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DEVELOPMENT OF LARGE-INTERNAL-SURFACE-AREA NICKEL METAL PLAQUES

by

J. McCallum, G. R. Schaer, D. G. Trevethan, and C. L. Faust

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Contract NAS 3-6003

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November 6, 1964

National Aeronautics and Space Administration
Lewis Research Center
Space Power Systems Procurement Section
21000 Brookpark Road
Cleveland, Ohio 44135

Attention John E. Dilley, MS 500-309
Contracting Officer

First Quarterly Progress Report
"Development of Large-Internal-Surface-Area
Nickel Metal Plaques"
Contract NAS 3-6003

This is your copy of the First Quarterly Progress Report on Contract NAS 3-6003. It covers the period between June 18 and September 18, 1964.

This report has been reviewed by Mr. William A. Robertson, as Technical Project Manager for your organization. Mr. Robertson authorized the reproduction and distribution of this report by telephone on October 23, 1964. Distribution of the report is in accordance with the instructions of your letter dated October 20, 1964, but incorporates the changes requested by Mr. Robertson on October 23, 1964.

We trust this report satisfies the requirements given in Articles IIA and IIB of our contract. We welcome comments from you or any of your associates.

Yours sincerely,

John McCallum

John McCallum
Project Leader

JM:so
Enc.

FIRST QUARTERLY PROGRESS REPORT

on

DEVELOPMENT OF LARGE-INTERNAL-SURFACE-AREA NICKEL METAL PLAQUES

September 22, 1964

by

J. McCallum, G. R. Schaer, D. G. Trevethan, and C. L. Faust

NASA-LEWIS RESEARCH CENTER
SPACE POWER SYSTEMS PROCUREMENT SECTION
Contract NAS 3-6003

(Period Covered: June 18 through September 18, 1964)

BATTELLE MEMORIAL INSTITUTE
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Columbus, Ohio 43201

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OBJECTIVES FOR THE PROJECT

This program at Battelle is part of NASA's continuing interest in the development of improved and novel electrochemical power-generating devices. The specific objective is the development of porous nickel metal plaques for the recharageable cadmium electrode to provide higher energy output per unit of weight than is presently available.

Presently used plaques, for the recharageable cadmium electrode, are made with sintered nickel carbonyl powders, having porosities on the order of 80 per cent. They have a range of random pore sizes and shapes. Not all of the pores can be filled with active cadmium and the impregnation procedures are lengthy and expensive. Active cadmium within the nickel pores tends to shift position during charge/discharge cycling. Effectiveness of contact between the uncharged cadmium oxide and the plaque is always uncertain because contacts are always changing.

Sealed nickel-cadmium batteries have shown themselves to be a remarkably useful invention. But, of course, there is always room for improvements. Greater and consistent contact area between plaque and active material would improve the reliability and/or constancy of voltage. Uniform pore sizes should increase the uniformity of battery performance. Controlled pore shapes should increase the effectiveness for using, and reusing, the active materials within the plaques. These, and other, improvements are continuously being sought by both the makers and users of sealed nickel-cadmium batteries.

NASA has started this research project to investigate new opportunities for improving nickel-cadmium batteries through the use of electroformed nickel plaques. These plaques are to be made from electroformed screens having from 62,500 to 1,000,000 pores per square inch. Such screens are available commercially. They approach perfection in that the size of pores can be identical with one another within a factor of twofold, or better, in screens up to more than 500 square inches in size. Figures 4 through 7, in the text of this report, show the uniformity of electroformed pores and Figures 2 and 3 show lack of uniformity of sintered-powder pores.

A first objective, therefore, is to explore the possibilities for stacking electroformed screens into plaque structures with a uniform pore distribution.

A second objective is to control pore shapes by selective electroforming and stacking procedures.

A third objective is to demonstrate the physical and electrochemical advantages for the controlled nickel plaques.

Within each of these broad objectives there are subsidiary objectives in each Task of the program.

SUMMARY

As the first Task toward achieving the above objectives, Classification of Raw Materials, Task A, has been completed. Electroformed nickel screens were compared as follows with commercial sintered nickel powder plaques.

Material	Average Pore Size, mils	Size-Tolerance Range of Diameters, mils, to Include		Shape of Hole
		99% of Volume	66% of Volume	
Powdered plaques	0.63	0.13 to <14	0.41 to 1.4	Random
1000-line screen	0.60	0.54 to 0.60	0.60 ± 0.01	Square
750-line screen	0.99	0.94 to 0.99	0.98 ± 0.01	Square
500-line screen	1.42	1.40 to 1.45	1.42 ± 0.01	Square
250-line screen	3.05	2.96 to 3.2	3.05 ± 0.01	Square

In the above tabulation, pore size for the powdered plaques is given as equivalent cylindrical diameters. For the screens, pore size is given as the width of square holes because this is the proper dimension for comparison. The smallest screen openings are about equal to the average diameter for powdered plaques. The range of pore sizes in the electroformed screens is within the range of pore sizes in powdered plaques. Electrodes made from the screens, therefore, will have pore sizes comparable with pores in commercial electrodes.

The feasibility of stacking screens into plaques with uniform pores of controlled shape was established during this work period. This feasibility resulted from a combination of studies made on Task B, Porous Mat Manufacture, and Task C, sintered Plaque Processing. A stacking machine was made and used to align screens, as part of Task B. The machine consists of a hollow punch, a die with removable bottom, a microscope for aligning screens, and the necessary supports, guides, and adjusting screws. The machine was used first to stack electroformed screens up to 5 mils of thickness. The resulting stack of 18 layers had 250 lines per lineal inch and about 10,000 square pores per square centimeter of electrode. The pores were straight through the electrode with parallel sides.

Techniques for electroforming special screen structures were commenced also as part of Task B. Four methods are receiving active consideration but significant accomplishments are yet to be achieved. In the meantime, work continues with commercial electroformed screens on Task B, Porous Mat Manufacture.

Conditions have been selected for Sintered Plaque Processing, Task C. Electroformed screens were stacked with a 45-degree orientation of wires and sintered into one-piece electrodes to establish the feasibility of making electrodes as planned. Research on Task C is considered to be completed.

Work commenced on Task D, Plaque Classification, toward fulfillment of the objective of demonstrating physical advantages for new plaques. First work evaluated

commercial sintered nickel powder plaques with respect to (1) electrical resistivity, (2) pore-size distribution, (3) pore shape, (4) internal surface area, (5) density and porosity, (6) measurement of interconnecting pores, (7) tensile strength, and (8) flexibility. The results obtained will provide reference points with which to compare new electrode structures.

Work was commenced also on Impregnation Procedures, Task E, and Electrochemical Evaluations, Task F. As with the above-mentioned work on physical properties, first work has established reference points for future studies. Chemical-impregnation studies duplicated results reported in the literature. Also, electrochemical studies on commercial batteries have shown that useful reference points can be achieved.

FUTURE WORK

Details of future work are given for each task of contract work within later sections of this report. In brief, the planned work is aimed toward the following accomplishments during the next monthly reporting period:

Task B – Porous Mat Manufacture

(a) Presently made stacked screens, having 45-degree orientation of wires, will be etched to greater than 60 percent porosity and physically prepared for impregnation and electrochemical evaluation.

(b) Superimposed screens with square holes and parallel sides, will be stacked to at least 5 mils of thickness and then be sintered together. Four sizes will be used: 250, 500, 750, and 1000 lines per lineal inch.

(c) New methods for making electroformed screens and pore structures will receive further consideration.

Task E – Impregnation Procedure

(a) Battelle's chemical-impregnation procedure will receive electrochemical evaluation.

(b) New electrode structures, prepared as part of Task B, will be chemically impregnated.

Task F – Electrochemical Evaluation

(a) Work on commercial battery plaques will be completed.

(b) New electrodes that were successfully impregnated, as part of Task E will receive electrochemical evaluations, as time permits.

EXPERIMENTAL RESULTS BY TASKS

Article IIB of the contract states (in part):

"The contractor shall submit quarterly reports describing all work accomplished during each three (3) month period of contract performance. These reports shall include factual data, analyses and interpretations of the results obtained, specific recommendations regarding the work to follow, diagrams, sketches, curves, photographs, tables and drawings in sufficient detail to convey the intended meaning.

The remaining sections of this report are arranged by Tasks and Work Items, as listed in the contract, in fulfillment of the above responsibility.

Task A – Raw-Material Classification

Objectives

Task A of the contract reads as follows:

"Item 1 – Average Size

Electroformed nickel screens shall be compared with commercially available sintered nickel plaques.

Average pore sizes shall be determined by using a mercury porosimeter and gas adsorption techniques for the sintered powdered plaques and the microscope for the electroformed screens.

"Item 2 – Size Tolerance

Size tolerance shall be determined. One brand of commercial sintered electrode shall be characterized for comparison with four (4) sizes of screens. The screens selected shall be between 250 and 1000 lines per lineal inch.

"Item 3 – Shape of Holes

The shape of holes shall be shown in photomicrographs."

The objective of this work is to compare the structure of commercial nickel powder plaques with the structure of electroformed screens that will be used to make new plaques.

Raw materials were interpreted to mean commercial materials that will be used in the preparation of plaques or used for comparison purposes. Further classifications will accrue, from time to time, as part of Task D.

Item 1 – Average Size

Powdered Plaques. For the purpose of comparing pore dimensions, random samples from commercially available sintered nickel plaques were used in a mercury porosimeter. A duplicate measurement gave essentially the same results shown in Figure 1. The plaques were 21 mils thick.

An average pore size of 16 microns or 0.63 mil was deduced from Figure 1 as an equivalent cylindrical diameter. This number means that one-half the pore volume has pores with diameters greater than 0.63 mil, and one-half has diameters less than 0.63 mil.

Electroformed Screens. As raw materials to prepare new plaques, commercially available electroformed screens were purchased from Buckbee Mears Co., St. Paul, Minnesota. The four sizes, 1000-, 750-, 500-, and 200-line-per-inch screens were checked microscopically for pore sizes.

These screens were remarkably uniform. Wire to wire spacing was excellent over all the surface and hole size was uniform over most of the surface. A linear accuracy of 0.01 mil over a length of 24 inches is reported by the manufacturer. The accuracy of screens was checked for distance from wire to adjacent wire and was found to be ±0.01 mil which is about the limit of the microscopic method used.

From measurement of hole sizes at several locations on the screens, the average widths of the square holes were estimated, as listed in Table 1. The size of the holes depended on the thickness of the nickel plate. Holes were larger in the center where the screen was thinner, and the holes were smaller at the edges where the screen was thicker than average.

TABLE 1. AVERAGE PORE SIZES FOR ELECTROFORMED SCREENS

Screen Size, lines/inch	Width Average Hole, mils
1000	0.60
750	0.99
500	1.42
250	3.05
Powdered plaque	0.63

Item 2 – Size Tolerance

Powdered Plaques. Pore-size distribution was deduced also from Figure 1. First of all, at least 99 percent of the interconnecting pore volume is covered by the range of equivalent diameters between a maximum of 4 to 14 mils and a minimum of 0.13 mil. Secondly, two-thirds of the interconnecting pore volume has pores of equivalent diameters between 0.41 and 1.4 mils. The maximum pore size that can be measured by the

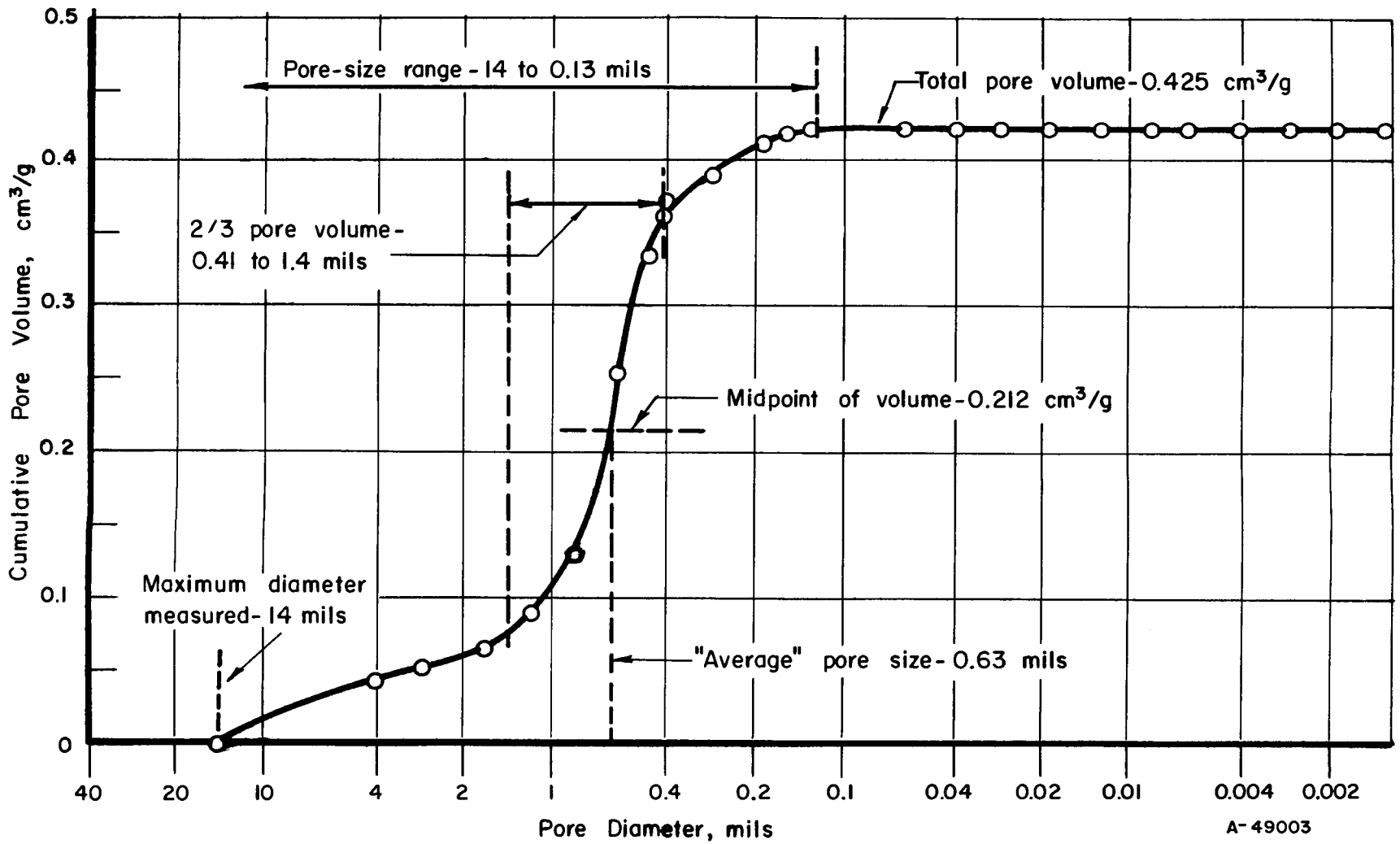


FIGURE 1. CUMULATIVE PORE VOLUME, CM^3/G MEASURED BY MERCURY POROSIMETER

mercury porosimeter is approximately 14 mils. Therefore, Figure 1 shows maximum diameter of 14 mils. During the measurement, the first diameter-volume point determined was 4 mils. This 4-mil value was selected for the comparison in Table 3, at the end of this section, because microscopic examination of the surface of the plaques showed no openings larger than about 4 mils' equivalent cylindrical diameter.

Electroformed Screens. Pore-size tolerance was +5 to -10 percent for the majority of the screen area as noticed earlier. Table 2 shows the minimum pore sizes at the edges of screens and the maximum pore sizes. The average pore size given in Table 1 includes more than two-thirds of the total area. Pore sizes for screens are widths of square holes and for powdered plaques are equivalent cylindrical diameters. The numbers are comparable because the width of a square hole is equal to the equivalent cylindrical diameter that would be measured by mercury porosimetry.

TABLE 2. PORE-SIZE TOLERANCE FOR ELECTROFORMED SCREENS

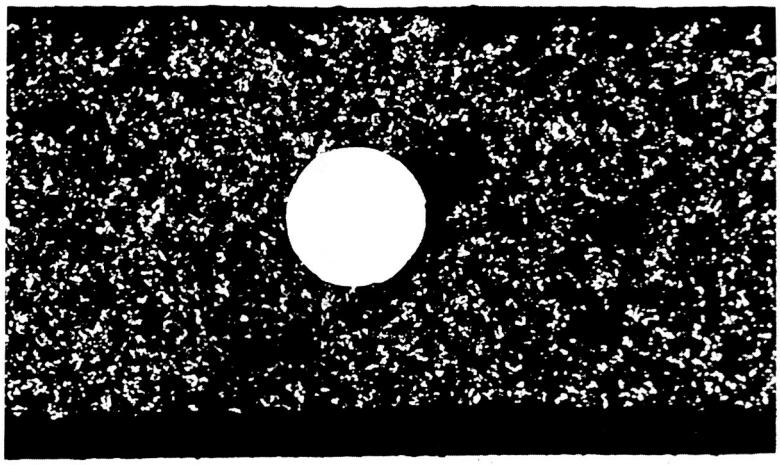
Screen Size, lines/inch	Minimum Wire Width, mil	Pore Size, mils	
		Maximum	Minimum
1000	0.37	0.63	0.54
750	0.34	0.99	0.94
500	0.55	1.45	1.40
250	0.80	3.2	2.96
Powdered plaques	--	<14.0	0.13

Item 3 – Shape of Holes

Powdered Plaques. The shapes of pores for the sintered powder nickel plaques are shown in Figures 2 and 3. The white areas in both figures are metallic nickel particles. The dark areas are the pores. The large white circle in Figure 2 is a cross section of one of the supporting grid wires in the commercial plaque. The shape of the pores is obviously random. Moreover, inspection of the dark areas shows the pore sizes have about the diameters predicted in Figure 1 from mercury porosimeter data.

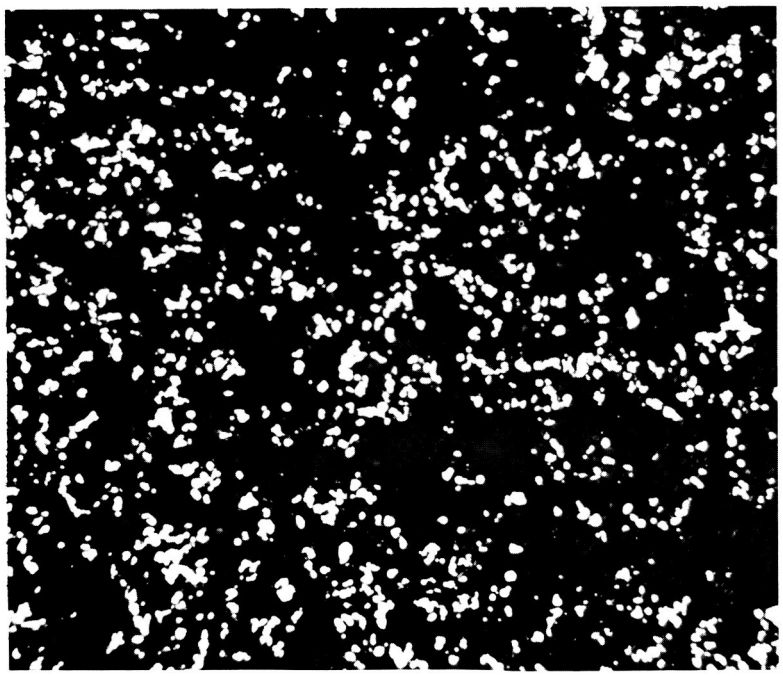
Electroformed Screens. The shape of the holes in the electroformed screens was square when viewed from the surface. The holes as seen in cross section were not rectangular because the wires became rounded and changed shape during the electroforming process.

Figures 4, 5, 6, and 7 show surface pictures of the center area of screens with 1000, 750, 500, and 250 lines per inch, respectively. Some of the holes near the edges of the screen have holes partly closed because of too much plate thickness. Figure 8 is a photomicrograph of the screen with the most severe hole closure of all those examined. Of the 16 corners examined on the screens shown in Figures 4, 5, 6, and 7, only four of the corners showed a measurable hole closure. Duplicate screens were purchased and examined and appeared to be accurate reproductions of the first four screens.



100X Polished Only 14027

FIGURE 2. CROSS SECTION OF COMMERCIAL SINTERED NICKEL PLAQUE



250X Polished Only 14029

FIGURE 3. CROSS SECTION OF COMMERCIAL SINTERED NICKEL PLAQUE

Black areas are voids; white areas are metallic nickel.

Photomicrographs of cross sections are shown in Figures 9, 10, 11, and 12, for 1000, 750, 500, and 250 lines per inch, respectively. Thicknesses of these screens were, respectively, about 0.2, 0.1, 0.2, and 0.3 mil. Sections were made near the corners and are on the same screens shown in Figures 4 through 7.

Comparisons. Table 1 shows that the smallest mesh of screen has an average pore size about equal to the average pore size of commercial powdered plaques.

The chosen electroformed screens have a range of average pore sizes slightly larger than the range of pore sizes that contain two-thirds of the voids in the nickel plaques, as follows:

Range of pore sizes in electroformed screens, mils: 0.60 to 3.05 equivalent cylindrical diameter.

Range of pore sizes in powdered plaques that contain two-thirds of the volume, mils: 0.41 to 1.4, equivalent cylindrical diameter.

A striking difference between hole shapes is seen by comparing Figure 2, for the powdered plaque, with Figures 4 through 7, for the electroformed screens. The powdered plaques have pores of completely random shapes. The electroformed screens have pores that are identical in shape – square.

The size-tolerance differences are also striking. These differences can be described as "percent variation" from average which is arbitrarily defined as follows:

$$\text{Positive percent variation from average} = \frac{\text{maximum diameter} - \text{average diameter}}{\text{average diameter}} \times 100$$

$$\text{Negative percent variation from average} = \frac{\text{average diameter} - \text{minimum diameter}}{\text{average diameter}} \times 100.$$

Results of such calculations are shown in Table 3.

TABLE 3. COMPARISON OF SIZE TOLERANCE

Screen Size, lines/inch	Average Pore Diameter, mils	Maximum Percent Variation From Average	
		Larger	Smaller
1000	0.60	+5	-10
750	0.99	0	-5
500	1.42	+4	-1
250	3.05	+5	-3
Powdered plaque	0.63	+535[a]	-80

(a) Assuming maximum size of 4 mils.

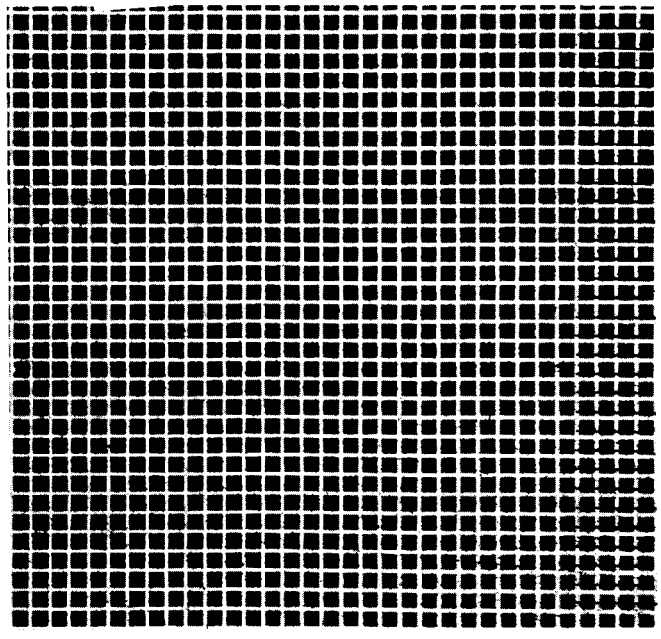


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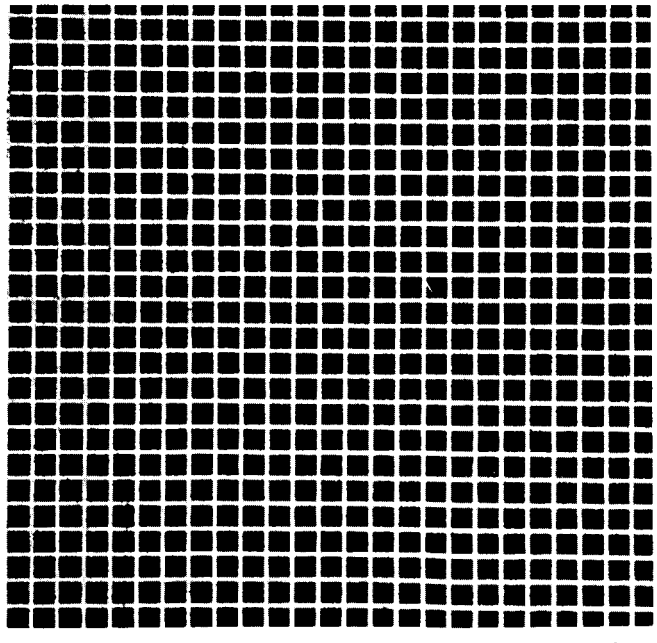
FIGURE 4. SURFACE VIEW OF CENTER OF 1000-LINE-PER-INCH ELECTROFORMED SCREEN



100X 13031

FIGURE 5. SURFACE VIEW OF CENTER OF 750-LINE-PER-INCH ELECTROFORMED SCREEN

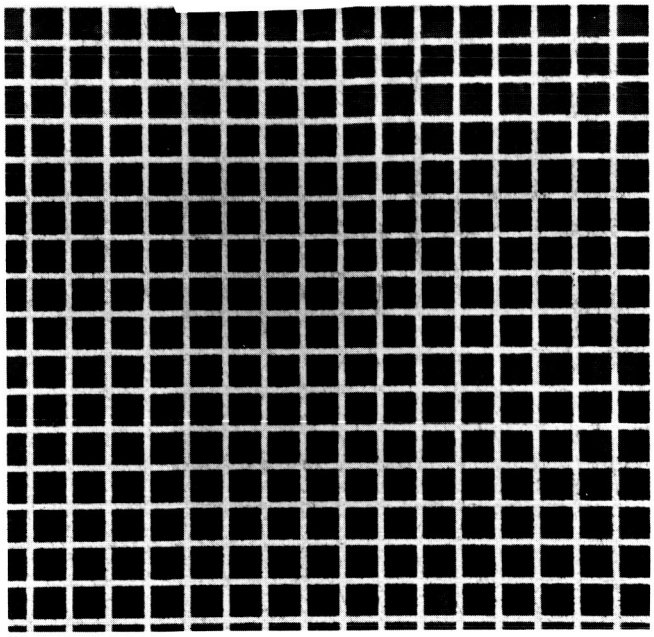


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FIGURE 6. SURFACE VIEW OF CENTER OF 500-LINE-PER-INCH ELECTROFORMED SCREEN

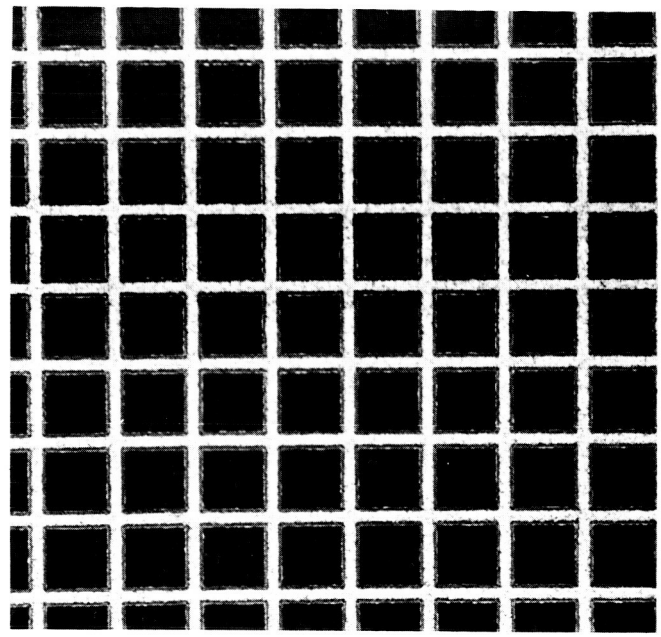

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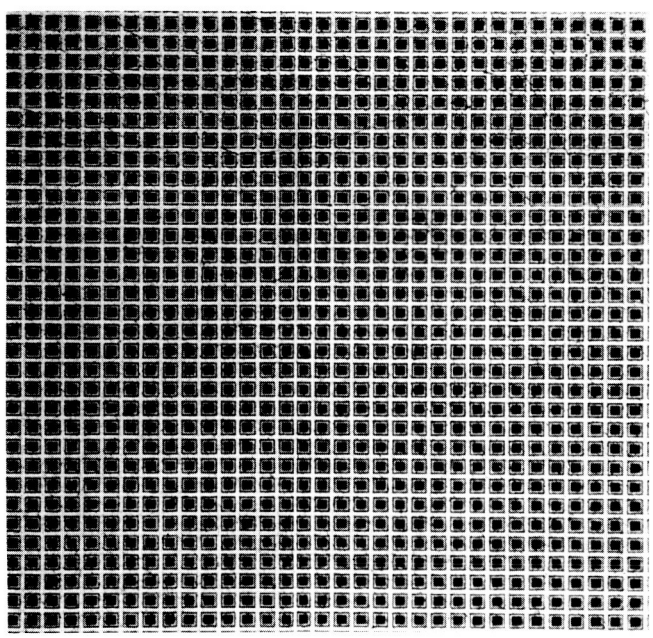
FIGURE 7. SURFACE VIEW OF CENTER OF 250-LINE-PER-INCH ELECTROFORMED SCREEN



100X 13034

FIGURE 8. SURFACE VIEW NEAR CORNER OF 1000-LINE-PER-INCH ELECTROFORMED SCREEN SHOWING THE MOST SEVERE HOLE CLOSURE

500X As Polished 13309

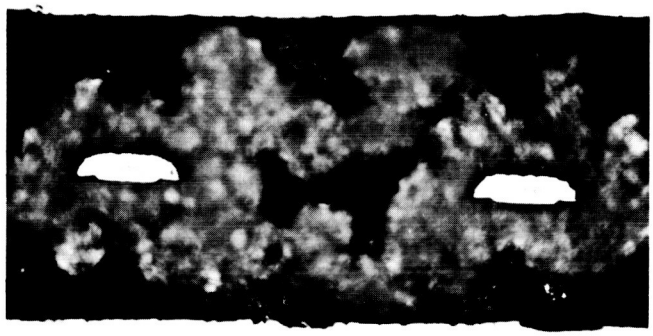
FIGURE 9. CROSS SECTION OF CORNER OF 1000-LINE-PER-INCH ELECTROFORMED SCREEN

500X As Polished 13308

FIGURE 10. CROSS SECTION OF CORNER OF 750-LINE-PER INCH ELECTROFORMED SCREEN

500X As Polished 13307

FIGURE 11. CROSS SECTION NEAR CORNER OF 500 -LINE-PER-INCH ELECTROFORMED SCREEN



500X As Polished 13306

FIGURE 12. CROSS SECTION NEAR CORNER OF 250-LINE-PER-INCH ELECTROFORMED SCREEN

Future Work

No further work is planned on Task A. Characterization of experimental plaques will be continued as part of Task D.

Location of Data

All experimental data on Task A are contained in Battelle Laboratory Record Book No. 21516, pp 24-28.

Task B – Porous Mat Manufacture

Objectives

Task B of the contract reads as follows:

"Item 1 – Thickness Ranges

Methods to construct porous plaques shall be investigated in the following thickness range.

a. 0.005 ± 0.001 of an inch
b. 0.010 ± 0.001 of an inch
c. 0.021 ± 0.001 of an inch
d. 0.035 ± 0.001 of an inch

"Item 2 – Techniques

Techniques shall be developed to electroform and superimpose electroformed screens.

"Item 3 – Methods

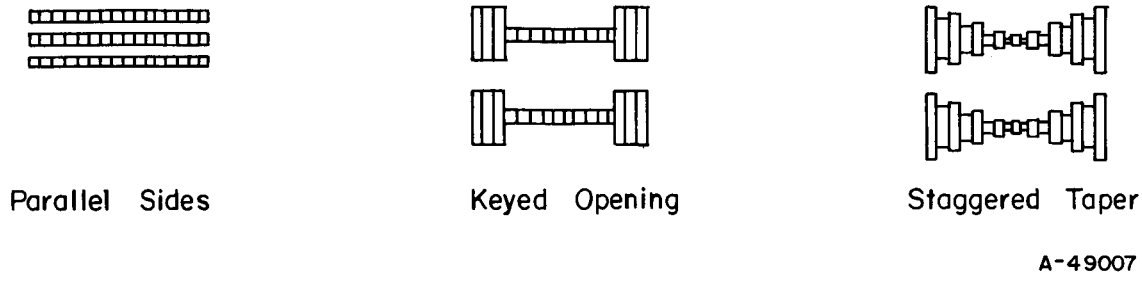
Methods to construct the plaques having the following three shapes shall be investigated.

a. Parallel Sides
b. Keyed Opening
c. Staggered Taper".

The objective of Item 1 is to provide electrodes that will allow determination of an optimum electrode thickness as a function of pore size. Nothing seems to be known about the relationship of electrode thickness to pore size because, heretofore it has not been possible to control the pore size. That is, sintered powder plaques have a range of random pore sizes, as described under Task A. The planned range of experimental thicknesses can be combined with the planned range of experimental pore sizes to permit reliable extrapolations to thinner and thicker electrodes having smaller and larger pores.

The main objective of Item 2 is to develop laboratory methods for electroforming and building porous plaques according to advance specifications. A second objective is to devise manufacturing methods which appear to be feasible for production use.

The objective of Item 3 is to provide a variety of predetermined pore shapes for experimental study. The three shapes mentioned in the contract should look about as follows:



Item 1 – Thickness Ranges

In 1956, Fleischer reported some studies on the effects of electrode thickness*. He prepared sintered plaques covering the range from about 6 to 60 mils and studied the efficiency of the impregnation process as a function of thickness. He reports

> "Impregnation by normal 4-cycle processing showed that at plaque thicknesses of 0.01 inch or less there was a marked decrease in the amount of active material introduced into the plates".

His results show that above 20 mils the weight gain by impregnation is proportional to the thickness. This implies that a plaque thickness of about 20 mils is a minimum useful thickness.

Fleischer is careful to point out,

> "Sintering of thin plaques required careful preparation of the carbonyl nickel powder, especially in eliminating coarse fractions and pre-annealing and flattening of grids".

This statement seems to be significant because a special screening of the starting powders should yield a different range of pore sizes. Moreover, elimination of large particles should lead to lower overall porosity. The result would be an apparent decrease in amount of material introduced in the thin plates, not because of the thinness, but because of lowered porosity.

From another viewpoint, it may be that the washing and handling procedures remove the outer 1 mil or so of active material. Such an effect can readily account for the results reported by Fleisher.

*A. Fleischer, Nickel-Cadmium Battery Company, NICKEL-CADMIUM BATTERIES, Proceedings of the 10th Annual Battery Research and Development Conference (May 23, 1956), pp 37-41.

The net effect is that both of the above uncertainties show the need for additional information on the relationship of plaque thickness to impregnation efficiency. The planned thicknesses of 5, 10, 21, and 35 mils with the planned controlled pore sizes from about 0.6 to 3.6 mils should provide the needed information about impregnation.

From an electrical viewpoint, Fleischer clearly shows an advantage for thinner plates. He shows results on two cells when both were discharged at 40 amperes. Cell 1 had 78-mil plates and was rated at 10 ampere-hours. Cell 2 had 25-mil plates and was rated at 5 ampere-hours. Cell 1 with the larger capacity (therefore actually less severely tested), but with the thicker plates, yielded only 25 percent of its rated capacity. Cell 2, with the thinner plates yielded 93 percent of its capacity. From a wattage viewpoint, the cell with twice the rated capacity yielded less than one-half the wattage. This result demonstrates the advantage for thin plaques for relatively high rates of discharge. The result also implies that plaques thinner than 25 mils would be more efficient at high rates of discharge.

Information is needed now to determine whether these electrical improvements are a function of electrode thickness or whether the important variable is pore size, pore shape, surface area, porosity, cell structure, or some unknown variable. The planned thickness range should permit experiments to provide the needed facts.

Item 2 – Techniques for Making Mats

Electroforming Screen Structures. Several methods for electroforming and improving screen structures have been considered. These include

(1) Electroforming on flat sheets with a photo-resist as a stop-off for the holes.

(2) Plating on one side of screens to increase thickness

(3) Electroform screens on plastic-filled mandrels

(4) Electroform corrugated strips for assembly into screen structures.

Method 1 – Photo-Resists. Buckbee Mears Company electroforms screens* on flat mandrels using a photo-resist to stop-off hole areas.

Figure 13 is a sketch of cross section of electroforming mandrels. The top section represents squares of photo-resist which have metal exposed between the squares. Screens are formed by plating on the exposed metal and peeling the plate from the mandrel. Their present quality requirement limits the use of one printed mandrel to about 20 cycles before the photo-resist fails in enough areas to be considered unusable. The exact causes for loss of photo-resist materials are unknown at this time. No work is planned at Battelle on ways to improve the life of photo-resist. However, an opinion will be sought from the Buckbee Mears Company about the feasibility of attaining hundreds or thousands of electroformed plaques from the same mandrel. Their wide experience in this area of technology should provide definitive answers.

*Buckbee Mears Company screens were used in stacking and bonding experiments.

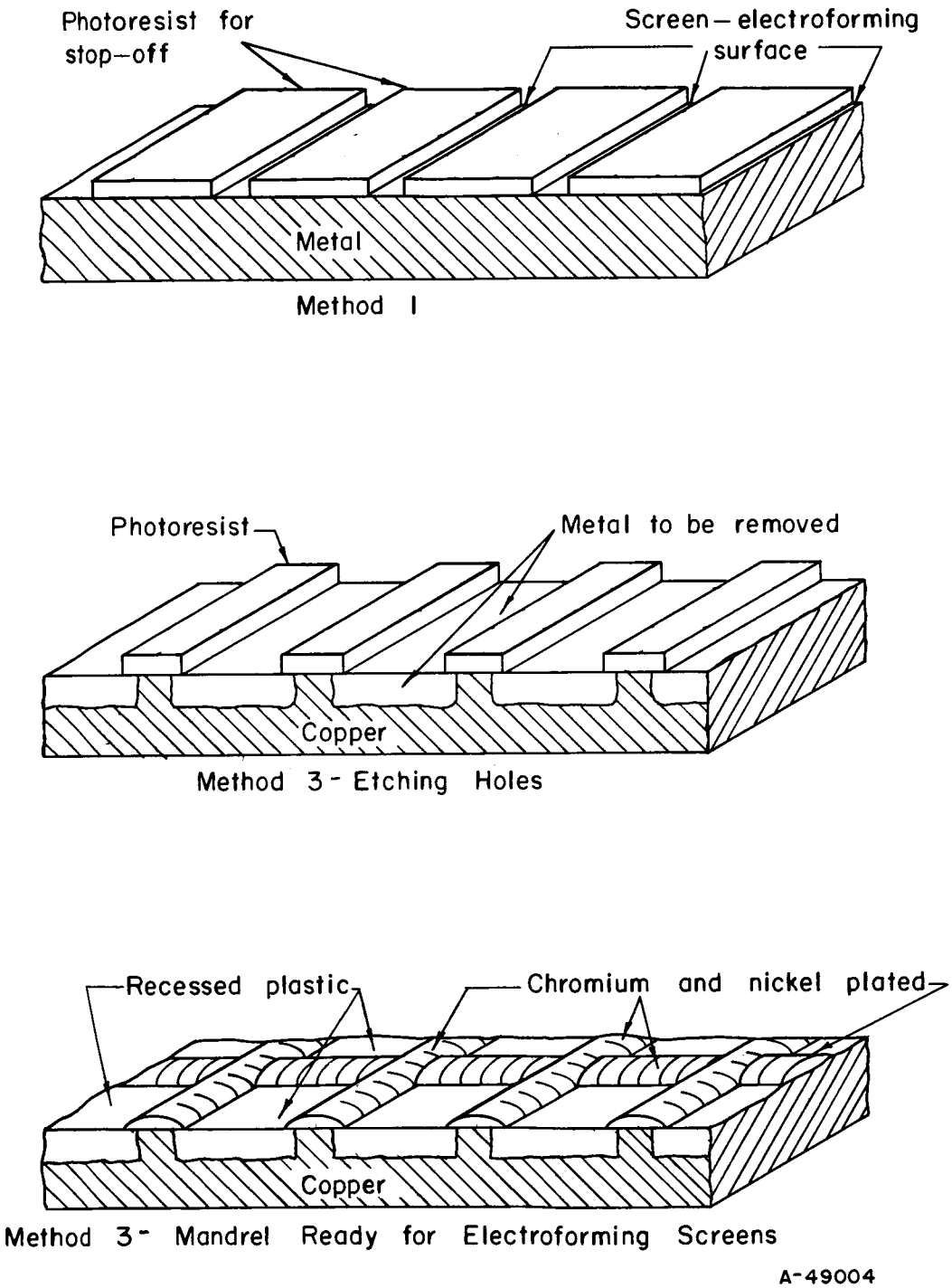
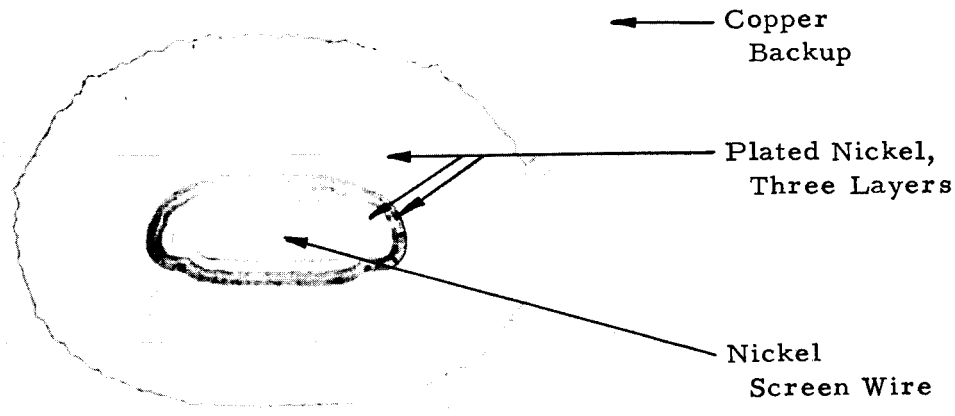


FIGURE 13. SKETCH OF CROSS SECTIONS OF SCREEN ELECTROFORMING MANDRELS

Method 2 – Increased Thicknesses. Plating on one side of electroformed screens, according to U. S. Patent 2,879,068, was evaluated for increasing the thickness of screens has been abandoned. The method was developed for increasing the strength of screens by plating on one side of the screen such that much less metal deposited in the holes than in conventional plating. Most of the work was done on screen with about 50 lines per inch and with a wire size of 10 mils wide and 5 mils thick. For this size of screen, plating could be concentrated on one side only.

The same process applied to a 250-line-per-inch electroformed screen did not limit the plate to one side only. Nickel was plated almost uniformly on all sides of the wires. Figure 14 shows a photomicrograph of one wire plated by the special technique. The elliptical area is nickel surrounded by copper for support for microscopic examination. Three layers of nickel were deposited on the wire under different conditions. The difference in the thickness of plate of the three layers was due to a different number of ampere-minutes used. The reason the process did not perform as expected on the fine 250-line-per-inch screen is probably because nickel baths have poor macrothrowing power but good microthrowing power. Throwing power of a plating bath is the relative ability to plate uniformly on all surfaces of a complex-shape cathode. Apparently, screens of 50 lines per inch fall in the poor macrothrowing-power range, whereas the 250-line-per-inch screen is in the macrothrowing power range.



1300X Etched-30% Lactic, 10% Nitric Acids 14033

FIGURE 14. CROSS SECTION OF ONE WIRE OF A 250-LINE-PER-INCH SCREEN PLATED WITH THREE LAYERS OF NICKEL PLATE TO INCREASE THICKNESS

Several conditions were identified which gave minor improvements in controlling the efficiency for plating on one side of the screens. These included operating the plating bath with (1) high pH, over 4.5; (2) high concentration, 90 g/l of nickel; (3) high temperature, over 120 F; and (4) low current density on the screen with high currents on the secondary cathode. A secondary cathode is placed behind the screen in this process so that current which would normally deposit on the sides and back of the wires is diverted to the secondary cathode.

Method 3 – Plastic-Filled Mandrels. Several reusable mandrels have been made with plastic filled holes and used for electroforming nickel screens. Copper plates were

etched using conventional photoengraving methods to produce a screen pattern that was a negative of the screen pattern in Method 1. Holes were filled with a plastic material to prevent plating in the holes. The excess plastic was removed from the raised wires, and screens were electroformed on these wires. The mandrel is illustrated in Figure 13. Three plastic materials, silicone rubber, polyvinyl chloride, and epoxy resins were checked. The epoxy was selected for use. However, the silicone rubbers, because of their flexibility, are still of interest.

A two-step technique was developed for filling the holes with plastic. A small amount of the uncured resin-catalyst mixture was forced into the holes with a flexible rubber blade, then more of the mixture was spread over the surface and the excess scraped off with a nonflexible blade to leave the holes filled.

To allow the electroformed screen to be removed from the mandrel, two surface conditions were checked. Nickel plated on the copper wires on the mandrel was oxidized anodically in an alkaline bath. This allowed an electroformed screen to be removed from the mandrel. However, when a second screen was formed on the mandrel without an intervening passivation, the screen stuck to the mandrel and could not be removed in one piece.

Chromium-plated mandrels were checked, as the second method, and successive screens were electroformed and stripped with ease. Therefore, subsequent mandrels will be chromium plated to aid removal of screens.

Method 4 – Corrugations. A method of making a screen structure has been conceived whereby corrugated strips of nickel would be electroformed continuously on cylindrical mandrels and assembled into a honeycomb to give a screenlike structure. Some advantages for this method would be (1) easy control of mat thickness because wire width would equal mat thickness, (2) no hole alignment problem because each ridge in the strip would form a continuous pore through the structure, (3) larger pore volumes could be obtained because strip thickness could be made small, 0.2 mil or less, and still be strong enough to handle. On the other hand, electroformed screens of the same thickness would be more difficult to handle because the holes significantly reduce the strength.

Superimposing Screens. A machine has been built for stacking screens, 1 by 1 cm, so that the holes in each stack will form a continuous path straight through the structure. In other words, a thick screen will be made by fastening together several thin electroformed screens.

The machine for superimposing screens consists of a hollow punch, a die with a removable bottom, a microscope for aligning screens, and the necessary supports, guides, and adjusting screws. In operation, an electroformed screen is aligned over the die by means of cross hairs in the microscope and a section of the screen is punched out and pushed into the bottom of the die. The microscope is mounted to view the screen through the center of the hollow punch. Every screen section can be checked for alignment before and after punching. Should a section of screen not align properly, it can be removed with adhesive tape. Alignment of each screen section will be considered satisfactory when each hole is within 90 percent of its intended position. The first stack of screens comprised 250-line-per-inch screens built up to 5 mils of thickness.

Five-mil-thick stacked screens have been made and diffusion bonded in which the holes were deliberately not aligned. The structure provided a good check on the sintering conditions that have been chosen. Instead of using a random orientation, alternate layers of screens were oriented about 45 degrees to each other. Electrochemical evaluation of this orientation should establish the merits of straight-through holes. The 45-degree orientation could have two functional advantages. More internal surface area is available because most of the area of all four sides of each individual wire is exposed, whereas with superimposed holes, only two sides of each wire are exposed. The second advantage is that the crisscross pattern of wires in successive layers could help hold the active material in place. Simplicity of stacking is increased also.

Item 3 – Methods for Controlling Pore Shapes

The first controlled shape to be produced is the parallel-side structure. After techniques have been developed for accurate stacking of screens, other pore shapes will be made and checked.

Future Work

(a) Presently made stacked screens, having 45-degree orientation of wires, will be etched to greater than 60 percent porosity and physically prepared for impregnation and electrochemical evaluation.

(b) Superimposed screens with square holes and parallel sides, will be stacked to at least 5 mils of thickness and then be sintered together. Four sizes will be used: 250, 500, 750, and 1000 lines per linear inch.

(c) New methods for making electroformed screens and pore structures will receive further consideration.

Location of Data

All experimental data on Task A are contained in Battelle Laboratory Record Books Nos. 12516, pp 1-23; 21595, pp 1-17; and 21652, pp 1-9.

Task C – Sintered Plaque Processing

Objectives

Task C of the contract reads as follows:

"Processing parameters for fastening superimposed screens together shall be determined.

"Item 1 – Temperature

Temperature and pressure for contact bonding;

"Item 2 – Time and Resistivity

Sintering time for high strength and low electrical resistivity.

"Item 3 – Atmosphere

Atmosphere to produce chemically clean surface areas and other special precautions that are required."

The objective of these items of work is to ascertain sintering conditions that are adequate to permit performance of the other tasks. By mutual agreement, between representatives of NASA and of Battelle, it was decided that research to optimize the sintering technique should be postponed until it was deemed necessary for more practical reasons.

Item 1 – Temperature and Pressure

Three randomly superimposed screens, 250 lines per inch and 1 by 1 cm, were sintered in tank hydrogen for 30 minutes at temperatures ranging from 1900 to 2300 F. Three die materials, Alundum, stainless steel, and oxide-coated stainless steel were investigated. Alundum weights of 6, 60, and 100 g/cm^2 supplied the pressure.

The temperature and pressure combination of 1950 F and 60 g/cm^2 was selected as best. This means that lesser temperatures and pressures failed to give satisfactory bonding. Greater temperatures and pressures gave obvious recrystallization and movement of material. These are qualitative measurements based on visual observations of technicians skilled in the art of sintering.

Item 2 – Time and Resistivity

A period of 30 minutes was selected arbitrarily as a convenient sintering time to achieve a sound structure. With complete sintering, electrical resistivity will be minimized.

Item 3 – Atmosphere and Special Precautions

Commercial tank hydrogen atmosphere was satisfactory for the reduction of any surface oxides on the screens.

Special precautions seem to be required for the die material. Oxide-coated stainless steel was satisfactory but the annealed nickel tended to bond to ordinary stainless steel. Oxide-coated stainless steel was prepared by heating stainless steel in air to form a tarnish. Oxide-coated chromium plate is considered to be equivalent to oxide-coated stainless steel and may be used in some instances.

Fingerprints and surface contaminations were avoided by handling all screens between plastic and with tweezers.

Summary

In summary, the following processing parameters for fastening superimposed screens have been selected for use in future preparations:

Atmosphere	-	Tank hydrogen
Time	-	30 minutes
Temperature	-	1950 F
Die Material	-	Oxide-coated stainless steel or its equivalent
Pressure	-	60 g/cm^2

Future Work

No further work is planned on Task C. If changes in sintering techniques appear necessary, such changes will be made. It seems unnecessary, at this time, to optimize sintering conditions.

Location of Data

All experimental data on Task C are located in Battelle Laboratory Record Book No. 21595, pp 1-17.

Task D – Plaque Classification

Objectives

Task D of the contract reads as follows:

"Processed plaques shall be tested to determine the following characteristics:

"Item 1 – Electrical Resistivity

Measurements shall be made in two directions; edge to edge and surface to surface.

"Item 2 – Pore Size Distribution

"Item 3 – Pore Shapes

"Item 4 – Internal Surface Areas

"Item 5 – Density and Porosity

"Item 6 – Measurement of Interconnecting Pores

"Item 7 – Tensile Strength

"Item 8 – Flexibility"

The objective of these items of work is to provide technical data with which interested persons might follow and evaluate the work. An attempt will be made to optimize internal surface area, Item 4, porosity, Item 5, and interconnecting pores, Item 6. The other items will be measured and compared with commercial sintered powder plaques before impregnation.

Item 1 – Electrical Resistivity

Electrical-resistivity measurements were made on commercial sintered powder plaques according to ASTM Standards A 344-60T. This test provides an edge to edge resistivity. The measured value was 159×10^{-6} ohm-cm. This resistivity is 23 times that of pure nickel* (6.9×10^{-6} ohm-cm), and indicates that the powdered plaque has relatively few good contacts.

Item 2 – Pore-Size Distribution

Results on commercial powdered plaques and commercial screens are described under Task A, Items 1 and 2.

Item 3 – Pore Shapes

Results on commercial powdered plaques and commercial screens are described under Task A.

Item 4 – Internal Surface Areas

Internal surface areas were measured by krypton adsorption on the commercial powder plaques described in Task A. The measurement was made by a modification of the conventional B. E. T. technique reported by Nelson and Eggertson,** employing a flaming gas stream. This method is particularly useful for small surface areas (less than 1 m^2/g) and is believed to be accurate to better than 20 percent of measured areas for sample sizes having a total area greater than 100 cm^2. The commercial powder plaques were found to have an internal area of 0.088 m^2/g. For 5-micron particles, the calculated area is 0.1 m^2/g. The photomicrograph of Figure 3 indicates the diameters are about 5 microns. For plaques with one-third weight of wire, the expected area would be 0.07 m^2/g. Measurements and calculations agree.

Surface areas of the commercial screens will be measured on stacked mats.

*Lange, N. A., Handbook of Chemistry, McGraw-Hill Book Company, Inc., New York, N. Y. (1961), p 105.
**Nelson, E. M., and Eggertson, F. T., Anal. Chem., 30, 1387-90 (1958).

Item 5 – Density and Porosity

Densities of commercial powdered plaques were measured and the density of the commercial screens were calculated assuming rectangular wires. The density values were used to calculate porosity using the following formula:

$$\text{Percent porosity} = \frac{\text{theoretical density - observed density}}{\text{theoretical density}} \times 100.$$

Results are shown in Table 4.

TABLE 4. DENSITY AND POROSITY OF COMMERCIAL ELECTROFORMED SCREENS AND COMMERCIAL SINTERED POWDER PLAQUES

Screen Size, lines/inch	Density, g/cm^3	Porosity, % voids
1000	5.33	40
750	3.56	60
500	3.47	61
250	3.60	64
Powdered plaque	1.85	79

As a check of the calculated porosity of the sintered powder plaques, the apparent percent volume of the nickel powder was checked from photomicrographs like Figure 3. A fine grid was printed on the pictures and the percentage of intersections that fell on the nickel (white color) phase was 14.3 percent of total number of intersections. Therefore, the approximate volume of voids was 86 percent calculated voids volume which does not include the wire screen in the powder plaque. The wire screen raises the net density and lowers the calculated void volume.

Item 6 – Measurement of Interconnecting Pores

All pores in the sintered powder plaques were interconnected because all the significant volume can be accounted for. The void volume measured by mercury porosimetry (Figure 1) was 0.425 cm^3/g. The void volume calculated from geometric dimensions of the plate was 0.413 cm^3/g and 0.427 cm^3/g for two determinations, or an average of 0.420 cm^3/g. The two independent measurements agree. The geometric measurement of gross volume was made by measuring the dimensions of a specimen and correcting micrometer thickness measurements by half of the surface roughness obtained with an electronic surface-roughness gage.

Item 7 – Tensile Strength

Tensile strengths of sintered powder plaques were measured with a substandard specimen 1/4 by 1 inch. The measured values are recorded in Table 5. Note that two end points were determined. The first was at the first visible cracking of the powder and the second was failure of the supporting screen.

TABLE 5. TENSILE STRENGTH OF COMMERCIAL POWDER PLAQUES

Sample	Tensile Strength, psi	Yield Strength (0.2% Offset), psi	Elongation (1-Inch Gage), %	Remarks
1	1650	1030	3	Tested to cracking of nickel powder
2	1700	912	3	
3	2320	997	15	Tested to failure of wire screen
4	2850	979	14	

Item 8 – Flexibility

The flexibility of sintered powder plaques was determined by the test described in ASTM Standards A-344-60T. In this test the specimen was bent over a 0.5-cm radius. Cracking of the powder was seen during the first 90-degree bend.

Future Work

Classification of commercial sintered powder nickel plaques has been completed except for surface to surface resistance. This measurement will be made. Promising screen plaques will be classified when identified experimentally in Task F, electrochemical evaluations.

Location of Data

Experimental data on Task A are contained in Battelle Laboratory Record Book No. 21595, pp 1-17.

Task E – Impregnation Procedure

Objectives

Task E of the contract reads as follows:

> "Electroformed nickel screens shall be impregnated with Cadmium Oxide. A minimum of two methods of placing the reactant material on the sintered plaque shall be investigated.
>
> a. Physical Packing
> b. Chemical Impregnation"

The objectives for this task are (a) to show that chemical impregnation procedures used at Battelle are comparable with impregnation procedures actually used in the manufacture of batteries, and (b) to ascertain whether simplified impregnation procedures might be used with the new electrode structures prepared as part of Task B.

A comparison of Battelle's impregnation procedure with commercial plaques seems necessary because commercial impregnation procedures are not known with certainty. There are occasional rumors that manufacturers have proprietary art that is superior in some way to the published procedures. There is, therefore, a need to show that commercial plaques can be duplicated in the laboratory.

Simplified impregnation procedures are desirable because impregnation seems to be a major item in the high cost of nickel-cadmium batteries. A 6-volt battery rated at about 1 ampere-hour, for example, costs a consumer about $20. This cost corresponds to more than 300¢ per watt-hour that requires less than 3¢ worth of active materials. A large part of this increased value would seem to be related to the costly impregnation procedures used in the manufacture of batteries. The controlled pore shapes, described in Task B, might lend themselves to a simple physical impregnation, as by rolling. The present work is intended to investigate such possibilities.

General Conditions

As a general scheme to accomplish the above objectives the following steps were planned:

Step 1 - Select the impregnation procedure most commonly indicated by the open literature.

Step 2 - Check weight gains observed in the laboratory against weight gains reported in the literature.

Step 3 - Check ampere-minutes per in.^{3} of plaques observed with laboratory electrodes against values observed for commercial plaques and against values reported in the literature.

Step 4 - Compare weight gains and ampere-minutes observed with new structures and new procedures against values obtained in Steps 2 and 3 above.

Step 1 – Selection of Procedure. The chemical impregnation procedure described by A. Fleischer* for the preparation of nickel-cadmium battery plates was selected for two reasons. First of all, the procedure has ample detail for duplication and comparison. Secondly, a review of the literature indicated that Fleischer's procedure is probably used by the battery trade with possible slight (but unknown) variations.

Fleischer's procedure consists of the following four steps:

(1) Vacuum impregnation in saturated nitrate salt solutions at about room temperature

(2) Precipitation of the hydroxides in the pores by cathodic polarization in 25 weight per cent potassium hydroxide

(3) Washing with water

(4) Air drying.

*Fleischer, A., J. Electrochem. Soc., 94, 293-296 (1948).

Repeat Steps (1) through (4) at least four times. The four steps combined is called one impregnation cycle. Each cycle required about 6 hours in the laboratory.

In commercial production, of course, the lengthy washing and drying steps might be greatly shortened. This would shorten a cycle to something on the order of 30 minutes, which is still a long time for commercial processing that requires four or more cycles.

Step 2 – Weight Gains. Commercial plaques have been impregnated at Battelle by using the procedure described above. Assuming the density of cadmium hydroxide to be 4.79 g/cm^3 (Fleischer also assumed this theoretical density), the volume of active material precipitated in the pores was calculated from the weight gain after each impregnation. The percentage of pore volume filled with active material was determined from the active material volume and the calculated pore volume. Battelle observations are given in Table 6. The results obtained compared favorably with those reported by A. Fleischer*, as shown in Figure 15.

TABLE 6. TOTAL AMOUNT OF CADMIUM HYDROXIDE PRECIPITATED AS A FUNCTION OF THE NUMBER OF IMPREGNATIONS

		Cadmium Hydroxide Precipitated		
Sample	Impregnation Number	Observed Grams	Calculated cm^3	% Pore Volume Filled
a	1	0.0262	0.00547	12.7
	2	0.0458	0.00957	22.2
	3	0.0600	0.0125	29.0
	4	0.0817	0.0171	39.6
b	1	0.0273	0.00570	13.2
	2	0.0478	0.00998	23.1
	3	0.0616	0.0129	29.9
	4	0.0799	0.0163	37.8

It should be noted that less than 40 percent of the pore volume was filled by four impregnation cycles, because this means that more efficient impregnation is possible.

Future Work

Future work will continue with Step 3, check ampere-minutes per in.^{3} of plaques, to ascertain the number of impregnation cycles required to duplicate the capacity of commercial plaques. New electrode structures, prepared as part of Task B, will be chemically impregnated.

Location of Data

All experimental data on Task E is contained in Battelle Laboratory Record Book No. 21685, pp 1-8.

*Fleischer, A., Nickel Cadmium Battery Corp. "Laboratory Investigations and Research Toward the Development of Nickel Cadmium Alkaline Storage Batteries", Quarterly Progress Report (Dec. 31, 1947).

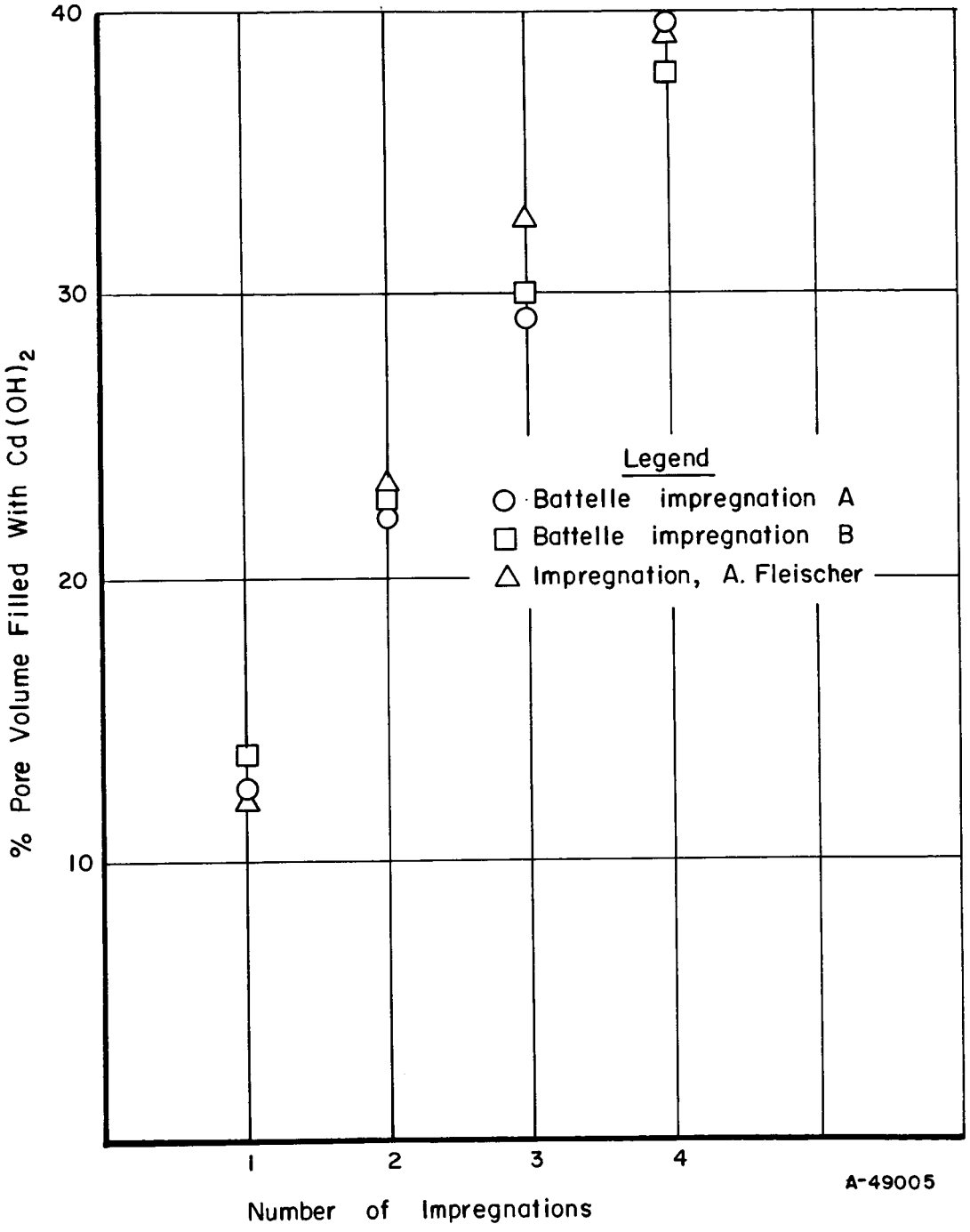


FIGURE 15. PER CENT PORE VOLUME FILLED WITH $Cd(OH)_2$ AS A FUNCTION OF THE NUMBER OF IMPREGNATIONS

Task F – Electrochemical Evaluation

Objectives

Task F of the contract reads as follows:

> "Charge and discharge data shall be obtained on single and multiple screen electrodes to obtain the following:
>
> Item 1 – Energy Output
>
> Energy output per weight and per unit volume of electrode as a function of current density, depth of discharge, pore size, pore shape and electrode thickness.
>
> Item 2 – Energy Input
>
> Energy input required to charge electrodes as a function of their charging current density, depth of discharge, pore size, pore shape and electrode thickness."

The objective of Item 1 is to show the conditions for increased energy densities on a weight and volume basis.

The objective of Item 2 is to show the conditions for minimizing energy input.

Item 1 – Energy Output

Units of Measurement. The ultimate units selected for evaluation of electrodes will be termed "Weight Improvement" and "Volume Improvement". These new terms are percentages defined as follows:

$$\text{Weight Improvement} = \frac{(WH/lb)_{Ex.} - (WH/lb)_{comm.}}{(WH/lb)_{comm.}} \times 100$$

$$\text{Volume Improvement} = \frac{(WH/in.^3)_{Ex.} - (WH/in.^3)_{comm.}}{(WH/in.^3)_{comm.}} \times 100$$

$(WH/lb)_{Ex.}$ = watt-hours per pound observed for experimental electrodes

$(WH/lb)_{comm.}$ = watt-hours per pound observed for commercial electrodes.

$(WH/in.^3)_{Ex.}$ and $(WH/in.^3)_{comm.}$ have identical definitions except that volume units are used in place of weight units. Precautions will be taken to make the two observations under comparable condition.

Relative units of measurement are necessary because standard tests, such as those proposed by the Sectional Committee on Dry Cells and Batteries-C18*, are not adaptable

*Specifications for Dry Cells and Batteries, Handbook 71, National Bureau of Standards, U. S. Department of Commerce (December, 1959).

to the single-electrode tests that must be used. Single-electrode tests, in turn, have meaning only when they can be related to the end product, that is, in a battery. The selected units will give practical meaning regardless of the means used to achieve their ultimate value.

Other units may be used as the work progresses. They will be explained as they are used and all the work will be directed toward achievement of values for the ultimate units of Weight Improvement and Volume Improvement.

Commercial Batteries. To give perspective to future experimental results, plaque data are being related to single-cell data and to battery data. This is necessary because new electrodes will be evaluated under special laboratory conditions but they will be used in assembled cells and batteries.

The general scheme is to show relative scale-up factors in going from experimental plaques to single electrodes, then from single electrodes to sealed cells, and, finally, from sealed cells to a battery.

A sealed commercial battery with flat plates was purchased. It had a nominal capacity of about 1 ampere-hour and contained five cells per battery. Physical dimensions were measured on the battery and on individual cells taken from the battery. Results are shown in Table 7. In Table 7, height includes the distance from the bottom of the battery to the top of the terminals. Width was measured parallel to the flat plates. Thickness was measured perpendicular to the flat plates. Measurements on other cells from the battery showed less than 1 percent variation from the values given in Table 7.

TABLE 7. PHYSICAL DIMENSIONS OF A COMMERCIAL NICKEL-CADMIUM BATTERY TO BE USED FOR COMPARISON PURPOSES

5-Cell Battery	
Height, inches	3.08
Width, inches	1.81
Thickness, inches	2.08
Total volume, cubic inches	11.6
Total weight, grams	403.2
Weight of Case Materials, grams	155.6
Volume of Case Materials, cubic inches	2.22
Individual Cells	
Height, inches	2.98
Width, inches	1.65
Thickness, inches	0.335
Total Volume, cubic inches	1.65
Total Weight, grams	49.3

The results in Table 7 indicate scale-up factors of about 38 weight percent and 19 volume percent in going from an individual cell to a battery. This particular battery had a metallic case with plastic-encased cells. This arrangement yields a relatively

large weight scale-up in going from cells to batteries. Some commercial batteries have plastic cases with metal encased cells. This alternate arrangement gives a smaller weight percentage scale-up, as low as 14 weight percent on another commercial model. Scale-up percentages will depend also on the size of the battery. An important point, however, is that at least one metallic case arrangement is required to contain the pressures that build up in sealed batteries during charging.

As a first approximation scale-up factors of 20 volume percent and 30 weight percent would seem quite reasonable.

Single Cells. A single cell was dismantled and dimensions are being obtained on the various parts. The plates were in the charged condition when the cell was dismantled. This gives some uncertainty to plate weight in the following tabulation:

Cell Part	Weight	% of Total
Case	11.93	24.3
Sealant	1.17	2.4
Terminals	5.78	11.7
Separators	0.61	1.2
Negative Plate	14.65	29.7
Positive Plate	10.07	20.4
Electrolyte	5.09	10.3
Total	49.30	100.3

In the tabulation, the first six items were weighed wet as dismantled from the cell. They were then washed and dried to constant weights to give the data tabulated. Losses of weight brought about by washing were added to the electrolyte weight, assuming 30 weight percent potassium hydroxide. The sealant item includes rubber grommets and a safety valve.

Plate weights are to be checked and volume dimensions are yet to be made. Tentatively, the data show that negative plates comprise about 30 percent of cell weight and the positive plates comprise about 20 weight percent. Both electrodes comprise about half the cell weight.

Electrochemical Data. A single commercial cell was charged at 60 milliamperes for 17 hours and discharged at 640 milliamperes to 0.5-volt cut-off for a number of cycles until reproducible ampere-hours were obtained on discharge. The above charging rate was used because it was recommended by the manufacturer. The charging coulombs capacity was 1.02 ampere-hours. The above discharge rate was used because it corresponded to a nominal 1-hour rate, based on the manufacturer's specifications. The first two cycles, shown in both parts of Table 8, represent the first two reproducible cycles. The results show an actual capacity of 0.95 ampere-hour instead of the 0.64 ampere-hour predicted from the manufacturer's brochure for a 1.0-volt cut-off.

After Cycle 2 the cell was dismantled and the electrodes were submerged in 30 weight percent potassium hydroxide. The objective was to determine whether the same results would be obtained with flooded electrolyte as was obtained with the sealed cell. There were three negative plates with two positive plates between them. Flooding the

electrodes in this manner caused them to flare out from the connecting terminals so that the electrode spacings became relatively large. Results are given in Table 8, as Cycle 3 and the discharge curves (Cycles 2 and 3) are shown in Figure 16. The voltage data in Figure 16 show that electrolyte resistance is negligible. The time data show that the ampere-hour capacity is practically identical in the sealed and flooded conditions.

TABLE 8. ELECTROCHEMICAL PROPERTIES OF ELECTRODES FROM A COMMERCIAL NICKEL-CADMIUM BATTERY

Input	Ampere-Hours
Per cell, sealed, Cycle 1	1.02
Per cell, sealed, Cycle 2	1.02
Per cell, flooded, Cycle 3	1.02
Per cell, flooded, Cycle 4	1.20
Per cell, flooded, Cycle 5	1.20
Output	
Per cell, sealed, Cycle 1	0.957
Per cell, sealed, Cycle 2	0.946
Per cell, flooded, Cycle 3	0.981
Per cell, flooded, Cycle 4	0.931
Per cell, flooded, Cycle 5	0.997

Because the charging coulombs were so close to output coulombs, Cycle 4 was performed to increase the charging coulombs to 1.2 ampere-hours. Output Cycle 4 actually showed fewer coulombs with the extra charging coulombs. Cycle 5 was, therefore, a repeat of Cycle 4 conditions. The result of Cycle 5 confirms the conclusions: (a) ampere-hour capacity is the same whether the electrode is flooded or sealed in a cell, and (b) the positive electrode had received full charge on all the cycles shown in Table 8.

A duplicate sealed cell was charged and discharged a number of times to determine the accuracy of results in Table 6. Measured values of 0.993 and 0.988 ampere-hour showed that cells from the battery can be considered to be identical.

Integration of the area under the curves in Figure 16 shows about 1.15 watt-hours. Combining the energy output with the weights given in Table 5 gives the following values:

$$(\text{WH/lb cell})_{\text{comm.}} = 10.6$$

$$(\text{WH/lb battery})_{\text{comm.}} = 6.46$$

The above battery value is significantly lower than values reported in the literature for nickel-cadmium batteries. Some of the values reported are

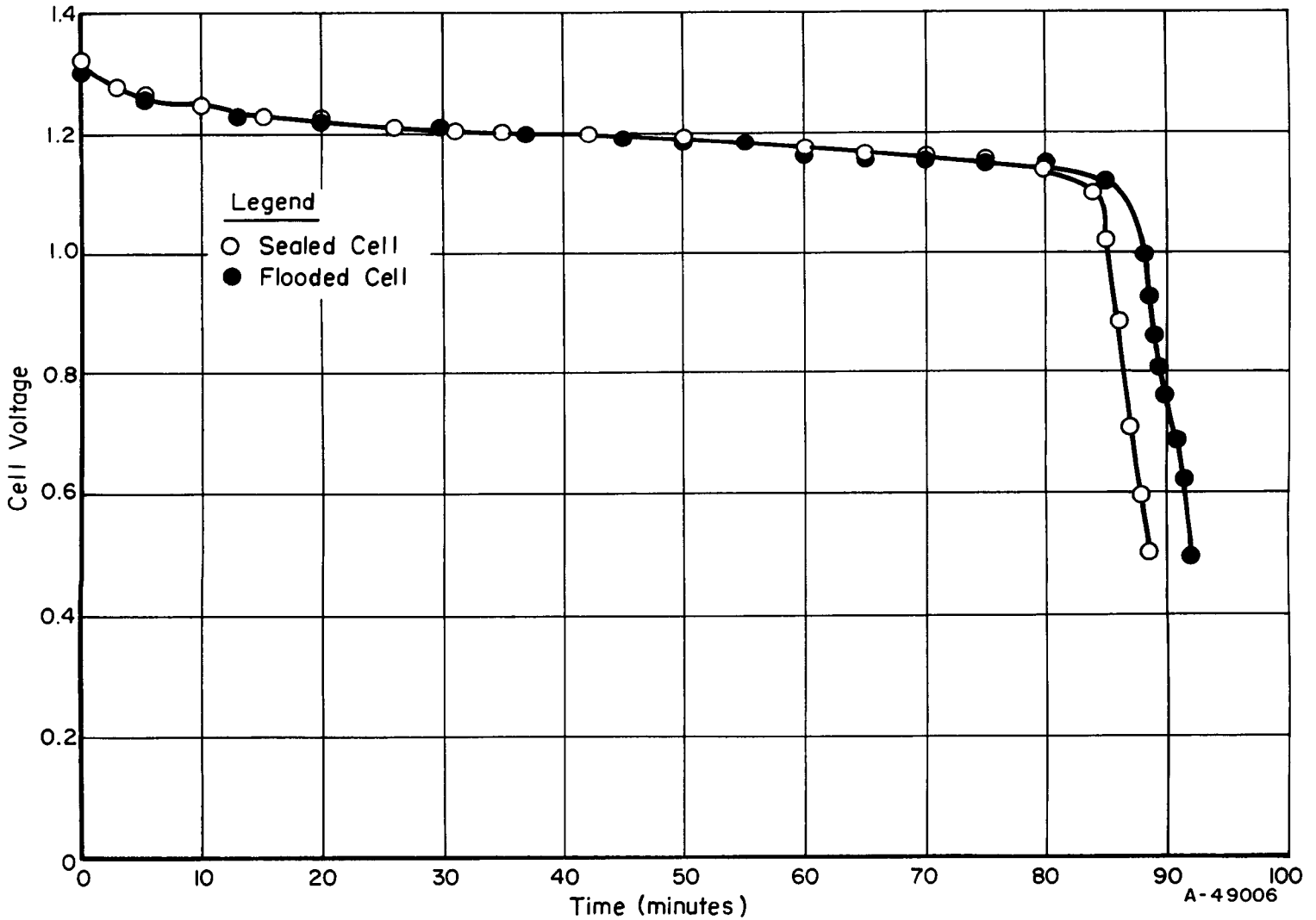


FIGURE 16. DISCHARGE DATA FOR COMMERCIAL NICKEL-CADMIUM ELECTRODES AT 0.640-AMP CONSTANT CURRENT IN A SEALED CELL AND UNDER FLOODED CONDITIONS

Reference	(WH/lb battery)$_{comm.}$
J. C. Duddy and A. J. Salkind, The Electric Storage Battery Company, HIGH ENERGY:WEIGHT RATIO, NICKEL CADMIUM CELLS, Journal of the Electrochemical Society, 108 (8), 717-719 (August 1961).	10
Paul L. Howard, Yardney Electric Corporation, WET-CELL BATTERIES FOR POWER, Product Engineering, 31 75-82 (February 15, 1960).	12
C. G. Grimes and W. S. Herbert, The Electric Storage Battery Company, THE CHOICE OF BATTERY SYSTEMS, SAE Preprint No. 269D presented at the 1961 SAE International Congress and Exposition of Automotive Engineering, Detroit, Michigan (January 9-13, 1961).	11.5
Howard T. Francis, Armour Research Foundation of Illinois Institute of Technology, SPACE BATTERY HANDBOOK, prepared under NASA Contract NASw-401 (April 15, 1963).	12
R. C. Shair, G. Rampel, and E. Kantner, Gulton Industries, Inc., HERMETICALLY-SEALED NICKEL-CADMIUM STORAGE BATTERIES, IRE Transactions on Military Electronics, MIL-6 (1), 67-71 (January 1962).	12

The most obvious explanation for the smaller value found at Battelle is associated with the smaller cells and higher rates used at Battelle. The literature values are for batteries on the order of 100-ampere-hour capacity discharged at 5- or 10-hour rates. The Battelle values are for 1-ampere-hour cells discharged at a 1.5-hour rate. The larger batteries have a larger percentage of electrode weight and smaller percentages of cases and adjunct parts. The weights of electrodes, electrolyte, and separators in the cells, checked at Battelle, for example, are only 39 percent of the total battery weight. In a large battery, these vital parts would exceed 60 percent of the total weight. Moreover, a lower discharge rate can be expected to increase WH/lb by 5 to 10 percent. The effect of rate will be studied in future work.

A significant point, from results to date, is that comparisons with commercial products will become more realistic as experimental conditions become identical for new and commercial electrodes. That is, comparison of a new single electrode with a commercial single electrode will be more meaningful than comparison of an electrode with a sealed cell and, in turn, much more meaningful than comparison of a single electrode with a commercial battery reported in the literature.

Item 2 – Energy Input

Cell-charging data have been obtained at constant current. An attempt has been made to use about 20 percent more charging coulombs than are obtained on discharge. In other words, energy input has not yet been made one of the experimental variables.

When energy input is to be minimized with future work, commercial plaques will continue to be the primary standard.

Future Work

During the next report period, through October 18, 1964, the following work should be completed:

(a) Weight data for single commercial cells

(b) Volume data for single commercial cells

(c) Electrochemical data for single commercial electrodes, including positive and negative electrodes

(d) Chemical analysis of a commercial negative electrode to provide an independent check on the accuracy of electrochemical data

(e) Electrochemical data for 1 cm^2 negative electrodes cut from commercial plates, to serve as primary reference standards.

In addition, electrochemical evaluation will start on the new screen electrodes described in Task B of this report. Evaluation of commercial battery plaques described in Task E will commence also.

Location of Data

All experimental data on Task F are contained in Battelle Laboratory Record Book No. 21685, pp 9-16.

Task G – Corrosion Stability

Objectives

Task G of the contract reads as follows:

> "Not more than six selected electrode structures that show promise under Task F shall be given potentiostatic test for corrosion. Commercial sintered nickel plaques shall be used as controls."

The objective of this work is to show the inherent stability of new electrode structures to electrochemical oxidation.

Future Work

Work on this task is postponed until the promising electrode structures are selected.

JM:so

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