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FABRICATION AND TESTING OF SILVER-HYDROGEN CELLS

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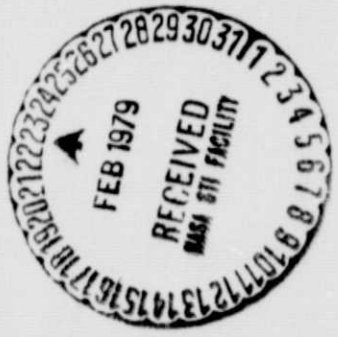

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NASA Lewis Research Center
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1. SUMMARY

This report covers the results and progress achieved on Contract NAS3-20805 for the development, fabrication and testing of Silver-Hydrogen secondary batteries. The program, which covered a ten month period, was a continuation of Contract NAS3-19780 for which a final report was delivered in October 1977.

The main thrust of the program was to improve the electrochemical utilization of sintered silver cathodes for use in Ag-H_2 aerospace batteries. To this end, most of the experimental effort involved fabrication and testing of silver electrodes which contained various additives. These electrodes were tested in single electrode cells and no major effort was made to optimize the other cell components throughout the program. This led to the use of a standard stack arrangement which featured the organic-inorganic separator developed by NASA. All cells were cycled in a regime designed to remove 75% of the cells nominal capacity based on 3.3 gms/AHr Ag utilization. In cases where performance degradation was observed, the main failure mode appeared to be corrosion of either the expanded silver current collector or the connection between the silver electrode and the electrode tab.

Promising silver electrodes from single electrode studies were used in the construction of 35 AHr Ag-H_2 cells. Two such cells were constructed during the course of the program and installed in heavy walled pressure vessels for testing. Based on the data obtained from all cells tested during the program, four lightweight 35 AHr cells were fabricated for delivery to NASA-Lewis. During acceptance testing these cells yielded an average gravimetric energy density of 30 WHr/lb.

2. INTRODUCTION

Many space probes and satellite systems employ rechargeable secondary batteries for peak power, eclipse portions of the orbit, and other auxiliary functions. Most systems employ nickel-cadmium or silver-zinc batteries for such applications. To achieve improvements, investigations are underway on nickel-hydrogen batteries. The silver-zinc cell system is limited in life due to the instability of the zinc electrodes, and the nickel systems are relatively heavy. This program is directed at the development of the silver-hydrogen battery system which represents the marriage of the silver electrode of the type used in silver-zinc cells and the hydrogen electrode of the type used in nickel-hydrogen cells. This combination of electrodes

offers a promising set of characteristics in terms of energy density and cycle life.

This development program which is a continuation of a previous contract NAS3-19780 is divided into the following tasks:

TASK I - SINGLE ELECTRODE STUDIES

A.1 Test Cells

The construction of ten boilerplate test housings for cycle testing of various single electrode combinations. The test housing design shall be such that a single electrode tested would be adaptable to cells of the 35 to 50 ampere-hour capacity range.

A.2 Component Optimization Studies

Construct single electrode test cells with an emphasis on optimizing the electrode separator configuration. Studies will be directed at maximizing the energy utilization of the silver electrode.

B. Series I - Life Test

Based on the screening investigations of the electrode configurations of the above Task, a number of configurations will be selected and subjected to life tests to evaluate the stability of the electrode matrix configuration.

C. Series II - Single Electrode Life Test

Based on the results of the above testing a selection will be made of most promising configurations and life tests will be conducted on a 1.2 hour discharge, 6.8 hour charge cycle at 75% depth. Cycle life goal shall be greater than 500 cycles with an extrapolated energy density of 77-88 watt hours per kilogram when the electrodes are scaled into full 35 to 50 ampere-hour cells.

TASK II - MULTI-ELECTRODE 35 AMPERE-HOUR CELL TEST

Four configurations from the above single electrode testing will be selected, constructed into 35 Ah stacks, installed in heavy walled test housings and feasibility testing shall be conducted. The two most promising configurations will then be selected and put on life test at 1.2 hour discharge, 6.8 hour charge for 500 cycles.

TASK III - FINAL CELL DESIGN

Based on the results of the boilerplate configurations of the above Tasks, an optimum design shall be selected for a lightweight 35 ampere-hour cell and a complete design will be prepared including detailed drawings.

TASK IV - CELL FABRICATION

Based on the above design, four 35 ampere-hour cells should be constructed and assembled in lightweight test housings. The cells shall be subjected to 5 acceptance cycles and then delivered to NASA-Lewis.

TASK V - RELIABILITY AND QUALITY ASSURANCE

Maintenance of a quality program in conformance with NASA, DOD requirements for all aspects of the program.

TASK VI - REPORTING REQUIREMENTS

The preparation of monthly summary reports, and a final report.

3. RESULTS AND DISCUSSION

3.1 TASK I - Single Electrode Studies

Single electrode studies were conducted to identify promising silver electrodes for use in 35 AHr Ag-H_2 cells. Seventeen (17) such cells were constructed during the contract period. In addition, life testing was continued on four (4) cells which had been assembled under contract NAS3-19780. All Ag electrodes were fabricated to give a theoretical capacity close to 3.5 AHr (7.0-7.5 gms Ag sinter) and a nominal thickness of 30 mils. Cells constructed under the previous program contained silver electrodes whose theoretical capacity was close to 4.0 AHr. The standard electrode shape and cell configurations are shown in Figures 1 and 2. All cells were charged at .33A for 6.8 hours and discharged at 1.7A for 1.2 hours yielding a 1.1:1 charge/discharge ratio. The resulting current densities experienced by the silver electrode during this cycle regime are 6.5 ma/cm^2 and 32.5 ma/cm^2 on charge and discharge respectively.

All silver electrodes used 1/4" wide 5 mil thick silver tabs except in cases where nickel expanded metal was used as a current collector. The silver tabs were spot welded to the silver sinter and then hot forged to assure good adhesion and electrical contact. In cases where nickel screen was used, the silver sinter was removed from the tab area and a nickel tab was spot welded to the silver sinter and then hot forged to assure good adhesion and electrical contact. In cases where nickel screen was used, the silver sinter was removed from the tab areas and a nickel tab was spot welded directly to the screen.

Other components for the single electrode tests were: H_2 electrodes (3 mg PT/cm^2 on nickel screen), NASA I/O separators (which were GFE), fuel cell grade asbestos, and Vexar gas diffusion screens. The electrode stacks were retained between 2 polysulfone endplates and compressed using stainless steel tie rods which also serve as electrical feed throughs. All tests were conducted in boilerplate test housings

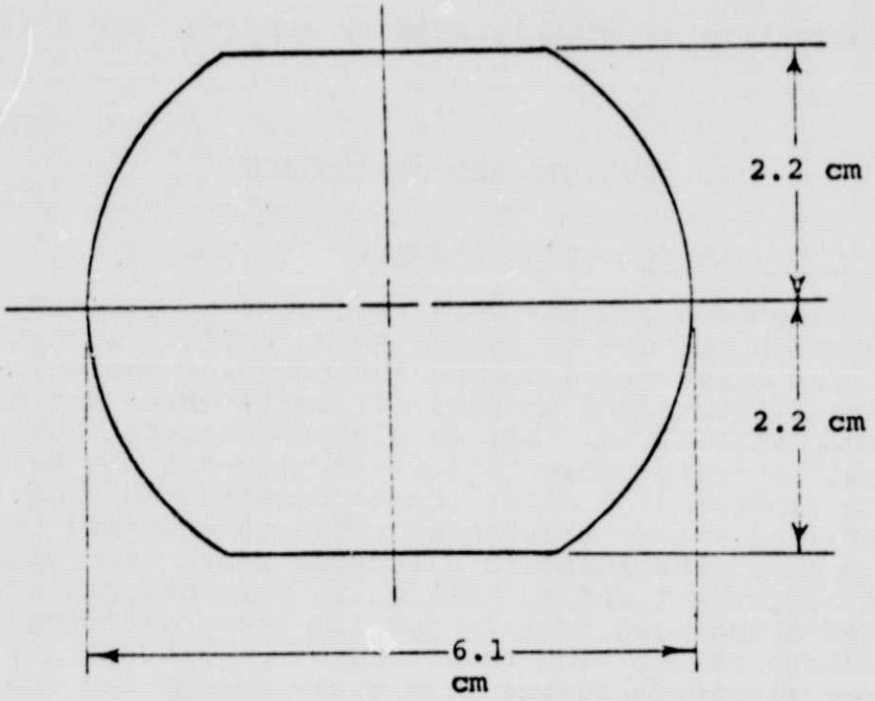


FIGURE 1

ELECTRODE SHAPE FOR SINGLE ELECTRODE CELL TESTS

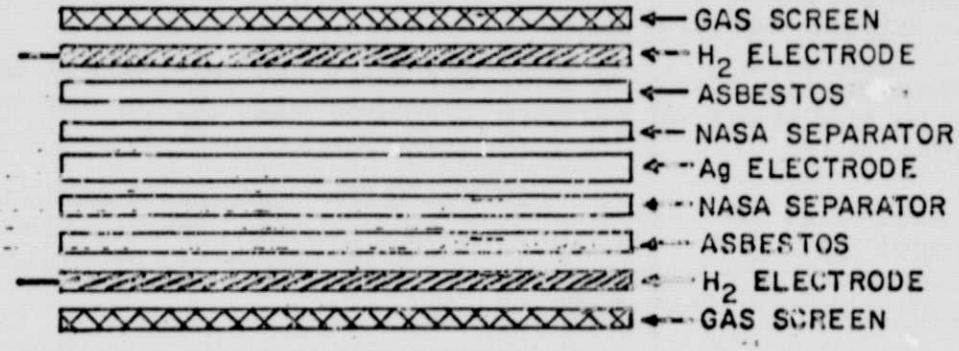


FIGURE 2 Ag-H_2 STACK ARRANGEMENT

which were proof and leak tested to 750 lbs. psig (Fig. 3).

Tables 1-3 show the construction variables of the test cells along with cycles accumulated as of the programs completion. All cells were impregnated by impregnating 30% KOH into the stack while in a vacuum environment. The immersed stack was then heated to 160°F and maintained at that temperature overnight. The discharged cell was then placed in the boilerplate housing with a 100 psi precharge of H_2. This precharge is added to make the cell positive limited and thus protect it should reversal occur.

Cell #6 contained a silver plate which had already accumulated 1000 cycles before failure. The electrode was charged and discharged in free electrolyte and then assembled with all new components. The cell cycled an additional 500 cycles before failing again. Apparently cycling in excess electrolyte allows the electrode to gas freely thus expelling any foreign material or isolated active material which may have accumulated in its pores. The failure at 1500 cycles can be attributed to a loss of electrical contact due to corrosion of the silver current collector.

Inspection revealed that the grid had completely disappeared and only the embossed pattern remained visible on the electrode surface.

Cell #14, containing an inorganic separator designated 2002 developed by E.R.C., cycled for 180 cycles. The cells capacity had been somewhat low since cycle 470 and use of this argentistatic membrane was not investigated further.

Cell #18 contained PT electrodes utilizing silver current collectors. The cell cycled well but began to polarize at about the 900 cycle point. The cell was given an extended overcharge and then reimpregnated in an attempt to restore capacity. Both methods having failed the cell was discontinued after 1100 cycles.

Cell #19 was a cell which contained no argentistatic membrane and only 2 layers of fuel cell grade asbestos were used as separators. In addition a carbon foam which is 1/8" thick and approximately 80% porous was used as a gas diffusion screen. This approach was tried since the use of carbon foam will increase the stack height which could aid in the thermal management of the cell. The cell cycled for 500 cycles but after that time failed to accept charge.

The first set of cells assembled under this contract were cells 20 through 25 which contained silver electrodes fabricated from Ag powders supplied by NASA-Lewis. These powders were co-precipitated with additives of potassium-titanate or carbon. None of the cells exhibited promising performance. Cell 21 developed an internal short on the first cycle and none of the other cells showed capacity in excess of 38% of theoretical. This is equivalent to approximately 5.3 gms Ag/Hr which is clearly unacceptable. Since the performance of these cells did not improve with continued cycling they were

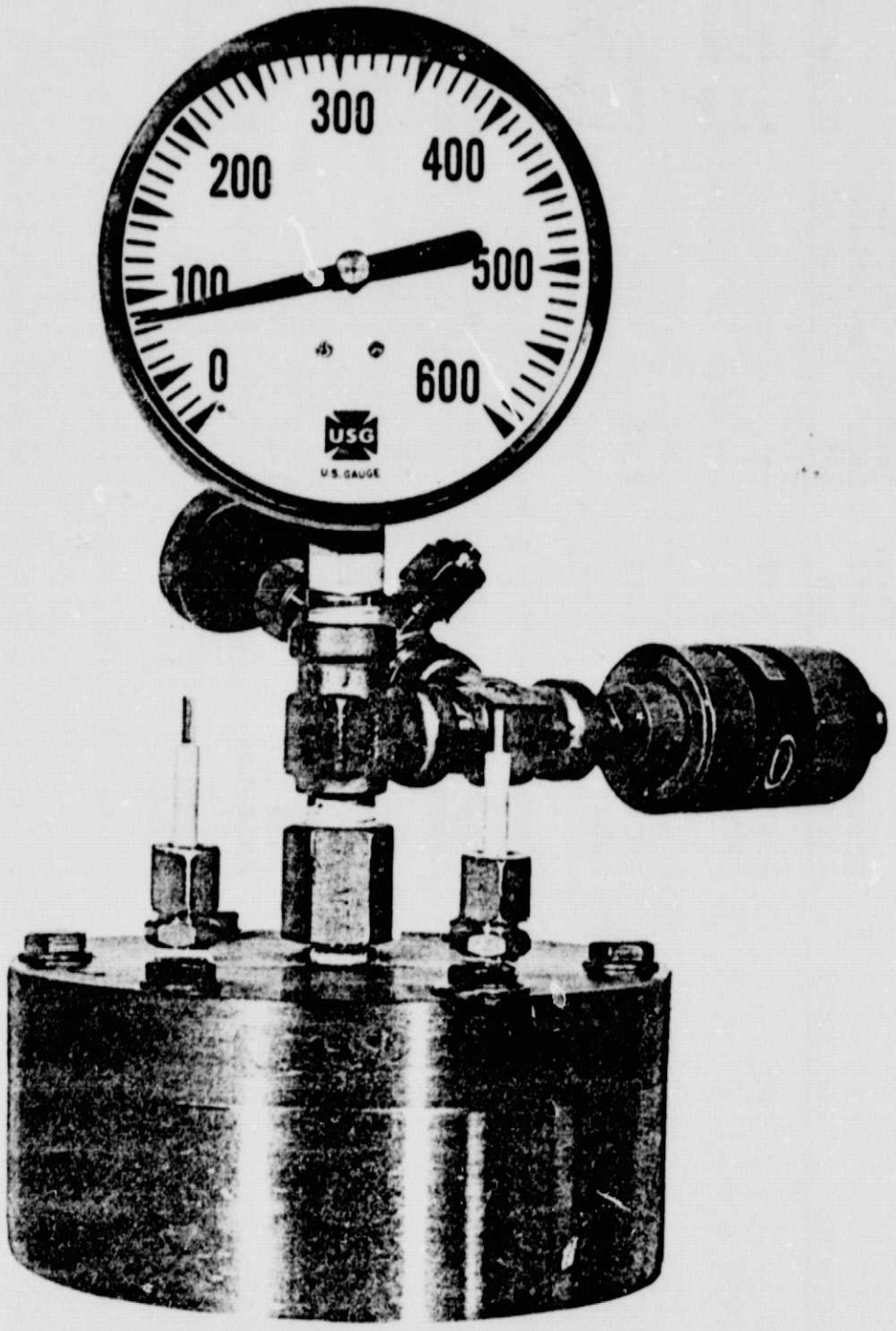


FIGURE 3 SINGLE ELECTRODE TEST CELL

TABLE I

SINGLE ELECTRODE STUDIES CONTINUED FROM PREVIOUS CONTRACT

CELL NO.	Ag ELECTRODE	H2 ELECTRODE	SEPARATOR LAYERS	THEO. CAP. AHR	INITIAL CAP. AHR	INITIAL Ag UTILI. AHR/Gm AG	COMMENTS
6	STD. .030"	PPF	NASA I/O Asbestos	3.99	--	--	Ag electrode cycled for 1500 cycles. See Text
14	STD. .030"	ERC	ERC 2002 Asbestos	4.10	2.75	0.34	Completed 780 cycles. Low capacity since cycle 400.
18	STD. .030"	ERC	NASA I/O Asbestos	4.10	2.25	0.27	Good performance thru 900 cycles. Discontinued after 1100 cycles.
19	STD. .030"	ERC	NASA I/O Asbestos	4.10	2.25	0.27	Failed after 500 cycles. Contained carbon foam as gas diffusion spacer.

TABLE II

SINGLE ELECTRODE STUDIES USING NASA-LEWIS SUPPLIED Ag/ADDITIVE POWDERS

CELL NO.	Ag ELECTRODE	H_2 ELECTRODE	SEPARATOR LAYERS	THEO. CAP. AHR	INITIAL CAP. AHR	INITIAL UTILI. AHR/Gm Ag	CAP. AT CYCLE 100	COMMENTS
21	E12 12.1:1 Ag/KT .028"	ERC Ag-Ex-met	NASA I/O Asbestos	3.27	Shorted	---	Terminated	---
22	E10 20:1 Ag/c .028"			3.32	1.25	0.19	Terminated	---
23	E13 20:1 Ag/KT .029"			3.14	1.00	0.16	Terminated	---
24	E14 20:1 Ag/KT .031"			3.56	1.25	0.18	Terminated	
25	E11 21:1 Ag/c .026"			3.24	0.75	0.12	Terminated	---
33	E16	ERC Ni Ex-met		3.33	2.00	0.30	1.50	DISCONTINUED AFTER 300 CYCLES

TABLE III

SINGLE ELECTRODE CELL TESTS

CELL NO.	Ag ELECTRODE	H_2 ELECTRODE	SEPARATOR LAYERS	THEO. CAP. AHR	INITIAL CAP. AHR	INITIAL UTILI. AHR/GM Ag	CAPACITY CYCLE 100 AHR	CAPACITY CYCLE 400 AHR	CAPACITY CYCLE 500 AHR	COMMENTS
26	1% CuO	ERC	NASA I/O Asbestos	3.51	2.50	0.36	2.50	2.00	1.76	Stable at program end
27	1% PbO			3.76	1.00	0.13	Termi-nated	--	--	Lead dendrite growth
28	5% CdO			3.62	3.25	0.45	2.00	1.67	Termi-nated	Resoaked at cycle 400
29	0.1% Au			3.51	3.00	0.43	2.50	2.25	2.18	Stable at program end
30	5% CeO_2			3.39	2.75	0.41	2.50	2.20	1.83	
31	1% HgO			3.67	2.25	0.31	2.00	1.33	2.32	Resoaked at cycle 400
32	1% PbO 1% HgO			3.44	1.75	0.25	Termi-nated	---	---	Lead dendrite growth
34	Std. w/ Ni Ex-met			---	Failed	---	---	---	---	Failure attributed to oxide film on nickel screen
35	Std. See Comments			3.57	2.50 see comments	0.35	Termi-nated	---	---	Ag electrode from cell 5-20 after 735 cycles completed
36	5% CeO_2 Ni Ex-met			--	--	--	2.33	---	---	Ag electrode sintered in H_2. Stable at program end.
37	Std. Ni Ex-met			3.40	2.40	0.35	2.50	---	---	Ag electrode sintered in H_2. Stable at program end.

terminated.

The second set of single electrode test cells (26 through 32) built under this contract contained E.R.C. Ag electrodes with various metal additives to aid in silver recrystallization and raise the O_2 overvoltage.

The seven cells were prepared concurrently and cycling was initiated. Cells 27 and 32 containing lead additions failed almost immediately completing less than 2 cycles. Dissection of the cells showed that lead had passed through the argentistatic membrane and had already begun to plate on the H_2 electrodes which were grey in color. Failure of these cells was attributed to lead dendrite growth and as such the cells were not rebuilt.

Cells 28 and 31 began to show low capacity (below 2 AHr) at about the 200 cycle point. At cycle 400 both cells were given an extended overcharge of approximately 50%. As their capacity did not improve the cells were removed from automatic cycling. At this point the cells were deliberately overdischarged to test their tolerance to reversal. Both cells were discharged to zero volts and then over-discharged at 1.7A for 4 hours. Time-voltage curves were taken during this test and the performance of Cell 28 is in Figure 4. No measurable pressure changes were observed during reversal. Upon termination of the overdischarge test the cells were fully charged and discharged. It should be noted that both cells showed capacities similar to those observed before reversal indicating the high tolerance of the Ag-H_2 cells to over-discharge.

In a final attempt to restore capacity, Cells 28 and 31 were reimpregnated with 30% KOH. After this treatment Cell 31, which regained an additional 4 grams of electrolyte showed improved performance and cycling was continued. The cell has completed 500 cycles as of the program's end and exhibits good capacity although its discharge voltage is somewhat low.

Cell #28, which did not improve after reimpregnation was disassembled and examined. Its separators were very discolored and the platinum electrodes were grey indicating metal penetration of the argentistatic membrane and platinum recrystallization.

Cell #26 which showed low capacity since cycle 200 showed signs of a slow recovery over the next 200 cycles. Its capacity increased 33% over the 200-400 cycle period. During the next 100 cycles it again showed signs of decay. The reason for this behavior is not known, and cycling was continued through the program's end to see if this phenomenon can be repeated.

Cell #29, which contained a silver electrode fabricated with .1% gold black, exhibited superior performance throughout the program. It showed a low capacity at cycle 318 which was attributed to H_2 starvation due to a slow gas leak on the boilerplate test vehicle. With this condition remedied the cell showed a silver utilization of

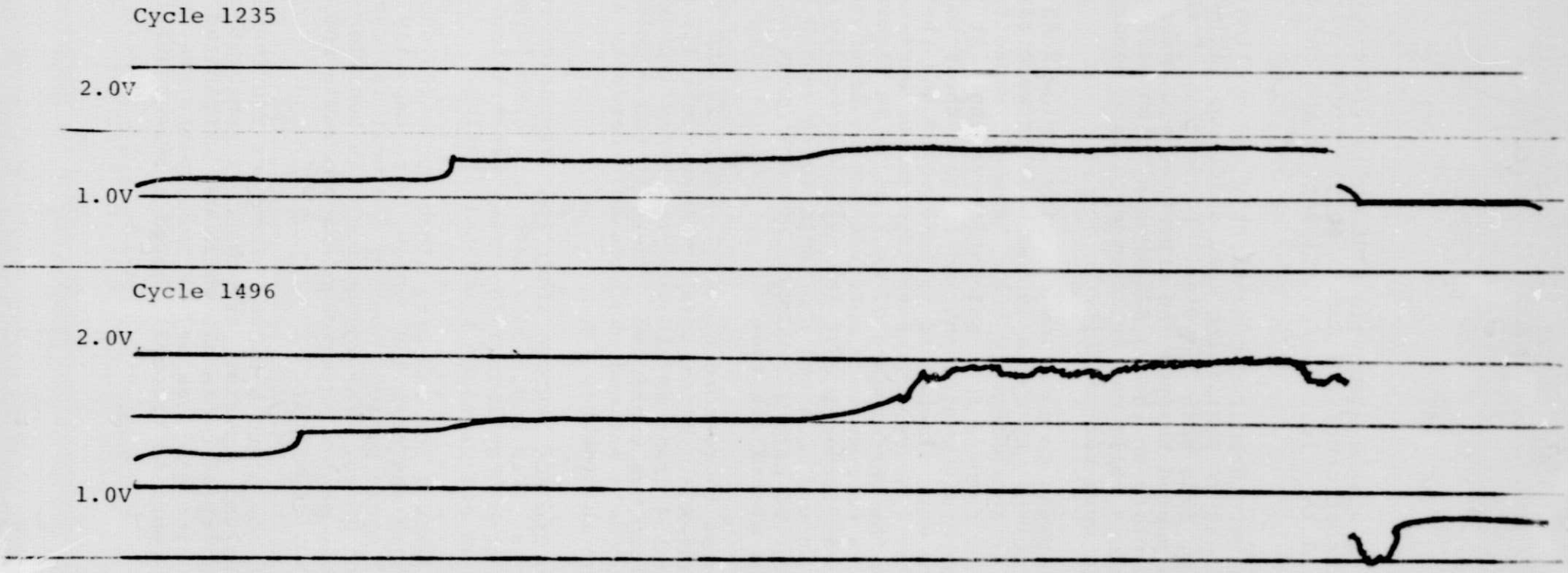


FIGURE 4

PERFORMANCE OF SINGLE ELECTRODE CELL #3

62% of theoretical (.31 Ah/g) at 500 cycles.

Cell #30 which contained 5% CeO_2 added to the silver powder showed the highest initial silver utilization of all cells tested. Its performance remained excellent for 400 cycles at which point its silver utilization was 65%. However, at the 500 cycle point it was found that its utilization had dropped to 54% or .27 AHr/gm Ag. Figure 5 shows Ag utilization as a function of cycle life for Cells 29 and 30.

Cell #33 which contained a silver powder designated Z16 supplied by NASA-Lewis was given 300 cycles before it was disassembled. The cell showed relatively low capacity (2 AHr) at its initial cycle and its performance deteriorated rather rapidly. The cell was overcharged and reimpregnated at the 300 cycle point with no improvement in performance observed. Upon inspection the stack appeared very clean and therefore its failure was attributed to a loss of capacity of the Ag electrode rather than a physical defect.

Cell #34 was built in an attempt to solve the problem of silver grid corrosion. This cell contained a silver electrode which utilized an expanded nickel current collector. The cell failed almost immediately and was rebuilt several times without success. Close inspection showed that the nickel screen had a blue coloring indicating oxidation which probably occurred during sintering. To alleviate this problem new electrodes were prepared and sintered in a hydrogen atmosphere. Cells 36 and 37 used silver electrodes prepared in this manner and both have completed 40 cycles as of the program's completion.

Cell #35 was assembled using a positive plate that had previously been used in 20 AHr Cell 5-20. The 20 AHr cell had completed 735 cycles prior to failure. In the single electrode cell, 124 cycles were accumulated before the stack was disassembled because of severe polarization on discharge. Disassembly revealed extensive grid and tab corrosion of the silver electrode. The area of the tab fastened to the plate showed considerable attack to the point where there was virtually no adhesion.

This completed the single electrode tests indicated under this program. Data obtained from these tests were used in the selection of electrodes for 35 AHr Ag-H_2 cells. (See Figures 6 through 9).

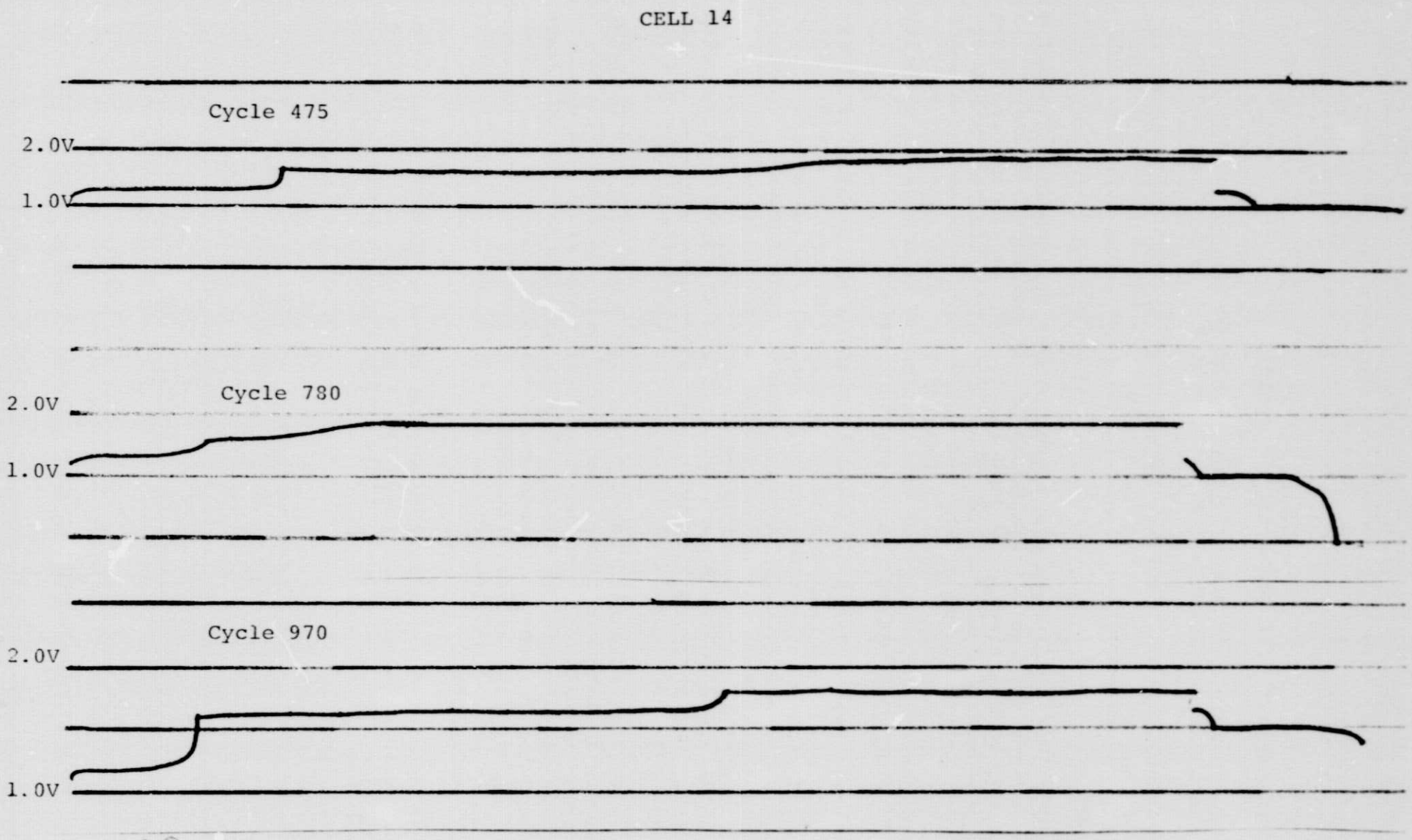


FIGURE 5
PERFORMANCE OF SINGLE ELECTRODE CELL #14

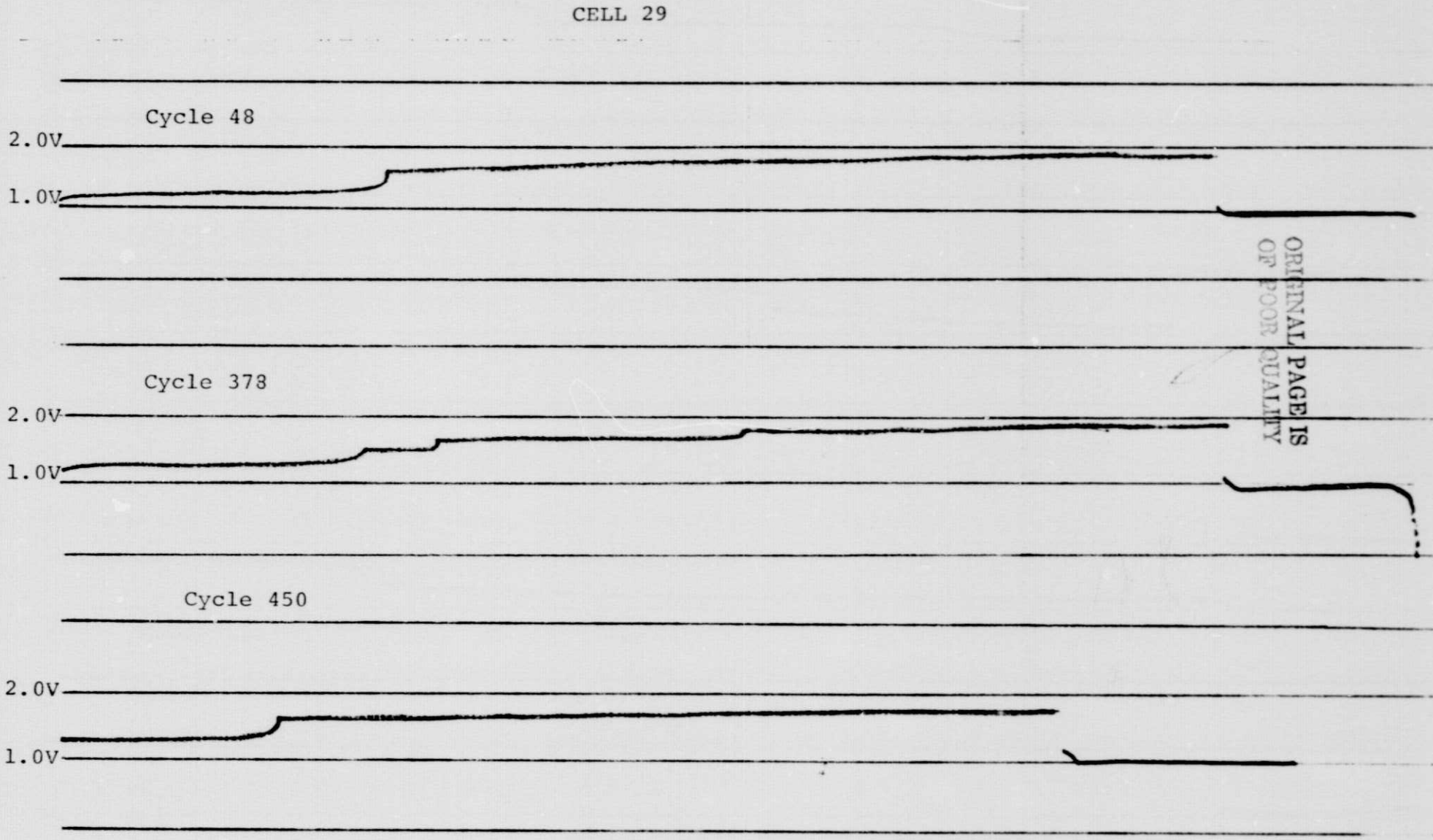


FIGURE 6

PERFORMANCE OF SINGLE ELECTRODE CELL #29

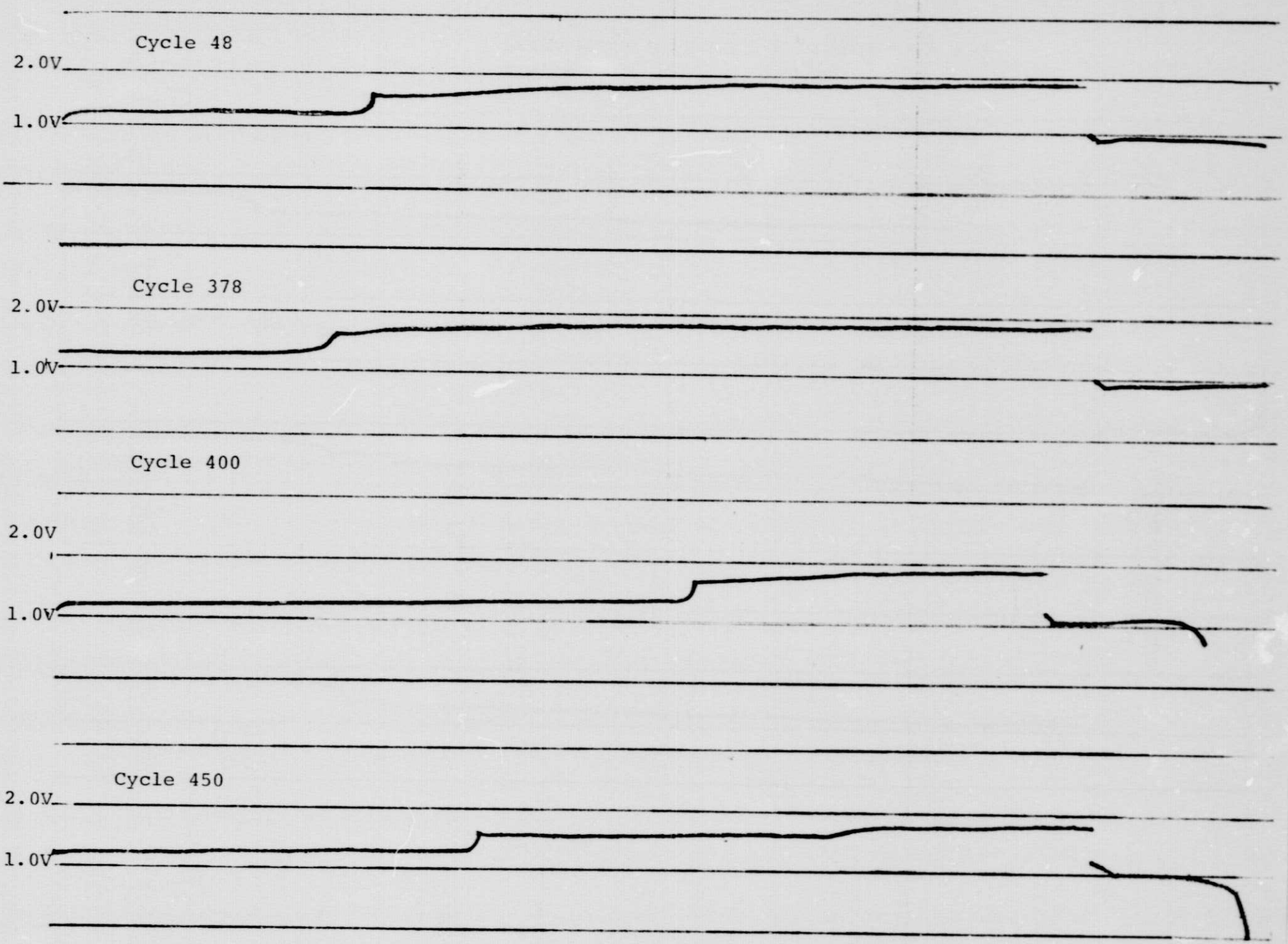


FIGURE 7 - PERFORMANCE OF SINGLE ELECTRODE CELL #30

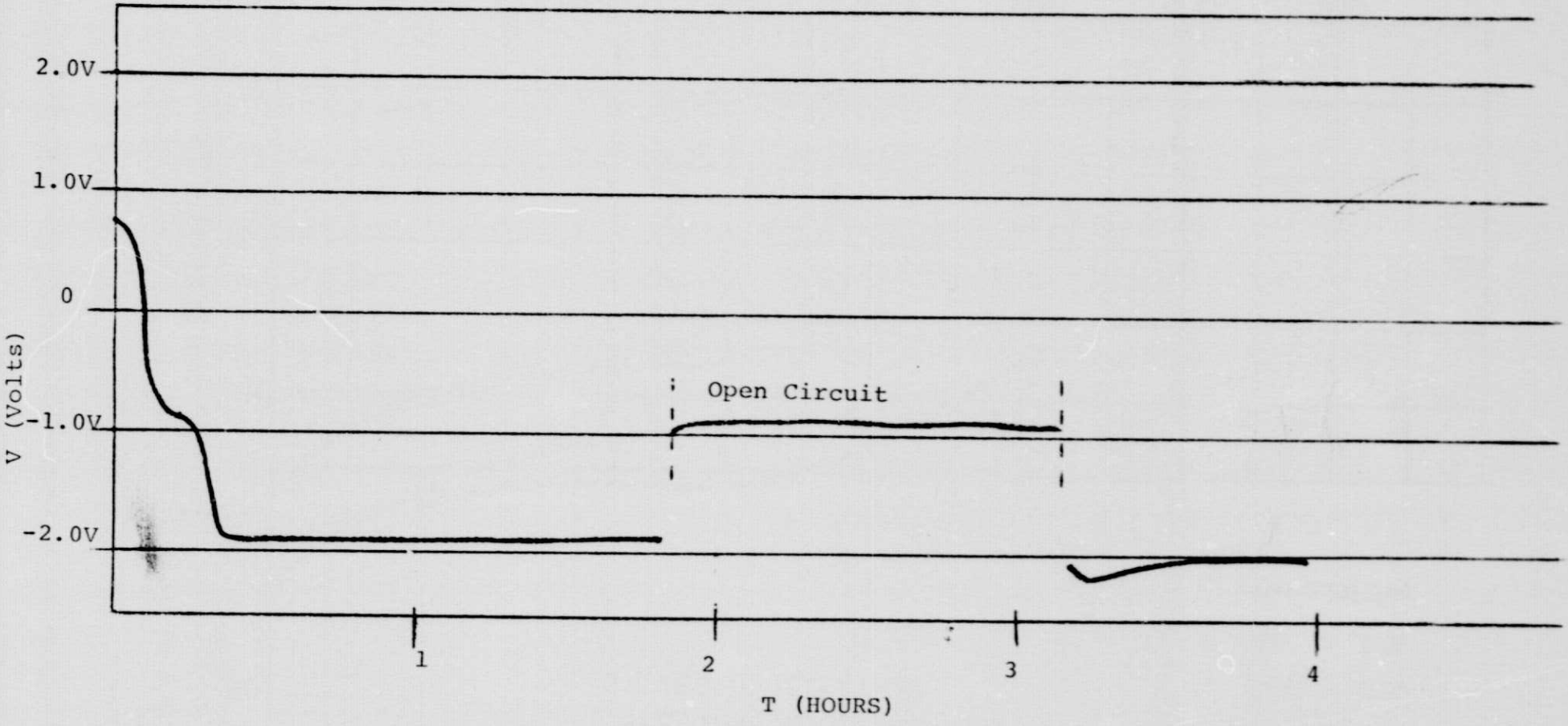
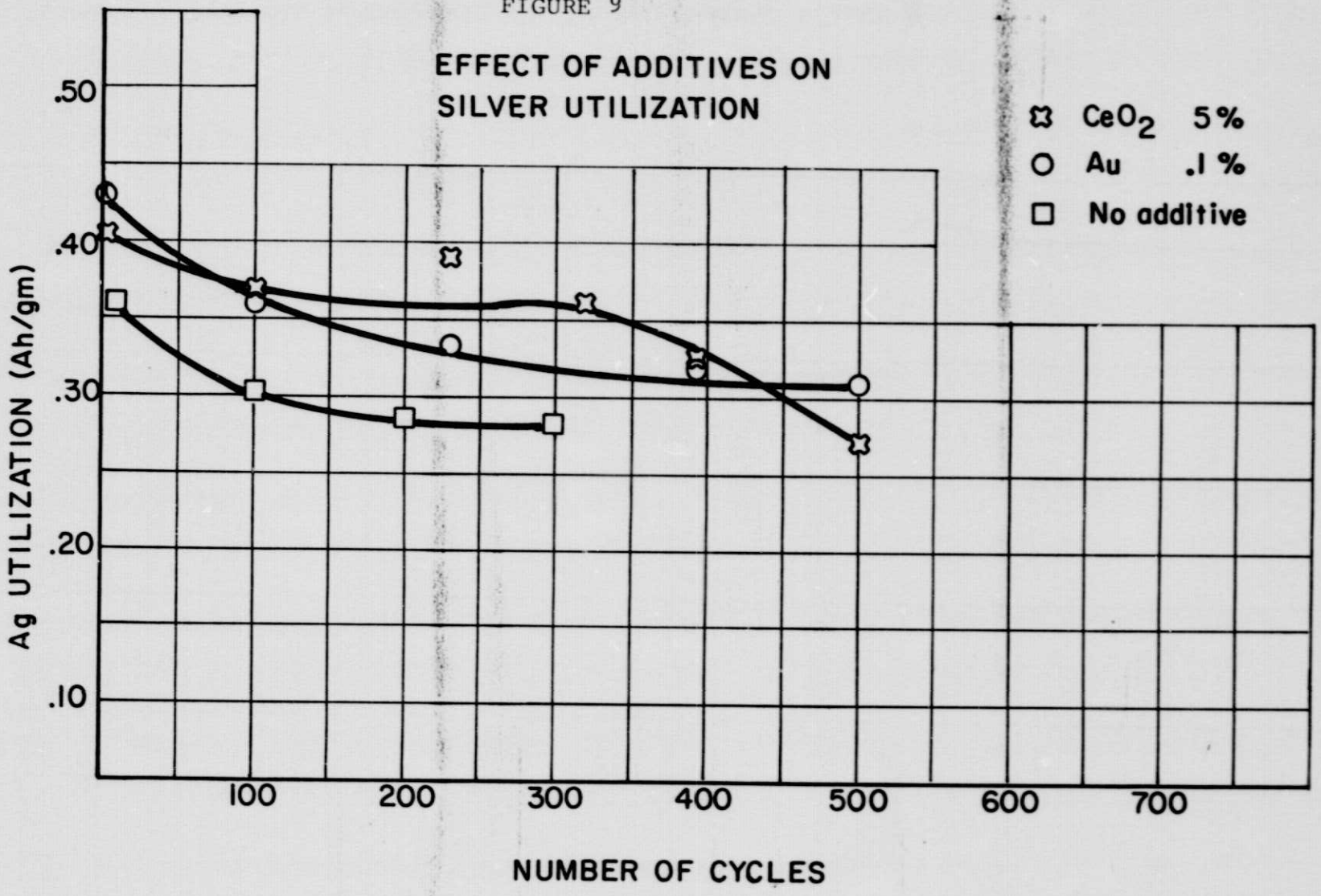


FIGURE 3 - REVERSAL OF CELL #28 (DISCHARGE CURRENT 1.7 AMP)

FIGURE 9


EFFECT OF ADDITIVES ON
SILVER UTILIZATION
CeO2 5%
Au .1 %
No additive
.50
.40
.30
.20
.10
Ag UTILIZATION (Ah/gm)
100
200
300
400
500
600
700
NUMBER OF CYCLES

3.2 TASK II - Multi-Electrode Test Cells

Three 20 AHr cells which were constructed under the previous Ag-H_2 contract continued cycling during this reporting period. These cells contained 10 positive plates with an average loading of 7 gms of silver sinter per plate.

Cell 5-20 which used Vexar as a gas diffusion screen and no alignment mandrels cycled successfully for 735 cycles at which point the cell failed. The cell was disassembled and one of its silver electrodes used in the construction of single-electrode Cell #35. Corrosion of the silver grid and tab on the silver electrode was observed.

Cells 11-20 and 12-20 were of the same basic construction except that the former used a central tie rod for alignment purposes and the latter used 2 tie rods. Both cells completed 900 cycles at which point they began to lose capacity rapidly. Once again the failure mode was corrosion of the silver grid and tab.

Two 35 AHr cells were constructed during the course of the program using the most promising electrodes from the single electrode test cells. The cells contained 14 positive plates with a nominal loading of 7 gms of Ag sinter per plate. Double tie rods were used for stack alignment. Component weights for these cells are shown in Table IV.

Cell 13-20 was fabricated with 5% CeO_2 added to the silver plates. During the initial deep cycles at 12A the cell yielded a capacity of 36 AHr or 75% of theoretical capacity. The cell was placed on automatic cycling at 3.8A charge for 6.8 hours and discharge at 21.3A for 1.2 hours. At the 100 cycle point the cells capacity had dropped to 27.4 AHr which is .29 AHr/gm Ag or 58% of theoretical. This is comparable to the performance of the CeO_2 single electrode cell at cycle 500. The reason for this rapid drop in capacity is not known but is probably related to heating effects encountered in scaling up to 35 AHr size cells. The cell appeared to stabilize at this utilization level and neither reimpregnation or overcharge improved this performance as the cell was still capable of delivering 75% of capacity. Automatic cycling was continued.

Cell 14-35 was identical in construction to Cell 13-20 except that the silver plates contained .1% Au black. This cell delivered 35 AHr during initial deep cycling which is 68% of theoretical and has completed 40 cycles as of the end of the contract period.

3.3 TASK III - Final Cell Design

At the program's outset a design analysis was undertaken to establish optimum cell diameter for a 35 AHr Ag-H_2 cell. Three designs were considered using 2-1/2", 3", and 3-1/2" as the cell's outer diameter. Since we expect the main problem of the 35 AHr cell to be heat transfer, the 2-1/2" diameter design was selected since it

TABLE IV

20 AH BOILER PLATE TEST CELLS

CELL NO.	SILVER ELECTRODE	HYDROGEN ELECTRODE	SEPARATOR	GAS DIFFUSION IER	CYCLES TO FAILURE	COMMENTS
1 (5-20)	STD 10 x .030	ERC	NASA I/O Asbestos	Vexar	735	Grid corrosion
5 (11-20)	STD 10 x .030	ERC	NASA I/O Asbestos	Carbon Foam	900	Single Alignment rod
7 (12-20)	STD 10 x .030	ERC	NASA I/O Asbestos	Carbon Foam	900	Double Alignment rods

provides the closest stack proximetry to the cell case.

Cells for delivery to NASA-Lewis will utilize Ag-positives to which 5% CeO_2 has been added. The current collector will be expanded silver metal to which a 5 mil thick 1/4" wide silver tab has been hot forged. The negative electrode will be of the standard ERC type; 3 mg PT/cm^2 with expanded nickel as the current collector and 5 mil thick 3/8" wide nickel tabs. Nickel was selected over silver for use in the H_2 electrode to avoid possible corrosion problems even though the use of nickel will have a slighly negative impact on cell weight. The H_2 electrode will have a porous TFE backing which is 2 mils thick.

Other components for the cell stack will be asbestos, NASA I/O separator and a gas diffusion screen. Two of the cells will use Vexar polypropylene as gas diffusion screens and two cells will contain carbon foam gas diffusion screen/spacers. The carbon foam acts as a stack extender and may help alleviate any heat dissipation problems by increasing the stack height. In addition, a polysulfone edge mask is utilized to protect all edtes from possible silver shorting. The asbestos - I/O separator combination is designed to provide a 1/16 inch average separator extension on the outside edge of the stack. The separator sequence will be arranged so that the inorganic side of the I/O faces the Ag electrode and the asbestos is placed facing the H_2 electrode. Alignment of the stack is accomplished by using 2 polysulfone mandrels. Finally, the stack is retained by 2 polysulfone endplates and compressed by attaching nuts to a threaded stainless steel rod which is located within the two polysulfone mandrels.

The cell stack will be contained within a 2-1/2" O.D. cylinder made of .016" Inconel type 718. The cylinder will be 5.8" long and will be fitted with 2 hemispherical end caps resulting in a total length of 8.5"; all seams will be electron beam welded. A schematic of the cell case is appended. Immobility of the stack in the vessel is accomplished by use of a ring that is welded to the can. The ring itself is fabricated with a crossmember which contains 2 holes. The same stainless rods that hold the stack under compression are fed through these holes and fastened with nuts. The center of the crossmember is cut away to accomodate the connection between the the positive leads and the terminal feed-throughs.

The terminal feed-throughs for the cell are of a plastic compression type known as the Zeigler seal. Electrical contact to the cell is provided by gold plated 3/16" copper-silver alloy rod which is compressed within the plastic of the seal. The rods are fastened to their respective leads with gold plated brass nuts. The negative terminal has a small hole drilled through it to allow for electrolyte filling and draining and the addition of a H_2 pre-charge.

TABLE V

COMPONENT WEIGHTS FOR 35 AHR CELLS

	13-20	14-35
Ag Sinter	95.95 gms	100.10 gms
Additive	5.05	0.10
Ag Screen & Tabs	7.80	7.80
PT electrodes & Tabs	49.50	49.50
Edge masks	3.08	3.08
Asbestos	17.36	17.36
NASA I/O	47.88	47.88
Gas screens	5.60	5.60
End plates	21.60	21.60
Alignment tubes	1.60	1.60
Rods & nuts	28.00	28.00
Misc.	1.28	1.28
Electrolyte	67.10	63.80
	351.80 gms	354.80 gms
Approximate total weight when installed in light-weight vessel	565 gms	568 gms
Formation capacity	36 AHr	34 AHr
Calculated Wt. Energy Density	70.1 WHr/Kg 31.7 WHr/lb	65.8 WHr/Kg 29.9 WHr/lb

The components of the cells and their weights are given in Table V.

4. CONCLUSIONS

It appears that significant improvements in the useable capacity of the silver electrode can be achieved by the addition of various additives. Utilization of about .30 AHr/gm Ag can be achieved over extended cycling with the use of CeO_2 or Au as an additive. Varying the percentage of the additive may possibly have beneficial effects and will be explored further during future study.

Silver electrodes exhibit performance degradation in the 900-1000 cycle range and this can be attributed to corrosion of the expanded silver current collector. Use of more inert metals or various silver alloys may alleviate this problem. In fact, nickel was tested as a current collector for silver electrodes and satisfactory performance was achieved. Although these cells have only accumulated about 50 cycles we do not expect grid corrosion to occur based on data gathered from Ni-H_2 cells which have cycled over 2000 times with negligible performance degradation.

Weight analysis of lightweight cells showed that the 35 AHr Ag-H_2 cell is capable of delivering 30 WHrs/lb. Improvement could be made by using a lighter weight pressure vessel. Designing components to minimize welding, possibly by hydroforming the whole cylinder and one end cap could allow for the use of lighter weight pressure vessels and boost energy density to the 35-50 WHr/lb. range.