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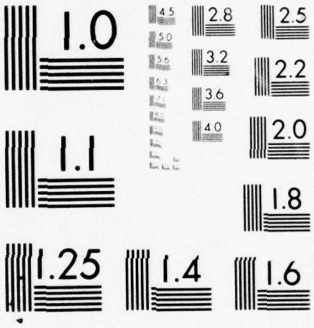

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PRODUCTION AND ENGINEERING METHODS FOR CARB-TEK® BATTERIES IN FORK LIFT TRUCKS

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OCTOBER 1976

INTERIM REPORT FOR PERIOD APR. 1976 - OCT. 1976

PREPARED FOR
U.S. ARMY MOBILITY EQUIPMENT RESEARCH & DEVELOPMENT COMMAND
FORT BELVOIR, VIRGINIA 22060

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This report describes the technological development of the Carb-Tek® Molten Salt Li/Cl system toward prototype production for eventual assembly into fork lift truck batteries. Engineering developments, cost reductions, and pilot line operations are described and discussed. Battery modules are being constructed.

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SUMMARY

The objective of this program is to produce a demonstration fork lift truck battery from the cell system $Li-Al/LiCl-KCl/C-TeCl_4$. A transition from the small 8 - 10 ampere hour cells to the production size 150 - 200 ampere hour cells has been accomplished. Problems were encountered with the physical and electrical characteristics of the cathode and with the non-wetting of the separator. Battery modules are being constructed.

PREFACE

This interim report was prepared by the Technology Center of ESB Incorporated under U. S. Army Mobility Equipment Research and Development Command (USAMERADCOM) Contract No. DAAK02-75-C-0035.

The objective of this program is to develop the manufacturing technology of the molten salt Li/Cl batteries. The goal is the demonstration of a molten salt battery meeting military specifications for fork lift truck applications.

Acknowledgement is made to Dr. T. Noveske for his development laboratory efforts, to J. Thompson, D. Mey, P. Struk, and R. Brown for their pilot line contributions, and to R. Baker and S. Torreso for their engineering efforts. Additional acknowledgement is also made for the many persons at the ESB Technology Center and Standard Oil Company (Ohio) Laboratories that have willingly assisted the program.

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INTRODUCTION

Historically, the Standard Oil Co. (Ohio) researched molten salt batteries in 1962 and developed the Carb-Tek® battery. The work continued until 1971. The technology was transferred to ESB Incorporated starting in 1973. Background information concerning prior theoretical and laboratory studies are reported in the literature.(8, 9, 11)

The objective of this program is to produce a demonstration fork lift truck battery from the molten salt cell system Li-Al/LiCl-KCl/C-$TeCl_4$. The Li-Al anode(1, 2, 3, 4, 10) is an alloy of about 16% lithium in aluminum. The LiCl-KCl electrolyte is the eutectic composition melting at about 355° C. The cathode is porous carbon impregnated with $TeCl_4$(2, 5) Cell separation is made by a BN fibrous mat.(3,6) Cell components are housed in a low carbon steel can(7) that is common to the anodes. A tungsten rod external lead connects the carbon cathode to the exterior. The voltage range is 3.2 to 2.4 volts. Cells operate at a temperature of about 450° C and develop 5 Whr/in^3 of cathode volume between 2.4 and 3.2 volts. The demonstration battery is expected to have the following characteristics:

Nominal voltage - 36 at full charge
- 30 at cut-off

Capacity when discharged from full charge to cut-off voltage -
- 720 Ahr at 6 hour rate

Recharge Rate - 1 hour to restore 80% capacity

Weight - about 1550 lbs.

Est. volume - 9.25 ft^3

A pilot line was assembled and qualified to produce cells measuring about 4" x 5" x 5/16" and to obtain basic information concerning production type problems.(8, 9) Engineering and process changes were made to enable a transition to the larger production size cathodes and cells measuring about 10" x 12" x 1". Work reported for this period covers the transition to the larger cells and the problems encountered. At the present time it appears that the transition problems have been solved, and the fabrication of cells and modules for batteries has begun.

INVESTIGATION

All voltage, current, and resistance measurements performed on this project are traceable through three Data Precision Company Model 1450 multimeters to the National Bureau of Standards.

The work activities are reported on the basis of cell construction and the steps followed in assembly. This should assist the reader in visualizing each component and its relationship to the cell.

Figure 1 is a diagram of the laboratory test unit utilized to qualify materials and to assist the pilot line in solving problems.

Figure 2 is an exploded view of the ESB cell as constructed at the conclusion of the report period. This figure will serve as the visual report outline.

A. CARBON CATHODES

1. Cathode Material

The cathodes are porous carbon slabs or blocks containing a graphite paper or cloth current collector through the central portion of the slab from the bottom to the top where it connects to the header. The graphite header holds a tungsten rod by press fit which extends through the cell can top and serves as the external cathode lead.

PCB grade carbon obtained from Calgon Corporation is fabricated into cathodes by Gaines Industries and is now standard. The determination of the appropriate carbon (PCB) was disclosed in the last interim report. Additional sources are being sought. Stackpole Carbon Company qualified a preliminary sample at the minimum value of 5 Whr/in^3. Further testing is anticipated.

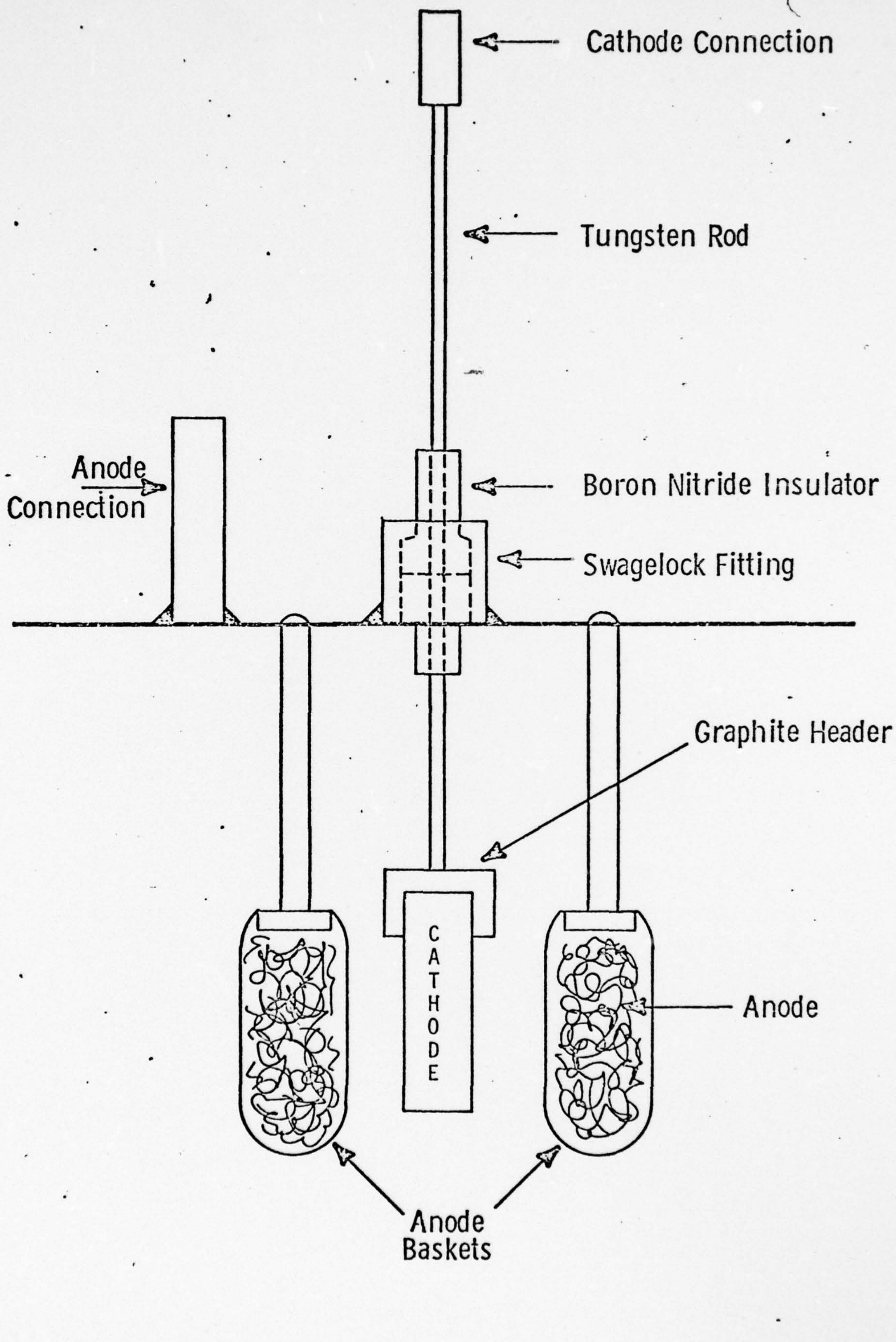


Figure I LABORATORY TEST UNIT

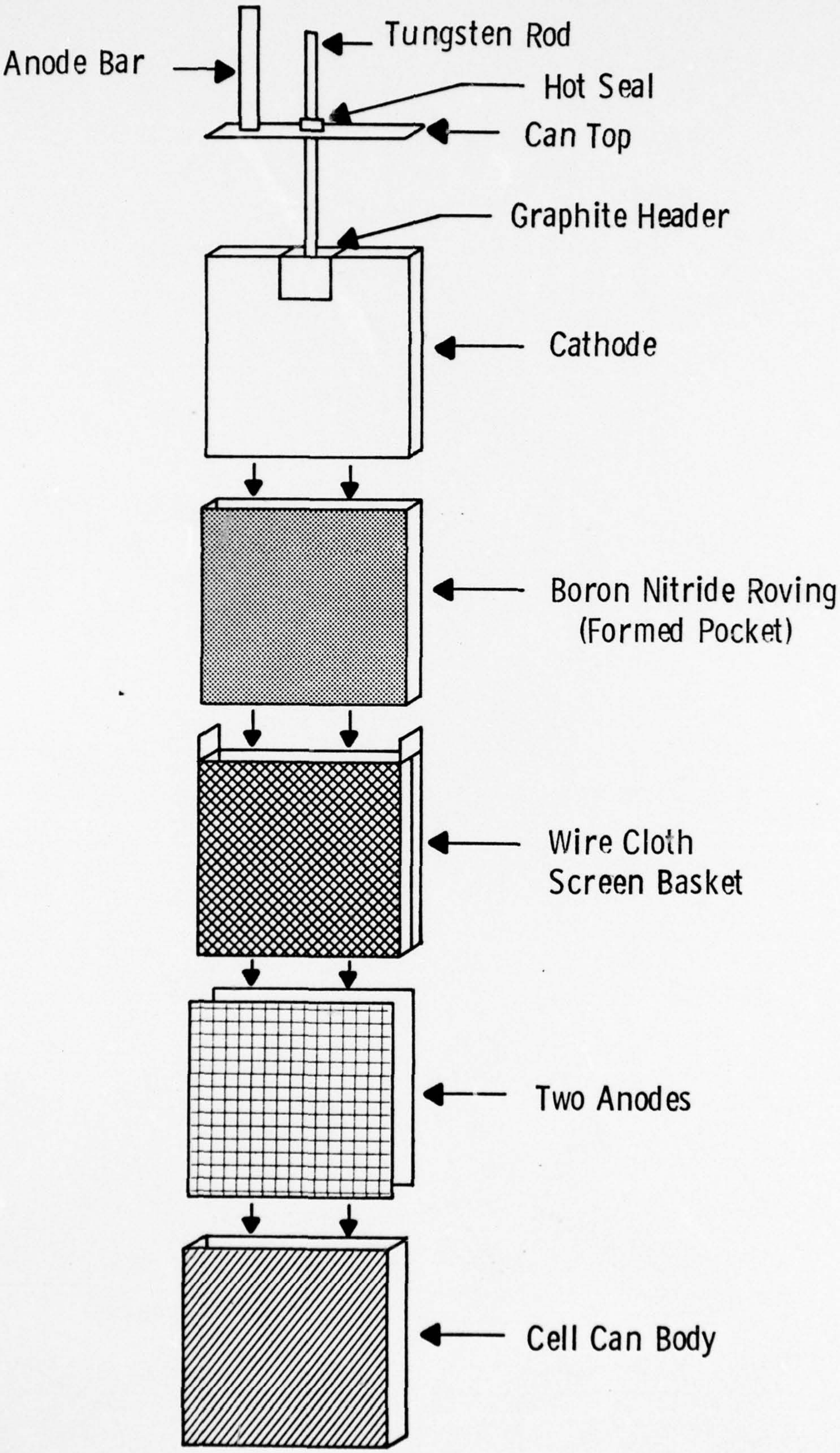


Figure 2 - ESB CELL CONSTRUCTION

2. Quality Control

a. Millivolt Drop Readings

Standard procedures prior to this report period required the measurement of millivolt drops between designated points on one face of a cathode and the header after the cathode carbon plate had been treated and impregnated with $TeCl_4$ (Figure 3). Figure 4 shows the method of attaching the positive and negative current leads to the cathode to be tested. The positive voltage lead is connected to the header while the negative voltage lead is free to make contact at the various test sites as previously shown in Figure 3. A current of 5.0 amperes D.C. is applied and the millivolt readings are noted as contact is made to the cathode at each of the indicated points. Readings obtained on the initial MM-1 production size cathodes were found to be much higher than those No. 6 size units used up to this time period (Table I). In both instances only one face was tested. The function of the measurement was to make a "go, no-go" test to determine whether it was reasonable to expect an acceptable cathode after the formation step.

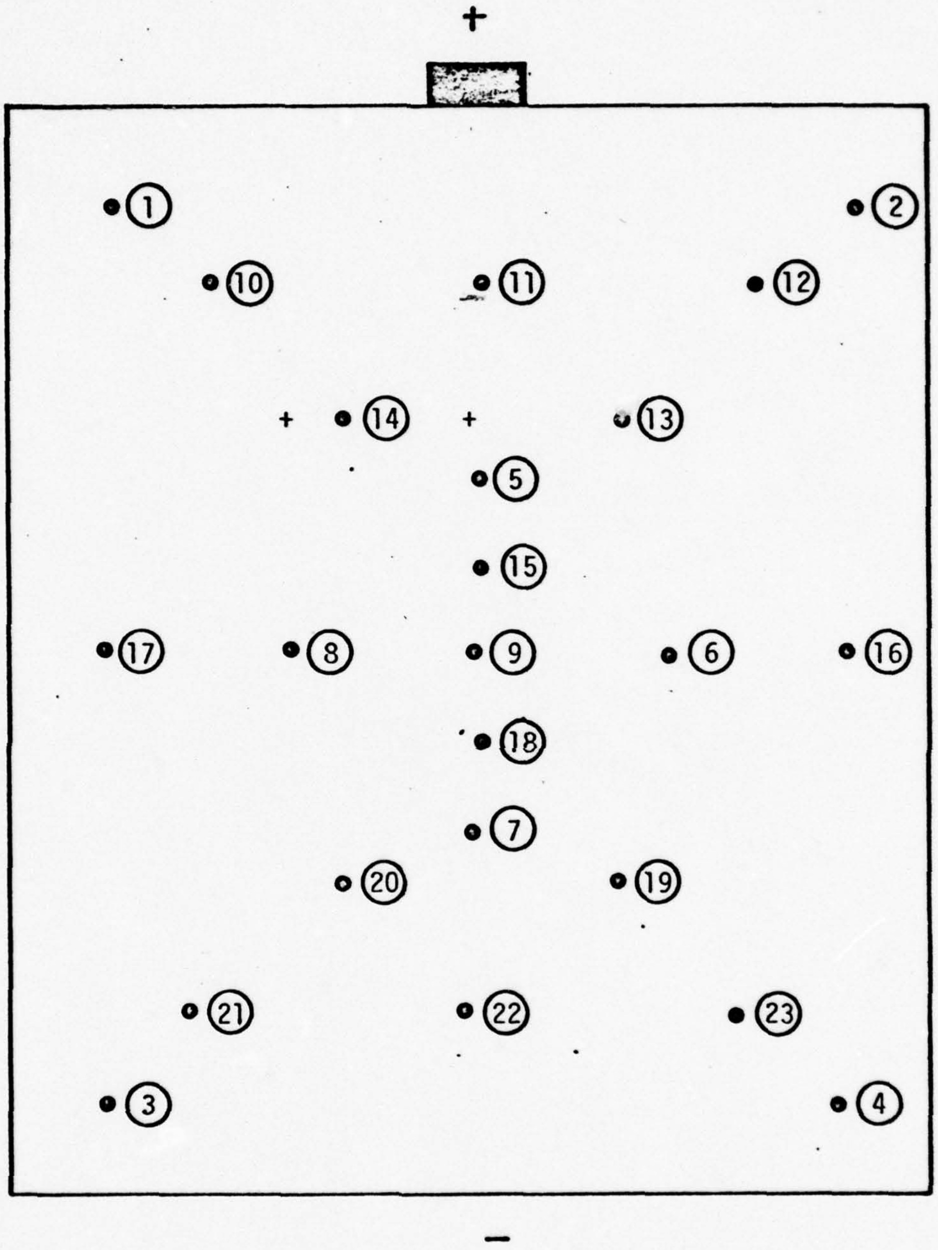
Readings were taken on both faces of a few MM-1 cathodes and were found to be within a few millivolts of corresponding points on the reverse side. The double readings, involving 9 points each, were continued as a routine check. Then it was noted that, on occasion, one side read considerably higher, indicating a separation or break between the carbon and the graphite cloth current collector (Table I).

The number of reading points was expanded to 23 for both sides and the time at which readings were taken was expanded to include: (1) as received; (2) after heat treatment; (3) after $TeCl_4$ impregnation; and (4) after tungsten lead attachment (Table I). These expanded readings made it possible to upgrade the quality and uniformity of the cathodes. By this technique it has

Cell Size	6	6	6	MM-1	MM-1	MM-1	MM-1	MM-1	MM-1	MM-1	MM-1
Cathode No.	121	137	148	22	31	38	45	47	49	53	54
Points/Reading	9	9	9	9	9	9	23	23	23	23	23
Avg. As Rec'd (1)	---	---	---	---	---	---	---	22.3	20.6	31.9	29.4
" " " (2)	---	---	---	---	---	---	---	23.8	20.3	30.3	28.1
Avg. Heat Treat (1)	---	---	---	---	---	---	---	18.7	17.7	15.1	18.0
" " " (2)	---	---	---	---	---	---	---	18.4	18.1	15.0	18.9
Avg. Impreg. (1)	13.5	15.1	17.6	33.7	96.0	148.8	35.8	18.6	18.0	14.6	19.2
" " (2)	---	---	---	---	---	87.0	35.3	19.2	17.4	14.9	18.0
Avg. Rod Insert (1)	---	---	---	---	---	---	---	21.3	19.3	16.6	13.0
" " " (2)	---	---	---	---	---	---	---	22.6	19.6	17.7	13.1
Range As Rec'd	---	---	---	---	---	---	---	(7-45)	(7-30)	(14-76)	(12-60)
Range H. Treat	---	---	---	---	---	---	---	(8-33)	(9-30)	(7-27)	(7-28)
Range Impreg.	(6.4-21.)	(7.6-22.)	(8.0-28.)	(23-43)	(74-115)	(76-160)	(10-64)	(8-31)	(9-30)	(6-26)	(5-30)
Range Rod Insert	---	---	---	---	---	---	---	(11-31)	(7-29)	(7-28)	(6-21)

MILLIVOLT DROP READINGS

TABLE I



Test locations Nos. 1-9 for one side only
Test locations Nos. 1-23 for two sides

Figure 3 - MILLIVOLT DROP TEST PATTERNS

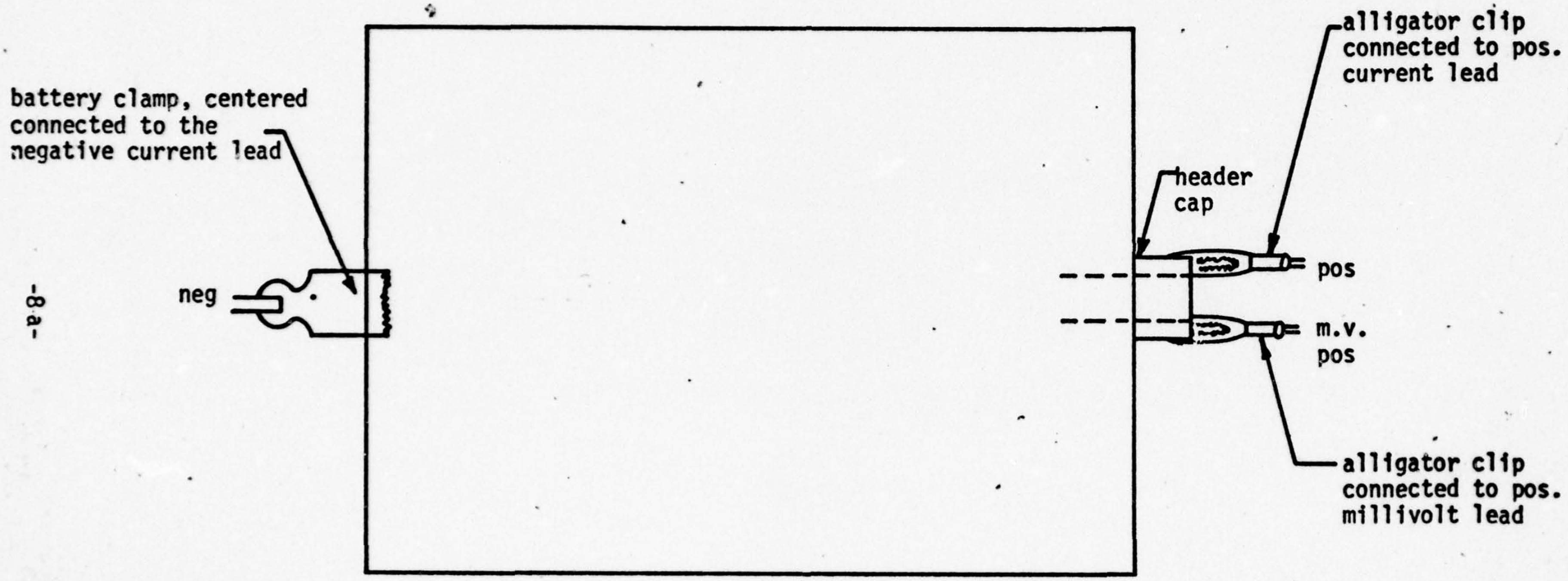


Figure 4 MILLIVOLT READING CONNECTION DIAGRAM

been shown that the re-designed graphite header resulted in millivolt drop readings of the large MM-1 cathodes equivalent to those of the small No. 6 cathodes. Table I shows the average of nine millivolt drop readings obtained with $TeCl_4$ including the range of the readings. (See Figure 3, test locations 1 through 9). The reported readings indicate an absence of internal problems. Unfortunately the 9 point-one side procedure only indicated the condition of the side being probed.

The Table also shows that the early MM-1 cathodes, Nos. 22 31, 38 and 45, were substandard to the No. 6 type cathodes. Cathode No. 38 illustrates the fact that reading both sides makes it possible to detect a side with differing characteristics and that there was a need for further improvement in the manufacturing techniques.

An example showing improved fabrication technique and greater consistency in quality between cathode sides is given by cathode No. 45. Additional indications of quality improvement can be seen in the corresponding values reported for numbers 47, 49, 53 and 54.

By expanding the number of readings from 9 to 23 (See Figure 3) for both sides of the cathodes and taking readings at each stage of cathode processing, it became obvious that each stage tended to cause a decrease in the average millivolt reading. These multiple readings also indicated a convergence of the readings with each step of the cathode development, showing an improvement in homogeneity.

This procedure is sufficiently effective to locate breaks in the graphite cloth current collector and graphite header. Cracks found in the carbon body do not affect readings or results provided the cracks do not cause a loss of contact of carbon with the current collecting graphite cloth. The explanation is that the carbon cathode is in effect two cathodes in parallel connected to a common conductor (graphite cloth). A crack propagated from the surface to the common conductor literally increases the number of parallel cathodes connected to the graphite cloth. However, a crack that separates a portion of carbon from the cathode body has no electrical contact and will not store energy. No repair procedures have been developed to correct the latter type breakage. No repair is needed for the former type of breakage.

The cathode header design was also a factor in the large voltage drops measured between the header and the control equipment. The initial design had the tungsten rod threaded into the graphite header to ensure that a good mechanical connection was made. This design was quickly abandoned in favor of a press fit when it was found that the graphite threads were often sheared or reamed out by the tungsten during assembly. Thus the total contact area across each thread to the mating thread was too low to be effective. The usual IR drop at this time was 0.45 to 0.50 volts. Changing to the improved header resulted in a lowering of the IR drop by 0.15 volts. The contact resistance of the various power supply contractors and connectors were found to be responsible for an additional lowering of the IR drop of 0.15 volts.

3. Cathode Impregnation

Tellurium tetrachloride is responsible for a capacity enhancement in the voltage range of 2.4 to 3.2 volts.

The purity of the compound has been found to vary considerably from supplier to supplier. Each supplier claims 99+% purity. Fresh 99.99% $TeCl_4$ was purchased from Great Western Inorganics for pilot operations while

The quality control laboratory ran experiments to determine the minimum acceptable purity using samples ranging from 99.0 to 99.99%. The criterion for acceptance was the attainment of better than 5 Ahr/in^3 from standard carbon cathodes in fresh electrolyte. It has been determined that a minimum grade of 99.70% will provide satisfactory cathode outputs. This represents a substantial reduction in material cost.

4. Cathode Formation

Prior to this report period it was the practice to place several cathodes in the molten salt formation tank at one time. Each cathode was connected to the other in parallel. As a result, all cathodes charged and discharged as a unit. With the assumption that each cathode reacted equally, the ampere-hour ratings were determined by dividing the total ampere-hours by the number of cathodes being formed, and that value was assigned to each cathode.

At one point, only two cathodes were formed simultaneously and were given an average value of 145 Ahr each. When each cathode was completed as a cell, one developed only 98 Ahr while the other developed 196 Ahr. Initially various reasons were contemplated to explain the 98 Ahr result until it was obvious that the average of the two cells was 147 Ahr -- a difference of only 2 from the average value obtained as cathodes in the formation tank. This indicated two things: (1) that the cell testing, BN wetting procedures, etc. were reliable, and (2) that it was necessary to monitor each cathode during formation. Individual power supplies, shunts, etc. are being installed to permit the monitoring of current and voltage for each cathode. These power supplies make it possible for each cathode to individually charge, set at open circuit, or discharge as programmed without regard to the state or condition of the other cathodes.

True ampere-hour ratings can now be assigned. Any cathode can be accepted or rejected at this point.

B. BN Separator

Numerous mechanical and electrical problems were experienced early in the scale-up to the production-size cathodes designated as MM-1's. These problems and their solutions are discussed in other sections of this report. The correction or elimination of the problems permitted the formation of cathodes in the range of 140 - 210 Ahr. Assembly of these early cathodes in cells showed capacities dropping to 15 to 50 Ahr. All cells appeared to take sufficient electrolyte during charging. Disassembly of the large MM-1 cells was indicated but was impossible in the molten state in the present dry box system. It was decided to immerse a fresh cathode assembly in the formation tank instead of into a cell can and to operate it as a cell and observe the performance. (The cathode assembly consists of the cathode completely surrounded by a BN mat which is inserted into a metal screen enclosure designed to hold the BN against the cathode surfaces.)

The test assembly was charged and discharged as per standard cell test procedures. The result was the same low ampere-hour value obtained for a completely assembled cell. It was reasoned that the electrolyte was not penetrating into the space between the BN and the cathode since it was known that there was adequate electrolyte between the BN and the anodes. The BN mat was cut at the top near the header to allow some salt to flow in between. The result was a minor increase in capacity. Additional efforts were made to fill the space between the BN separator and the cathode without success. From this test and others, it was obvious that so-called filled cells did not have electrolyte between the separator and the cathode.

To determine whether non-wetting of the BN was the basic problem, the cathode assembly was slowly and deliberately opened. The sections at the top where the mat had been forcibly separated from the cathode contained salt. The salt had not spread. The BN mat was sectioned, was found to be unwetted, and had the appearance of fluffy cotton. The total BN area capable of conducting current by virtue of being wetted was no more than 10 percent.

Various attempts to increase the wetting of BN were pursued.

1. One approach was to coat the BN with salt prior to forming the mat. After the BN fibers are cut to length, they are sparged in water to effect a random orientation of fibers before forming the mat. A saturated solution of LiCl-KCl was substituted for the water. This improved the wetting but did not guarantee that each cell would operate at capacity.

2. Same as No. 1 but with the absence of LiCl. Results showed no increase in wetting;

3. Reduction of BN mat thickness. This was helpful. Process changes (1) and (3) improved wetting but did not ensure that each cell produced would operate; and

4. Dipping of cold cathode in molten salt after formation. This procedure was found to be highly successful.

Methods 1, 3, and 4 were adopted. Table III given in the "Production and Cell Testing" section shows the correspondence between the formation tank values and those for the completed cells.

C. Anodes

The sandwich-type anode disclosed in the last Interim Report is the standard anode for cells and formation tank. As the overall size increased, certain minor problems became apparent. To appreciate the problem it is necessary to understand the formation of the alloy.

Temperature control during the anode alloy formation reaction is important. It is necessary to reach the temperature at which diffusion is rapid without melting the lithium. The contact area between the aluminum and the lithium is also important since the reaction is exothermic. Conduction of the heat to an area of poor contact results in melting lithium which can flow to an adjacent area to give a fast reaction and further uneven heating. As a result, complete homogeneity is not always achieved. Some areas tend to rise, producing an anode of uneven thickness which requires an increase in cell thickness.

Anodes formed with irregular composition are of variable strength and are difficult to handle. To eliminate the handling, the unreacted anode materials, as a sandwich, are placed in the cell can and reacted in situ. This procedure does not solve the irregular thickness problem. To attempt to solve this problem, a graphite mold has been fabricated. The expectation is that the large graphite mass will maintain an even temperature during the alloying and result in more uniform anodes. Laboratory tests of small anodes indicate improvement. Successful full-size tests are expected and will ensure better handling characteristics.

D. Production and Cell Testing

Table II lists the MM-1 cathodes produced to date. The comment column shows the initial problem and the gradual shift to acceptable cathodes.

Table III lists the MM-1 cells produced to date. It relates to Table II in that the cathode and cell numbers are the same and the comments show the gradual shift to acceptable cells.

E. Cell Failure Modes

1. Oxygen Concentration

Oxygen is a detrimental contaminant whether present as elemental gas or as a soluble oxide. Soluble oxides can be electrolyzed in the cell to form free oxygen. Oxygen can be continuously recycled in the cell causing a resultant partial loss of power. Elimination of the sources of oxygen is an on-going effort, and one of the effective methods for monitoring is the neutron activation analysis procedure.

During this report period it was possible to have various samples analyzed, such as BN roving, anodes, salt, etc. The results are tabulated in Table IV. It can be seen that the levels of oxygen contamination are about a factor of 10 less than previous report periods. These present levels are markedly improved but still need further reduction.

TABLE II

CATHODE HISTORY

Item #	Cathode*	Max. Ahr Rating/Cathode	Comments
1	12, •14, 17	30	Lack of wetting
2	16, •20, 23	153	
3	•7, •9, •18	210	
4	19, 22, •24	164	
5	•10, •21	154	
6	25	--	Graphite Sheet Collector No Grafoil, High IR Drop
7	31, 37		Headers loose
8	38, 41	--	Press fit connection replaced threaded header. Headers fragile - broke off
9	40	--	Fragile - broken header
10	42	--	Cathode cracked during degassing
11	43	100	Slightly cracked
12	44	--	High IR drop
13	45	122	High IR drop
14	47, 48	146	Fabrication de-bugged
15	49	160	Fabrication de-bugged; #42 repressed
16	50, 51	136	Fabrication de-bugged
17	52, 54	145	Fabrication de-bugged
18	53	160	Fabrication de-bugged
19	57	100	Fabrication de-bugged
20	55	145	Fabrication de-bugged
21	59	156	Fabrication de-bugged
22	56, 58	159	Fabrication de-bugged

Notes:

Cathodes 27, 28, 29, 30, 39, 46 were not received from vendor.

Cathodes 1-6, 8, 9, 11, 13, 15, 26 broke during processing prior to cathode formation.

* Cathodes in each item run in parallel.

• Cathodes broken after formation step.

TABLE III

CELL HISTORY

Cell #	Formation Rating	Cell Rating	Comments
12	30	--	High IR drop due to threaded connection + un-wetted BN separator
17	30	--	"
16	153	--	"
23	153	--	"
19	164	--	"
22	164	--	"
45	122	--	High IR drop due to un-wetted BN separator. Confirmed by dissection
47	146	122	Wetting
48	146	43.66	Large IR due to unwetted separator Gradual failure
R-49 (Run 1)	160	166	Achieved in formation tank Assembled with separator
R-49 (Run 2)		--	Failed due to assembly problems
50	136	175	Cathode dipped in clean eutectic prior to assembly
52	145	196*	Cathode dipped. Electrical connections improved on cyclers
54	145	95*	Failed - High IR drop - Causes unresolved

* These cells were formed together with an average value of 145 Ahr. After cell fabrication the average was 145.5. No. 54 was a poor cathode.

TABLE IV
OXYGEN CONCENTRATIONS

Previous %	Sample No.	Identification	Oxygen Conc. %	Precision + or -
--	1	Li-Al Sandwich Type Anode	.177	.012
.265-.40	2	Li-Al Cast Anode	.125	.009
4.45-5.1	3	BN Roving (as received)	.958	.150
6.9	4	BN mat (Eutectic-H_2O Treated)	1.720	.170
--	5	Baked-Out Carbon Cathode	1.400	.070
3.97	10	Purchased LiCl-KCl Eutectic	.448	.026
.68	11	Formation Tank Eutectic Salt	.314	.025
3.82	12	BN Rod (as received)	6.790	.390
1.93-2.5	13	Cathode Carbon with Salt	2.700	.130

With improved procedures on the part of the vendors and the project total oxygen has been significantly reduced except in the case of the BN rod. It also appears that the cathode carbon collects oxygen and retains it to some degree. The BN rod result has been investigated by the vendor and it has been reported to have been a bad lot and that all BN rod is less than 2% oxygen.

2. Holding Current*

Operable cells have a relatively low holding current value initially. As the cell is charged and discharged repeatedly the current necessary to prevent further discharge gradually becomes larger until it approaches the charging current. Holding currents are read after the current values on constant voltage charge have become essentially constant.

An example is given by cell No. 167Q (No. 6 size). (Data obtained at Fort Belvoir, Va.)

TABLE V

Cycle No.	Ahr Rating	Holding Current (A)
1	6.26	.085
40	5.19	.230
80	4.56	.268
120	4.27	.309
160	3.87	.355
200	3.65	.355
241	3.48	.395
280	3.38	.378
291	3.34	.401
313	3.28	.408

* The parameter previously referred to as leakage current is now referred to as holding current.

Now that cell manufacture is consistent it is possible to study various parameters and their effects on cell performance. A significant effort has been initiated to define the problem and provide a solution.

F. Battery Design and Construction

1. Heaters

The battery must be at least 355° C to operate and preferably in the range of 400 to 450° C to avoid salting out due to composition changes during charge and discharge.

For the initial heating or any possible auxilliary heating, the battery will be fitted with two plate heaters per module. There will be a cell then a heater, then two cells, a heater, and a final cell for each module. With this arrangement any given heater will be radiating to two cells.

2. Bus Bar

The demonstration battery will be composed of 12 modules of 4 cells each. Each module will have the 4 cells connected in parallel. The modules will be connected in series to provide the necessary voltage range of 30 - 36 volts.

Bus bars and connections cannot be exposed copper because of gradual oxidation at the operating temperature. It is planned to use oxygenfree high conductivity (OFHC) copper clad with 430 stainless steel for protection. Nickel plated or nickel clad copper are also being investigated for suitability. The relative conductivity of stainless steel is about 2.4% of copper while nickel is about 22% of copper. It is obvious that the stainless or nickel must be very thin to reduce the electrical resistance through the clad layer.

3. Hot Seals

A short external cathode lead is necessary to provide the electrical connection between the carbon cathode inside the cell and the bus bar on the outside in order to obtain a battery of a practical size. To accomplish this objective a seal must be resistant to molten salt electrolyte and must prevent air and moisture penetration from the atmosphere. It must be able to withstand thermal cycling between room temperature and cell.

Graded glass seals placed in the annular space between the tungsten lead and the metal sleeve surrounding the tungsten have been studied. At best they are only short-lived seals since they are subject to attack by the electrolyte.

A better match of thermal expansion properties could be achieved by using molybdenum instead of tungsten, but the molybdenum is not stable in LiCl-KCl in the voltage range of this cell system.

Work is proceeding with General Electric and Duramic Products toward obtaining a satisfactory seal. Preliminary samples for testing are expected about the time of publication of this report. A report will be made on the use of these seals in the next report.

G. Facilities

1. Formation Tank Covers

Purchased reagent grade salts contain appreciable amounts of moisture. The removal of the moisture requires heat and vacuum followed by electrolysis. Electrolysis generates oxygen and hydrogen, with oxygen being detrimental to the operations conducted in the dry boxes. To prevent the oxygen entering the dry box via the formation tank, lids were placed over the tank to form a seal. A slight vacuum was created in the tank to evacuate the oxygen and hydrogen. The lids are heavy and awkward to handle.

It has been possible to eliminate the purification procedures by purchasing LiCl-KCl electrolyte drawn from Lithium Corporation's lithium metal production cells. The awkward covers were eliminated in favor of sectional lightweight units for individual cathode attachment and easy handling.

2. Formation Tank Anodes

Previous tank anodes were held against flat plates by retainer screens welded in place. Removal of anodes was impossible.

The utility of the tank anodes was extended by the use of boxes or baskets into which slabs and/or pieces of anode stock could be added as desired. These boxes are adaptations of the familiar anode baskets used by the electroplating industry for electrodeposition of nickel from pieces of rod, buttons, rounds, etc. The total amount of anode material was also increased.

3. $TeCl_4$ Impregnation

Once the cathode carbons are baked-out or heat-treated, they are never exposed to air. The bake-out is accomplished in a vessel similar to an autoclave. After the bake-out, the vessel and its contents are transferred to the inside of a dry box to permit removal of the cathodes and measurement of millivolt drops. The cathodes are then placed into the $TeCl_4$ impregnation vessel which is then sealed. The Te-vessel is removed to the outside atmosphere and placed in a furnace where the $TeCl_4$ is sublimed to impregnate the activated carbon cathodes. This excessive handling of heavy equipment and the danger of insecure closures allowing air contamination needed improvement or elimination. Evidence of leakage was provided by a gradual decrease in $TeCl_4$ pickup dropping from about 85 - 90% to about 80 - 85%. Intrusion of oxygen due to improperly seated seals permits oxygen to change the partial pressures in the vessel thereby reducing the pickup of $TeCl_4$ by the carbon and the probable conversion of a minor portion of the $TeCl_4$ to TeO_2. Either action reduces the effectiveness of the cathode.

To eliminate the danger to operators and the possible cathode contamination, a dry box section has been remodeled for conducting the impregnation step within the confines of the dry box system.

4. Dump Tank

The drying and purification of electrolyte salts was discussed in another section. The retention of water in the salts prior to purification caused the failure of the dump tank. The attack above the level of the molten salt was due to the formation of hydrochloric acid. Installation of the replacement dump tank is essentially complete at this time.

CONCLUSIONS

1. Cathode improvements, necessary to make a viable production size cell (MM-1), were made in current collection as evidenced by the low millivolt drop data and the strength was increased as evidenced by the fact that cells were completed without cathode breakage.
2. Effective wetting of the BN separator with electrolyte was accomplished as evidenced by the equal ampere-hour data from the cathode formation step and the cell test station.
3. All pilot line systems are operating and assembly of modules can begin.
4. Monitoring of individual cathodes during formation is being established to permit adequate quality control to avoid the assembly of low capacity cathodes into cells.
5. Studies are continuing to define the problem behind the eventual increase in holding current.

PLANS FOR FUTURE WORK

1. Assemble and test modules of four cells each including heaters.
2. Assemble appropriate modules into a battery.
3. Continue the study of holding current to define the problem.
4. Design and build a suitable battery charger.
5. Complete the design for a battery production plant.
6. Complete the battery production cost estimate.

LIST OF REFERENCES

1. E. S. Buzzelli, U. S. Patent No. 3,607,413, dated 9/21/71
2. Rightmire, et al, U. S. Patent No. 3,567,516, dated 3/2/71.
3. H. Adams, U. S. Patent No. 3,428,493, dated 2/18/69.
4. Metcalfe, et al, U. S. Patent No. 3,762,954, dated 10/2/73.
5. Rightmire, et al., U. S. Patent No. 3,770,506, dated 11/6/73.
6. Selover, et al., U. S. Patent No. 3,816,242, dated 6/11/74.
7. T. Hacha, U. S. Patent No. 3,645,792, dated 2/29/72.
8. J. Schaefer, "Characteristics of the Molten Salt Carb-Tek® Battery," Paper No. 750148 SAE Congress Detroit, Michigan, 2/26/75.
9. J. E. Metcalfe, "Production and Engineering Methods," AD 006 548, Contract DAAK 02-73-C-0493, Dec. 1974.
10. J. Schaefer, U. S. Patent No. 3,981,743, dated 9/21/76.
11. J. Schaefer, "Production and Engineering Methods for Carb-Tek® Batteries in Fork Lift Trucks," Interim Report dated March 1975 - March 1976.