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SIXTH QUARTERLY REPORT
SECONDARY ZINC-OXYGEN CELL FOR SPACECRAFT
APPLICATIONS

(23 September 1967 - 23 December 1967)

CONTRACT NO. NAS-5-10247

for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
GODDARD SPACE FLIGHT CENTER
GREENBELT, MARYLAND

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UNION CARBIDE CORPORATION
CONSUMER PRODUCTS DIVISION
RESEARCH LABORATORY - PARMA, OHIO

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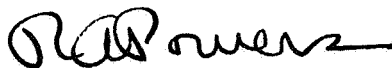
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APPROVED BY:



R. A. Powers, Director

CONSUMER PRODUCTS DIVISION
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TABLE OF CONTENTS

	<u>Page</u>
ABSTRACT	ii
INTRODUCTION	1
DISCUSSION	2
A. Experimental Unit Cell Construction	2
B. Zinc Anode Shape Change	3
C. Anode Shape Change Versus Discharge Depth	3
D. Electroosmosis and Cycling of the Unit Cell	4
E. Oxygen Electrodes	5
1. Union Carbide "Thin Fixed Zone" Oxygen Electrode	5
2. American Cyanamid LAB-40 Oxygen Electrode	5
F. Constant Current-Voltage Limiting Trickle Charging of Unit Cells	6
G. Zinc-Oxygen Unit Cell Performance at 25 °C	7
1. Performance of a Unit Cell Employing a 10 Ampere-Hour Anode	7
2. Performance of a Unit Cell Employing a 20 Ampere-Hour Anode	7
3. Performance of a Unit Cell Employing a 15 Ampere-Hour Anode	9
4. Cycle Life to 0.8 Volt Versus Zinc Discharge Depth	9
5. Cycling of American Cyanamid LAB-40 Electrode Cell	13
6. Polarization Characteristics of a Union Carbide "Thin Fixed-Zone" and an American Cyanamid Oxygen LAB-40 Electrode as a Function of Cycle Life	13
H. Zinc-Oxygen Unit Cell Performance at 40 °C	13
1. Performance of a Unit Cell Employing a 10 Ampere-Hour Anode	13
J. Zinc-Oxygen Unit Cell Performance at 0 °C	13
1. Performance of a Unit Cell Employing a 10 Ampere-Hour Anode	13
NEW TECHNOLOGY	18
PROGRAM FOR THE NEXT REPORTING INTERVAL	18
CONCLUSIONS AND RECOMMENDATIONS	18
BIBLIOGRAPHY	19

LIST OF ILLUSTRATIONS

<u>Figure No.</u>		<u>Page</u>
1	Cycling of 10 Ampere-Hour Anode Cell at 26% Zinc Discharge Depth at 25°C	8
2	Cycling of 20 Ampere-Hour Cell at 13% Zinc Discharge Depth at 25°C	10
3	Cycling of 15 Ampere-Hour Cell at 17% Zinc Discharge Depth at 25°C	11
4	Comparison of 10, 15, and 20 Ampere-Hour Anodes Discharged at 26%, 17%, and 13% Depth	12
5	Cycling of American Cyanamid LAB-40 Electrode Cell	14
6	IR-Free Cathode Polarization - American Cyanamid LAB-40 Vs. Union Carbide T-2	15
7	40°C Performance of 10 Ampere-Hour Cell Discharged at 26% Zinc Depth	16
8	0°C Performance of 10 Ampere-Hour Cell Discharge at 26% Zinc Depth	17

ABSTRACT

Cycling of zinc-oxygen unit cells operating at a discharge current density of 25 mA/cm^2 has been demonstrated. This represents discharge to the 25 percent depth based upon the theoretical capacity available from the actual amount of zinc present in the anode. As of this time, the cells have delivered 56, 108, and 56 cycles at 0° , 25° , and 40°C , respectively, on a 2-hour discharge/4-hour charge test regime. Polarization data indicate that cells whose construction has been modified for high rate discharge possess the capability of discharging at current densities up to 70 mA/cm^2 (70 percent zinc depth). Cycle life, however, is increasingly limited as the depth of zinc discharge increases by redistribution of the zinc anode mass resulting in the gradual movement of zinc with successive cycles from the top of the electrode to the bottom. Anode constructions are being explored in an effort to maximize cycle life and discharge depth.

* * *

INTRODUCTION

Work conducted during the previous quarter showed that the rechargeable zinc-oxygen unit cells are capable of delivering over 300 cycles at room temperature when discharged to the 13.5 percent depth based upon the theoretical capacity available from the actual amount of zinc present. This corresponds to a current density of 11.5 mA/cm². The performance of such cells was duplicated during the present report period.

Effort this quarter was concentrated upon improving the high rate discharge capability of the cells. Polarization data indicated that the cell capability was severalfold greater than the 11.5 mA/cm² current density at which they had delivered 168, 356, and 252 cycles at 0°, 25°, and 40°C, respectively, on the 2-hour discharge/2-hour charge cycle. Modified cells were found to successfully cycle at a current density of 25 mA/cm² on the 2-hour discharge cycle with good voltage regulation. A 4-hour charge time was used. The theoretical capacity of zinc present in the cell anode was 10 ampere-hours and total discharge represented a zinc discharge depth of 25 percent. As of the present time, the cells have cycled 56, 108, and 56 cycles, respectively, at 0°, 25°, and 40°C. A redistribution of the zinc anode mass with cycling has been observed. This redistribution results in the gradual motion of zinc from the top of the electrode toward the bottom of the electrode as cycling progresses. In effect, this reduces the cross sectional area of the zinc electrode and thereby increases the current density at which the electrode operates. The rate at which the zinc redistribution occurs is accelerated by increasing discharge depths and limits the total number of cycles obtained from the cell.

An evaluation of PERMION 1770C was necessitated inasmuch as the supplier, RAI Research Corporation, no longer produces the film membrane used in previous cells, PERMION 110. A single layer of the PERMION 1770C was found to be equally effective in resisting dendrite penetration and oxygen permeation as the PERMION 110. PERMION 110 was reported to be a radiation-grafted cross linked low density polyethylene-acrylic acid copolymer, while PERMION 1770C is stated to be a chemically-grafted polyethylene-acrylic acid film.

The feasibility of using an American Cyanamid LAB-40 electrode both as the oxygen electrode and as the charging electrode was established. A problem of apparent deterioration of the American Cyanamid LAB-40 electrode, however, has resulted in cycle life limitation. This will be examined further in subsequent work.

A second set of five experimental rechargeable zinc-oxygen cells was constructed and delivered to NASA during this report period.

DISCUSSION

A. Experimental Unit Cell Construction

Construction of the zinc-oxygen rechargeable experimental unit cell was described and shown schematically in the Fifth Quarterly Report. The construction of both a three-electrode cell and a two-electrode cell was shown.

The three-electrode cell employed a Union Carbide T-2 oxygen electrode utilizing a separate charging electrode. The two-electrode cell employed an American Cyanamid oxygen electrode. A separate charging electrode was eliminated as the zinc was charged directly off the oxygen electrode.

The only change made within the cell components was the substitution of the PERMION 1770C separator for the PERMION 110 separator, which is no longer available from the supplier, RAI Research Corporation. The PERMION 110 was reported to be a radiation-grafted polyethylene-acrylic acid film, while the PERMION 1770C is chemically grafted. To date, PERMION 1770C has demonstrated the ability to resist dendrite penetration and oxygen permeation equally as well as PERMION 110. A comparison of their resistivity and dendrite penetration resistance is shown in Table I.

TABLE I

Separator	Specific Resistance (ohm-cm)		24 Hour Typical Values			Time Required to Grow a Dendrite Through Separator (1/16" between electrodes) (C. D. at start 250 mA/cm ²)
	5 min.	24 hrs.	Measured Resistance	Thickness		
				Dry	Wet	
PERMION 110	41	41	0.2 ohm	.001 in.	.0015 in.	Could not traverse membrane
PERMION 1770C	37	37	0.3 ohm	.0015 in.	.002 in.	Could not traverse membrane

Reference is made to the Fifth Quarterly Report for a description of the test procedure used in obtaining the data for Table I.

B. Zinc Anode Shape Change

It has been demonstrated that the zinc-oxygen unit cell is capable of rechargeability and continuous operation after occasional very deep discharge. Performance of a cell that was completely discharged and returned to the normal cycling schedule was shown in the Fourth Quarterly Report.

Repeated deep discharges caused severe anode shape change and associated problems. Impaired anode mass-collector contact and a loss of capacity associated with the continually diminished geometric surface area was the primary mode of failure of all cells consistently discharged at a current density of 25 mA/cm² to 25 percent theoretical depth in two hours.

The active anode area of dissected cells has decreased by as much as 70-80 percent. As a result, the current density imposed on both the anode and the cathode was continually increased as cycling progressed. An initial current density of 25 mA/cm² on a fresh cell becomes over 100 mA/cm² after 75 percent zinc reorientation has occurred.

C. Anode Shape Change Versus Discharge Depth

Over 300 cycles were delivered from a unit cell employing a 10 ampere-hour zinc anode. This cell was discharged at a current density of 11-12 mA/cm² to 12-14 percent zinc depth in two hours. Low discharge voltages associated with 22-25 mA/cm² discharging of this cell necessitated modifications to improve the anode-collector contact and to decrease the cell internal resistance.

These modifications improved the discharge performance to the extent that a discharge current density of 22-25 mA/cm² could be attained at essentially the same voltage level as the 11-12 mA/cm² discharge current density in the cell from which over 300 cycles were delivered.

Doubling the rate of discharge, while maintaining the discharge time constant, correspondingly doubled the discharge depth (12-14 percent to 25-30 percent depth of a 10 ampere-hour anode). Severe anode shape change occurred early in the cycle life of cells discharged to this depth. Failures due to problems associated with 70-80 percent zinc slump were not uncommon after 50-60 cycles.

Over 300 cycles could be obtained from a 10 ampere-hour cell subjected to 12-14 percent depth of discharge in two hours, while only 50-60 cycles could be expected from a cell consistently subjected to 25-30 percent depth of discharge. This observation suggested the feasibility of retarding anode slump and correspondingly increasing cycle life by limiting the discharge depth to a value comparable with that at which the previous cell delivered over 300 cycles. Two cells were built, both employing anode structures identical to the aforementioned 10 ampere-hour cells, but with increased zinc capacity. One of these cells contained a 20 ampere-hour anode and the other a 15 ampere-hour anode. Both of the cells were discharged across a fixed load of 0.75 ohm at an average current density of 23 mA/cm². The theoretical zinc depth per discharge on the 20 ampere-hour cell and the 15 ampere-hour cell was 13 and 17 percent, respectively. Both of these cells are still cycling after 108 cycles. Approximately 10 percent slump has occurred at the anode; whereas, 70-80 percent anode slump was observed in unit cells subjected to 25-30 percent zinc depth discharges after 50-60 cycles.

D. Electroosmosis and Cycling of the Unit Cell

Electroosmosis hindered cycling of zinc-oxygen cells on a 2-hour discharge/ 4-hour charge schedule to a theoretical output/input balance of 2.5-3.0 ampere-hours. This was not a problem when cells were cycled on a 2-hour discharge/2-hour charge schedule to a theoretical output/input balance of 1.2-1.4 ampere-hours.

As cycling progressed, the electrolyte level in the anode compartment would rise during charge and was accompanied by a corresponding lowering of the electrolyte level in the cathode compartment during charge. This electrolyte would not return to the cathode compartment during discharge. This was not a simple problem of electrolyte level fluctuation in the respective electrode compartment during cycling which is normal in cells employing a membrane separator.

A gelled anode was used in the cells in which this problem occurred, thus the viscosity of the anolyte increased after overnight contact with the gelled anode. The viscosity of the catholyte was not affected by the anode binder, since the anode was sealed off in a separate compartment on the other side of the PERMION 110 membrane separator. During discharge the more viscous anolyte would not move to the cathode compartment as would normally be expected. However, the catholyte would move to the anode compartment during charge and mix with the more viscous anolyte. The catholyte involved, which was now also viscous, would not return to the cathode compartment. The net effect was overflowing of electrolyte from the anode compartment and ultimate evacuation of the cathode compartment.

E. Oxygen Electrodes

1. Union Carbide "Thin Fixed-Zone" Oxygen Electrode

The Union Carbide "thin fixed-zone" oxygen electrode continues to demonstrate stability of performance when repeatedly discharged. There was very little increase in polarization at this electrode when higher current densities were imposed on it as a result of shape change occurring at the anode. However, deterioration of this oxygen electrode structure takes place when oxygen is evolved from the catalyzed carbon surface. Hence, a separate charging electrode is needed.

2. American Cyanamid LAB-40 Oxygen Electrode

Investigation of the American Cyanamid LAB-40 oxygen electrode as a means of eliminating the separate charging electrode was continued.

Although the feasibility of using this electrode to charge the zinc was demonstrated, its apparent deterioration during cell operation limits cycle life. Both flooding and polarization of the electrode occurred during cell operation.

Six cells were constructed using American Cyanamid LAB-40 oxygen electrodes. These cells were activated with electrolyte and left to soak overnight before testing. No back pressure was applied to the oxygen electrodes during the soak period. The oxygen electrodes were flooded on all six cells by the following morning.

Subsequent cells employing American Cyanamid oxygen electrodes were activated only after oxygen back pressure was applied. The applied back pressure was sustained during cell operation. Although leakage through the electrode was retarded, it still occurred shortly after cycling began.

Discharging of cells employing an American Cyanamid LAB-40 oxygen electrode and a 10 ampere-hour anode can be limited by both electrodes when subjected to 26 percent discharge depth. With the present anode structure, anode polarization can occur as a result of slump, while cathode polarization can occur as a result of apparent deterioration of the active layer. Higher current density imposed on the cathode as a result of zinc slump can also cause problems due to overloading of that part of the cathode facing the reduced zinc area.

F. Constant Current-Voltage Limiting Trickle Charging of Unit Cells

A constant current-voltage limiting trickle charge was investigated during this report period as a means of controlling hydrogen evolution. This type of charge incorporates two steps:

1. The current is preset at a constant value such that the input is balanced with the output in the required time (depending on the cycling schedule) to a preset voltage (below hydrogen evolution).

2. When the preset voltage is reached, the charge is switched to constant voltage and the amount of current flow is determined by the difference in voltage between the power supply and the test cell.

The feasibility of charging the zinc by means of this type of charge was not established during this report period because of the unstable end-of-charge voltage associated with zinc shape change with the present anode structure. The cells reached the voltage limit before they were sufficiently charged, and the end-of-charge voltage became progressively higher as cycling continued. Zinc mass-collector contact and polarization of the zinc anode, both associated with anode shape change, were the main causes of the end-of-charge voltage fluctuation that prohibits full utilization of a constant current charge with a voltage limit or a voltage limiting trickle charge unless the charge time sufficiently is extended.

G. Zinc-Oxygen Unit Cell Performance at 25°C

1. Performance of a Unit Cell Employing a 10 Ampere-Hour Anode

Cycling of a unit cell at 25°C to 26 percent zinc discharge depth based upon the actual amount of zinc present progressed to 108 cycles on a 2-hour discharge/4-hour charge schedule. The discharge current density was approximately 22 mA/cm²; the charge current density was approximately 11 mA/cm².

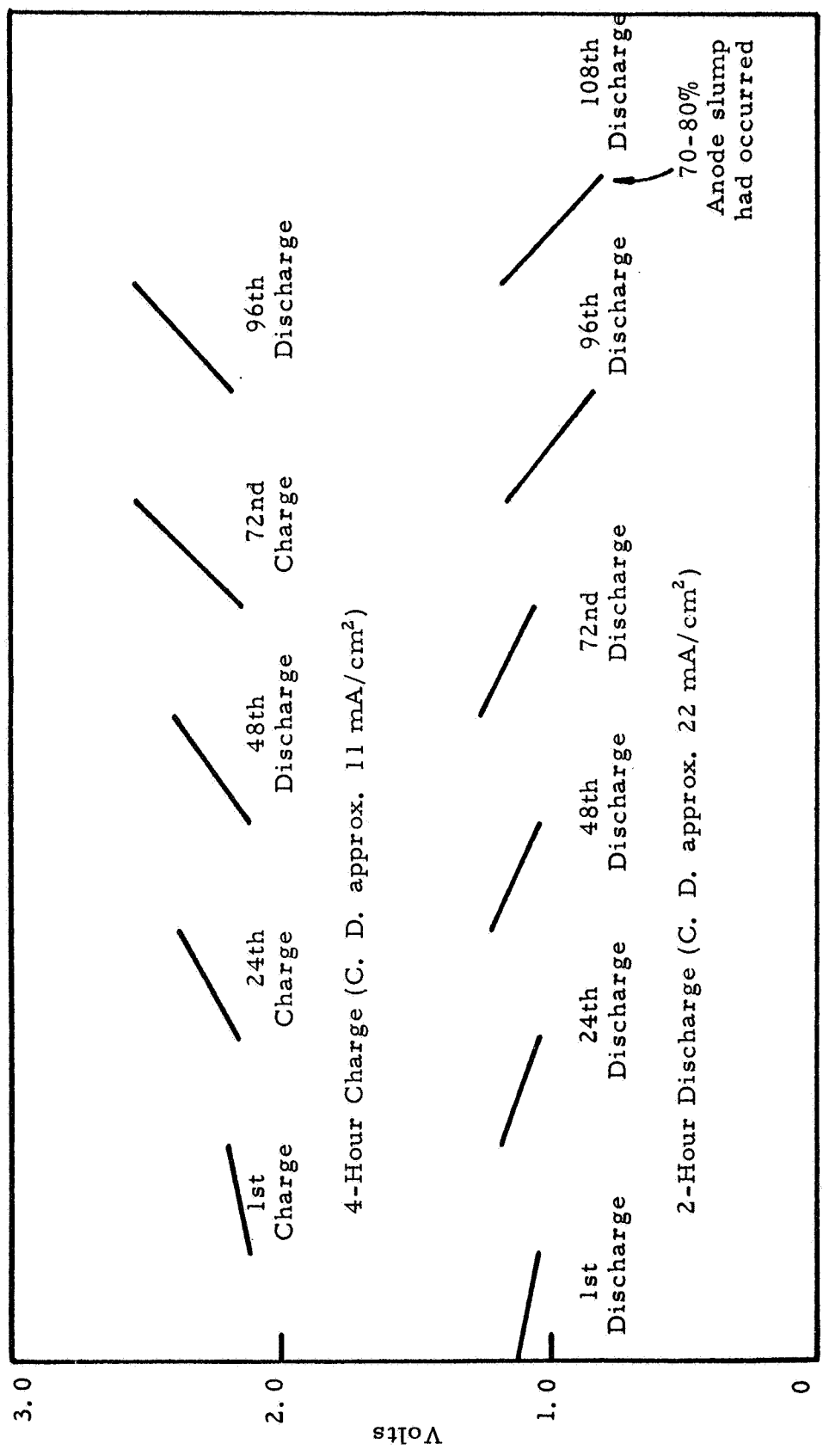
The result of degradation of the zinc anode as cycling progressed was a continually declining discharge voltage level. Testing was terminated when the end-of-discharge voltage reached 0.8 volt, which value was associated with 70-80 percent anode slump in practically every cell cycled to 26 percent depth in two hours. Fifty to sixty cycles were generally obtained from cells repeatedly discharged to this depth in two hours. However, 108 cycles were delivered by the cell shown in Figure 1.

2. Performance of a Unit Cell Employing a 20 Ampere-Hour Anode

Over 350 cycles were delivered by a unit cell, when the theoretical zinc discharge depth was limited to 12-14 percent. The discharge current density was 11-12 mA/cm². When unit cells employing identical anode structures were consistently discharged at a current density of 22 mA/cm² to 26 percent zinc depth, cycle life was drastically reduced by severe anode slump. The fact

FIGURE 1

Cycling of 10 Ampere-Hour Anode Cell at 26% Zinc Discharge Depth at 25°C



that cycle life was so drastically reduced with identical anodes by deeper discharge suggested the possibility of minimizing anode slump and thereby extending cycle life. This possibility could be realized in cells consistently discharged at a current density of 22 mA/cm² if the zinc discharge depth was limited to a level comparable to that at which the cell delivering over 350 cycles was discharged.

The anode in this cell contains 20 ampere-hours of zinc capacity. A two-hour discharge is equivalent to approximately 13 percent zinc depth. The discharge current density is 22 mA/cm² and the charge current density is 11 mA/cm². To date, this cell has delivered 108 cycles and is still cycling with good voltage regulation. Performance of this cell is shown in Figure 2.

3. Performance of a Unit Cell Employing a 15 Ampere-Hour Anode

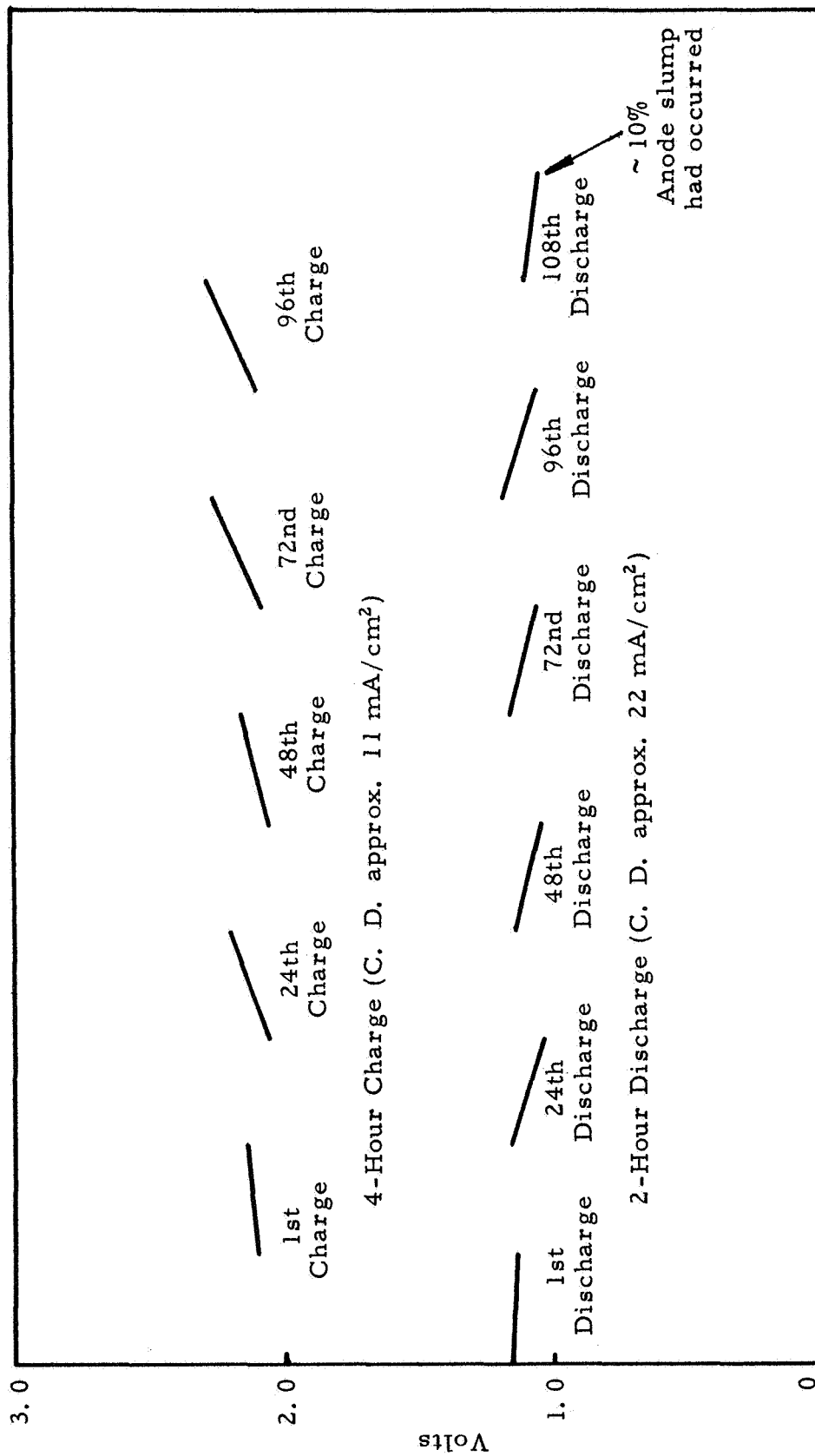
Cycling of a cell made with a 15 ampere-hour anode at 25°C was part of the experiment designed to determine the effect of zinc discharge depth on the rate at which anode slump occurs and on cycle life extension. A two-hour discharge is equivalent to approximately 17 percent zinc depth. The discharge current density was 22 mA/cm² and the charge current density was 11 mA/cm². This cell has delivered 108 cycles to date and is still cycling with good voltage regulation. Performance of this cell is shown in Figure 3.

4. Cycle Life to 0.8 Volt Versus Zinc Discharge Depth

Figure 4 shows a comparison of cycle life of cells employing anodes containing 10, 15, and 20 ampere-hours of zinc. Discharging of cells employing 10, 15, and 20 ampere-hours of zinc was conducted at a current density of 22 mA/cm² and was to 26, 17, and 13 percent zinc depth, respectively, based upon the capacity of actual amount of zinc present. These cells were discharged to an 0.8 volt cutoff. Anodes in most cells that were subjected to 26 percent zinc depth discharge had slumped to 20-30 percent of the original area after 50-60 cycles. However, one cell delivered 108 cycles. One hundred and eight cycles were delivered from cells discharged to 13 and 17 percent depth. These cells are still cycling and approximately 10 percent anode slump has occurred.

FIGURE 2

Cycling of 20-Ampere-Hour Cell at 13% Zinc Discharge Depth at 25°C



C-4204

FIGURE 3
Cycling of 15 Ampere-Hour Cell at 17% Zinc Discharge Depth at 25°C

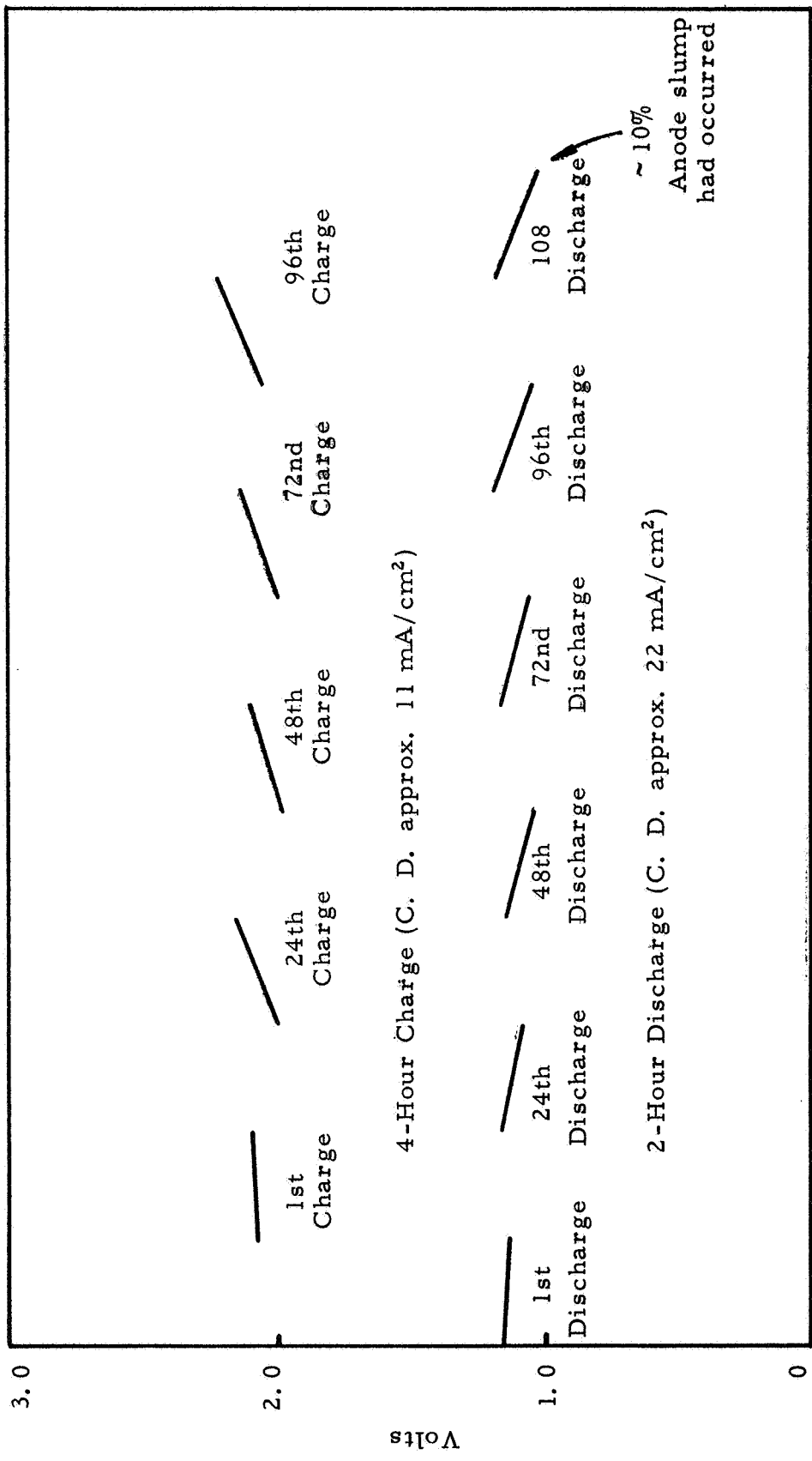
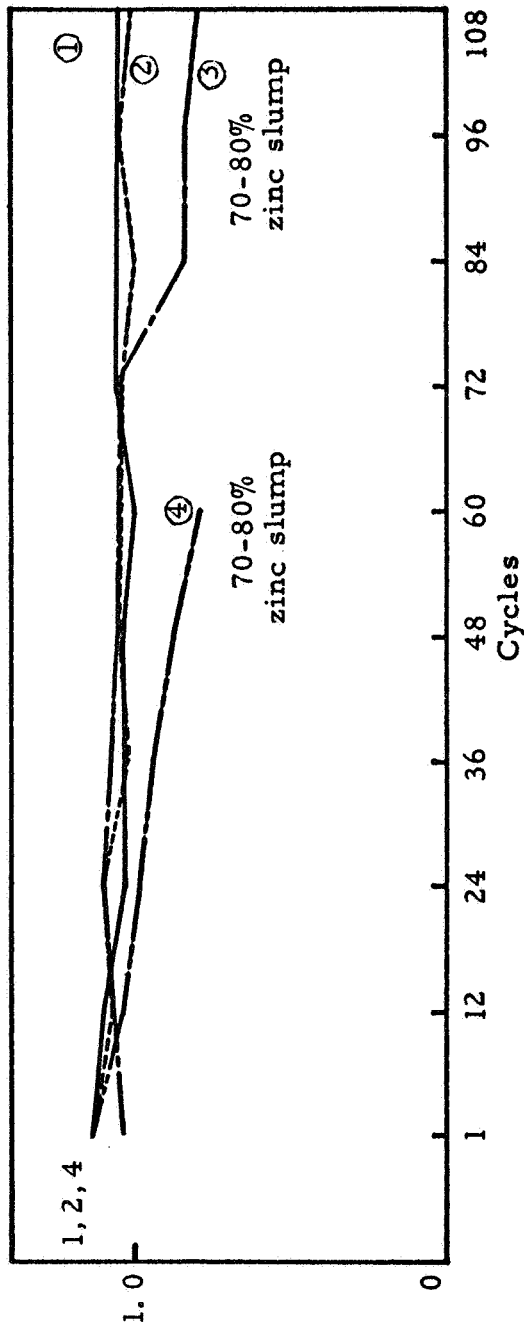


FIGURE 4

Comparison of 10, 15, and 20 Ampere-Hour Anodes Discharged at 26%, 17% and 13% Depth

Cell No.	Capacity	Discharged	Discharge C. D.	% Zn Depth per Discharge	No. Cycles to 0.8 Volts
1	20 amp-hrs	2 hrs	22 mA/cm ²	13%	*
2	15 amp-hrs	2 hrs	22 mA/cm ²	17%	* 1 cell
3	10 amp-hrs	2 hrs	22 mA/cm ²	26%	108
4	10 amp-hrs	2 hrs	22 mA/cm ²	26%	50-60 Average of 10 Cells

* Cells 1 and 2 are still cycling after 108 cycles. Approximately 10% anode slump can be observed.



5. Cycling of American Cyanamid LAB-40 Electrode Cell

The feasibility of using an American Cyanamid electrode to recharge the zinc was established. Apparent deterioration and flooding of this electrode however limited cell life to 36 cycles as shown in Figure 5.

6. Polarization Characteristics of a Union Carbide "Thin Fixed-Zone" and an American Cyanamid Oxygen LAB-40 Electrode as a Function of Cycle Life

Figure 6 shows a comparison of the polarization characteristics of the Union Carbide "fixed-zone" oxygen electrode and the American Cyanamid LAB-40 oxygen electrode as cycling progresses.

Performance of the Union Carbide "fixed-zone" electrode actually improved over 56 cycles whereas the LAB-40 electrode became worse after 36 cycles. The improvement seen in the performance of the Union Carbide electrode was due to improved wetting of the electrode active layer as cycling progressed. Although the voltage of the LAB-40 oxygen electrode versus a zinc reference electrode was higher than that of the "fixed-zone" oxygen electrode on a fresh cell, the LAB-40 electrode flooded and suffered apparent degradation of its activity. The LAB-40 electrode was used to recharge the zinc. The zinc electrode in the cell employing a "fixed-zone" oxygen electrode was charged by a separate charging electrode.

H. Zinc-Oxygen Unit Cell Performance at 40°C

1. Performance of a Unit Cell Employing a 10 Ampere-Hour Anode

The 1st, 12th, 24th, 36th, 48th, and 56th cycles of a cell discharged at a current density of 22 mA/cm² to 26 percent depth is shown in Figure 7. Cycling was on a 2-hour discharge/4-hour charge schedule. This cell employed a 10 ampere-hour anode and was susceptible to the same problem of anode slump as other cells.

J. Zinc-Oxygen Unit Cell Performance at 0°C

1. Performance of a Unit Cell Employing a 10 Ampere-Hour Anode

The 1st, 12th, 24th, 36th, 48th, and 56th cycles of a cell cycled at 0°C on a 2-hour discharge/4-hour charge schedule are shown in Figure 8. The discharge current density was 21 mA/cm².

FIGURE 5
Cycling of American Cyanamid LAB-40 Electrode Cell at 31% Zinc Depth

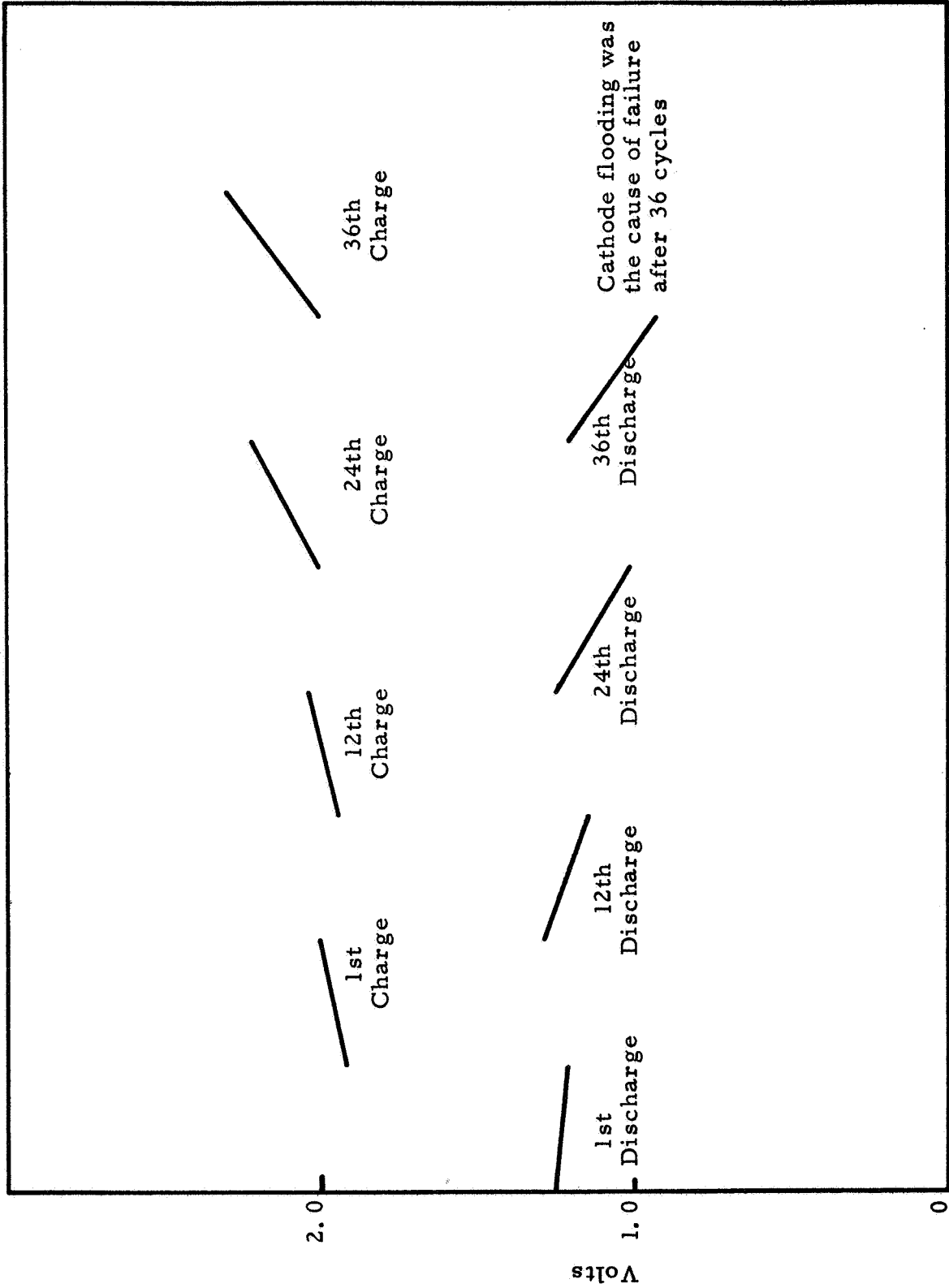
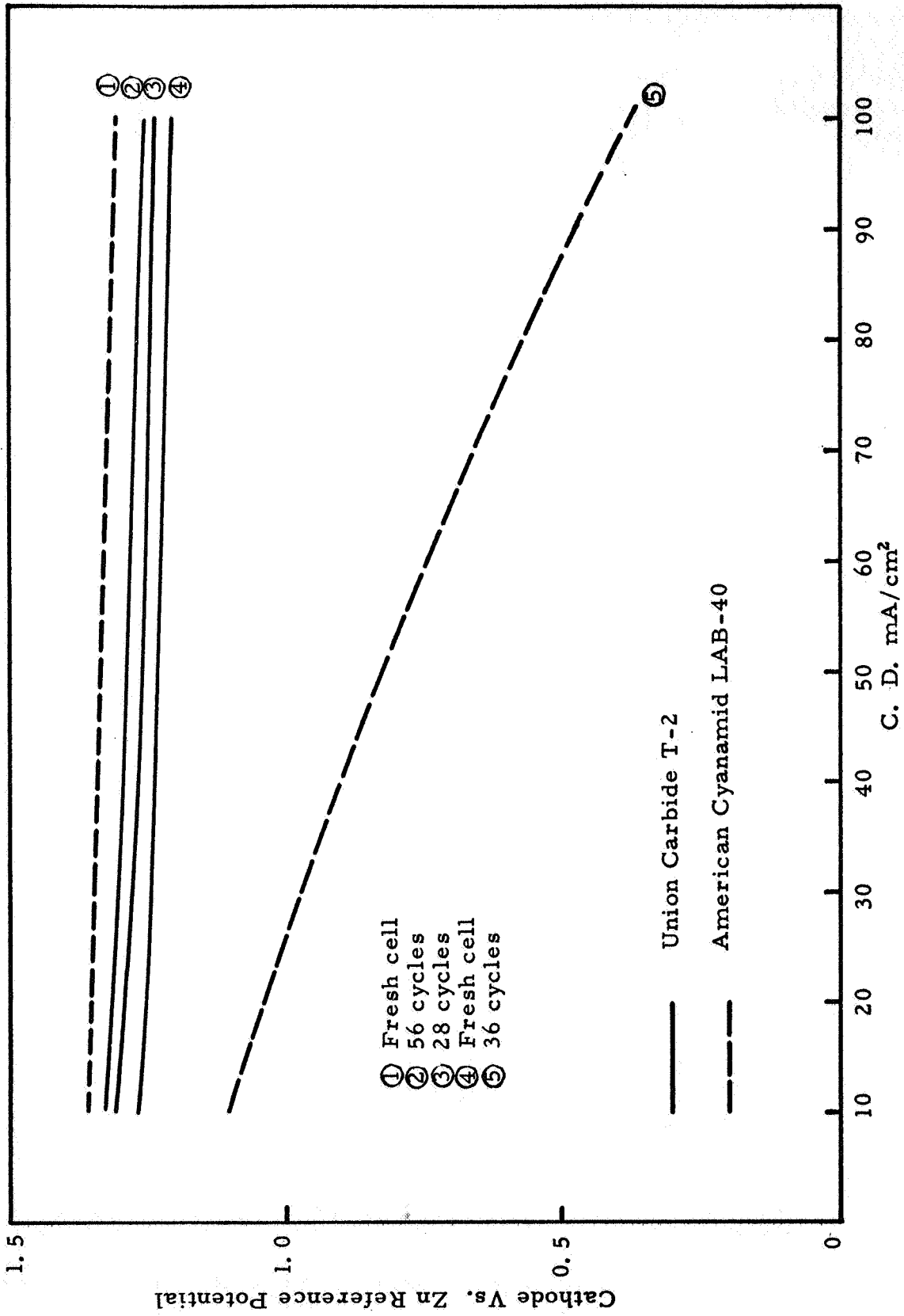


FIGURE 6
IR-Free Cathode Polarization - American Cyanamid LAB-40 Vs. Union Carbide T-2



C-4208

FIGURE 7

40°C Performance of 10 Ampere-Hour Cell Discharged at 26% Zinc Depth

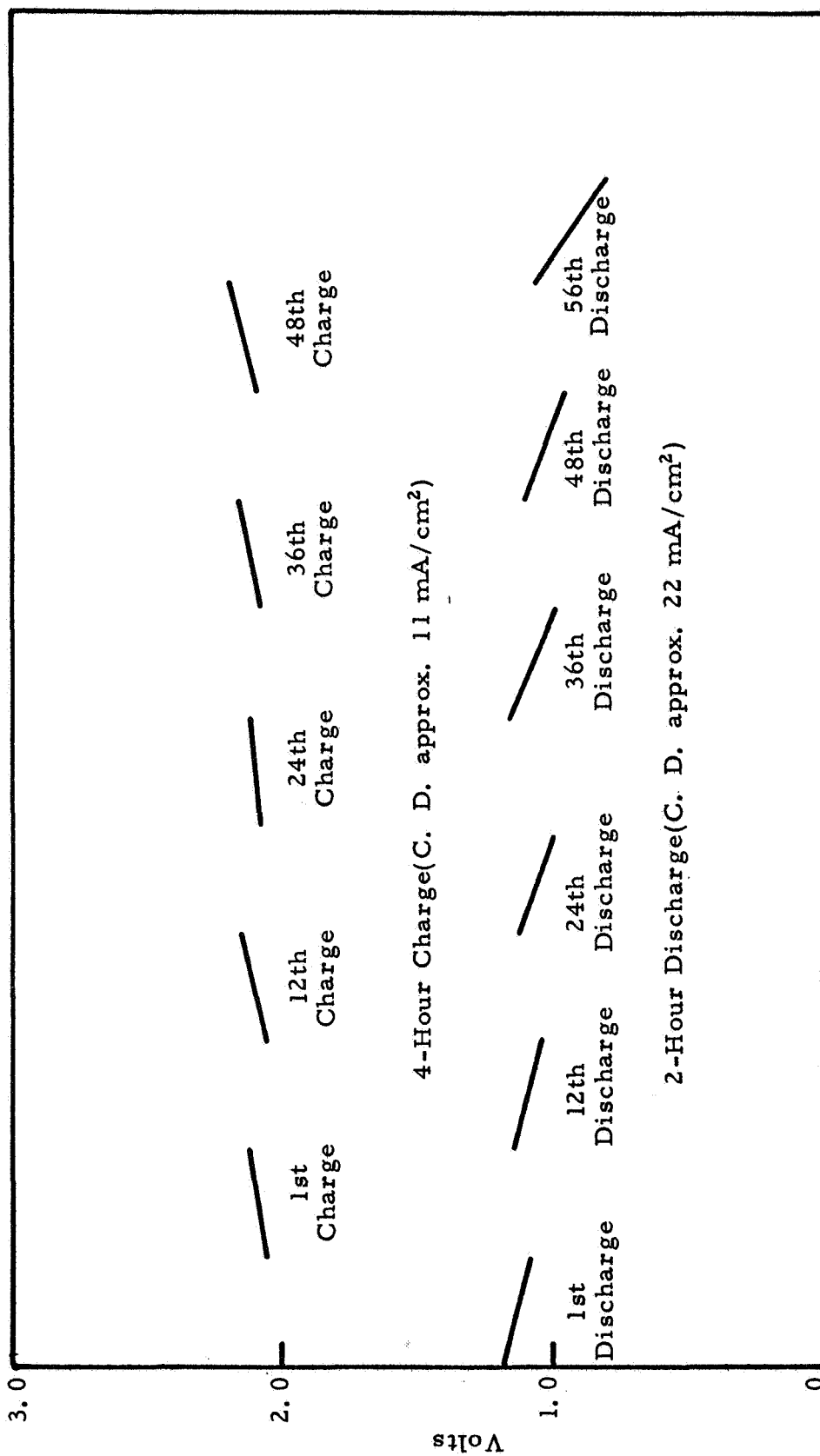
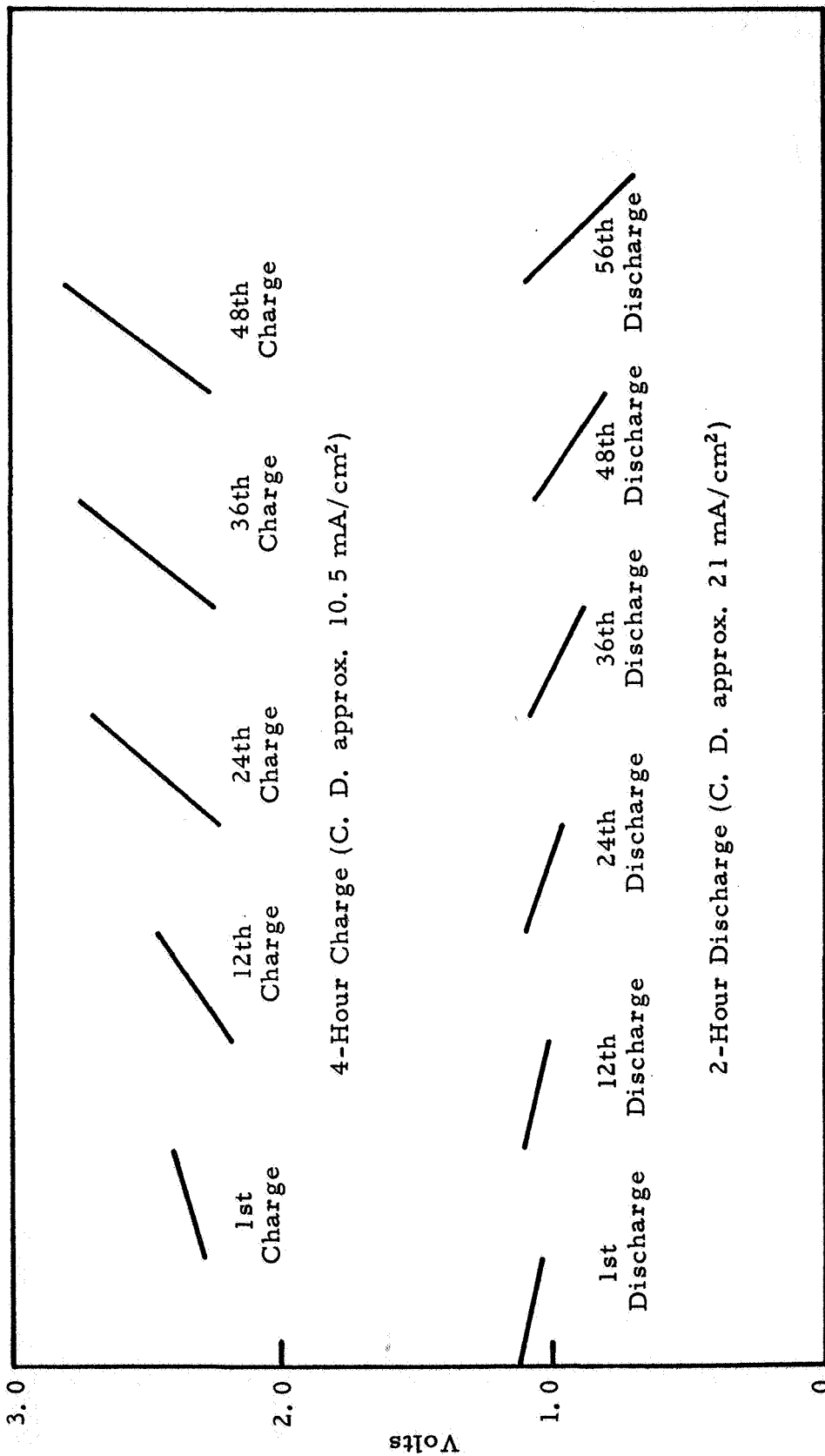


FIGURE 8
0°C Performance of 10 Ampere-Hour Cell Discharge at 26% Zinc Depth



NEW TECHNOLOGY

There are no new technological advances falling within the scope of this contract to be reported at this time.

PROGRAM FOR THE NEXT REPORTING INTERVAL

Continued exploration of means for reducing the zinc redistribution upon cycling from the upper portion of the electrode toward the bottom of the electrode will be emphasized. This work will include the evaluation of anode structures other than those presently being used. It is hoped that as a result cell cycling will be attained at discharge current densities even greater than 25 mA/cm² on the 2-hour discharge/4-hour charge regime.

The possibility of eliminating the separate charging electrode from the present cell construction through the use of the American Cyanamid LAB-40 oxygen electrode will be further investigated.

A solution to the problem of unstable electrolyte levels during cycling will be sought through redesign of the electrolyte expansion chambers at the top of the unit cell.

CONCLUSIONS AND RECOMMENDATIONS

Cycling of the zinc-oxygen unit cells at a discharge current density of 25 mA/cm² has been demonstrated. As of this time, cells have delivered 56, 108, and 56 cycles, respectively, at 0°, 25°, and 40°C. The cycle regime was a 2-hour discharge followed by a 4-hour charge. This corresponded to discharge of the zinc anode to a 25 percent depth based upon the theoretical capacity available from the actual amount of zinc built into the anode.

Redistribution of the zinc mass within the anode as cycling progresses resulting in the transfer of zinc from the top of the electrode to the bottom of the electrode was recognized as the limiting factor in determining cycle life. The rate at which the redistribution occurs was found to be a function of the depth to which the zinc electrode was discharged. For a zinc electrode of given capacity, therefore, the deeper the discharge per cycle, the greater was the redistribution of the zinc mass. The disadvantage of such redistribution is obvious. As the electrode moves toward the bottom of the anode

cavity, it becomes smaller in apparent cross sectional area and the current density both during discharge and charge becomes increasingly greater not only at the zinc electrode but also because of the redistribution in shape of the zinc electrode upon the oxygen electrode as well. Increasing the built-in capacity of the zinc electrode while maintaining a higher discharge rate of 25 mA/cm², as was the case in the cells reported above, results in a decrease of shape reorientation with attendant improvement in discharge voltage and cycle life. It is recommended that the construction of the zinc electrode be further explored with the objective of attaining the minimal amount of shape change as a function of discharge depth controlled through the total amount of zinc built into the electrode for a given higher current density discharge. Discharge current densities of 25 mA/cm² and higher should be explored in this experimental work.

PERMION 1770C was evaluated as a membrane separator and found to be the apparent equivalent to the previously used PERMION 110 both with respect to zinc dendrite penetration resistance and oxygen transfer resistance. This evaluation was made necessary by the fact that PERMION 110 is no longer available from the supplier.

The feasibility of using an American Cyanamid LAB-40 electrode both as a charging electrode and as the oxygen electrode was demonstrated. Apparent deterioration of this electrode during cell cycling, however, appears to be a limiting factor. It is recommended that further work be done in order to determine the extent to which such cycle life limitation can be minimized.

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Department of Chemistry
Pocatello, Idaho 83201

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