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HIGH ENERGY DENSITY PELLETIZED ALUMINUM CHLORIDE THERMAL BATTER--ETC(U)
APR 77 J C NARDI, J K ERBACHER, C L HUSSEY

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FRANK J. SEILER RESEARCH LABORATORY

HIGH ENERGY DENSITY PELLETIZED ALUMINUM CHLORIDE THERMAL BATTERIES: PART II. CATHODE SCREENING

APRIL 1977

AIR FORCE SYSTEMS COMMAND

UNITED STATES AIR FORCE

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Pelletized single cell experiments have been conducted to develop a family of potential cathodes for a new, low temperature thermal battery system. The cells utilize a 60.2 a/o lithium-aluminum alloy anode and a molten salt electrolyte of NaCl-saturated $AlCl_3$. They operate at temperatures between 175 and 250°C.

A total of 40 sulfides, oxides, and halides were evaluated and compared to the molybdenum(V) chloride and copper(II) chloride cathodes previously studied. Of this group, three halides, WCl_6, $FeCl_3$, and $TeCl_4$, exhibited long discharge

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lifetimes, high available energy density, and high voltages under load. Various brands of graphite, acetylene black, and various types of metallic current collectors also were evaluated for performance enhancement.

HIGH ENERGY DENSITY PELLETIZED ALUMINUM CHLORIDE THERMAL BATTERIES: PART II. CATHODE SCREENING

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DIRECTORATE OF CHEMICAL SCIENCES
FRANK J. SEILER RESEARCH LABORATORY
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FOREWORD

This report was prepared by the Directorate of Chemical Sciences, Frank J. Seiler Research Laboratory, United States Air Force Academy, Colorado. The work was initiated under Project No. 2303, "Chemistry," Task 2303-F2, "Physical Chemistry and Electrochemistry," Work Unit No. 2303-F2-07, "Pelletized Thermal Batteries."

The report covers work conducted from January 1976 to April 1977. The manuscript was released by the authors for publication in April 1977.

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This technical report has been reviewed and approved.

BEN A. LOVING, LtCol, USAF
Director, Directorate of Chemical Sciences

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INTRODUCTION

Many compounds exist which theoretically would make good thermal battery cathodes. The object of this second cathode screening study was to select those materials compatable with the electrolyte (NaCl-saturated $AlCl_3$) and anode (60.2 a/o LiAl alloy) which would warrant further, more detailed investigations. Compounds investigated during this study were identified as potential cathodes during experiments conducted after the completion of the first study in this laboratory in 1976 (1). The electrochemical system utilizes a NaCl-saturated $AlCl_3$ electrolyte, m.p. 158°C. This electrochemical system is a candidate to replace or complement current thermal battery systems operating at much higher temperatures (2). Previous work in this laboratory (3,4) revealed that molybdenum(V) chloride and copper(II) chloride were potential cathodes for pelletized thermal batteries utilizing this electrolyte. The performance of these cathodes will be used to provide a "baseline" for the present study.

A total of 40 inorganic compounds was tested: this included 24 halides, 9 sulfides, 6 oxides, and elemental sulfur. Various brands of purified and unpurified graphites, acetylene black, and several metallic compounds were used as current collectors and were evaluated as to performance enhancement.

The main criteria that were considered in our evaluation were:

1. A reproducible high open circuit voltage, OCV.
2. Thermal efficiency.
3. Chemical compatibility with the electrolyte.
4. Coulombic efficiency.

5. Low solubility of the cathode material.

6. Long discharge life to 80% of the initial closed circuit voltage, ICCV.

7. Low polarization as evidenced by a low iR drop under load.

In a typical operational battery configuration, voltage constraints imposed by the electronic circuitry often require voltage cutoffs of 80% of peak voltage. Based on this criterion, the compounds tested in this study were ordered according to their delivered energy density to this voltage.

The results of this study are preliminary in that the cell configuration tested had been optimized specifically for the $MoCl_5$ cathode, which is not necessarily the optimum for other cathodes. Those compounds showing promise will be optimized in a manner similar to that undertaken in the $MoCl_5$ study (4).

EXPERIMENTAL

Electrolyte preparation, pellet fabrication, and cell discharge experiments were described previously (1). The individual anode and separating electrolyte weights were identical to those used in the LiAl/$NaAlCl_4$/$MoCl_5$ optimized single cell (4) and were unchanged for all tests (see Table I). The graphite and electrolyte content in the cathode compartment were also identical to those in the $MoCl_5$ cell. To facilitate direct comparison with cells containing a $MoCl_5$ cathode, the same amount of each new cathode material, 3.44 millimoles, was used. Several of the compounds evaluated were known to be semiconductor materials and were tested both with and without the addition of powdered graphite. A complete pelletized single cell is illustrated in Figure 1.

Anode: 60.2 a/o lithium-aluminum powder was obtained from the Foote Mineral Company and used without further treatment.

Cathode: The Results and Discussion section contains information on the source and purity of the cathode materials tested. Those compounds not specifically discussed were a minimum 99.0% pure and obtained from the Alfa-Ventron Corporation or the Research Organic/Inorganic Chemical Corporation (ROC/RIC). Graphite (grade #38) and acetylene black were used as obtained from the Fisher Scientific Company. Superior graphite flakes were purified at 600°C under a chlorine atmosphere using a procedure developed in this laboratory (1). Some of the Fisher graphite samples were purified by boiling for one-half hour in concentrated HCl. This graphite was then washed with distilled water, suction filtered and dried in an oven.

Current Collectors: Five mil nickel foil (Alfa-Ventron), 99.98% pure,

Table I. Typical Single Cell Composition

Section	Weight (g)	Component
Anode	0.27	60.2 a/o LiAl
Separator	0.78	EB Mixture[a]
Cathode	0.64	EB Mixture[a]
	b	Cathode Material
	0.23	Graphite

[a]EB Mixture: Electrolyte (49.85 m/o $AlCl_3$ - 50.15 m/o NaCl) + Binder (10 w/o Cab-O-Sil)

[b]3.44 millimoles of cathode material

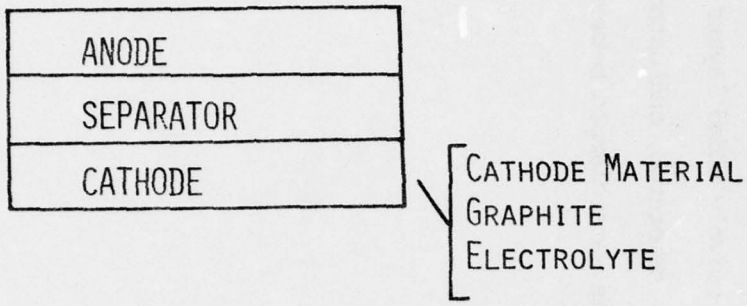

Figure 1. Typical Trilayer Single Cell Configuration

was cut into 1.125 inch diameter circles with tab. The collectors were burnished with emery paper, washed with distilled water and acetone, stored under dry argon, and again lightly burnished before use.

Pellet Fabrication: Three-layer pellets were formed in all cases. Pressures used to compact the successively added layers were 16,700, 23,000 and 29,200 lb/in^2 for anode, separator, and cathode layers respectively. The assembled pellets were clamped between the platens of the cell tester at 4.5 lb/in^2.

RESULTS AND DISCUSSION

The data obtained during this investigation are summarized in Tables II through IX and Figures 2 through 10. The tables give the open circuit voltage (OCV), initial closed circuit voltage (ICCV), the active lifetime to 80% ICCV, and delivered energy density at 80% ICCV. The accompanying figures represent the discharge curves to zero volts for each of the top ten cathodes as determined in this evaluation, based on the 80% ICCV lifetime criteria. The $MoCl_5$ and $CuCl_2$ cathode discharge curves as investigated in previous studies (3,4) are shown in Figure 2 for comparison.

An important consideration when analyzing the experimental data is the application intended for the particular electrochemical system. For example, $MoCl_5$ with its high OCV could be used as a high energy output thermal battery whereas $CuCl_2$ would make an excellent low energy output, long lifetime thermal battery as evidenced by its lower OCV but long, flatter discharge plateau. To obtain the advantages of both cathodes, a thermal battery could be developed using a mixture of $MoCl_5$ and $CuCl_2$ as the cathode. Mixed cells of this type will be examined in future studies.

In view of their noticeably superior performance, the following cathodes were investigated in detail.

$\underline{WCl_6}$: The WCl_6 cathode (ROC/RIC, 99% pure) gave a cell OCV which was between that of $MoCl_5$ and $CuCl_2$. The initial iR drop for a cell containing WCl_6 was similar to these cells, but a steeper drop in the cell voltage discharge curve led to a shorter life and available energy density to 80% ICCV at 175°C and 15 mA/cm^2. At 250°C and 15 mA/cm^2,

Table II. Discharge Results for the Metal Chlorides at 175°C and 15 mA/cm^2

Chloride	OCV (Volts)	ICCV (Volts)	80% ICCV Lifetime (Min)	80% ICCV Energy Density (W-hr/lb)
$MoCl_5$	2.40	2.31	30.0	16.3
$CuCl_2$	1.83	1.72	36.5	13.8
WCl_6	2.16	2.05	23.7	9.1
$FeCl_3$	2.29	2.17	13.3	7.9
$TeCl_4$	1.73	1.66	15.7	6.0
$NbCl_5$	1.70	1.59	11.0	3.8
$HgCl_2$	1.78	1.68	9.7	3.8
$CoCl_2$	1.14	0.86	10.9	2.5
$MoCl_3$	2.11	1.97	3.6	1.6
VCl_3	1.99	1.80	3.4	1.6
$BiCl_3$	1.33	1.13	5.0	1.23
$CrCl_3$	1.64	1.33	2.2	0.76
$TaCl_5$	1.58	1.29	2.9	0.73
$RuCl_3$	1.72	1.57	1.6	0.64
$NiCl_2$	1.35	1.01	2.0	0.52
$CuCl$*	1.85	1.76	0.7	0.37
	1.07	0.86	45.6	12.2
$CrCl_2$	1.20	1.11	0.6	0.18
$ZrCl_4$	1.14	0.87	0.4	0.10
$MnCl_2$	1.39	1.08	0.02	0.05
$FeCl_2$*	1.64	1.50	0.01	0.04
	0.63	0.50	25.0	3.56
$TiCl_3$	1.18	0.94	0.01	0.02

*Double entry discussed in text

Table III. Discharge Results for Metal Oxides at 175°C and 15 mA/cm^2

Oxide	OCV (Volts)	ICCV (Volts)	80% ICCV Lifetime (Min)	80% ICCV Energy Density (W-hr/lb)
Fe_2O_3	1.86	1.72	10.8	5.0
CuO	1.85	1.76	8.3	4.5
Cu_2O	1.09	1.00	5.5	1.6
V_2O_5	2.20	2.01	1.9	1.0
Fe_3O_4	1.59	1.44	1.8	0.6
Sb_2O_3	0.45	0.12	0.3	0.0

Table IV. Discharge Results for Metal Sulfides and Sulfur at 175°C and 15 mA/cm^2

Sulfide	OCV (Volts)	ICCV (Volts)	80% ICCV Lifetime (Min)	80% ICCV Energy Density (W-hr/lb)
Cu_2S	1.43	1.35	7.0	2.50
CuS	1.68	1.58	3.9	1.81
S_8	1.48	1.10	3.2	0.77
TiS_2	1.43	1.33	2.0	0.70
MnS	1.21	0.96	2.2	0.60
TaS_2	1.23	1.05	0.8	0.21
Tl_2S	1.41	1.21	1.0	0.21
ZrS_2	0.74	0.62	1.0	0.14
FeS_2	0.98	0.71	0.2	0.03
FeS	0.94	0.77	0.1	0.03

Table V. Discharge Results for the Metal Florides and Bromides at 175°C and 15 mA/cm^2

Halide	OCV (Volts)	ICCV (Volts)	80% ICCV Lifetime (Min)	80% ICCV Energy Density (W-hr/lb)
SbF_5	2.18	2.00	2.2	1.09
VF_4	1.88	1.66	1.4	0.63
VBr_3	1.60	1.44	0.1	0.26
$MoBr_2$	1.38	1.01	0.1	0.12

Table VI. Discharge Results for the "Top Ten" Potential Cathodes at 175°C and 15 mA/cm^2

Cathode	OCV (Volts)	ICCV (Volts)	80% ICCV Lifetime (Min)	80% ICCV Energy Density (W-hr/lb)
$MoCl_5$	2.40	2.31	30.0	16.3
$CuCl_2$	1.83	1.72	36.5	13.8
WCl_6	2.16	2.05	23.7	9.1
$FeCl_3$	2.20	2.17	13.3	7.9
$TeCl_4$	1.73	1.66	15.7	6.0
Fe_2O_3	1.86	1.72	10.8	5.0
CuO	1.85	1.76	8.3	4.5
$NbCl_5$	1.70	1.59	11.0	3.8
$HgCl_2$	1.78	1.68	9.7	3.8
Cu_2S	1.43	1.35	7.0	2.5

Table VII. Cathode Performance Comparison to 80% ICCV as a Function of Temperature (Discharge Rate = 15 mA/cm^2)

	175°C		250°C	
Cathode	Time (Min)	Energy Density (W-hr/lb)	Time (Min)	Energy Density (W-hr/lb)
$MoCl_5$	30.0	16.3	12.3	6.80
$CuCl_2$	36.5	13.8	5.33	3.25
WCl_6	23.7	9.10	6.33	2.41
$FeCl_3$	13.3	7.90	12.3	7.61
$TeCl_4$	15.7	6.00	7.33	2.65

Table VIII. Single Cell Performance for Various Internal Current Collectors ($MoCl_5$ Cathode at 175°C and 15 mA/cm^2)

	80% ICCV	
Current Collector	Time (Min)	Energy Density (W-hr/lb)
Graphite (grade #38)	30.0	16.3
Nickel (5 micron)	1.0	0.6
Molybdenum (200 mesh)	0.2	0.2

Table IX. Single Cell Performance for Various Types of Graphite ($MoCl_5$ Cathode at 175°C and 15 mA/cm^2)

	80% ICCV	
Graphite	Time (Min)	Energy Density (W-hr/lb)
Fisher (HCl boiled)	31.7	17.1
Fisher (unpurified)	30.0	16.3
Superior (purified)	24.3	13.2

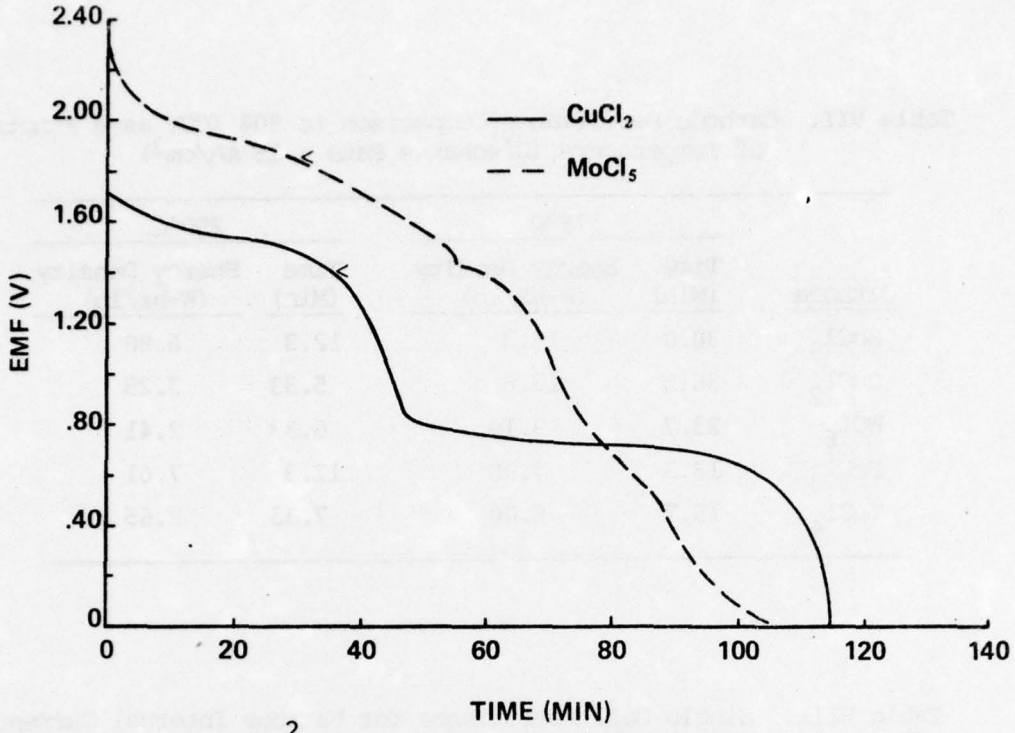

Figure 2. 15 mA/cm^2 Constant Current Discharge Curves for $MoCl_5$ and $CuCl_2$ Cathodes at 175°C. <, 80% ICCV.

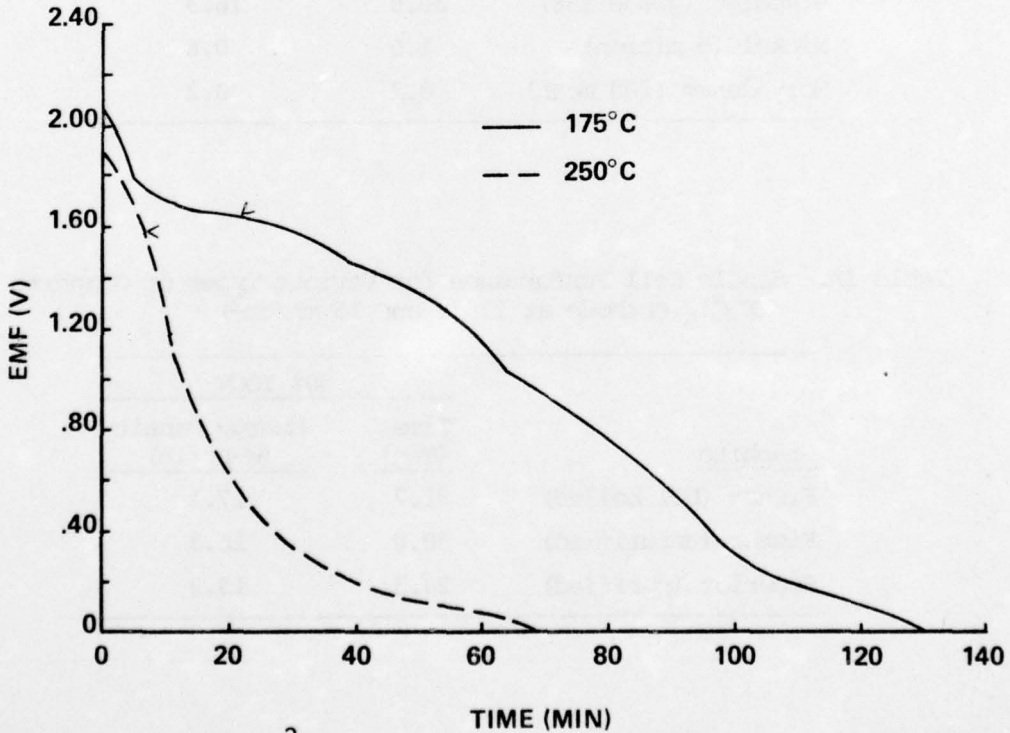

Figure 3. 15 mA/cm^2 Constant Current Discharge Curves for the WCl_6 Cathode at 175 and 250°C. <, 80% ICCV.

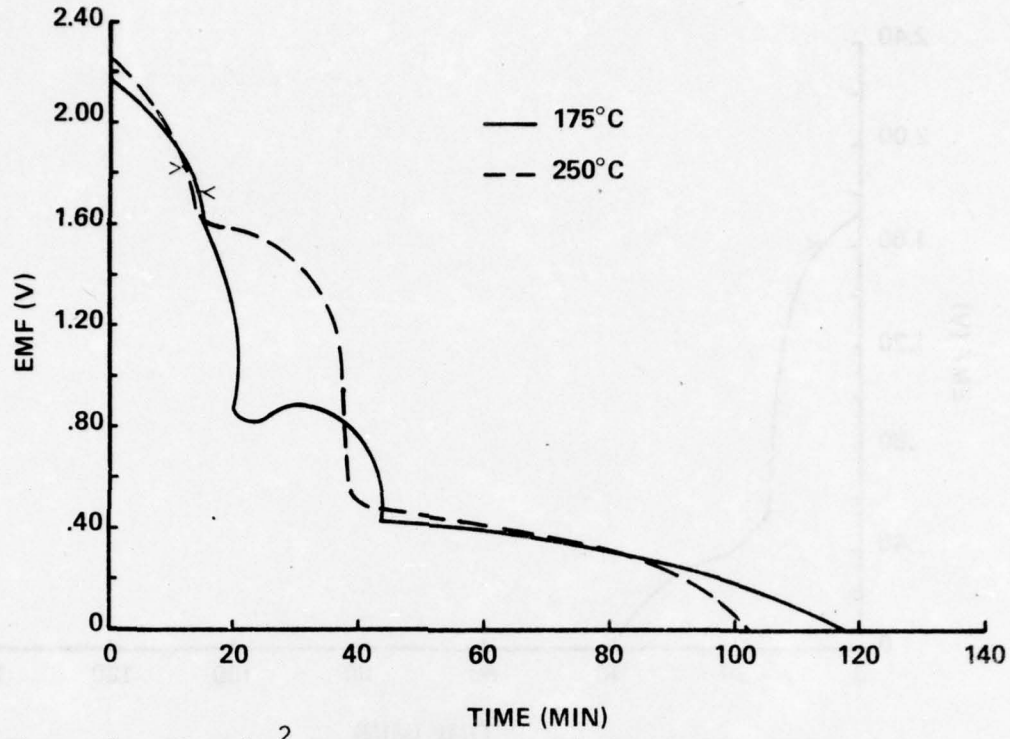

Figure 4. 15 mA/cm^2 Constant Current Discharge Curves for the $FeCl_3$ Cathode at 175 and 250°C. <, 80% ICCV.

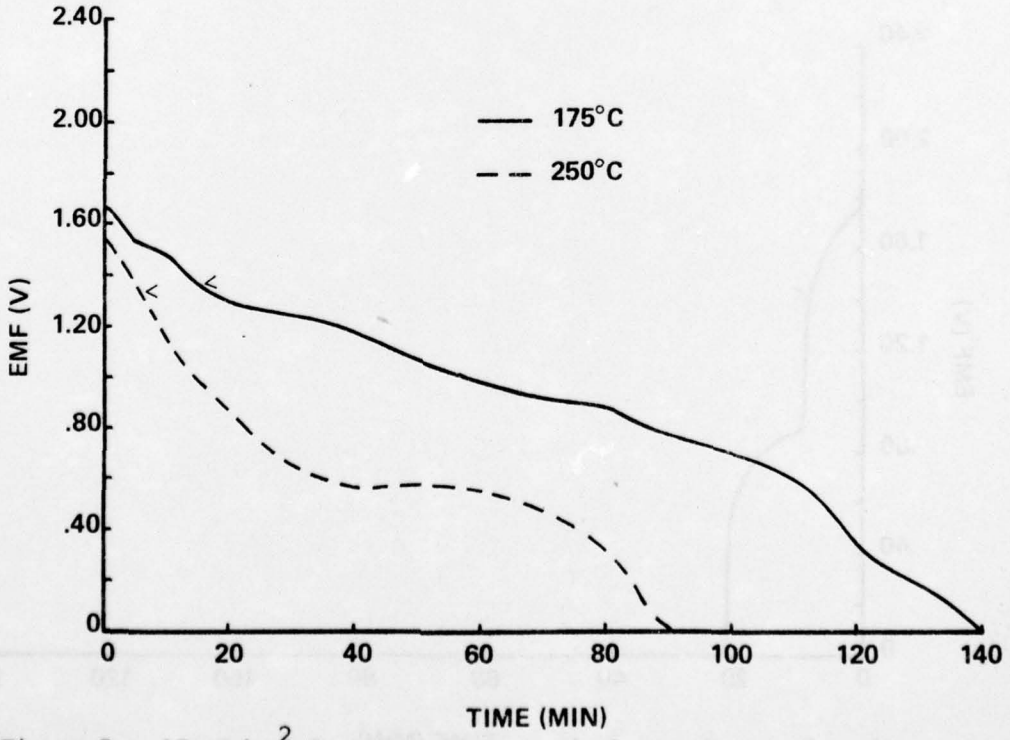

Figure 5. 15 mA/cm^2 Constant Current Discharge Curves for the $TeCl_4$ Cathode at 175 and 250°C. <, 80% ICCV.

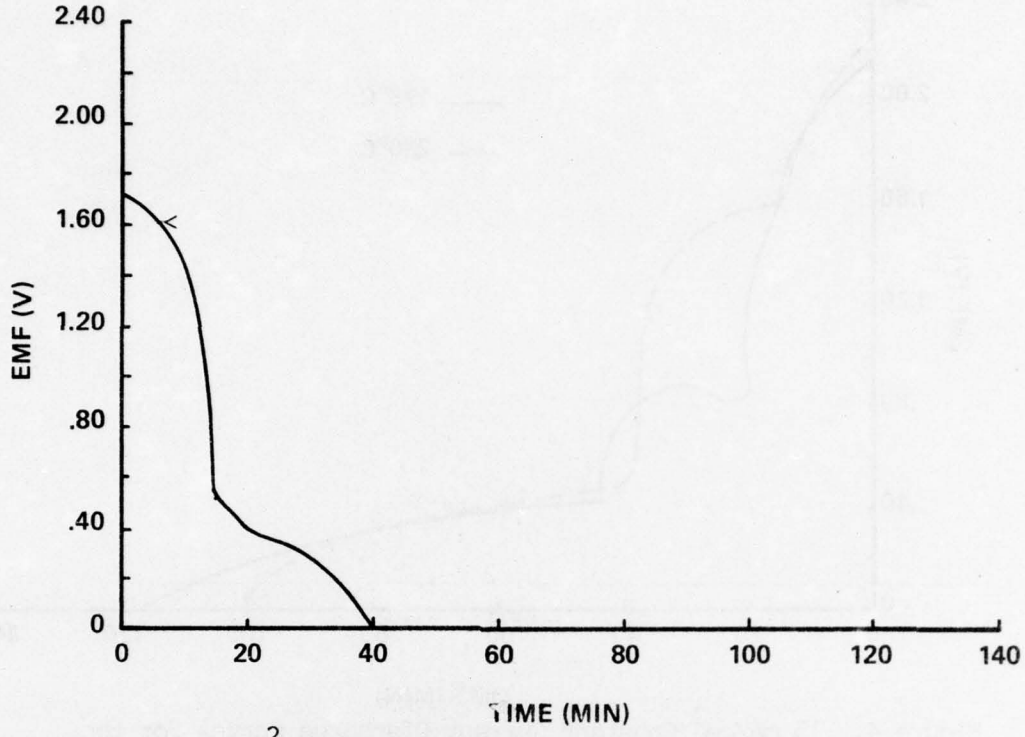

Figure 6. 15 mA/cm^2 Constant Current Discharge Curve for the Fe_2O_3 Cathode at 175°C. <, 80% ICCV.

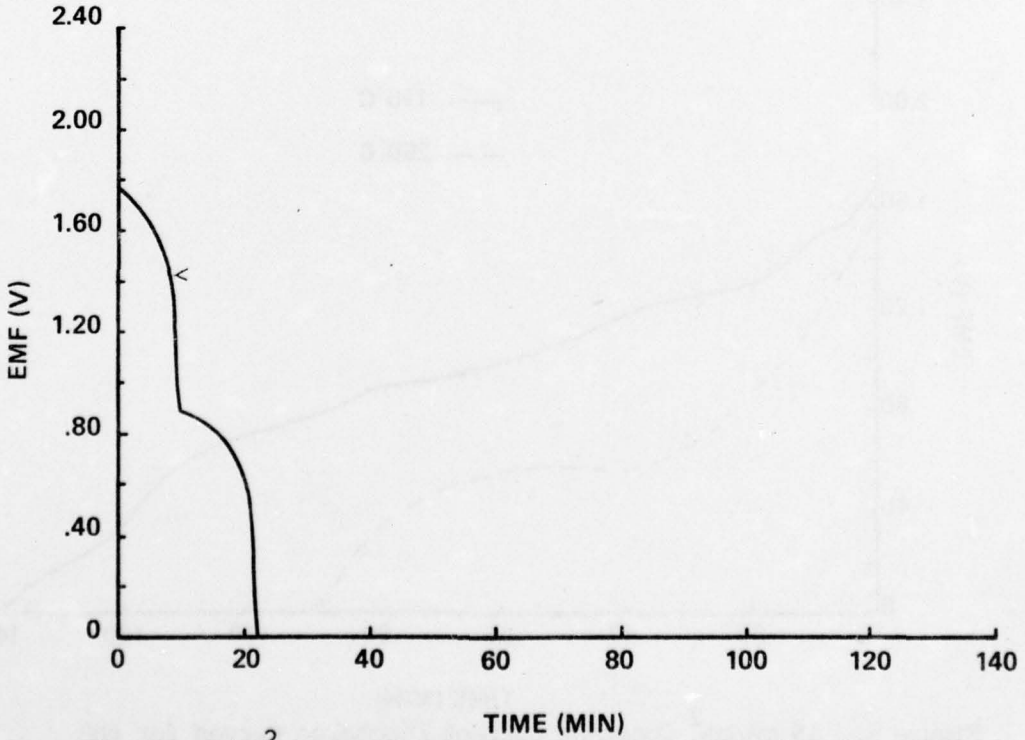

Figure 7. 15 mA/cm^2 Constant Current Discharge Curve for the CuO Cathode at 175°C. <, 80% ICCV.

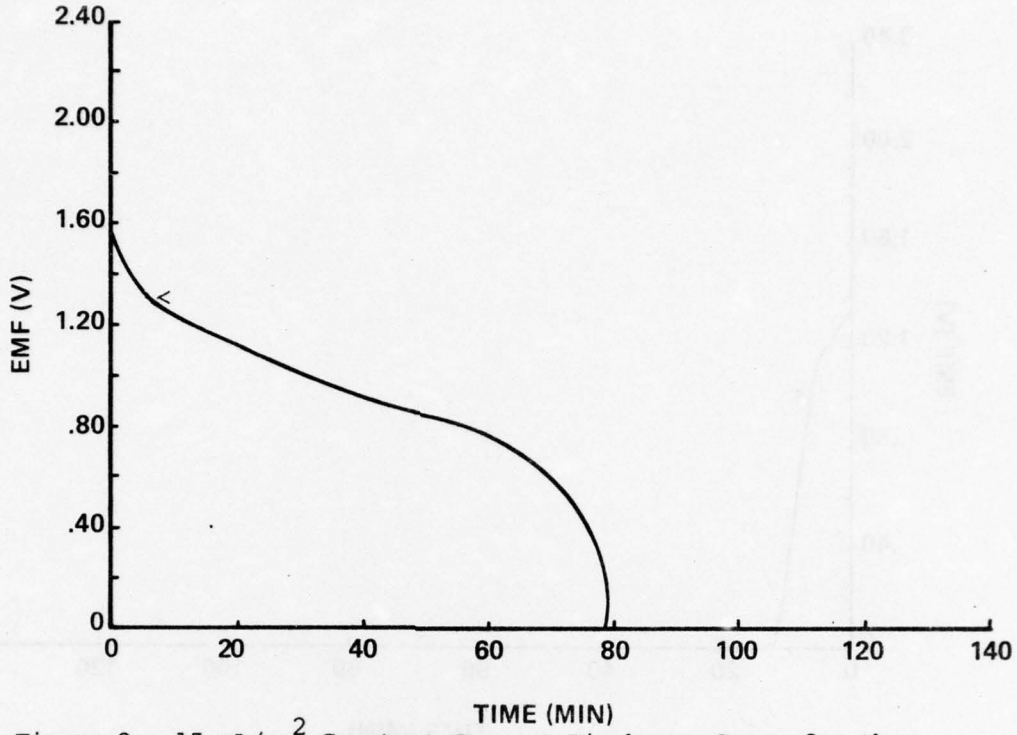

Figure 8. 15 mA/cm^2 Constant Current Discharge Curve for the $NbCl_5$ Cathode at 175°C. <, 80% ICCV.

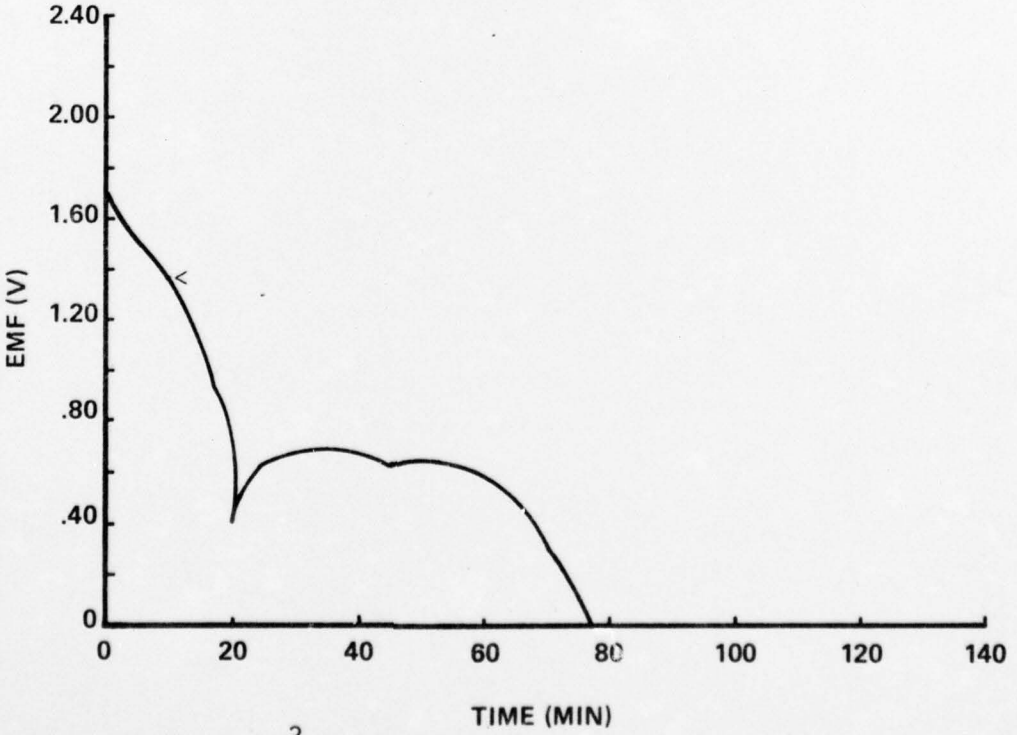

Figure 9. 15 mA/cm^2 Constant Current Discharge Curve for the $HgCl_2$ Cathode at 175°C. <, 80% ICCV.

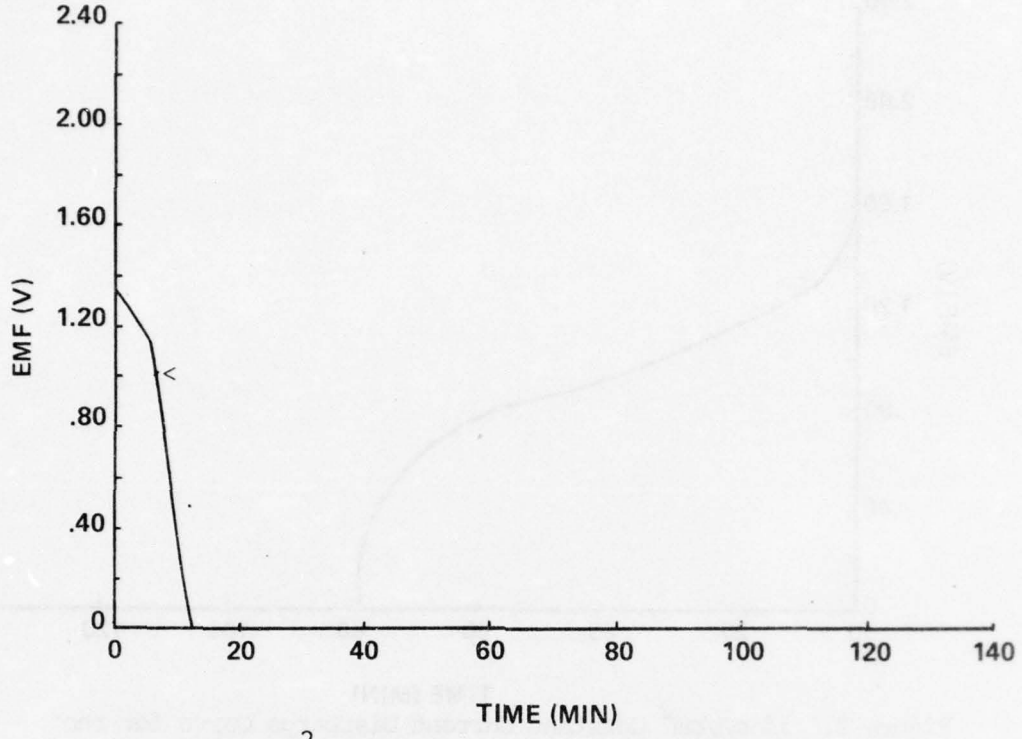

Figure 10. 15 mA/cm^2 Constant Current Discharge Curve for the Cu_2S Cathode at 175°C. <, 80% ICCV.

the performance deteriorated drastically, as shown in Figure 3 and Table VII.

$\underline{FeCl_3}$: The $FeCl_3$ (MC/B, 99% pure) had a very small iR drop from a relatively high open circuit potential of 2.20 volts. It has the additional advantage of being cheap and readily available. The $FeCl_3$ discharge curve (Figure 4) at 175°C and 15 mA/cm^2 showed a voltage increase after the first discharge plateau had been attained. We suggest that this increase is due to the dissolution of an insoluble $FeCl_2$ surface film formed on the cathode during the initial discharge (6). At temperatures of 250°C and above, the discharge curve (Figure 4) is flatter with no voltage irregularities and there is only a slight decrease in the 80% ICCV performance. Because there is little degradation in performance at higher temperatures, the $FeCl_3$ cathode can be operated over a wider temperature range. This would simplify the manufacturing and quality control of $FeCl_3$ thermal batteries, especially in the selection of heat paper of a suitable caloric value.

$\underline{TeCl_4}$: The $TeCl_4$ cathode (ROC/RIC, 99% pure) gave an extremely flat discharge curve (Figure 5), the flattest yet observed with the exception of $CuCl_2$ (Figure 2). It also had the longest lifetime to zero volts, 140 minutes. However, the 80% ICCV performance data suggest an inferior output as compared to the WCl_6 or $FeCl_3$ cathodes at both 175 and 250°C (Figure 5). In addition, $TeCl_4$ is a highly poisonous chemical and its use as a cathode may present manufacturing and handling problems for production thermal batteries.

The discharge behavior of the other cathodes evaluated fell well below the performance of the above mentioned cathodes and thus will not be discussed in any detail. However, an explanation of the double

entries in Table II for the $FeCl_2$ and CuCl cathodes is necessary.

The discharge curve of CuCl (Baker, 96.4% pure) shows a significant voltage drop upon application of a 15 mA/cm^2 load. As a result the 80% ICCV lifetime and energy density were very low. If, however, the 80% ICCV performance is measured from an OCV value determined in an electrochemical study of copper in the tetrachloroaluminate melt (6), the 80% ICCV output is more respectable. The 80% lifetime was 45.6 minutes and energy density was 12.2 W-hr/lb. The OCV of copper(I) chloride which was observed in this study was similar to that of copper(II) chloride, indicating the probable presence of copper(II) as an impurity in the commercially obtained copper(I) chloride. If all of the copper(II) could be removed, a cathode consisting of only copper(I) chloride would make an excellent long life, low output thermal battery.

A similar argument can be made for the iron(II) chloride cathode. The large voltage drop gave a poor 80% ICCV performance. However, the correct Fe^{2+}/Fe^0 potential was reported to be 0.63V (6). Use of the latter value leads to an 80% ICCV lifetime and energy density of 25.0 minutes and 3.56 W-hr/lb respectively. The actual 1.85V OCV indicates the probable presence of trace quantities of iron(III) in the cathode material. Removal of the iron(III) impurity would improve the performance of the $FeCl_2$ cathode in the same manner as would removal of $CuCl_2$ from CuCl.

Current Collector: The use of molybdenum (Alfa-Ventron, 99.7% pure) or nickel (Alfa-Ventron, 99.5% pure) powders in the place of graphite (Fisher, untreated) as an internal current collector in the cathode did not improve single cell performance, but drastically reduced the 80% ICCV output as shown in Table VIII. Graphite has proven

to be a superior internal current collector and because of cost and purity requirements of the other metallic current collectors tested will continue to be used.

Small quantities (no more than 1% of total pellet weight) of acetylene black in the $LiAl/NaAlCl_4/MoCl_5$ pelletized single cell produced a 2.5% increase in output as compared to a similar cell not containing this material. However, use of acetylene black in the WCl_6 single cell showed no increase in output. This suggests that the optimization of individual cathodes in our electrolyte for use in pelletized single cells in apparently unique. A single cell formula eventually may be deduced after further studies are undertaken.

The absence of graphite when using the semi-electronic conductors ZrS_2 and TiS_2 as cathodes showed no increased performance, as evidenced in the short lifetimes and low energy density at 80% ICCV of these single cells (Table IV). It was anticipated that these cells containing known semi-electronic conductors as cathodes (7), would give better energy density output due to their lower pellet weight with the absence of graphite. The use of graphite with these two compounds also gave poor 80% ICCV results.

Tests were also made to evaluate the effectiveness of various methods to purify graphite. Unpurified graphite and graphites purified by several methods were incorporated into single cells containing $MoCl_5$. Table IX contains the results of these experiments. Fisher graphite purified by treatment in boiling HCl gave the best output at 175°C and 15 mA/cm^2. Untreated Fisher graphite, known to contain impurities, gave the second best output. The poorest performance was graphite obtained from the Superior Graphite Company and subsequently purified,

The use of the purified Fisher graphite did not show a performance increase over the untreated Fisher graphite greater than the relative error established in a previous study (4). However, use of the purified Superior graphite did result in at least a 20% drop in performance.

FUTURE WORK

The three best cathodes selected in this study, WCl_6, $FeCl_3$, and $TeCl_4$ will be investigated in greater detail. Tests will be conducted at different current densities and temperatures to determine whether these potential cathodes might be advantageous for certain thermal battery applications. After optimization of these cathodes, a statistical analysis will determine if a general single cell formula for $AlCl_3$ based pelletized single cells can be formulated.

The role of acetylene black and the effectiveness of various methods to purify graphite will also be studied in greater detail.

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