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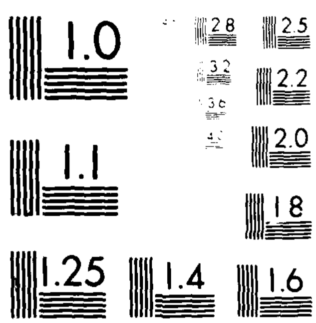

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HIGH EFFICIENCY LITHIUM-THIONYL CHLORIDE CELL

Dr. N. Doddapaneni
Honeywell Power Sources Center
104 Rock Road
Horsham, PA 19044

January 1982

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DCASMA, PHILADELPHIA
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Cathode polarization and discharge performance studies at -20 and -40^{o}F showed that the limiting currents of $Li/SOCl_2$ cells decreased linearly with reciprocal temperature and an Arrhenius temperature dependence of limiting current was not observed between the temperatures -40 and 75^{o}F.

Systematic evaluation of cathode variables such as carbon substrate, thickness and density on the cathode performance was carried out at 0^{o}F —

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and 75°F. Significant improvements in both cathode polarization and performance were achieved with catalyzed cathodes containing 5% Teflon binder.

TABLE OF CONTENTS

LIST OF FIGURES

ABSTRACT

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Systematic evaluation of cathode variables such as carbon substrate, thickness and density on the cathode performance was carried out at 0°F and 75°F. Significant improvements in both cathode polarization and performance were achieved with catalyzed cathodes containing 5% Teflon binder.

I. INTRODUCTION

The $Li/SOCl_2$ system (1-4) has the potential to be one of the best primary batteries having combined characteristics of high rate and high energy density capability, long shelf-life and wide operating temperature range. However, many of these advantages have not been fully derived. The operating capabilities of $Li/SOCl_2$ batteries are limited, to a large extent, by the Teflon bonded porous carbon electrode commonly used as a cathode. Cell failure at high discharge rates and/or low operating temperatures has, as one of its main causes, the high cathodic overpotential resulting from non-uniform current distribution over the porous electrode.

The porous carbon cathode, where the reduction of $SOCl_2$ occurs, has a limited capacity for retaining solid lithium chloride as it precipitates in the pore structure. As the lithium chloride accumulates, the porosity of the electrode is reduced to where mass transport, particularly of the cathode depolarizer, can no longer be maintained at a rate sufficient to support the required current density. When this happens, polarization becomes excessive and denotes the end of useful battery life.

At high rate discharges and low operating temperatures, the cathode polarization problem becomes very severe. Analysis of the porous electrode shows that, at high rate discharges, only a small part of the available surface participates in the electrochemical process.

Cathode polarization and the reaction zone thickness strongly depend on electrode reactions, cathode thickness and composition. Minimization of the effects of these variables on overpotential is essential in order for $Li/SOCl_2$ batteries to be viable electrochemical devices for many of the high rate/low temperature applications.

The objectives of this program, therefore, are to:

a) Evaluate the polarization characteristics of Teflon-bonded porous carbon cathodes.

b) Improve cathode performance at high discharge rates and low operating temperatures.

Polarization characteristics will be determined as a function of cathode thickness, composition and density, applied current density and temperature (-40°F to 75°F). Several experimental techniques such as half-cell measurements, discharge performance characteristics, impedance measurements, and cyclic voltammetry studies will be employed to evaluate the extent of each variable's contribution to the cathode polarization.

Improvement in cathode performance will be made by the use of electrocatalysis. Three catalysts to be employed in this program (5) have shown marked improvements in both cell voltage and cathode capacity. They are*,

Catalyst A = Cobalt Phthalocyanine monomer, CoPc
Catalyst B = Iron Phthalocyanine monomer, FePc
Catalyst C = Polymeric Cobalt Phthalocyanine, $(CoPc)_n$

During the first quarter of this program, we examined:

a) The overpotential of cathodes with and without Catalyst B and Catalyst C, over a temperature range of -40 to 75°F,

b) The effects of catalysts and temperatures on cyclic voltammograms in $LiAlCl_4/SOCl_2$ electrolyte solutions.

c) The effects of operating temperatures (-40 to 75°F) on the conductivity and viscosity of the electrolyte solutions ($LiAlCl_4/SOCl_2$).

During this reporting period, we have completed the overpotential measurements at -20 and -40°F on our present baseline cathodes with and without catalysts. These cathodes contained 5% Teflon binder and had a thickness of 0.020 inch. Furthermore, we have systematically evaluated the effect of cathode thickness, amount of Teflon binder and cathode substrates, on the cathode performance. The two types of carbon substrates examined were:

a) 50% compressed grade Shawinigan Acetylene Black (50% SAB), and
b) 100% Compressed grade Shawinigan Acetylene Black (100% SAB).

In addition, the effect of catalyst doping in 100% SAB was evaluated.

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* Patents pending

II. EVALUATION OF CATHODE OVERPOTENTIAL

A. LABORATORY CELL MEASUREMENTS

1. Experimental

The laboratory cell design and the description of cell components used in the evaluation of cathode overpotential presented here are described in the first quarterly report (6).

2. Half-Cell Measurements

The effect of operating the $Li/SOCl_2$ cells at -20 and -40°F on the cathode overpotential was examined. In addition, the effect of catalyst doping [FePc and $(CoPc)_n$] on the cathode polarization was evaluated. The potential dependence of the current was determined by measuring the steady-state potential versus the lithium reference electrode at various controlled currents.

The polarization characteristics of three electrodes (one baseline and two catalyst doped) were examined at operating temperatures of -20 and -40°F and the results were plotted in Figures 1 and 2, respectively. Electrolyte insoluble $(CoPc)_n$ catalyst doping was achieved by impregnating the catalyst onto 100% SAB from a concentrated H_2SO_4 solution whereas FePc doping was carried out by dissolving it in the electrolyte before $Li/SOCl_2$ cells with baseline cathodes were activated. The cathode density and surface area for baseline cathodes and FePc doped cathodes in $Li/SOCl_2$ were, therefore, equal. However, only a slight difference in cathode density and surface were observed with $(CoPc)_n$ doped cathodes when compared to the baseline cathode.

Catalyzed cathodes reduced the cathode polarization significantly. This effect was anticipated and the activation polarization associated with $SOCl_2$ adsorption and electron transfer processes was minimized by electrocatalytic reduction of $SOCl_2$. However, the electrode polarization increases as the operating temperature decreases. And also, $Li/SOCl_2$ cell limiting current decreases with decreasing temperatures. The relationships between the limiting current, limiting current density and operating temperatures were plotted in Figures 3 and 4, respectively.

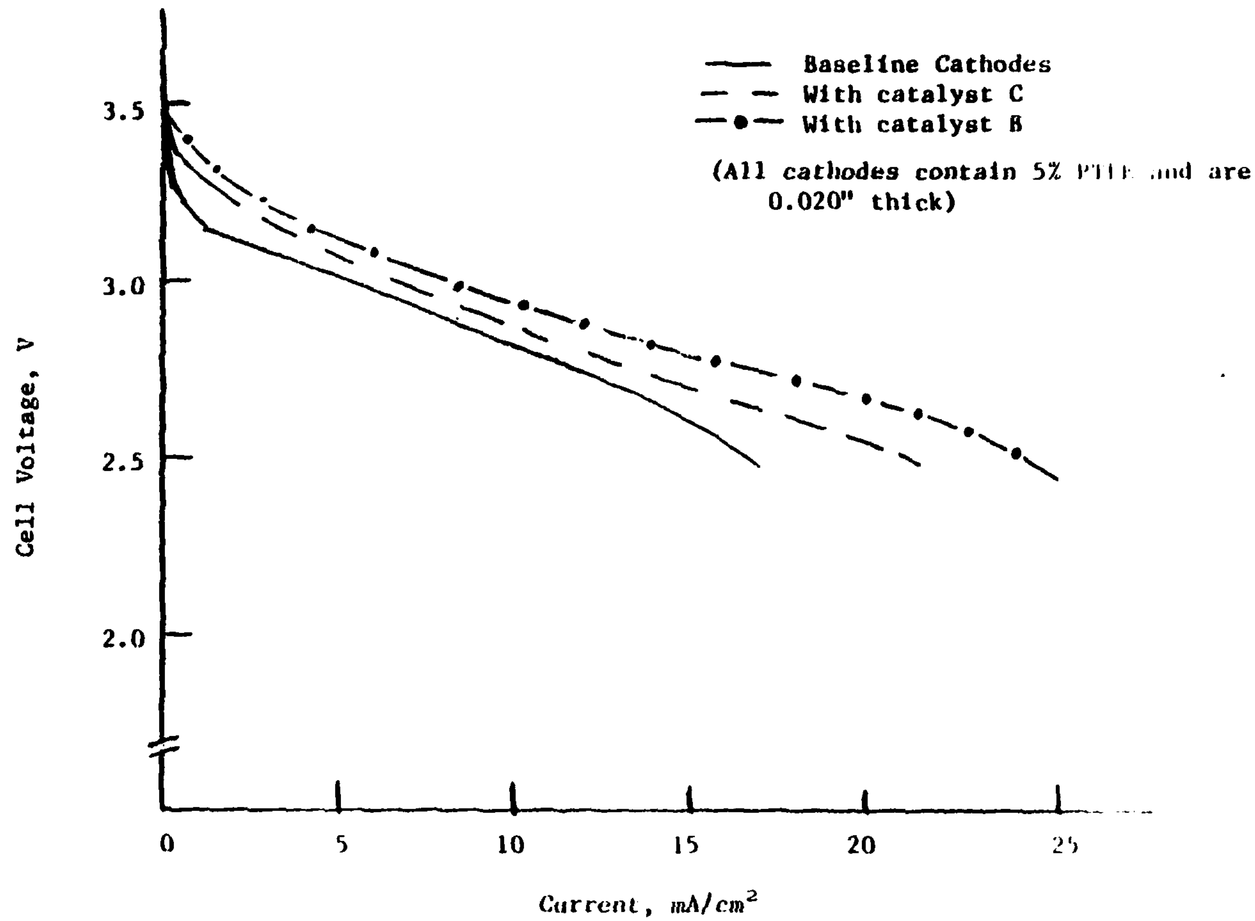

Figure 1. Polarization Characteristics of $Li/SOCl_2$ Cells at $-20^{\circ}F$.

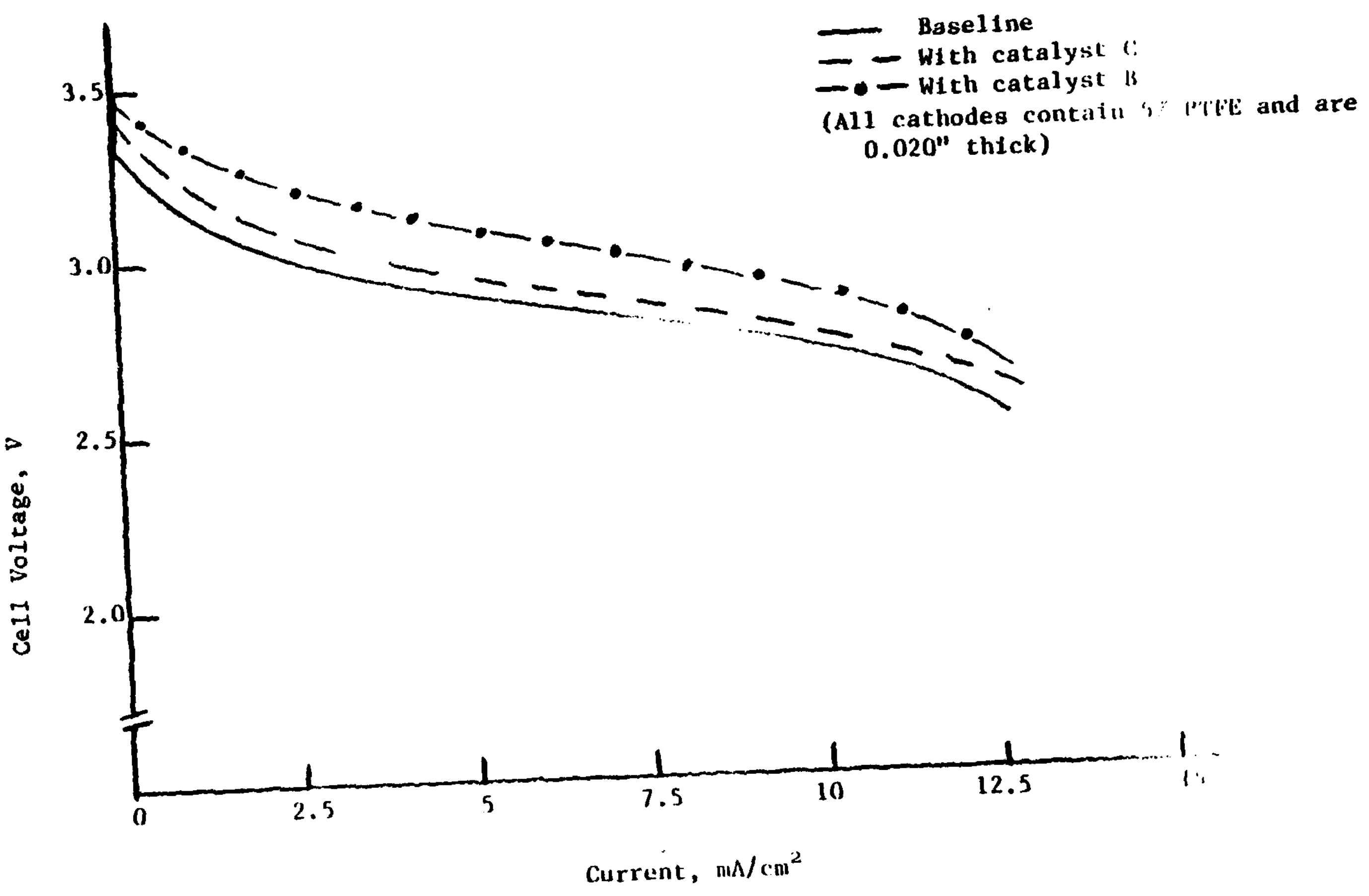

Figure 2. Polarization Characteristics of $Li/SOCl_2$ Cells at $-40^{\circ}F$

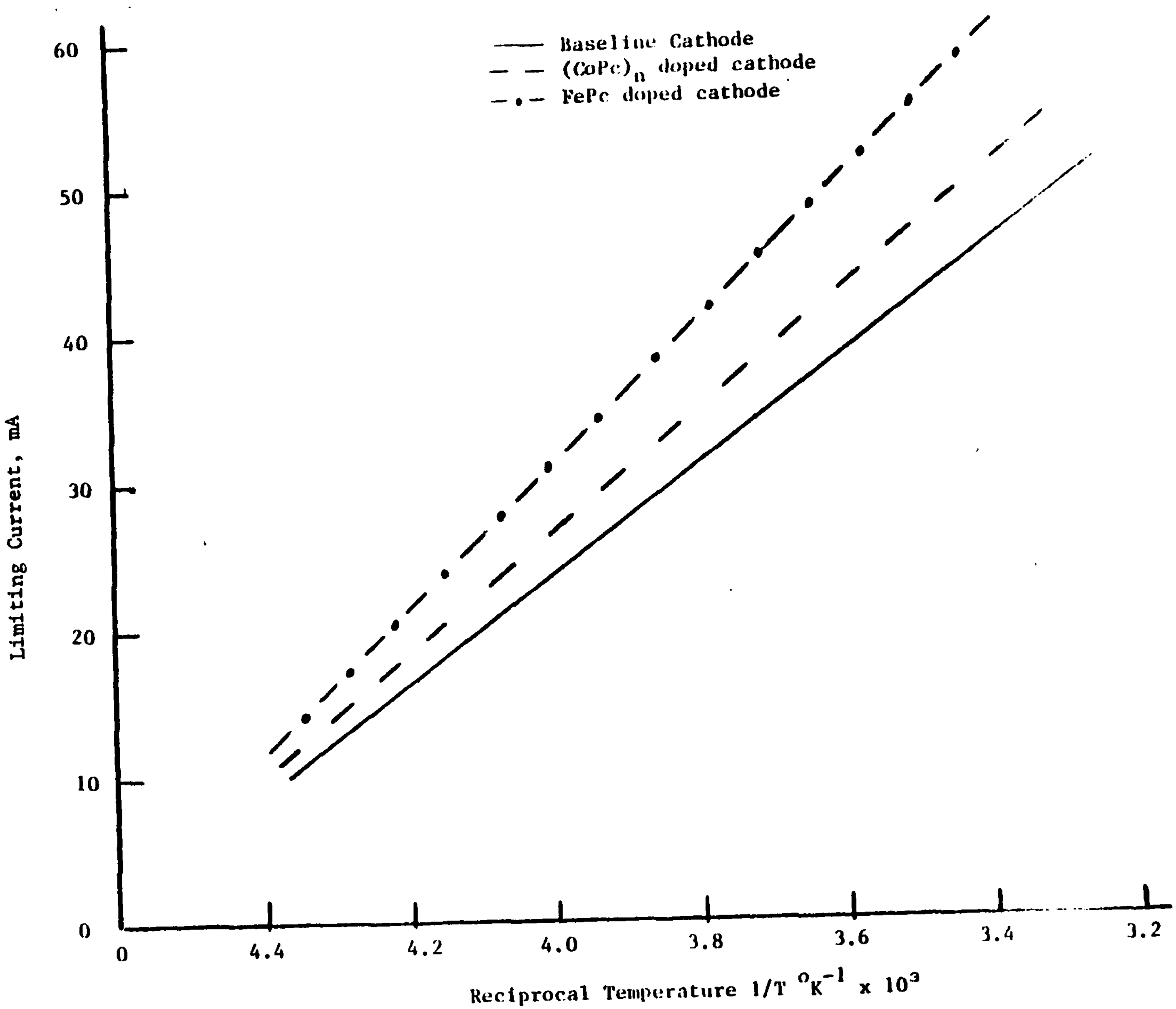

Figure 3. Effect of Operating Temperature on Laboratory $Li/SOCl_2$ Cell Limiting Current

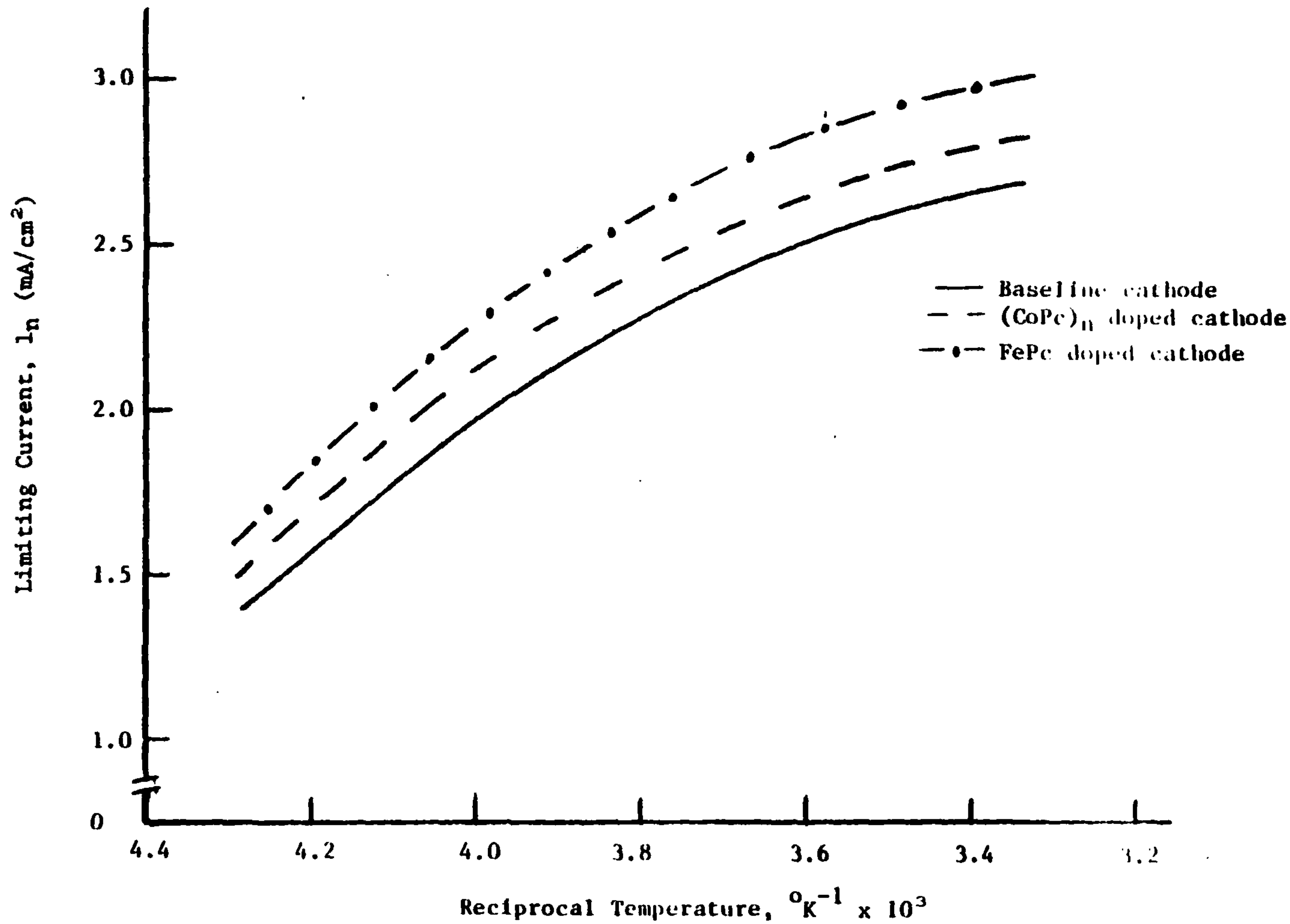

Figure 4. Effect of Operating Temperature on Ln of Cell Limiting Current Density

A linear relationship between limiting current and reciprocal temperature (Figure 3) exists for all three types of cathodes. It is interesting to note that a similar relationship between electrolyte conductivity and reciprocal temperature exists as reported in the first quarterly report. An Arrhenius-type temperature dependency of limiting current was not observed as shown in Figure 4. As the temperature decreases, the slope of the curve increases indicative of changes in the mass transport rates. Beside mass transport problems of cathode depolarizer, electrochemical product adsorption on the carbon surface and glass mat separator porosity contribute to the lowering of $Li/SOCl_2$ limiting current at low operating temperatures.

3. Discharge Characteristics

Discharge performance of $Li/SOCl_2$ cells at -20 and -40°F were studied. Cells were activated at room temperature and then cooled to appropriate temperatures. During cooling period, the cells were kept under a constant current load of 0.2 mA/cm^2 in order to avoid passivation problems.

In Figure 5, the discharge characteristics corresponding to current density of 20 mA/cm^2 in $Li/SOCl_2$ cells using three different cathodes were compared. Cathode performance at 5 and 10 mA/cm^2 at -20°F was reported in (6). A significant cathode polarization is observed without catalyst at 20 mA/cm^2 rate.

At operating temperature of -40°F, the $Li/SOCl_2$ cells with baseline cathodes cannot support a discharge current density of 20 mA/cm^2 for more than a few minutes. For this reason, the cells were discharged at 5 and 15 mA/cm^2. Dramatic improvements in cathode performance and cell voltage were obtained with catalyzed cathodes as shown in Figures 6 and 7.

B. KINETIC AND MECHANISTIC STUDIES

Cyclic voltammetric and rotating disc electrode studies in 1.0M $LiAlCl_4/SOCl_2$ solution at various cathode surfaces were continued. Initial results obtained with rotating disc electrode (glassy carbon) showed problems with reproducibility. Therefore, it is planned to employ pressure annealed pyrolytic graphite and platinum as rotating disc electrodes. Presently these electrodes are being fabricated and further studies will be made in the next quarter.

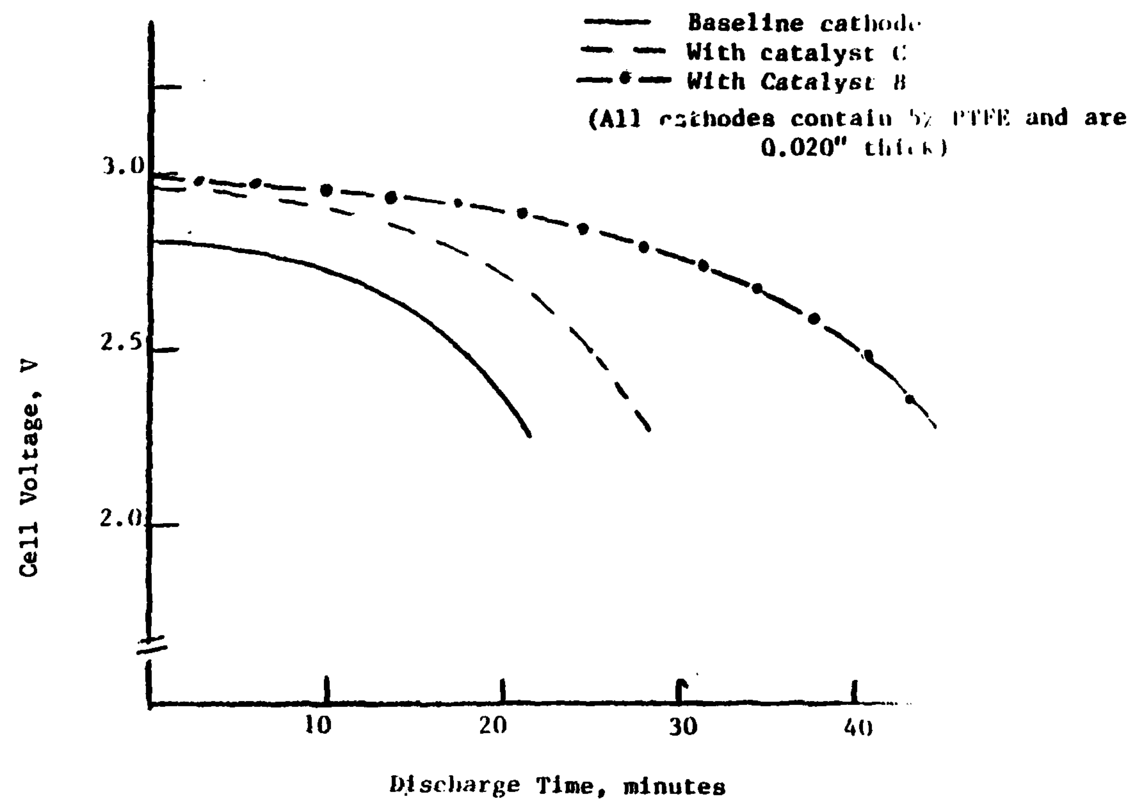

Figure 5. Discharge Characteristics of $Li/SOCl_2$ Cells at 20 mA/cm^2 and -20^{o}F

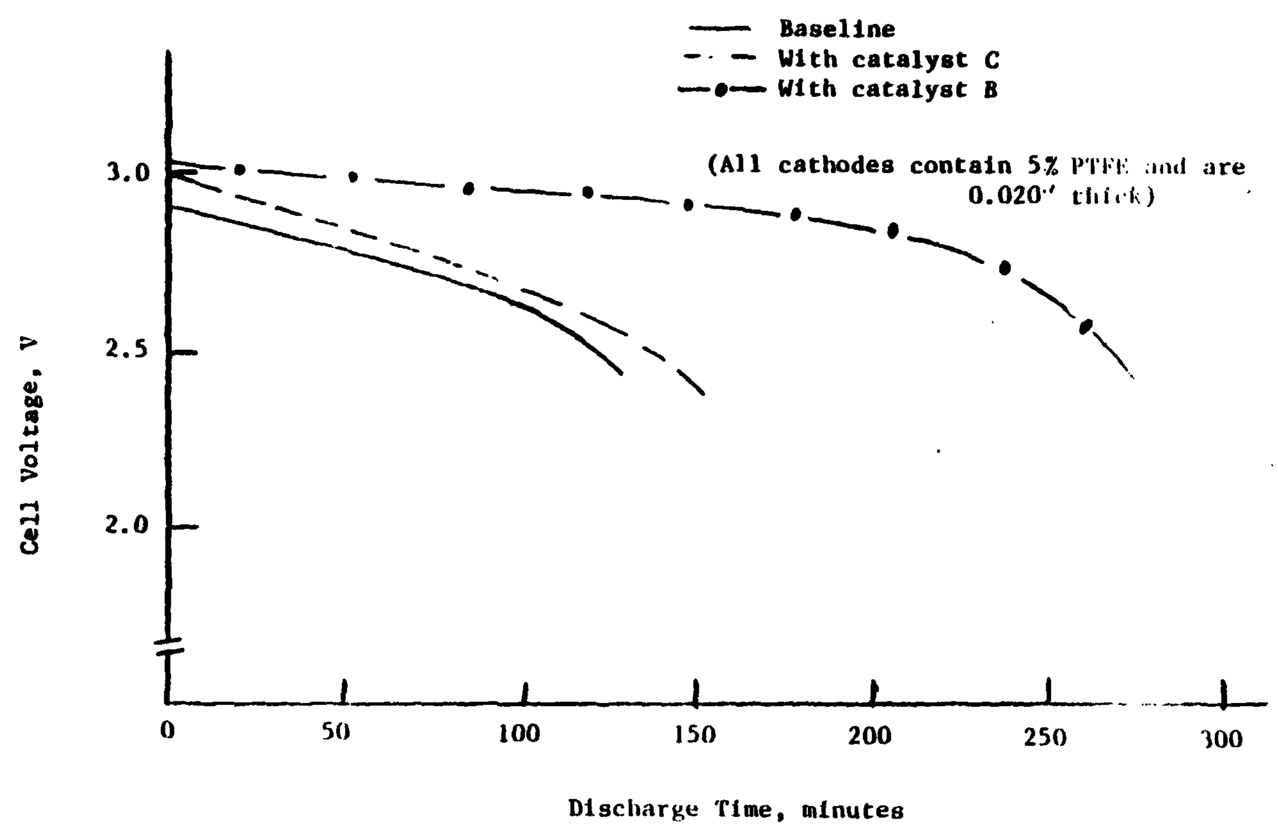

Figure 6. Discharge Characteristics of $Li/SOCl_2$ Cells at 5 mA/cm^2 and $-40^{o}F$.

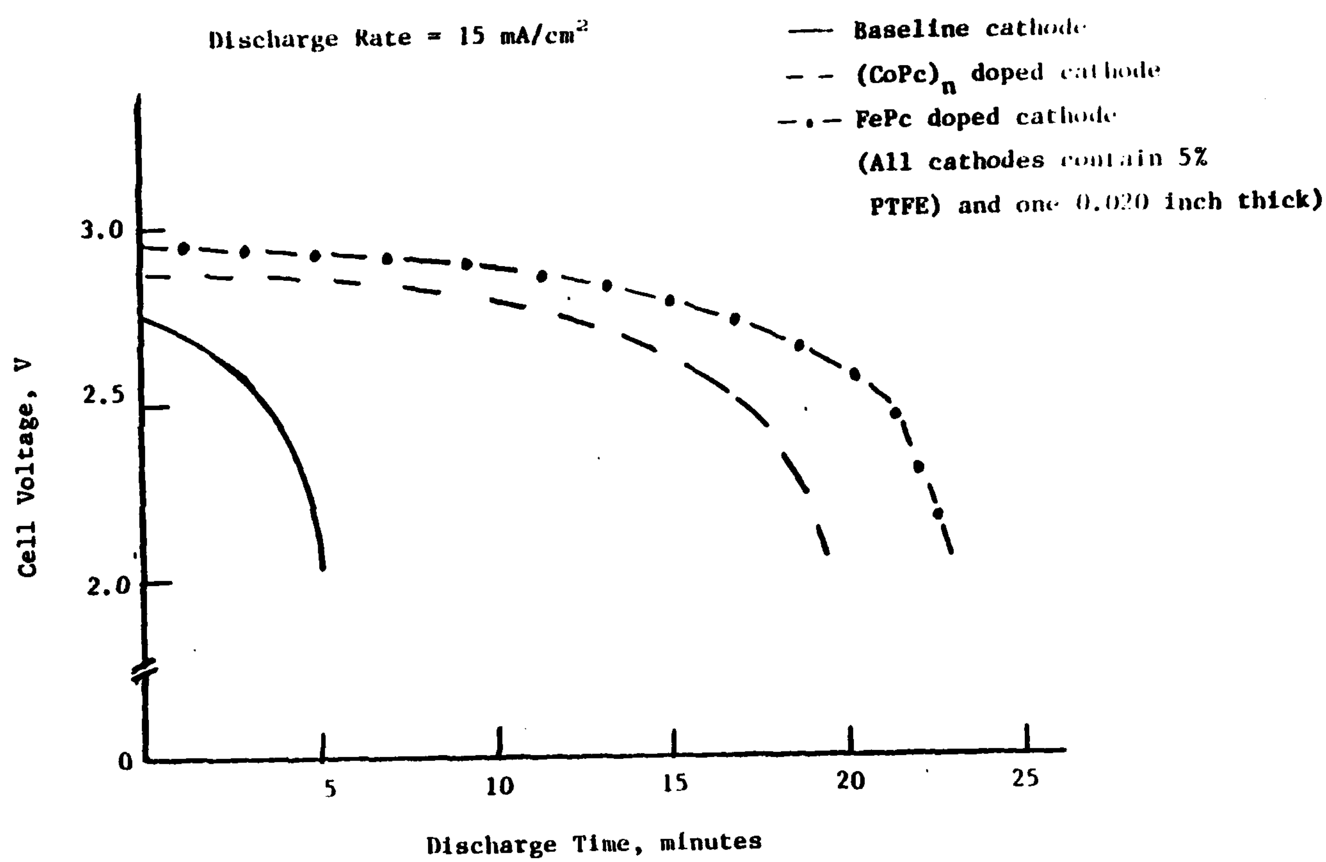

Figure 7. Discharge Performance of Laboratory $Li/SOCl_2$ Cells at 15 mA/cm^2 and -40°F.

III. CATHODE PERFORMANCE IMPROVEMENTS

A. INTRODUCTION

The electrochemical reduction of thionyl chloride takes place at the cathode surface and the reduction rate is known to depend strongly on the cathode material. The widely accepted cathode reaction

$$SOCl_2 + 2e^- \rightarrow 2\ Cl^- + SO$$

proceeds smoothly at low rates upon various metals and non-metals, but it has been found that high rates can be achieved only by using carbon black substrates doped with or without electrocatalysts (5). Properties such as surface area, particle size, density, conductivity, etc., of the carbon influence, to a varying degree, its ability to enhance the rate of $SOCl_2$ reduction.

Cathode polarization and the electrochemical reaction zone thickness strongly depend on electrode reactions, cathode thickness and composition. Minimization of the effects of these variables on cathode polarization and performance are essential in order for $Li/SOCl_2$ batteries to be viable electrochemical devices for many of the high rate/low temperature applications. Therefore, during this reporting period, three cathode variables were studied to optimize the cathode performance. They are:

1) Carbon substrates
2) Cathode thickness
3) Cathode density

1. *Carbon Substrates*

Since it is known that acetylene black carbons catalyze the $SOCl_2$ reduction better than any other carbon reported in the literature, we examined both 50% and 100% compressed grade Shawinigan acetylene blacks (SAB) for their effect on cathode performance. In addition, the catalytic effect of $(CoPc)_n$ and FePc catalysts were examined. These catalysts were doped only on 100% SAB because of its superior performance observed over 50% SAB. The catalyst doping method was described elsewhere(6).

2. Cathode Thickness

At high discharge rates, only a small part of the available cathode surface participates in the electrochemical $SOCl_2$ reduction process at porous teflonated carbon cathodes. This part of the electrode structure is often referred to as the reaction zone. The reaction zone thickness is influenced by both kinetics and mechanism of electrochemical reduction. In addition, cathode thickness effects the reaction zone (depth of penetration). Therefore, the effect of cathode thickness on both voltage and performance of cathode were studied. Cathodes of thickness, 0.010, 0.015, 0.020, 0.030 and 0.040 inches were fabricated for each substrate described above. The standard cathode fabrication process was summarized in Figure 8.

3. Cathode Density

The cathode in the $Li/SOCl_2$ system uses porous teflonated carbon electrodes. The amount of Teflon binder influences the cathode density, conductivity, surface area, etc. Therefore, in order to optimize the Teflon amount on these physical properties which influence the cathode performance, cathodes were fabricated containing 5, 10 and 15% by weight Teflon binder. In Figure 9, cathode density dependence on the Teflon content was plotted for three different carbon substrates. Approximately 20% increase in cathode density is observed with 100% SAB compared to that of cathode with 50% SAB substrate. The drop in density with 5% $(CoPc)_n$ doped 100% SAB might be due to porosity created during heat treatment at 550 - 600°C.

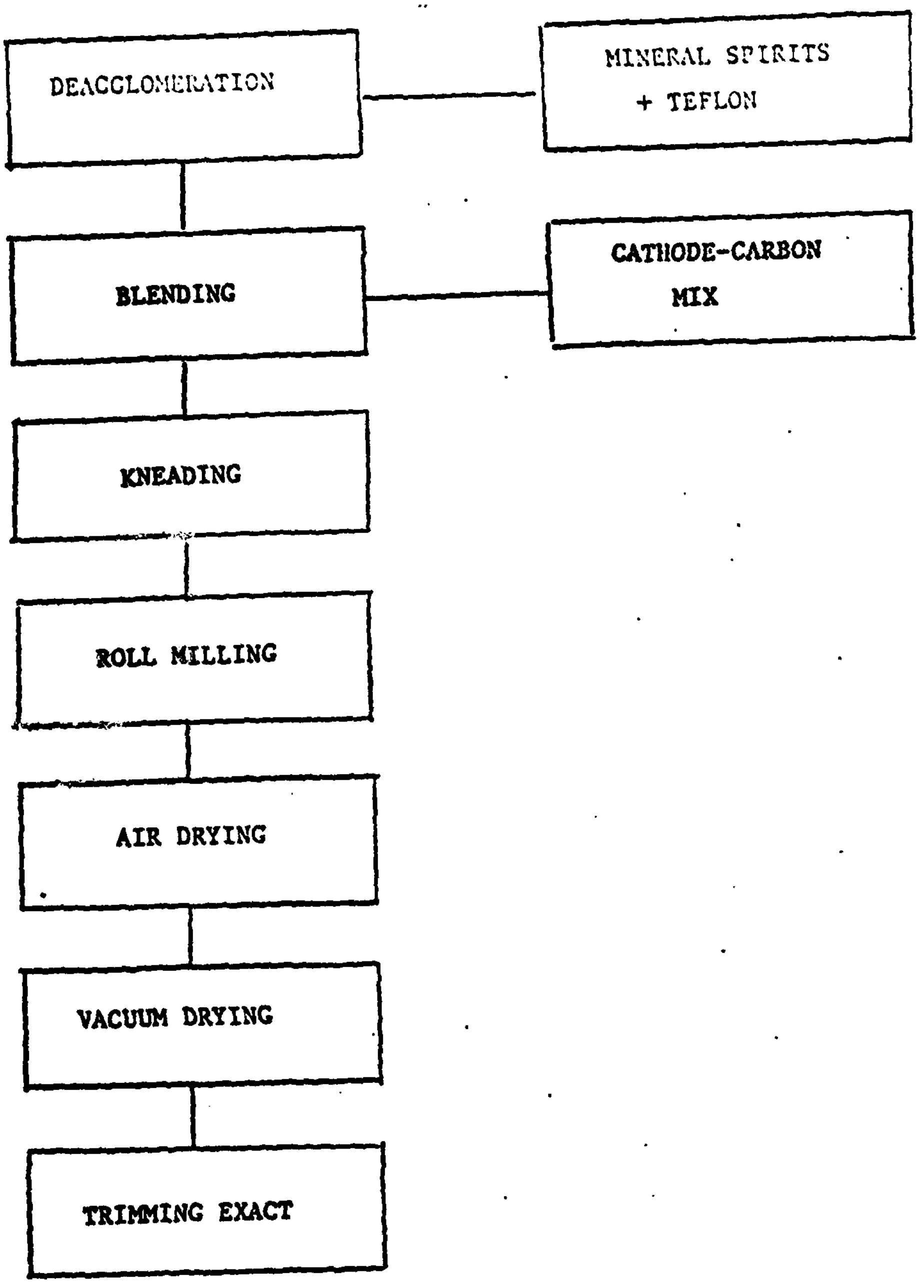

Figure 8. Flow Diagram of Cathode Fabrication Process. Workability of the process not restricted to specific carbon type. Rolling incurs excellent mechanical integrity on the cathode.

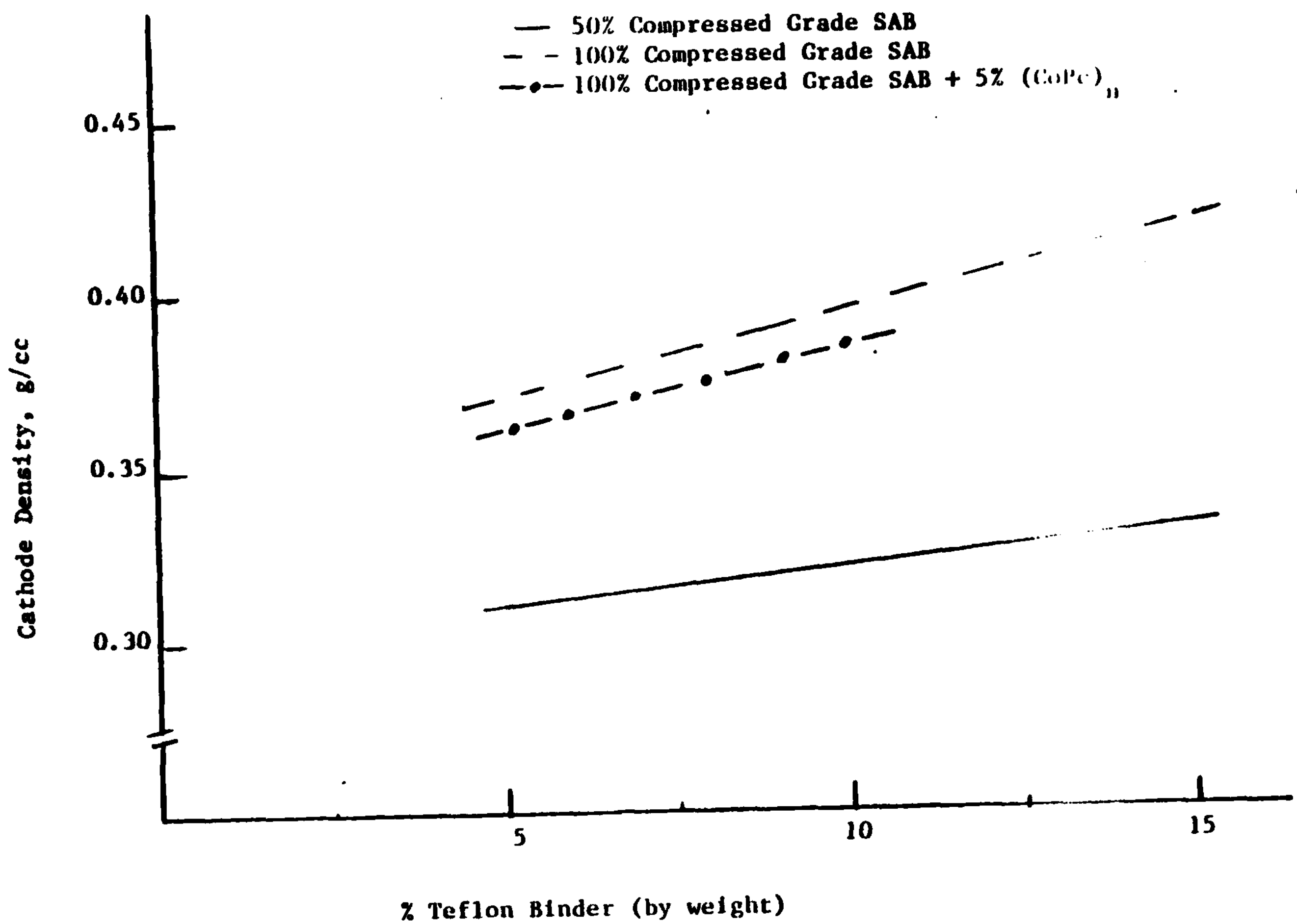

Figure 9. Effect of Teflon Content on the Cathode Density

B. EVALUATION OF CATHODE PERFORMANCE

1. Introduction

Cathode performance was evaluated by discharging laboratory $Li/SOCl_2$ cells at a fixed 10 mA/cm^2 current rate. The effect of cathode substrate, thickness and density, and the temperature on the discharge performance were examined. Performance evaluation of two operating temperatures ($0^{o}F$ and $75^{o}C$) was carried out during the reporting period. All cells were activated at ambient temperature and cooled to appropriate temperature with a constant current load equivalent to 0.2 mA/cm^2 to avoid passivation problems associated with $Li/SOCl_2$ system. All experiments were duplicated and if a variation was found, it was repeated until reproducible results were obtained. A total of 40 cathodes were fabricated and evaluated.

2. Discharge Performance

Discharge characteristics were studied in our standard laboratory cell fixture. Cells were discharged with a constant current load equivalent to 10 mA/cm^2 using a Hewlett-Packard DC current source (#6181B). In order to maintain identical experimental conditions, cells containing cathodes of constant density were built and discharged together for every operating temperature. For example, cathodes having thicknesses of 0.010, 0.015, 0.020, 0.030 and 0.040 inches, fabricated from a carbon mix containing 5% Teflon binder and 50% SAB, were evaluated in duplicate together at $0^{o}F$. This procedure maintained a constant cooling time and exposed the cells to the same fluctuations in temperature and discharge rate.

In Figures 10 - 12, the effect of cathode density on the discharge performance of cathodes fabricated from 50% SAB, 100% SAB and 5% $(CoPc)_n$ doped 100% SAB were given, respectively. No significant change in performance was observed (Figure 10) with 50% compressed grade Shawinigan acetylene black cathode substrate containing 5 - 15% Teflon binder. However, cathodes fabricated with 100% SAB with and without $(CoPc)_n$ catalyst showed best performance with 5% Teflon binder (Figures 11 and 12). Similar behavior was observed for FePc catalyzed cathodes.

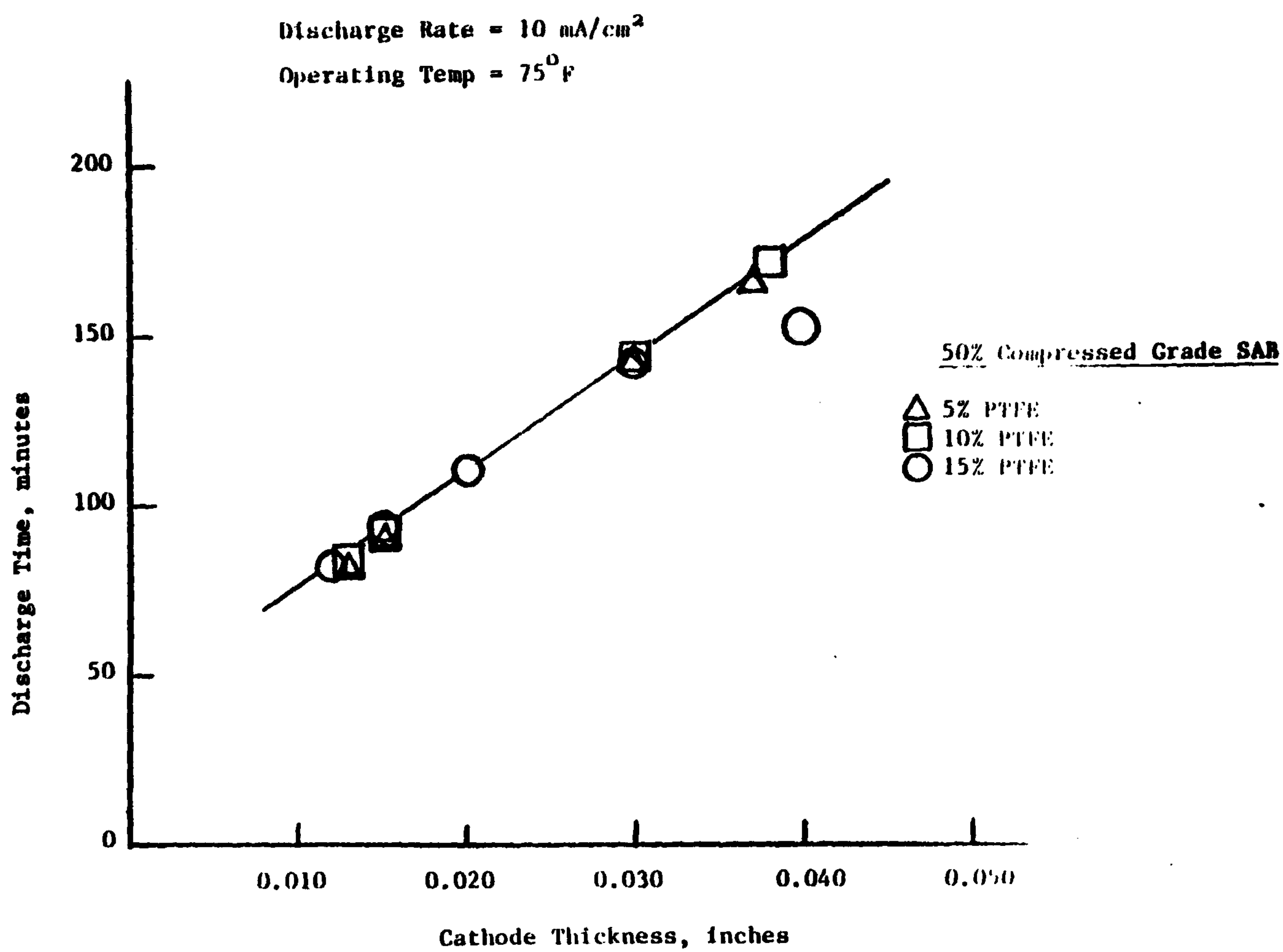

Figure 10. Effect of Cathode Variables (Teflon Content) on the Discharge Performance of $Li/SOCl_2$ Cells with 1.5M $LiAlCl_4/SOCl_2$ Electrolyte

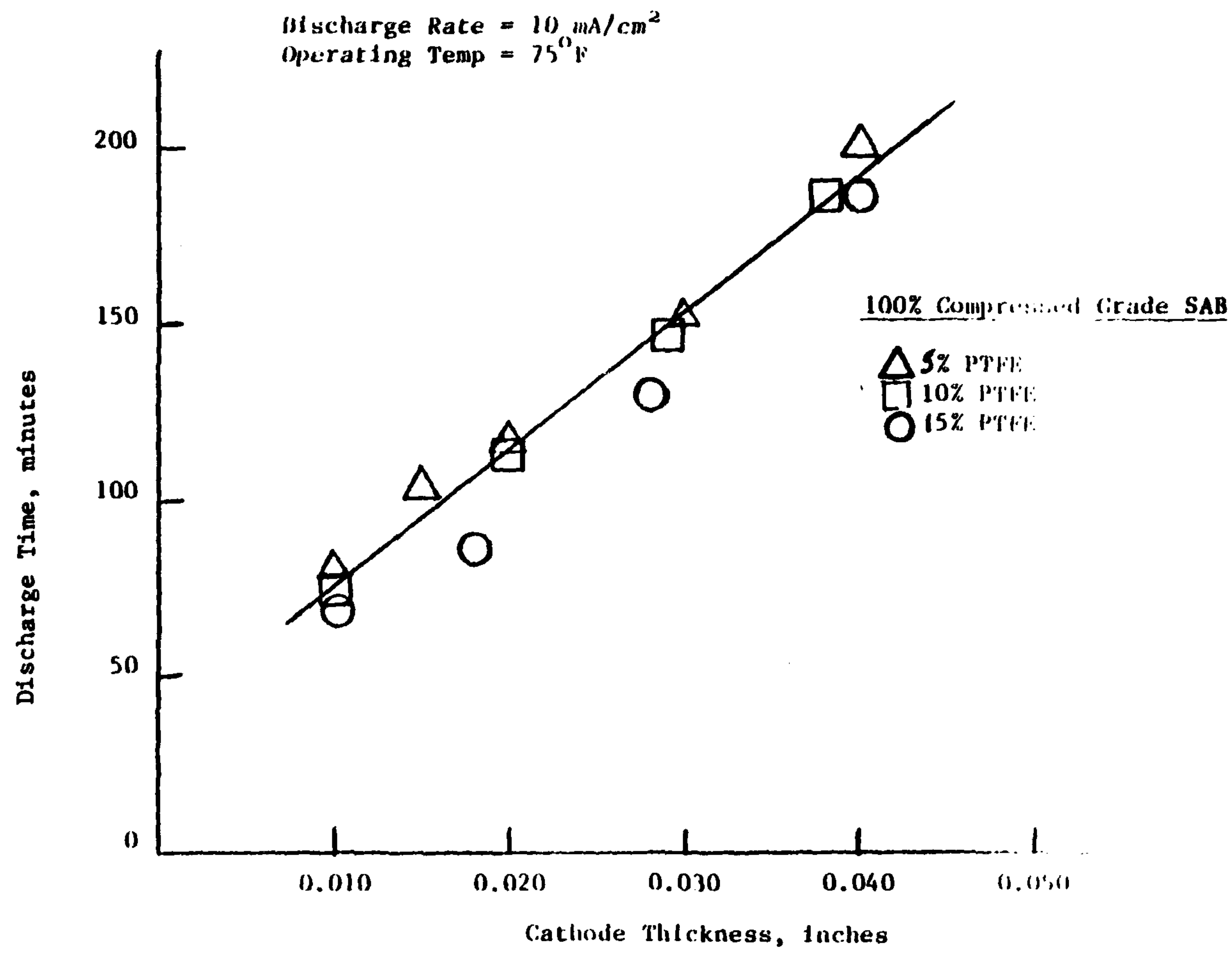

Figure 11. Effect of Cathode Variable (Teflon Binder Content) on the Discharge Performance of $Li/SOCl_2$ Cells with 1.5M $LiAlCl_4/SOCl_2$ Electrolyte

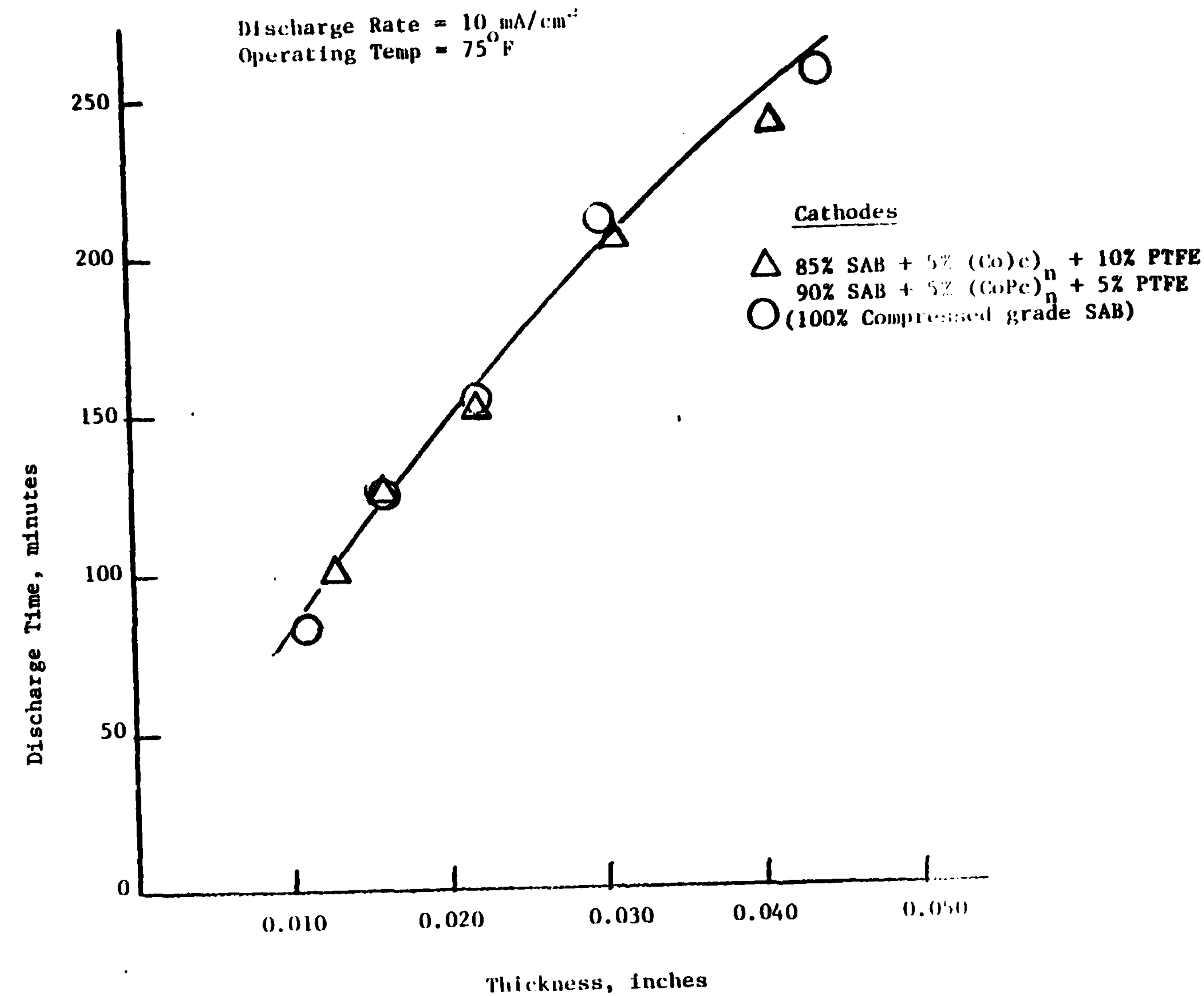

Figure 12. Effect of Cathode Variables on the Discharge Performance of $Li/SOCl_2$ Cells with 1.5M $LiAlCl_4/SOCl_2$ Electrolyte

The discharge performance of cathodes fabricated from various substrates was compared in Figure 13. All the cathodes contained 5% Teflon binder and the cells were discharged at ambient temperature with a constant current load equivalent to 10 mA/cm^2. Significant performance improvements were achieved with catalyzed cathodes. As the cathode thickness increases, the improvement in cathode performance increases with catalyzed cathodes. However, the slope decreases with cathode thickness regardless of cathode substrate.

In Figure 14, the cell voltage at 50% depth-of-discharge with respect to cathode cell thickness was compared for the cells described in Figure 13. The cell voltage remained constant for cathodes containing 50% SAB whereas all other increased with cathode thickness. Dramatic voltage improvements were achieved with FePc catalyzed cathodes.

Low temperature ($0^{\circ}F$) performance of cells described in Figure 13 was given in Figures 15 and 16. In general, lowering the temperature resulted in a decrease in electrochemical cell performance irrespective of cathode thickness and cathode substrate (Figure 15). However, with the uncatalyzed cathodes, the cell voltage improved significantly (Figure 16) with cathode thickness. Furthermore, the cell voltage of $(CoPc)_n$ catalyzed cathodes exhibited a lesser decrease. This can be seen when both Figures 14 and 16 are compared. The significance of this observation will be better understood when the performance is evaluated at -20 and $-40^{\circ}F$.

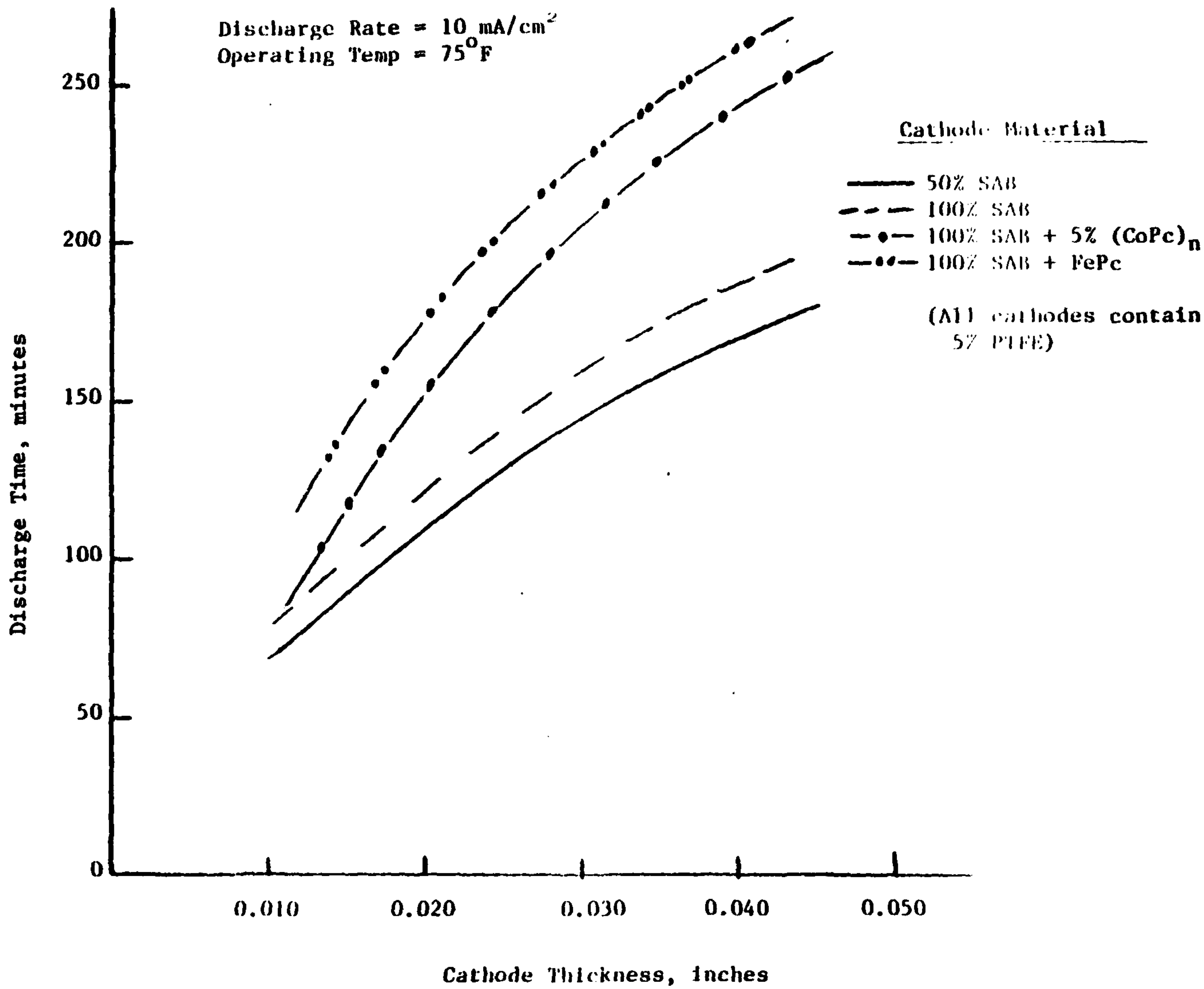

Figure 13. Effect of Cathode Variables on the Discharge Performance of $Li/SOCl_2$ with 1.5M $LiAlCl_4/SOCl_2$ Electrolyte

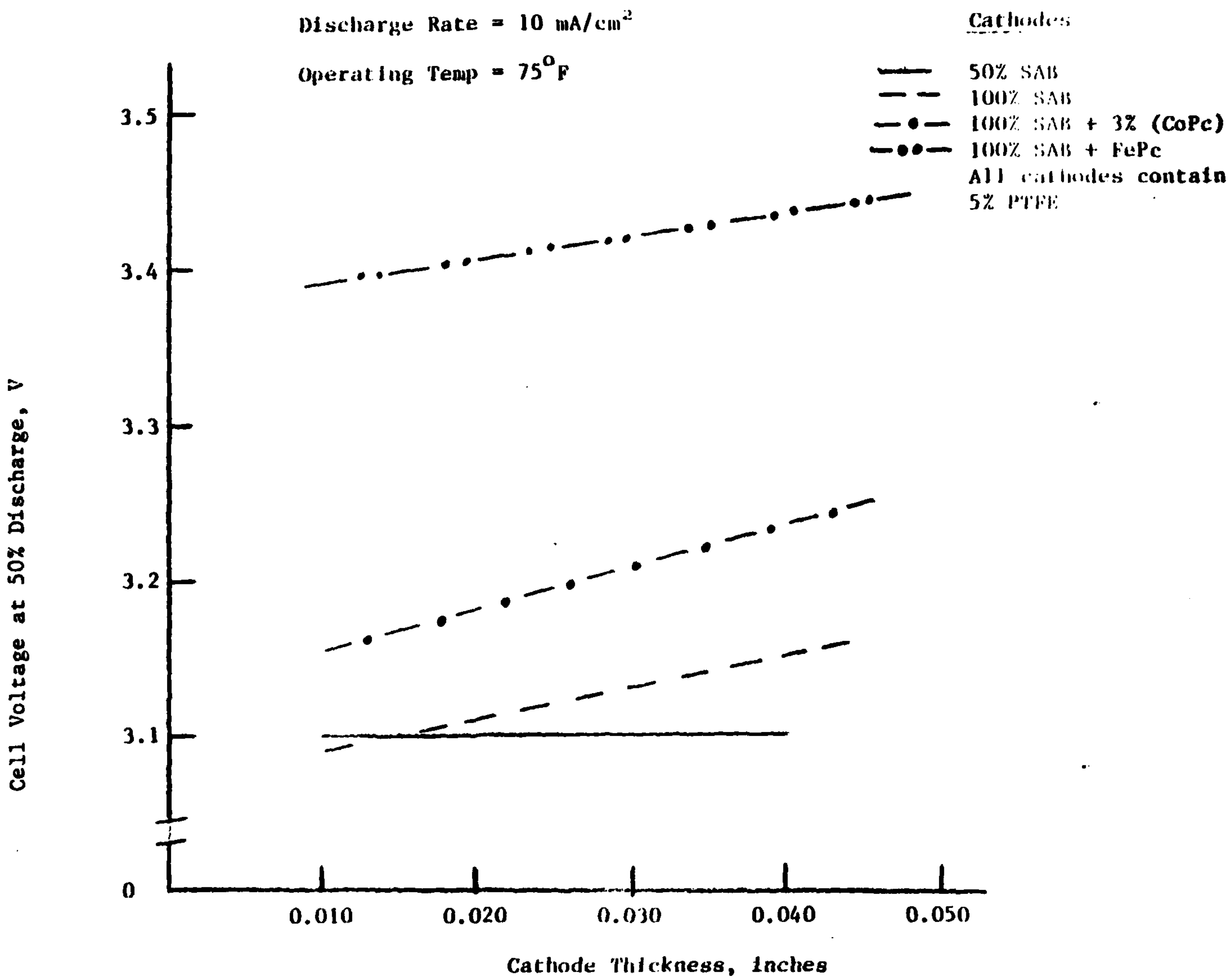

Figure 14. Effect of Cathode Variables on Voltage of $Li/SOCl_2$ Cells with 1.5M $LiAlCl_4/SOCl_2$ Electrolyte at 10 mA/cm^2 and 75°F.

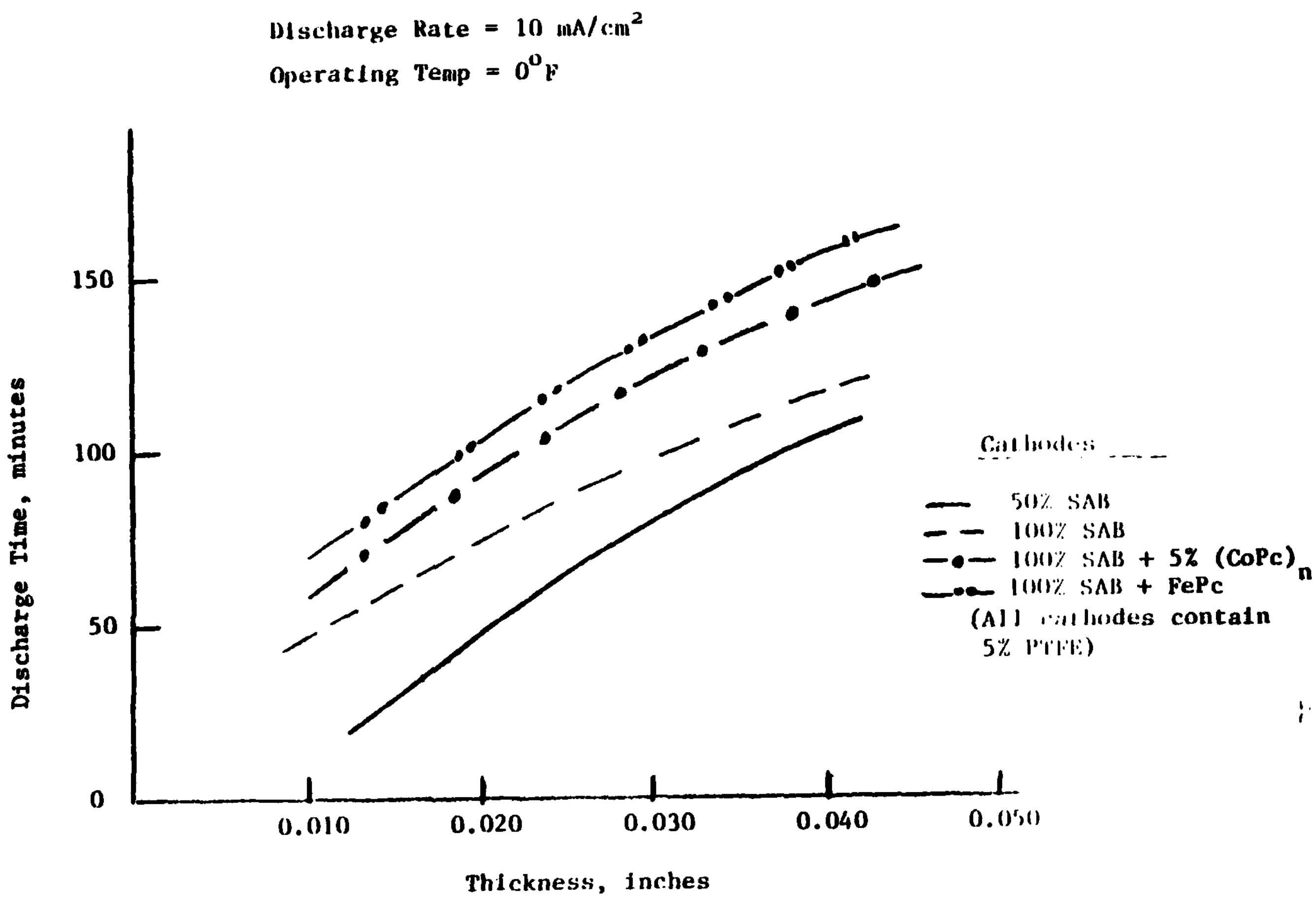

Figure 15. Effect of Cathode Variables on the Discharge Performance of Li/SOCl Cells with 1.5M $LiAlCl_4/SOCl_2$ Electrolyte at 10 mA/cm^2 and 0^oF.

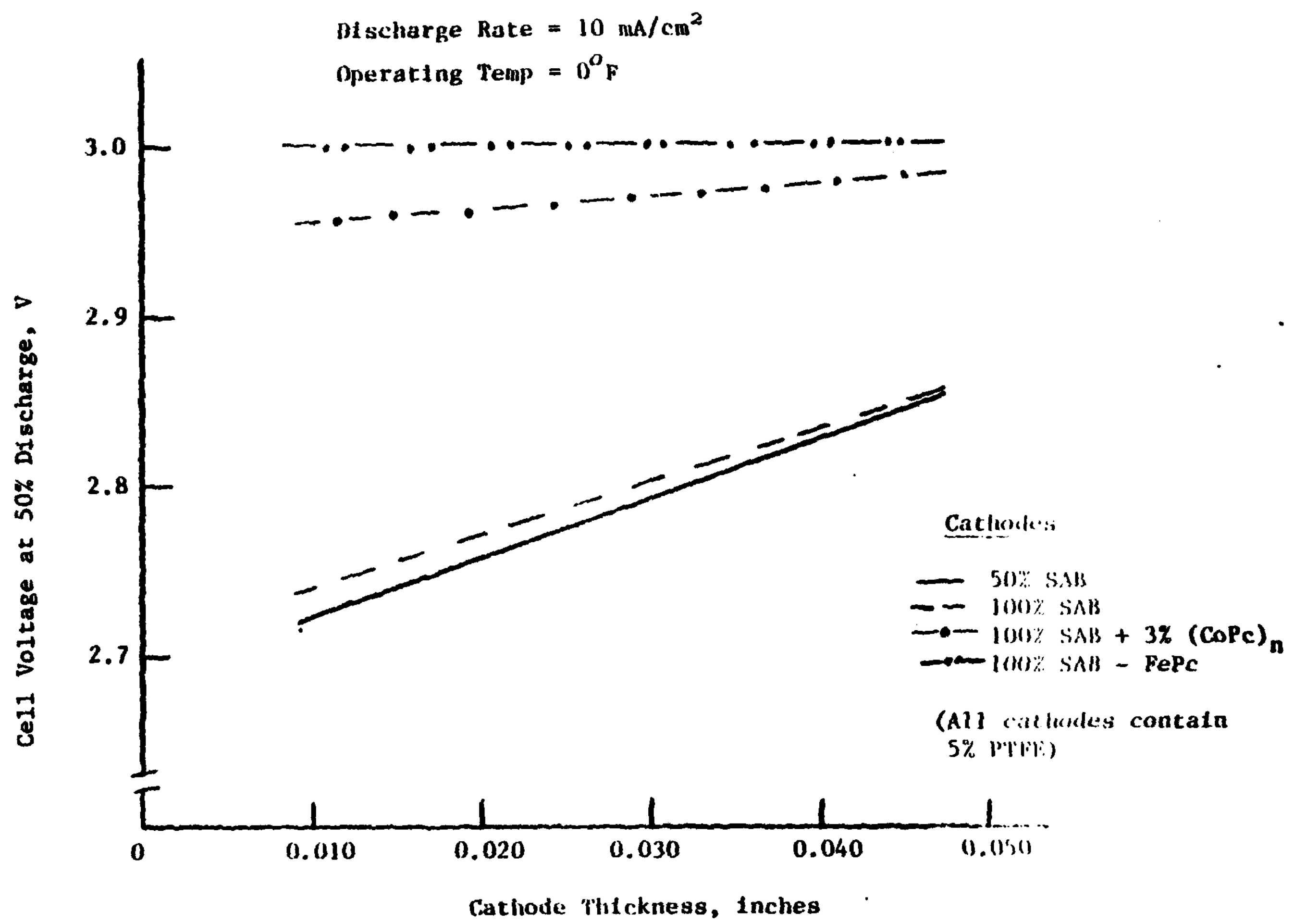

Figure 16. Effect of Cathode Variables on Voltage of $Li/SOCl_2$ Cells with 1.5M $LiAlCl_4/SOCl_2$ Electrolyte at 10 mA/cm^2 and 0°F.

IV. SUMMARY AND FUTURE WORK

Polarization and discharge performance characteristic of our best baseline cathodes with and without catalysts such as $(CoPc)_n$ and FePc were studied at operating temperatures of -20 and -40°F. Both cathode overpotential and performance were affected by the operating temperature and a linear relationship between cell limiting currents and reciprocal temperature was observed.

The effect of cathode substrate material, thickness and density on discharge performance were evaluated at 0°F and 75°F. It was found that 100% compressed grade Shawinigan acetylene black (SAB) is superior to 50% SAB. Furthermore, catalyzed cathodes improved both cell voltage and cathode performance. Irrespective of cathode substrate material, the cathodes containing 5% Teflon binder performed better.

During the next quarter, effect of cathode variables on discharge performance will be evaluated at -20 and -40°F. Kinetic and mechanistic studies of $SOCl_2$ reduction will be continued. Impedance measurements will be made on best cathodes selected after our cathode variable study.

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Argonne National Laboratories
9700 South Cass
Argonne, IL 60439
ATTN: Dr. E.C. Gay (1)

GTE Sylvania, Inc.
77 A Street
Needham Heights, MA 02194
ATTN: Mr. Richard Pabst (1)

General Motors Corp.
Research Laboratories
General Motors Technical Center
12 Mile and Mounds Roads
Warren, MI 48090
ATTN: Dr. J.L. Hartman (1)

Union Carbide Corporation
Parma Research Center
P.O. Box 6116
Cleveland, OH 44101 (1)

Duracell International, Inc.
S. Broadway
Tarrytown, NY 10591
ATTN: J. Dalfonso (1)

North American Rockwell Corp.
Atomics Internation Division
Box 309
Canoga Park, CA 91304
ATTN: Dr. L. Heredy (1)

General Electric Research &
Development Center
P.O. Box 8
Schenectady, NY 12301
ATTN: Dr. Stefan Mitoff (1)

University of California
Department of Science & Research
Santa Barbara, CA 93100
ATTN: Dr. J. Kennedy (1)

Gulton Industries, Inc.
Metuchen, NJ 08840
ATTN: Mr. S. Charlip (1)

INCO Research and Development
Center
Sterling Forest
Suffern, NY 10901
ATTN: Nehemiah Margalit (1)

Director
Propulsion and Power Division
Mail Code EP5
NASA-Johnson Space Center
Houston, Texas 77058
ATTN: Mr. B. J. Bragg (1)

GTE Laboratories, Inc.
520 Winter Street
Waltham, MA 02154
ATTN: Dr. Ronald McDonald (1)

Electrochimica
2435 Charleston Road
Mountain View, CA 94040
ATTN: Dr. Eisenberg (1)

Energy Storage & Conversion Dept.
TRW Systems
One Space Park
Redondo Beach, CA 90278
ATTN: Dr. H.P.Silverman (1)

Sanders Associates, Inc.
24 Simon Street
Mail Stop NSI-2208
Nashua, NH 03060
ATTN: J. Marshall (1)

Power Conversion, Inc.
70 MacQuesten Pkwy
Mount Vernon, NY 10550
ATTN: Stuart Chodosh (1)

Portfolio Manager
Hooker Chemicals & Plastics Corp.
M.P.O. Box 8
Niagara Falls, NY 14302 (1)

G207
S.R.I.
Menlo Park, CA 94025
ATTN: Dr. Leonard Nanis (1)

Bell Laboratories
600 Mountain Avenue
Murray Hill, NJ 07974
ATTN: Dr. J.J.Auborn, Rm 1A-317 (1)

Jet Propulsion Laboratory
4800 Oak Grove Drive
Pasadena, CA 91103
ATTN: Mr. Harvey Frank
Mail Stop 198-220 (1)

Naval Surface Weapons Center
White Oak Laboratory, Code R-33 (M/S A026)
Silver Spring, MD 20910
ATTN: Dr. D. Ernst (1)

Energy Conversion Branch Code 3642
Naval Underwater Systems Center
Newport Laboratory
Newport, RI 02840
ATTN: Mr. J. R. Moden (1)

NASA Lewis Research Center
Mail Stop 6-1
21000 Brookpark Road
Cleveland, OH 44135
ATTN: Dr. Stuart Fordyce (1)

Naval Undersea Center
Code 608
San Diego, CA 92132
ATTN: Mr. Joe McCartney (1)

EIC, Inc.
Newton, MA 02158
ATTN: S.B. Brummer (1)

Altus Corp.
440 Page Mill Road
Palo Alto, CA 94306
ATTN: Douglas Glader (1)

MS 488
NASA Langley Research Center
Hampton, VA 23665
ATTN: J. Bene (1)

Research and Development Division
The Gates Rubber Co.
999 S. Broadway
Denver, CO 80217
ATTN: Mr. Eddie T. Seo (1)

Mail Stop 8C-62
Boeing Aerospace Company
P.O. Box 3999
Seattle, WA 98124
ATTN: Mr. Sidney Gross (1)

Honeywell Technology Center
10701 Lyndale Avenue, South
Bloomington, MN 55420
ATTN: Dr. H.V. Venkatasetty (1)

Jet Propulsion Laboratory
M.S. 198-220
4800 Oak Grove Drive
Pasadena, CA 91103
ATTN: Mr. Aiji Uchiyama (1)

Naval Surface Weapons Center
White Oak Laboratory, Code R-33
Silver Spring, MD 20910
ATTN: Dr. Frank Bis (1)

Dr. Jerry J. Smith
ONR Chemical Program
Arlington, VA 22217 (1)

Saft Corporation of Aermica
711-A Industrial Blvd.
P.O. Box 1284
Valdosta, GA 31601
ATTN: Mr. Lou Adams (1)

Gould Laboratories-Energy Research
40 Gould Center
Rolling Meadows, IL 60008
ATTN: Dr. J. Hall (1)

Electrochem Industries (E-I) Inc.
9990 Wehrle Drive
Clarence, NY 14031
ATTN: Dr. C. Liang (1)

GTE Laboratories Incorporated
40 Sylvan Road
Waltham, MA 02254
ATTN: Dr. C. Shlaiker (1)

Harvey N. Seiger Associates
8 Beacon Hill Drive
Waterford, CT 06385
ATTN: Harvey N. Seiger, Ph.D. (1)

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2-8