	37.0

(1) Cold rolled 50 PSI, 4 dir

(2) +5 Ni7-2/0 steel fiber charging current collector (3.75 g)

TABLE A-4B

Full Cell (100 cm²) Air Electrode Construction

Electrode Identi- fication	Current Collector			Active Layer			Hydrophobic Layer			Fabrication Process	Final Process Conditions			Thickness (mils)
	Type	No.	Wt. (g)	No.	Type	Wt. (g)	No.	Type	Wt. (g)		Temp. (°C)	Load (Ton)	Time (Min)	
B-1	Ni Fiber ⁽¹⁾	2	5.6	2	NN, OO	7.8	1	PP	3.0	Dry Powder + Preform	357	36	10	37.0
B-2	Ni Fiber- 5 Ni7-2/O	2	10.8	2	NN	4.4	1	BB	4.5	Dry Powder	357	36	10	-
B-3	7 Ni12-2/O	2	20.0	2	NN	4.4	1	BB	4.5	Dry Powder	357	36	10	-
B-4	5 Ni7-2/O ⁽²⁾	1	-	1	NN	1.3	1	BB	3.0	Dry Powder	357	36	10	-
B-5	7 Ni12-2/O ⁽³⁾	1	12.4	1	QQ, RR	25.0	1	SS	3.0	Wet Pasting	357	36	10	29.4
B-6	8 Ni15-2/O ⁽³⁾ 5 Ni7-2/O	2	16.15	1	TT, UU	9.7	1	VV	3.0	Dry Powder	343	12	10	41.1
B-7	Ni Fiber- 5 Ni7-2/O ⁽³⁾	3	15.90	1	LL, TT	4.8	1	VV	3.0	Preform	343	12	10	34.8
B-8	5 Ni7-2/O ⁽³⁾	1	5.40	1	WW, XX, YY	5.2	1	ZZ	3.0	Dry Powder - Preform	343	10	10	39.6
B-9	5 Ni7-2/O	2	4.6	1	AAA	7.6	1	BBB	7.6	Dry Powder	370	36	10	-
B-10	5 Ni7-2/O	2	4.6	1	AAA	7.6	1	BBB	7.6	Dry Powder	370	36	10	-

(1) 1 piece

(2) Coated with Ni powder

(3) Coated with Ni-Fe-WC-CO powder mixture

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