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**ANNUAL REPORT FOR 1978 ON
RESEARCH, DEVELOPMENT AND DEMONSTRATION OF
NICKEL-IRON BATTERIES FOR
ELECTRIC VEHICLE PROPULSION
Contract No. 31-109-38-4292**

by

Eagle-Picher Industries, Inc.

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ARGONNE NATIONAL LABORATORY, ARGONNE, ILLINOIS
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Prepared for
The Office for Electrochemical Project Management
Argonne National Laboratory
Under Contract No. 31-109-38-4292

By

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Joplin, Missouri

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October 1979

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ANNUAL TECHNICAL STATUS REPORT

Contract 31-109-38-4292

Report No. 1

Fiscal Year 1978

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1.0 INTRODUCTION

The objective of this program is to develop a Nickel-Iron battery suitable for use in electric vehicles. Ultimately, it is expected that a number of these batteries will be demonstrated under the Electric & Hybrid Vehicle Act of 1976.

The technical program goals from the Argonne National Laboratory (ANL) Statement-of-Work (SOW) and the Eagle-Picher Design and Cost Study Program report, ANL Contract 31-109-38-3709, are tabulated below:

	<u>ANL SOW</u>	<u>EPI REPORT</u>
Battery Capacity, Kw·hr-100% rated	20-30	28.8
Specific Energy, w·hr/kg	60	60
Specific Power w/kg-Peak (30 sec)	100	100
-Sustained	20	30
Duty Cycle Charge (hr)	4-8	4-8
Discharge (hr)	2-4	2-4
Lifetime - Deep Discharges	2000	2000
Price/Energy, \$/Kw hr	60	115
Energy Efficiency, %	60	70
Typical Installation Voltage	90-120V	96V

This report presents the technical approach and a summary of the progress that was achieved under the contract. Work began 1 May 1978. This report covers the period through September 1978.

1.1 Technical Approach

The fundamental difference between this battery concept and Edison's original Nickel-Iron battery is in the plate construction. The Edison battery plates contained the active material inside perforated metallic packet and tube structures. The negative plates contained specially prepared iron powder in flat metal packets. The positive plates required reinforced nickel-plated iron tubes to resist the expansion of the material during operation. EPI will adapt an iron electrode developed for alkaline iron-air batteries to marry with a positive nickel electrode which is usually used for nickel-cadmium batteries. Both electrodes are sintered metal structures with inherently good conductivity. This feature is especially important for relatively small, high-discharge-rate batteries. In addition, the positive plate will be electrochemically impregnated. When compared to the usual nickel plates made by a chemical soak and precipitation process, the electrochemical plates exhibit remarkable dimensional stability. This feature

will be especially important to this program which is directed toward a long life, deep-cycle battery. The battery will then consist of plates which are the products of a more modern technology which should exhibit the desired electrical and physical performance.

1.2 Development Locations

Eagle-Picher Industries, Electronics Division (Joplin) has elicited the services of the Swedish National Development Co. (SU) to develop and make the iron negative electrode for this battery; to select a suitable separator, and to conduct many of the characterization experiments on the battery. The positive plate for the battery will be developed by the Eagle-Picher Industries, Colorado Springs plant. Their total output is nickel-cadmium batteries. They should be qualified to develop a positive electrode for this battery. EPI Joplin will retain all program responsibilities with respect to cell and battery design as well as the coordination of all program activities.

1.3 Baseline Objective, Cell Design

The EPI Design and Cost Study Report detailed a cell design; the first objective of the program. The cell description includes dimensions 5.5 cm X 17.5 cm X 28 cm. The electrodes and separators are:

	<u>Ni Electrode (Pos)</u>	<u>Fe Electrode (Neg)</u>	<u>Separator</u>
Size, H & W	18 cm x 16 cm	18 cm x 16 cm	19.5 cm x 17 cm
Thickness	2.4 mm	1.2 mm	0.9 mm
Area	288 ² cm	288 ² cm	332 ² cm
No. per Cell	10	9	18
Weight	227 gms	116 gms	4.2 gms
Capacity	30.1 AH	41.8 AH	

2.0 SUMMARY

2.1 SU Performance Summary

The Swedish National Development Company program is on schedule. They have been successful in scaling down their electrode from 3.2 mm thickness to 1.2 mm. At this time the test electrodes are 12.8 x 14 cm in size. Their output is about 0.9 AH/cc --close to the 1.0 AH/cc performance goal. The electrodes have begun their characterization tests. The first results indicate good performance; outputs near 0.9 AH/cc, for rates from C/5 up to C/1. Performance is, however, temperature dependent. At 0°C battery temperature, there was no useful

performance even at the C/5 rate. Details concerning these first electrodes are presented in the body of the report.

SU has made a tentative recommendation regarding the type separator which should be used. They have determined that a microporous PVC sheet separator appears satisfactory. The film barrier separators of the "Celgard" type by Celanese Corp. offer no advantage for the battery. These are thin microporous polypropylene films. If dissolved iron is a problem, these separators would not be an effective solution. They would merely delay, not solve the problem. The "Celgard" separators retard the migration of iron ions in the electrolyte but do not stop it. This design nickel-iron battery requires a substantial electrolyte gap for both heat storage and gas separation during charge. Consequently, the thin Celgard films could not be used to any advantage in this battery.

As part of the 1.2 mm thick plate development SU compared the expanded iron substrate with a woven iron screen of equal weight per unit area. The expanded and flattened iron proved to be the better conductor. Plates made this way had lower voltage drop. SU has decided to continue with the expanded metal grid.

Some preliminary cycling tests on small cells began. The tests compared nickel positive plates against iron and nickel counter electrodes. Cells with a great excess of iron are compared with others which are more nearly balanced. Early results, to about 300 cycles, show a definite capacity fade especially in the cells with the smaller amount of iron and the nickel counter electrode.

2.2 EPI Colorado Springs (C.S.) Performance Summary

The C.S. contribution is to be the development of a practical positive electrode of the sintered type. To be practical, the plate should exhibit some specific physical as well as electrochemical properties. The desired positive plate characteristics were described briefly in section 1.3. The main reason for selecting a plate thickness of 2.4 mm is that it should strike a good balance between operating current density and cell volumetric efficiency. The area gives an operating current density of 30 mA per cm² at the two hour discharge rate. This is a good, high range for the plate. To achieve the 300 AH desired cell capacity requires that the plates yield 0.116 AH/cm². This equates to a chemical loading of 0.402 gms/cm² or 1.67 gms/cc of plate. This is a practical loading for a thin plate. In effect the 2.4 mm thick plate appears to be a good choice; it will result in a cell design with relatively few plates and which operates at a reasonable current density. The required cell volume is less than what would be the case with a larger number of thinner plates.

The task at C.S. resolves itself into two efforts:

1. Produce a thick, 0.24 cm, sinter.
2. Impregnate, by electrochemical means, that same sinter.

2.2.1 Sinter Development

Sinter for nickel plates (at Colorado Springs) is made by two different processes: the dry-powder and nickel-slurry techniques. In the dry-powder method, nickel powder is sprinkled over the grid material substrate in a shallow mold. The nickel is leveled; the mold frame is then removed; and the material is carefully placed on the chain belt for a pass through a sintering furnace. This produces a most desirable sinter because it is inherently pure nickel. No foreign material was added to aid the process. It is not as desirable for making sinter when production is considered. Each plaque is hand made, and its quality is dependent largely upon operator skill. C.S. attempts at producing 2.4 mm plaque by this method were unsuccessful, regardless of the substrates used. The 2.4 mm sinter is three times as thick as that which had been produced previously. At this thickness, shrinkage was a problem. The plaque was always severely cracked or it had shrunk away from substrates that were used. In addition, the fact that the dry powder process is not so desirable from a production standpoint encouraged early abandonment of these experiments.

The nickel slurry method for making plaque is a more desirable production process. It is a continuous, automated process which maximizes the output of the sintering furnace. One operator can monitor the complete process where at least two operators are needed for the dry powder process. For this reason, more effort was expended in inventing a method for making 2.4 mm thick slurry process plaque.

Briefly the slurry process consists of preparing a very viscous mixture of nickel powder, water, alcohol, and thickeners (such as "Methocel" and "Polyox"). The mud like mass is held in a hopper through which a nickel screen substrate is passed under a roller which is submerged in the slurry. The mix fills the screen and clings to form a wet sheet. The wet sheet is then sized to a desired thickness by passing it vertically between a set of scraper blades which hold back all excess material. The wet, sized sheet immediately passes into a vertical pass, infra-red drying oven. The dried sheet (green plaque) then passes through the sintering furnace and is rolled up on an automatic take-up spool. The simplicity of the process makes it a desirable

candidate for serious experimentation. During this time period C.S. was not successful in preparing plaque thicker than 1.3 mm. The severe shrinkage caused mud cracking when thin substrates were used. With thicker substrates, shrinkage would separate the sinter from the substrates. Honeycombs, expanded, and stretched material were tried in these experiments.

Later in this reporting period, work was initiated on a "reslurry" process. In this case a typical thin (0.8 mm), sintered plaque is made by the technique which has just been described. The sintered plaque is then passed back through fresh slurry with new, wider settings on the scraper blades. This process results in a double-pass sinter which appears to offer promise for yielding a 2.4 mm sinter.

Little effort was expended upon experimentation in impregnating thicker plaque because practically none was available from the sintering experiments. However, a limited study compared the results of two prominent methods of electrochemical impregnation. In this study, the performance of 0.75 mm thick plates made by the "Pickett" (USAF) process was compared to similar plates made by the Joplin method. The comparison study was made because it was noticed that there was a difference in average voltage level from supposedly equivalent plates. The two electrochemical processes are supposedly alike; both precipitate nickel hydroxide from a nickel nitrate bath directly in the pores of the sintered plaque. In the USAF method the nickel nitrate bath includes ethanol to lower the boiling temperature and facilitate nickel ion diffusion at a temperature of about 70°C. This process also uses active nickel anodes. As a consequence of the chemistry, acid is generated at the anode, however, the apparent pH increases as the process progresses; therefore, excess nickel must be dissolved. Nitric acid is added to maintain pH at about 2.7, the desired level.

In the Joplin process the nickel nitrate is dissolved in water only, giving a boiling temperature of about 105°C. This higher temperature is required to maintain ion mobility. The anodes in this case are platinum plated titanium. In operation the pH is driven lower, but the acidity is neutralized by adding a buffer.

The actual chemical reactions are varied and complex for either method. A simplified view of the overall result is to consider that as nitrate is reduced at the cathode, hydroxide ions (OH^-) and oxides of nitrogen are created within the pores of the plaque. When the concentration of hydroxide is sufficiently high, $\text{Ni}(\text{OH})_2$ will be precipitated. At the anode, oxides of nitrogen and water create the equivalent of nitric acid which balances the overall reaction. The differences between the two processes deal with how the solution is maintained in proper balance. The USAF process uses active, dissolving, electrodes to replenish the nickel and neutralize the acid.

In the EPI process, fresh nickel nitrate is added and a buffer neutralizes the acid which is produced.

The early experimentation favored the Joplin processed plates because they operated at a 10 to 50 mV higher voltage level during discharge. This would be considerable in an 80 cell battery. It would require three extra cells in the battery to compensate for a 50 mV difference in average voltage. Graphical data is presented in the body of the report.

3.0 TECHNICAL PROGRESS

3.1 EPI Joplin

3.1.1 Cell Development

The cell development has made good progress in all areas except for the positive plate. At this time the design of para. 1.3 is still considered valid and will be pursued. Figure 1 is a pictorial presentation of that design. A tabulated physical description is as follows:

Size:

Positive Plates	10 @ 2.4 mm ea.	= 24 mm thickness
Negative Plates	9 @ 1.2 mm ea.	= 10.8 mm thickness
Separator	18 @ 0.85 mm ea.	= 15.3 mm thickness
Allowance	1.5 mm	1.5 mm
Case Walls	2 @ 2.25 mm ea.	= 4.5 mm
Cell Thickness	TOTAL	= 5.61 cm
Cell Height		28 cm
Cell Width		17.52 cm

Weight:

Positive Nickel	2270 gms
Electrodes	
Negative Iron	1044 gms
Electrodes	
Separators	76 gms
Electrolyte	1230 gms
Cell Case	245 gms
Cell Cover	27 gms
Terminals	95 gms
Vent	6 gms
Current Collectors	125 gms
	<hr/>
	5118 gms

The design of Figure 1 includes more metal in the current carrying circuit than was proposed in the Design and Cost Study Report. This is not wasted in a high rate battery. The tab and comb design is also a good rigid structure as required for supporting heavy

VNF 300
NICKEL-IRON CELL
SECTION

FIGURE 1

plates. The electrical circuit has been shortened compared to a conventional individual cell construction. The design is similar to recent Lead Acid battery practice of making intercell connections under the battery cover.

3.1.2 Positive Electrode

In addition to the C.S. effort on positives, EPI, Joplin has also conducted positive plate experiments. Their performance was compared with plate processing as the only independent variable. Figure 2 and 3 are typical results. The phenomenon was observed repeatedly. Plates from the EPI Joplin beaker process consistently operated at higher voltage levels. The USAF process, which is the only electrochemical process in use at C.S., did, however, indicate higher utilization. There is a large uncertainty factor in the theoretical capacity of either plate. This is calculated from the apparent weight gain during impregnation. In the USAF process there is a possibility that more corrosion occurs due to a much longer process time. Consequently, until the plate capacity goals are more nearly approached by either method, detailed calculated predictions regarding which offers the highest specific energy are not warranted. In thin, 1 mm, sections EPI Joplin positive plates have indicated 100% utilization.

3.2 Negative Electrode and Separator

EPI Joplin has subcontracted portions of the development program to the Swedish National Development Company (SU). They have experienced little trouble in their portion of the job.

3.2.1 Negative Electrode

SU first used their negative electrode in an Iron-Air battery system. They used electrodes 3.2 mm thick in order to maximize energy density. For our purposes, sufficient capacity should be attainable using electrodes 1.2 mm thick. As might be expected, reducing the thickness was no problem. These plates are made by the reduction of iron oxide in a hydrogen atmosphere. The resulting iron powder agglomerates are then placed under and over an iron grid in a shallow mold. The powder is compressed to the desired density and sintered in a reducing atmosphere.

NICKEL-IRON CELL
DISCHARGE CHARACTERISTICS

C/4 CHARGE, 5 HRS.
C/3 DISCHARGE

ELECTROCHEMICAL POSITIVE PLATES
PROCESS COMPARISON
EPI JOPLIN - THEORETICAL CAPACITY = 18.23 AH
C.S. USAF - THEORETICAL CAPACITY = 15.4 AH

FIGURE 2

NICKEL-IRON CELL DISCHARGE CHARACTERISTICS

C/4 CHARGE, 5 HRS.
C/3 DISCHARGE

ELECTROCHEMICAL POSITIVE PLATES
PROCESS COMPARISON
EPI JOPLIN - THEORETICAL CAPACITY = 18.23 AH
C.S. USAF - THEORETICAL CAPACITY = 15.4 AH

FIGURE 3

3.2.1.1 Negative Electrode Testing

The performance testing of these electrodes has begun. SU reports the data in terms of AH/cc or AH/gm. The electrodes under test are all 1.2 mm thick, but may deviate in size--either 9 x 12 cm or 12.8 x 14 cm is used. Half cell tests use iron electrodes vs. solid nickel sheet counter-electrodes. This should give the best performance possible because there can be no concern or claim that cross contamination might influence the results.

Tests on half cells at C/1 and C/2 rates are continuing. The average performance of 12 cells is presented below:

<u>Cycle No.</u>	<u>Discharge Rate</u>	<u>Capacity Results</u>	
		<u>AH/g</u>	<u>AH/cc</u>
12	C/2	0.34	0.95
19	C/1	0.32	0.91
26	C/2	0.33	0.92

There are too few cycles to make predictions concerning electrode life. The performance is as expected. The output is about 90% of the original program goals.

The following tabulation shows the average results of a set of 6, three-plate half cells. The iron plates, 1.2 mm x 9 x 12 cm is size, were built into cells with sheet nickel on either side.

IRON ELECTRODE
ELECTRICAL PERFORMANCE VS. COMPACTION FORCE

FIGURE 4

Iron Electrode
Performance vs. Temperature

The cycling profile used was:
Charge C/5
Discharge C/2 for 80% of nominal capacity
Return factor 120%

Cycle No.	Temp. °C	Capacity		
		Average AH/ccm	Maximum AH/ccm	Minimum AH/ccm
23	22	0.84	0.87	0.82
28	40	0.95	0.96	0.94
32	24	0.78	0.82	0.76
36	0	-	-	-
41	23	0.94	0.98	0.92
43	5	0.60	0.70	0.52
44	5	0.42	0.55	0.25
46	10	0.62	0.69	0.50
55	23	0.70	0.78	0.58

Besides the temperature performance, which this test series was to demonstrate, the data shows some cycle performance data. The data are alarming because in only 55 cycles vs. sheet nickel counter electrodes the 23°C capacity did not return to its former level at cycle 23 and 41. This test will continue regardless of the fact that it has already demonstrated that performance at low temperature is very poor. SU will continue its investigation into the possibility of improving this characteristic.

The temperature performance relationship investigations continued with a series of four cells. These were charged at one temperature and discharged at another. The data are tabulated here:

Iron Electrode
Performance vs. Temperature

Charge		Discharge		Capacity (AH/ccm)		
Current	Temp	Current	Temp	Mean	Max	Min
C/5	20°C	C/2	0°C	0.54	0.60	0.48
C/5	20°C	C/2	10°C	0.79	0.80	0.78
C/5	0°C	C/2	20°C	0.58	0.59	0.56
C/5	10°C	C/2	20°C	0.78	0.79	0.77

There is now an indication of at least partial performance at 0°C regardless of whether it was charged or

discharged at that temperature. However, the iron electrode is most sensitive to cold discharge. Note that the lowest performance, and the greatest deviation in performance, occurs when the cells are discharged at 0°C. This in combination with the fact that the iron electrode does not accept a charge well at 0°C (the next lowest performance) indicates why performance was nil when both the charge and discharge occurred at 0°C as indicated in the previous test set.

The data of these cells is also presented in graphical form in Figure 5. It shows all of the last two sets of data presented. This is most descriptive and indicates that low temperature performance needs further research work.

The combination of rate and temperature is similar to other battery systems. Higher rates require a warmer battery to support performance near equal to normal temperature operation. Figure 6 shows this interrelationship. The same data is presented twice in two families of curves. The top graph shows capacity vs. discharge current for a family of temperature curves. Note that the +10°C curve is diverging from the others as the current increases. The other set of curves is more descriptive. The near vertical approach to zero performance at the C/5 rate shows the performance of the present iron electrode at 0°C and higher discharge rates. Here again, there is an indication that low temperature operation will need to be improved. Alternately, the battery system might be made tolerant of a cold ambient temperature by use of heaters during charging, giving at least partial practical operation down to 0°C.

The next three graphs, Figures 7, 8, and 9 are the discharge curves from which Figure 6 was generated. These figures show Hg/HgO reference electrode potentials during half cell operation. The discharge was interrupted just at the point where the second discharge plateau was about to begin.¹ Figure 7 shows the 40°C test results. Apparently the higher temperatures are beneficial to the iron electrode since an improvement at the C/5 rate is evident. The 10°C graphs show the severity of the C rate at this temperature. The graphs of Figure 6 were made with variations in the end-of-test voltages. As the temperature was decreased the end voltage was lowered to improve the apparent capacity. This is the reason that in Figure 6 the C/1 and C/2 curve appeared inconsistent with the C/5 performance. The temperature problem is now recognized and will be the subject of further investigations.

Figure 10 shows some data on the iron electrode recharge capability. It appears that the 20% extra return from 110% to 130% yields very little in extra capacity. At the C rate the

Temperature dependence. Capacity versus temperature. Charge C/5, Discharge C/2, Return Factor 120%

NICKEL/IRON CELLS.

FIGURE 5

IRON/NICKEL SHEET HALF CELL

Capacity as a function of current and temperature respectively.
Average of 6 iron electrodes.

FIGURE 6

Discharge curves at 40°C
IRON/NICKEL SHEET HALF CELLS
Charge C/5 for 6 Hrs.

FIGURE 7

Discharge curves at 24°C

IRON/NICKEL SHEET HALF CELLS

Charge C/5 for 6 Hrs.

Potential vs. Hg/HgO (V)

FIGURE 8

Discharge curves at 10°C

IRON/NICKEL SHEET HALF CELLS

Charge C/5 for 6 Hrs.

Potential vs. Hg/HgO (V)

IRON ELECTRODE

Capacity versus Return Factor for
different charge rates.
Discharge current at C/2

FIGURE 10

AH/cc changed from 0.78 to .83, or about 6.5%. This was after an extra input of 18%. This is only about 30% efficiency on charging over 110%. At the C/5 charge rate the capacity increased from .9 to .94 AH/cc when the return was increased from 110% to 130%. This condition is worse; the extra yield was only 4.4% for the 18% extra input, or 24% efficiency. When the average voltage on charge vs. discharge is considered, the energy efficiency is also poor. The nickel-iron battery should be used in its most efficiency yield/input mode if it is to be a creditable energy efficiency system. Recharge should be the minimum to achieve the required performance.

3.2.1.2 Negative Electrode Grid Material

Part of SU's program in iron plate development is the evaluation of expanded metal vs. screen. In this study, it was assumed that a cell would maintain 1 volt for 30 seconds at the 2C rate. This is equivalent to about 67 amps per negative plate. SU assigned 50 mV as the maximum IR loss for an acceptable plate structure. The tests were conducted on 9 x 12 cm electrodes. Both grid materials were equal in weight per unit area, 15 g/dm². The substrates are shown in Figure 11. The flattened expanded metal is .5 mm thick. These grids were built into finished plates 1.5 mm thick. The method for determining the voltage drop is shown with the performance graphs in Figure 12. The expanded metal proved to be the superior conductor in this test. Since capacity is taken to an end voltage, the plate with the lower losses should also yield higher performance. This is also indicated by the data of Figure 12.

Figure 13 tabulates the voltage drop through a full size plate while it was carrying the equivalent of a C/1 current. In this case the gridwire, 20 mesh with double wire to aid conduction toward the plate tab was compared to the 0.5 mm thick expanded metal. Plates with single and two layers of the woven wire screen were made. The important feature of the data is that there is equal metal in the two screens and one expanded metal grid. The expanded metal again yielded the lowest losses. These measurements indicate no reason to change from the expanded metal.

3.2.2 Separator Evaluation

Several separators were evaluated in this portion of the program. Their evaluation was performed using various methods.

3.2.2.1 Barrier Type Separators

There were four different barrier separators tested. Three are inert microporous films, cellophane is an organic fine pore membrane. Barrier separators were tested because iron contamination might be detrimental to the positive electrode. The materials which were tested and the preliminary results which were obtained are tabulated below:

NEGATIVE PLATE SUBSTRATES

Iron Screen
15 g/dm²

Expanded Iron
0.5 mm thick
15 g/dm²

FIGURE 11

Voltage drop as a function of the discharge current for expanded metal and woven metal as gridwire.
Average of three electrodes.

FIGURE 12

FULL SIZE IRON PLATE

Voltage drop between the current collector and defined points on the electrode surface when sending 32.5 A from bottom to top.

Voltage Drop (mV)	A	B	C	D	E	F	G	H	I
1 Woven metal grid	4.4	27.7	35.7	33.2	37.9	41.2	52.0	53.3	53.0
2 Woven metal grids	3.1	20.9	27.0	23.1	27.3	30.3	36.5	38.0	37.3
expanded metal grid	2.5	18.6	23.7	21.5	24.9	27.5	34.1	35.5	35.2

FIGURE 13

	Barrier Separators	Cycle 130	
	Resistance $m\Omega / cm^2$	Fe ppm Inside	Outside
Celgard 3401 (Celanese)	388	13.5	0.5
Yumicron Y3115 (Yuasa)	22	7.5	1.0
Yumicron YS-UE-02 (Yungfer)	1039	--	--
Cellophane (Pudo 300) EPI	52	--	--

Only the Celgard 3401 and Yumicron Y3115 were considered suitable for cell tests. The YS-UE-02 was too high in resistance. The Cellophane is too fragile to survive the gassing in the nickel-iron battery system. The data indicate these separators are effective in retarding iron ion migration when they are used to "bag" the iron electrode. Further separator tests indicated that bagging the electrodes was detrimental to the operation of the cell. The probable cause is that bagging traps the gas which is evolved from electrode. Trapped gas will prevent good battery operation due to current density distortion.

3.2.2.2 Mechanical (Physical Space) Separators

There is limited data from this portion of the program. Some separator test results in cells are available. The 4 types of cells tested are described in Figure 14. They are nominal 24 AH units with .9 mm thick positives. The data for cells after 294 cycles are tabulated here.

Mechanical Separator Study

24 AH Cells, 0.9 mm Pos. Plates 9 x 12 cm
 Cell arrangement per Figure 14
 Charge C/3
 Discharge C/2, 80% of Nominal C
 Return Factor 120%

Cycle No.	TYPE 1 2 cells (AH)	TYPE 2 2 cells (AH)	TYPE 3 1 cell (AH)	TYPE 4 1 cell (AH)
30	26.4	23.2	24.7	25.8
61	26.3	22.5	23.2	24.6
107	25.5	21.7	21.8	24.8
155	23.9	-	-	-
175	-	18.0	22.4	22.8
182	23.7	-	-	-
210	-	18.0	-	23.0
217	23.7	-	-	-
277	-	17.7	-	17.4
294	23.0	-	-	-

IRON ELECTRODE ELECTRICAL PERFORMANCE VS. COMPACTION FORCE

FIGURE 14

A nonwoven separator is wound around the positive electrode, a ribbed sintered PVC-separator around the negative except for type 4 where a PVC gridwire is used around the positive electrode.

The capacity data indicate trends other than what might be an effective separator:

1. Compare Type 1 cells to Type 2 units. It is observed that the capacity of the positive plates is higher when they are operated individually rather than in pairs.

2. Type 1 cells consistently yielded higher capacity when compared to Type 3 and 4 cells. In this case, the Type 1 cells are particularly interesting because at least 300 cycles have been logged with little capacity fade.

3. The separator, nonwoven fabric on the positive plate, tends to keep the surface of the plate moist regardless of the gassing. Nonwoven separators allow the transmission of gas. This has been demonstrated in sealed Ni-Cd batteries. The evolved gas can escape into the open ribbed space next to the negative plate. Operating voltage data is required before a judgement can be made regarding the performance of this separator system vs. any others.

Other separator/construction combinations have been tested. A limited experiment with microporous plain PVC sheet and Nylon thread was conducted. The positive plates were nickel counter electrodes. The iron was first wrapped with the Nylon thread, 0.2 or 0.55 mm diameter, and then bagged in the sheet PVC. One cell with no gas space formers (Nylon thread) failed to operate with any consistency. This data is presented in Table 1. It appears that 0.5 mm spacing is sufficient to insure release of the gas from the iron electrode. The spacing needs to be kept at the minimum acceptable in order to help keep a high battery specific energy.

Separator combinations with the microporous PVC Jungfer material are designated as Type 5, 6, and 7.

Type 5: 0.2 mm/flat around both electrodes

Type 6: 0.2 mm/flat around iron
Nonwoven FT 2112 around Ni

Type 7: 0.2 mm/0.55 mm ribs toward the Ni

The designation 0.2 mm/flat indicates 0.2 mm ribs on one side of the flat PVC sheet. The 0.2 mm/0.5 mm material has ribs on both sides, the ribs being 0.2 mm and 0.5 mm.

The nominal capacity of these cells with 1.2 mm nickel oxide electrodes was 32 AH. The results of these cells are presented below:

TABLE 1

Table 1.1 Capacity and potential for varying distances between electrodes and separators

nylon cord diameter	potential vs counter electrode	capacity		
		average	max	min
mm	mV	Ah/ccm	Ah/ccm	Ah/ccm
0	-	-	-	-
0,2 ¹⁾	525	0,54	-	-
0,5	405-460	0,79	0,86	0,70
0,8	380-405	0,80	0,86	0,74
¹⁾ only one half cell still in the test				

Table 1.2 Capacity [Ah/ccm] for different separator configurations.

dis- tance	tube	bag	M
0,55 mm	0,93	0,84	0,89
0,2 mm	0,76	0,68	0,72
$\bar{M}$	0,85	0,76	

Cell and Separator Tests

32 AH (Nominal) cells
Charge C/5
Discharge C/2 80% of nominal
Return factor 120%

	Cycle No.	Capacity (AH)		
		Type 5	Type 6	Type 7
GROUP 1 1 cell/type	1	27.5	27.5	27.5
	2	29.1	28.8	29.1
	29	25.9	24.0	29.3
	47	24.3	22.2	28.5
GROUP 2 3 cells/type	11	28.6	26.5	29.4
	29	27.6	25.4	28.1

The type 7 cells are yielding better results than the other separator combinations. They have the 0.2 mm/0.5 mm Jungfer PVC separator material. The data are preliminary; however, it shows the beginning of a capacity fade.

3.3 Technical Progress EPI C.S. - Positive Electrode Development

3.3.1 Preparation of Sinter

There is little more that can be added to the summary report of para. 2.2. Nearly all of the results of the development efforts were unsatisfactory because of severe cracking of the thick sinter, regardless of the method by which it was prepared. However, even negative results are a form of progress since they redirect efforts toward new thought channels.

3.3.1.1 Dry Powder Technique

C.S. investigated several methods of making 2.4 mm sinter by the dry powder process before proceeding to the slurry.

First, the direct approach as if a thick sinter could be hand made like the 0.5 mm sinter was attempted. Based on known percentage shrinkage factors, the moulds were set at 3.5 mm and filled with powder at 0.76 gm/cc. The results were unsatisfactory because of cracking, curling and brittleness. When the plaques were sufficiently sintered, they were curled and cracked. At higher belt speeds, up to 46 cm/min rather than the usual 25 cm/min, the plaque was weak because it was not sufficiently sintered. Resintering this material caused it to become extremely brittle. No proper sintering scheme was discovered for the direct approach.

The next series of attempts involved corrugating the 20 mesh nickel wire cloth. In these experiments the corrugated (mesh) reinforced the sinter throughout its thickness. Furnace belt speeds from 13 cm/min to 46 cm/min were used. The results were as before; severe cracking, curling, and extreme brittleness. At this point the attempts at dry sintering were essentially terminated. Only occasional tests with exceptional substrates such as honeycomb were tried. Work with the nickel slurry proved so promising in outward appearance that dry powder work ceased.

3.3.1.2 Wet Slurry Plaque

The first work with the wet slurry method for making nickel sinter began simply. Three layers of 0.8 mm green plaque were stacked and passed through the sintering furnace. In each trial there were several areas where the separate layers adhered to each other, the sintered contact areas were sporadic and not sufficient to be practical. Experiments in which the several layers were weighted with top plates to cause more uniform bonding gave better results. The plates were more pliable but badly discolored. This was attributed to the cover plates interfering with the furnace atmosphere access to the sponge. The three individual layers naturally lead to attempts to make a thicker slurry sponge. C.S. plan was to attempt a 1.2 mm thick plaque; then two of these when impregnated and used back-to-back would be equivalent to a 2.4 mm electrode. These plates could be used to further cell design, while work on achieving a single 2.4 mm thick plate would continue.

In June of 1978, the first runs to make 1.2 mm thick plaque were attempted. Since the process is a continuous one, the complete system speed has to be synchronized. The system had been established for 0.8 mm plaque, the common Ni-Cd battery plate. To make 1.2 mm plaque required complicated and delicate adjustments to the process.

First, the furnace speed must be correct to properly sinter the nickel at the 920°C peak temperature setting. Tabulated below are the results of the first runs:

Sintered Sponge Thickness

Nominal Belt Speed cm/min	Sample ave. Thickness mm	Thickness Range mm
13	1.26	1.23-1.28
	1.21	1.18-1.23
	1.26	1.23-1.28

Sintered Sponge Thickness (Continued)

Nominal Belt Speed cm/min	Sample Ave. Thickness mm	Thickness Range mm
18	1.33	1.31-1.36
	1.41	1.36-1.46
	1.43	1.41-1.46
20	1.38	1.36-1.38
	1.33	1.31-1.38
	1.33	1.31-1.33
28	1.43	1.43-1.46
	1.36	1.36-1.38

The actual sinter was unsatisfactory because of severe cracking. The best belt speed found for sintering was 13 cm/min. The slow speed allowed too much time in the wet sheet drying oven. The rapid drying caused mud cracking. At higher belt speeds the wet sheet (now much thicker than normal production) would not dry in one pass through the drying tower. It then stuck to the rolls and delaminated before it reached the sintering furnace. Experimentation was continued to slow the drying of the wet sheet to prevent mud cracking while maintaining sintering at 13 cm/min. This would be accommodated by reducing the power to the vertical pass infrared drying oven.

The physical properties of the 1.2 mm plates was satisfactory-except for the cracking. Porosity was measured at about 77 to 79%. Development of this thickness plaque continued until satisfactory sinter was produced. The 1.2 mm thickness was the maximum which could be made without slumping of the wet sheet before drying. The introduction of the double pass (reslurry) technique eliminated further experimental work to attempt 2.4 mm wet slurry plaque in one pass.

3.3.2 Electrochemical Impregnation

The very first C.S. attempts at impregnating sinter by the USAF process were eminently successful. At that time only a "Pilot Plant facility" was operational for use on the readily available 0.9 mm plaque. The parameters for impregnation/formation were held as close as possible to the following specification:

1. Nickel Nitrate Concentration 1.3 M
2. Cobalt Nitrate Concentration .2 M
3. Operating pH $3.0 \pm .5$
4. Operating Temperature $78 \pm 2^{\circ}\text{C}$
5. 50/50 D.I. water and ethanol for solution
6. Seven formation cycles in KOH after impregnation

The results, weight, gain vs. impregnation time, are tabulated here for the 0.9 mm sinter:

<u>Time</u>	<u>Wt. Gain</u> <u>gms/cm²</u>	<u>Loading</u> <u>gms/cc</u>
180	0.13	1.45
240	0.15	1.64
300	0.16	1.80

These first results were promising for attaining 1.67 gm/cc loading in the 2.4 mm plates. Impregnation of thicker development sinter in the Pilot facility resulted in loading of 1.47 gms/cc. Performance of these 0.9 mm plates was only about 75% of expectations. However, some samples were loaded to 1.67 gm/cc again yielding low performance, about 83% of expectations.

In this reporting period the positive plate development has shown itself to be the area of major concern. The problems in producing satisfactory plates have not been resolved and will be one of the major areas of continuing work during the upcoming fiscal year.

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