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ANNUAL REPORT FOR 1978 ON RESEARCH, DEVELOPMENT AND DEMONSTRATION OF NICKEL-IRON BATTERIES FOR ELECTRIC VEHICLE PROPULSION

Contract No 31-109-38-4141

by

Westinghouse Electric Corporation

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Prepared for

The Office for Electrochemical Project Management
Argonne National Laboratory
Under Contract No. 31-109-38-4141

By

Westinghouse Electric Corporation
Pittsburgh, Pennsylvania

October 1979

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ABSTRACT

The program has advanced to the level of full-size, prototype cell fabrication and evaluation. EPP nickel electrodes are now being prepared of up to 24 Ah/plate (at C/3 drain rate) at design thickness (2.5 mm). Iron electrodes of the composite-type are deliveryying 24 Ah/plate (at C/3) at target thickness (1.0 mm). Both plates are displaying good capacity stability at 130 to 175 test cycles, respectively, in some of the 3 plate cell tests. Finished cells are delivering up to 58 Wh/Kg at C/3, based on projected weight of the finished cell and in the actual designed cell volume. Reduction in cell resistance, reduction in nickel plate processing time and swelling on cycling are areas of major effort to reach the final battery objectives. Thermal nickel electrodes are showing promise in full size plate tests (up to 22 Ah in a plate of only 2.3 mm thickness) and will be evaluated in finished cells as soon as the technology shows repeatable results in full-size test plates.

CONCLUSIONS

1. Fiber grid structures have been selected for the iron and nickel electrodes. A nickel-plated steel wool having a steel current collector tab, is used for the nickel electrode. Unplated steel Exmet, sandwiched on either face with unplated steel wool and having an unplated steel current collector tab, serves as the iron plaque.

2. The electroprecipitation process has been used to produce, to date, 24 Ah full size nickel electrodes at target thickness (2.5 mm). EPP electrodes demonstrate cyclic stability up to ~130 test cycles on the best plates and up to 200 cycles on a lower capacity plate to date.

3. Thermal nickel electrodes have demonstrated ~22 Ah in full size electrode tests at ~50 test cycles to date.

4. Briquet full size iron electrodes have been prepared and demonstrated, in initial attempts, 23 Ah in full size plates, although some degradation (~25%) has been experienced in up to ~200 test cycles to date.

5. Composite-type, full size iron electrodes have been prepared and demonstrated at target thickness (1.0 mm) about 21-26 Ah. From 0 to 12% degradation has been experienced in these plates in cyclic tests up to ~175 cycles to date.

6. Full size iron-nickel cells have been constructed and tested that demonstrate 48-58 Wh/Kg at the C/3 drain rate. Degradation noticed in some of these cells seems to be related to nickel electrode swelling during cycling.

7. Accidental voltage reversal (for 35 hours) in 3 plate cell tests, due to a tester malfunction, has caused degradation in iron and nickel plates but, suprisingly, no catastrophic failures. This condition is worse than can ever be experienced by a plate in a battery-theoretically no more than ~3 hours of discharge.

8. The separator system has been identified as the major source of cell resistance and efforts will be concentrated on modification of the system to reduce overall cell resistance to the level desired to insure good power characteristics.

1.0 PROGRAM MANAGEMENT

In addition to providing technical and financial control of program activities, the program management tasks include preparation of special reports required by the Statement of Work. Since the technical and financial reporting elements are covered in other sections of this report, the information presented in this section will deal with the status of the special reports and other designated activities.

1.1 PROGRAM MANAGEMENT REPORT

A program plan was submitted on schedule, reviewed by ANL personnel and found acceptable with a few revisions and clarifications required. The revised plan incorporated the suggested revisions, was submitted in May and found acceptable by ANL personnel.

The detailed program plan for fiscal year 1979 which deals primarily with revisions to the Cost Plan (DOE Form 533P) and the Manpower Plan (DOE Form 534P) is currently being prepared with a planned submission to ANL by September 1, 1978.

1.2 QUALITY ASSURANCE (QA) PLAN

A draft QA Plan was submitted to ANL/OEPM in April and reviewed by ANL personnel. Several elements of the plan required additional clarification. A meeting was held with Mr. Erv Peterson, ANL QA/QC engineer, on August 2, to review the plan and discuss the specific elements requiring additional clarification. All questionable elements were resolved and a subsequent submission of the draft QA Plan is planned for September.

1.3 MATERIAL REPORT

A draft Material Report was submitted on schedule to Dr. John Barghusen the ANL/OEPM official administering the study. The report includes manufacturing process flow sheets, a complete breakdown of materials used in the manufacture of iron-nickel batteries (Kg/Kwh), an estimate of quantities of materials that could be recycled, projected market volumes and a forecast of battery characteristics throught the year 2000. The final report will be submitted on schedule (August 31).

1.4 RECYCLING STUDY

Several commercial vendors have been contacted to discuss the feasibility of recycling materials from used iron-nickel batteries and chemical compounds derived during the manufacturing process. Alternative materials reclamation procedures are being evaluated which range from a complete separation of cell components and subsequent raw material reclamation to total cell processing in a melt furnace with only a ferro-nickel alloy as the reclaimed material. The metals market supply/demand relationships between scrap and virgin metals appears to be the dominant factor in assessing financial feasibility of a suitable reclamation process.

Nickel is the major material of concern for reclamation. Three vendors have been contacted to evaluate feasible reclamation processes:

(a) National Nickel Alloy Corporation, Greenville, PA, is currently interested in processing only the nickel electrode as a ferro-nickel alloy for nickel reclamation. A sample nickel electrode was sent to them for materials evaluation and recycling feasibility.

(b) INMETCO, Ellwood City, PA, is a wholly owned subsidiary of INCO established to reclaim nickel and other precious metals. Mr. Todd Grant, President, met with Westinghouse personnel in August to discuss metals reclamation. INMETCO has the capability to reclaim nickel from ferro-nickel compounds as either nickel or to refine the scrap to other ferro-nickel alloys. Process developments are currently being undertaken to remove other tramp metals, such as cobalt, from scrap compounds. The plant is currently under construction and is expected to be operational by late 1978. Sample electrodes were sent to INCO Research and Development Center for materials evaluation.

(c) The Pesses Company, Solon, Ohio, is currently the major recycler of nickel-cadmium and nickel-iron batteries in the United States. A meeting was held with Dr. Marvin Pesses, President, to discuss recycling the Westinghouse nickel-iron battery. Edison-type iron-nickel batteries are presently smelted as a unit with no attempt to reclaim materials other than ferro-nickel alloy obtained. The cells are bought on the basis of contained nickel at approximately 50% of the current nickel market price.

Nickel-cadmium batteries are handled differently. The tops are cut off the cells and plates are separated. The nickel and cadmium electrodes are processed separately to give high purity ingots which are sold as such. An effort is currently underway to wash the thermoplastic case material and sell it as regrind. No attempt is made to reclaim the electrolyte.

In the case of the Westinghouse iron nickel cell design, either of the above processes is viable, dependent on the final design. The following criteria would determine the process:

(1) If copper terminals are used, the cell would be cut and stripped to remove the copper since it is considered an unwanted contaminant in ferro-nickel alloys.

(2) If cobalt is used at the current ratio in the nickel electrode they may also be required to strip the cell and process to remove the cobalt. Since a bulk of the ferro-nickel alloys sold by them to stainless steel manufacturers end up in nuclear equipment applications, the cobalt is an unwanted "contaminant". Dr. Pesses stated that most of the cells they currently recycle have from trace amounts of cobalt to 1-2%.

If the cell electrodes were sold separately, the nickel would be worth approximately \$1.30/# contained nickel (with the current market price of nickel at ~\$1.90/#), the iron would be worth a few cents a pound.

Dr. Pesses indicated a strong willingness to work with Westinghouse in the future and stated they would buy scrap cells in small lots for initial feasibility studies.

Since the above three companies are interested in primarily the metals reclamation and have no current interest in plastics, the case/cover and separator materials reclamation will be investigated with appropriate thermoplastic processors and manufacturers. Recycling applications for cell electrolyte and spent electrolyte used in the EPP process will also be determined.

1.5 ENVIRONMENTAL IMPACT STUDY

A computer assisted literature search was done on the Lockheed data base, centering on the following segments: APTIC, Chemical Abstracts, Energyline, Enviroline, Compendex, NTIS, and Pollution Abstracts. Words searched were selected with a view towards environmental impact statements and included components of the battery, compounds used in its manufacture and use, toxicity thereof, safety precautions and disposal methods. The abstracts were evaluated and pertinent abstracts noted. Relevant information is planned to be retrieved from the Key abstracts.

The regional Federal EPA offices and Pennsylvania Department of Environmental Resources office have been contacted for appropriate effluent information and other relevant environmental standards. Currently no standards or guidelines exist on the state or federal level for nickel battery facilities. It appears to be the consensus of those involved that if a standard were imposed it would closely adhere to the electroplating industry guidelines which are currently published in EPA document 440/1-75/040.

The major concern is currently concentrated on nickel electrode processing and subsequent effluent treatment requirements.

An August meeting has been established with ANL personnel from the Biological and Medical Research Division and Environmental Impact Studies Division to discuss the Westinghouse iron-nickel battery manufacturing processes relative to their health assessment program on near-term batteries.

1.6 SAFETY ANALYSIS

The battery system safety analysis information is being collected for the preliminary report due at the first design review. Experiences obtained as a result of previously operated electric vehicle battery systems and applicable safety standards followed by other industries utilizing battery systems will form the basis for the analysis.

1.7 SYSTEM SPECIFICATION

The preliminary specification was submitted on schedule in June. The final specification will be delayed until after an ANL/OEPM near-term contractors meeting to be held in September to discuss battery specification guidelines, testing procedures and reporting requirements.

The Initial Iron-Nickel Battery Specification consists of four sections. The first section relates Cell and Battery Specifications to the initial near-term target of 50 Wh/Kg (23 Wh/lb), as well as the final goal on the contract of 60 Wh/Kg (27 Wh/lb). The second section details pertinent performance information, particularly cell charge, discharge, power and temperature characteristics. The third section addresses potential performance limiting characteristics in the cell and battery. Important factors here appear to be charge energy efficiency and total electrolyte weight. The fourth section lists 11 battery tests that are planned to be performed on the iron-nickel battery. These tests will establish efficiency as a function of drain rate, charge acceptance, voltage characteristics, internal resistance behavior, open circuit capacity losses, temperature effects on capacity and life, gassing, electrolyte displacement and short circuit currents.

The data presented have been compiled from several sources: (1) extensive, in-house background experience and data obtained at Wetsinghouse, (2) recent development test results on small and full-size test electrodes, and (3) engineering projection based on the present state of technology.

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2.0 TECHNICAL STATUS

The technical effort on the program during this year is aimed at meeting the objectives of the System Development Task of the program.

2.1 SYSTEM DEVELOPMENT

The objective of the System Development task of this Fe-Ni battery program is to demonstrate improved performance capability in the initial prototype cells. These improvements include attaining 50 Wh/Kg and 100 Wh/l in the overall cell, under 4-8 h charge and 2-4 h discharge conditions, while exhibiting short-term, stable cycling capability.

The System Development task encompasses 5 major subtasks:

(1) the nickel electrode
(2) the iron electrode
(3) cell components
(4) cell testing
(5) pilot plant operations

The objectives of each of these subtask areas and their present status of progress are now described.

2.1.1 Nickel Electrode

The nickel electrode development consists of:

- controlling electrode swelling during charge/discharge cycling
- improving active material utilization
- improving the loading factor and porosity.

Concurrently all these developments are aimed at reduced materials, processing and capital costs.

The development approach to solving these problems consists of three interrelated activities: (1) grid structure, (2) electroprecipitation process (EPP) and (3) thermal nickel development.

2.1.1.1 The Grid Structure Development

The goal of the grid structure development is to provide the structural basis on which the optimal mix of loading, active material utilization, and final electrode porosity can be achieved. This is being accomplished by investigating variations of starting grid density and thickness and their effects on electrode loading, loaded electrode thickness and performance. The feasibility of eliminating or reducing high cost items (as in nickel plating the grid prior to loading) will be tested. In addition, the ability of the grid structure to control the nickel electrode swelling without external aids will be investigated.

The effort to date has been to produce fiber metal grids, using state of the art technology, and to establish the properties of the grids and the plates, when loaded, with respect to the design parameters. Here double-plaques, with the current collector tabs, have been fabricated, nickel plated, and used in the nickel plate development work.

New fabricating techniques, aimed at reducing costs and improving plaque uniformity, are also being investigated. Comparison of plaques made by these techniques with the state-of-the-art will dictate the candidate process to be pursued later in the program.

2.1.1.2 Nickel Plaque Preparation

The current collector and container for the active nickel oxide is a porous structure formed from sintered steel fibers. Fabrication of a plaque begins by assembly of a number of steel wool battes into a given fiber density. The plaques are heated in a hydrogen atmosphere causing the fibers to be sintered together in contact areas. The sintered plaque is then sized to the required thickness by a rolling operation. The plaque is trimmed and a steel tab-bus is seam welded to the plaque.

Corrosion protection is provided to these steel fibers by depositing nickel, uniformally, around each fiber by electroplating in nickel sulfamate. Figure 2.1 is a cross-section of a nickel-plated plaque, showing the excellent throwing power of the sulfamate bath. The nickel plating covers the steel fibers attached to the tab in the center of the

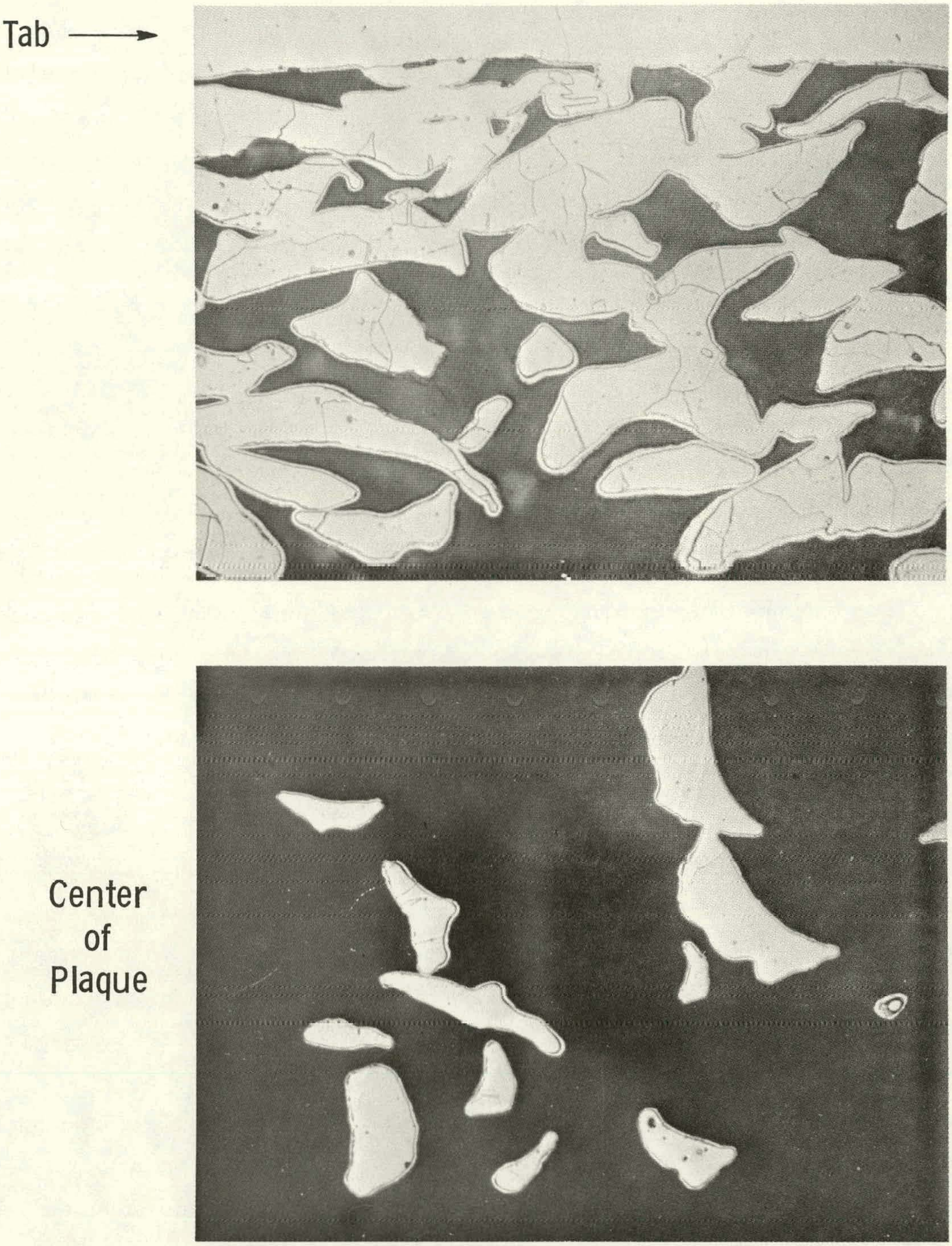

Fig. 2.1 — Ni-Plated steel fiber plaque structure of approximately 10% density - (1/0 fibers). (500 x)

plaque. The sinter bonds formed between fibers in contact during the sintering operation is also evident in these photomicrographs.

The present cell design dictates a final plate thickness of 2.5 mm. Plaques used with the EPP procedure are sized after the first sintering operation and are found to expand to approximately 2.5 mm, as a result of EPP processing and cycling in a finished cell. On the other hand, grid structures used with the thermal nickel process have a starting thickness of at least 4.4 mm and are sized to less than 2.5 mm in the pasting operation associated with this process.

Fibers used in these plaques are fabricated from wool having a designation of 1/0. As noted in Figure 2.1, their cross-section is irregular in shape, but are typically about 0.025 mm on an edge. Plaques have been prepared with fibers designated as 3/0, which have a smaller cross-section and, therefore, higher fiber surface area for attachment of the nickel oxide in the pores of the plaque, at a given weight of fiber.

In the needle punching process, a plate containing a large number of needles, ~1000 per 750 cm^2, is pressed vertically into the unsintered fiber plaque. Barbs on the side of the triangular shaped needle end, or a fork or groove cut into the end of another needle type, push some fibers down through the fiber structure. These fibers tend to lock the structure, causing the plaque to be reduced in thickness. The final thickness of the plaque can be controlled by varying the depth of penetration, from one or two sides of the plaque. Experiments to date, have shown that when the needle just penetrates to the base of the plaque, resulting in a compaction thickness of 7.5 mm, the uniformity of the fiber structure is maintained. Perforations where the needles had passed through the structure are evident but there is no gross tearing of fibers evident, as when the needle penetrates excessively and the plaque is caused to move slowly through the perforating area. A laboratory model of a needle-punching machine (Fiber Locker) is shown in Figure 2.2. The uncompacted fiber lay-up and needle-punched sections of plaques cut from two different trials through this machine are shown in Figure 2.3.

Experimental trials, to date, have been concerned with establishing compacting behavior of these plaque lay-ups under different machine conditions of needle-penetration and draw speeds, using the barbed

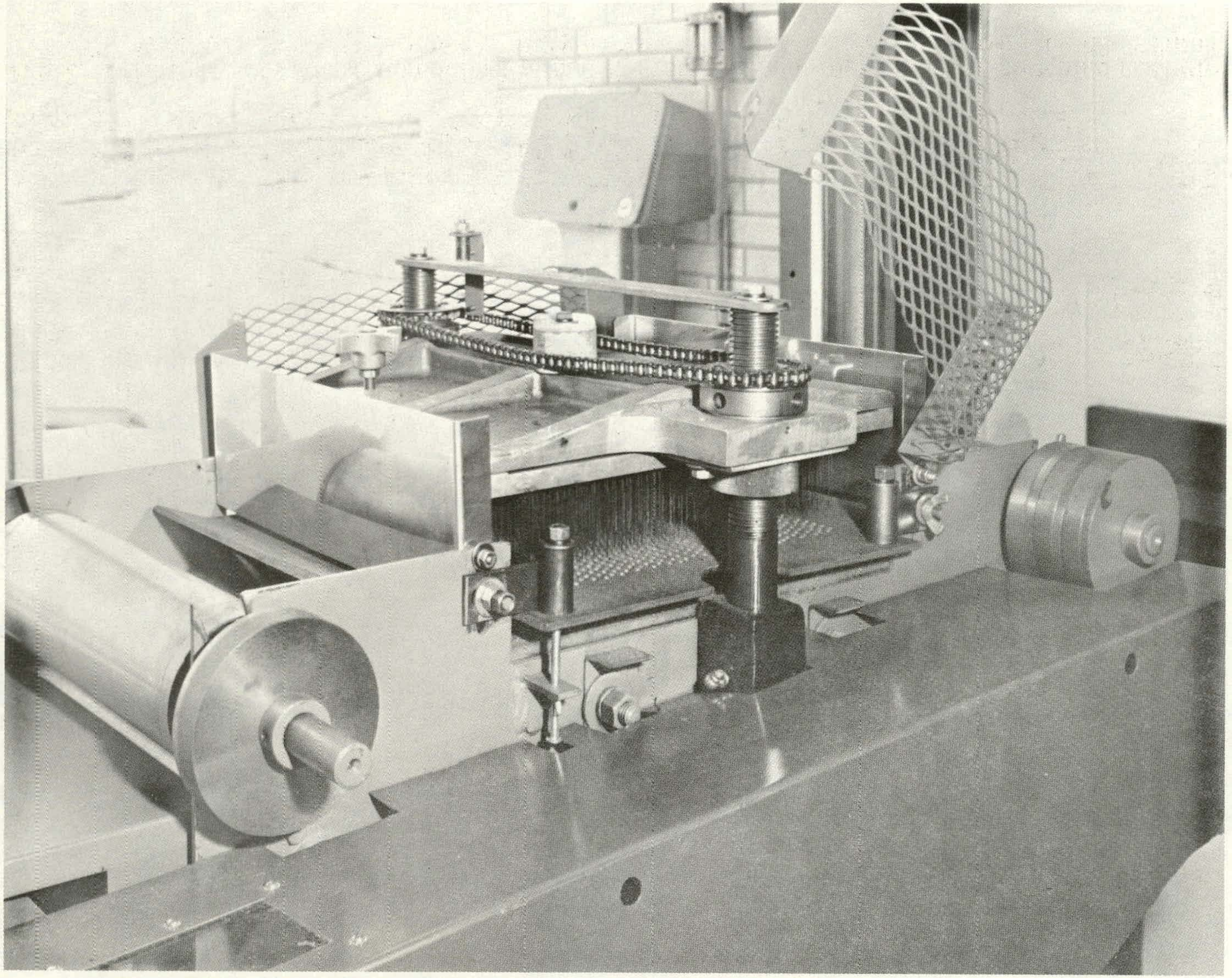

Fig. 2.2 — Laboratory model of a needle-punching machine

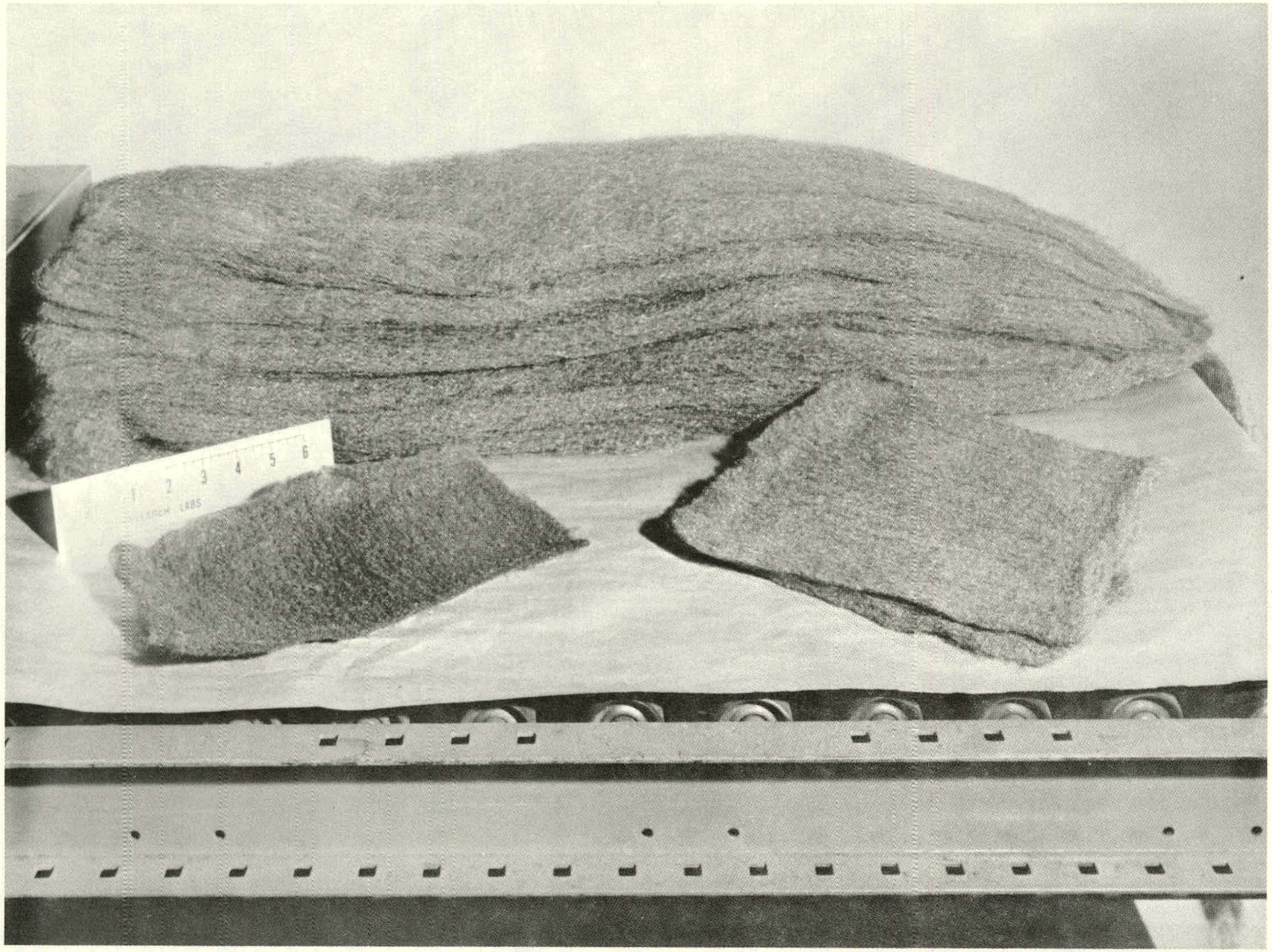

Fig. 2.3 — Fiber lay-ups and section of fiber plaque after processing through a needle-punching machine

needles. Some of these experimental conditions will also be performed with the fork needle.

Expansion growth of the plaque during EPP processing or cycling service of the plate is not readily prevented by the typical fiber structure because of the small contact regions between fibers in the thickness direction of the grid structure. With the needle-punching operation, fibers are mechanically bent 90° from their axis, causing a greater tensile strength in this thickness dimension. In preliminary experiments to develop this feature, it was found that while the fibers had interlocked and caused a permanent compaction of the unsintered fibers, there were very few fibers in the needle-punched area which had maintained their original length after being pulled down through the plaque. Since these machined fibers are highly stressed and notch sensitive, it is probable that this attempt to perform a dual function in one needle-punching operation is undesirable. Experiments are to be performed to establish a procedure which will develop the desired fiber structure.

2.1.1.3 The Electroprecipitation (EPP) Process

The objective of this subtask is to improve upon the well-established Westinghouse EPP process for preparing nickel electrodes. Specifically, the work is aimed at developing nickel electrodes with 0.25 Ah/g active material, 0.14 Ah/g total electrode and 0.075 Ah/cm^2 all at the 3h discharge rate. These goals are to be attained in plates of about 2.5 mm maximum thickness. The EPP process technology, at the onset of this program made 343 cm^2 plates of the following specifications: 0.20 Ah/g active material, 0.12 Ah/g total electrode and 0.07 Ah/cm^2 at the 3h discharge rate, ~3.3 mm in thickness.

In addition, process techniques are being investigated both to improve plate dimensional stability and eliminate the need for the nickel plating step, presently used on steel wool.

Ultimately on the program, the EPP process is to be further refined to meet the technical requirements of the nickel plate in the Fe-Ni cell, while minimizing process time and material losses.

Initial investigations in the small tank experimental line* involved tanks with Ni 200 anodes in a high gravity bath (maintained above 50°C). Cathode inefficiency of this temperature resulted in hydroxide ion buildup in the baths. Coupled with this, the high efficiency of the anode (due to the 50°C temperature) produced Ni++, and no acid, and soon turned the bath into an impossible sludge, even though, in initial runs, using a fresh bath, high capacity plates were produced.

Thus it was decided to revert to a room temperature bath. Depolarized anodes were also installed when it became apparent that Ni 200 anodes were unpredictable, since they could be easily passivated.

Therefore, the mini-line** was set-up with depolarized anodes, Ni/Co Nitrate bath above 2.5 pH and above 1.50 gravity.

An analysis of the nickel plate loaded in the mini-line showed a linear distribution of loading with the bottom plaque about 30% higher in weight gain than the top. This was attributed to the OH diffusing out from the plaques, producing colloidal $Ni(OH)_2$ in the bath which slowly floated upward. The colloidal $Ni(OH)_2$ clouds were not electrically conductive and thus the upper plates were in a zone of continually decreasing conductivity. The $Ni(OH)_2$ ended up as foam on top of the bath. Stirring completely eliminated this problem, as it intermingled enough of the acid bath to prevent formation of this colloidal $Ni(OH)_2$. However, stirring was found to be a variable in the process and experiments were conducted in which stirring was done only during the rest periods between pulses, as well as only during the pulse (loading) periods. Procedures have been developed whereby full size nickel plates have been produced with 24 Ah capacity at about 2.5 mm in thickness. Correlation between capacity, as determined during the formation discharge

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*Here, a single, double-plaque can be processed.

**The mini-line processes a rack of 5 double-plaques, sufficient to produce 10 finished nickel plates for a full-size iron-nickel test cell.

and actual capacity of the electrode in 3 plate* bench test cells, under conditions of limited electrolyte and tight packing, is excellent.

The efficiency of deposition of $Ni(OH)_2$ has been improved by a method involving control of two parameters: 1) applied voltage and 2) visual monitoring of evolved gases. The method is designed to control and limit undesirable side reactions, which not only divert the applied current into unproductive reactions, but also can block and create inaccessible pore regions, due to gas buildup.

Experimentation has demonstrated that 2.5 mm plates having 24 Ah capacity can be produced more efficiently using voltage control, coupled with visual gas monitoring. Plates heretofore produced with constant current techniques have required 90 Ah of EPP processing. We now produce, with voltage control and minimum outgassing, 24 Ah plates with less input EPP processing.

It is expected that, with future adjustment of NH_4^+ content in the EPP solutions, amine-complex inhibition will be eliminated, resulting in the production of plates having capabilities in the 26-27 Ah range at 2.5 mm thickness.

2.1.1.4 Thermal Nickel Plate Development

The technical goals for the thermal nickel electrode, in full-size plates, are the same as those specified for the EPP nickel electrode (see Section 2.1.1.3). The potential advantages offered by the thermal process are a reduction in active material preparation time and cost and a significant decrease in capital equipment expenditure. The EPP process, however, is in an advanced state of development, whereas the thermal nickel process is still in the early stages of scale-up. Although the various parameters for optimizing the thermal nickel process have been investigated on a small scale, methods for continuous large batch preparation have not been developed experimentally. Therefore, our effort during the past months has been to explore these possible preparation

---

*A nickel electrode with two iron counter electrodes.

methods. An equally important task is to formulate an optimum active material paste composition for incorporation into the fiber metal plaque.

All of the experiments previously conducted were done in a resistance heating oven, using small crucibles of either nickel or alumina. The three ingredients required, e.g., nickel oxide, cobalt oxide, and sodium peroxide, were blended thoroughly and heated slowly to the desired temperature. Time and temperature were measured using a standard themocouple-recorder assembly. This preparation scheme could easily be adapted to scale-up, using larger ovens and crucibles but it was felt that it could not be designed to produce material continuously. Therefore, other approaches with potential for continuous production were attempted.

The first apparatus designed and constructed was analogous in make-up and operation to a cupola. The reaction vessel consisted of an alumina tube with a hemispherical bottom. A hole was drilled in the bottom of the tube to allow molten reaction product to drain slowly into a collecting pan. Heating was accomplished using a circular Nichrome coil. The entire apparatus was encased in a transite cabinet. A thermocouple, touching the outer wall of the alumina, allowed accurate measurement of the temperature in the reaction zone. A well blended mixture of nickel oxide-cobalt oxide and sodium peroxide was poured slowly into the alumina tube, so that the reaction zone was filled. Some of the molten reaction product dripped from the tube but most was retained due to solidification blockage. The apparatus was cooled to room temperature and the material remaining inside was removed by hydrolysis. The material was washed to neutral with tap water, ball milled and loaded into a fiber metal plaque for testing. Maximum electrochemical utilization obtained for this material was 0.21 Ah/g at the C/3 drain rate.

Observation of the experiment indicated that several shortcomings were apparent in this method. Small cracks developed throughout the alumina tube surface, particularly at the junction of the reaction and cold zones. Also, significant degradation of the tube was observed in the reaction zone. It was estimated that a 5% weight loss was experienced in the reaction zone of the container.

Concurrent with the above experiments, several thick-walled nickel crucibles of 400 cm^3 volume were prepared as containers for both induction and resistance heating experiments. Five of these are currently available and can produce enough material in a single run for construction of sufficient nickel electrodes (20) for two full-size cells. Initial experiments using these crucibles were done using an induction heating apparatus. Several variables were explored including temperature rise rate, time at the reaction temperature and cooling sequence. An active material giving an electrochemical output of 0.25 Ah/g (small plate test) has been obtained. The temperature rise rate and cooling sequence did not seem to affect active material performance. Encouragingly, experiments using resistance heating, produce a material which is comparable in performance to that produced using induction heating.

The target performance of nickel electrodes in the iron-nickel battery is 0.14 Ah/g of finished plate at the C/3 discharge rate. In order to achieve this goal, both the active material utilization and plaque structure must be optimized. Electrochemical utilization of thermally prepared nickel active material in various sintered plaque structures has been determined in order to obtain the optimum porosity required for best finished electrode performance. Surveys were done using foam metal and cross-hatched fiber plaques with sinter porosities varying between 70 and 93 percent. Results for both plaque types surveyed indicate that substitution of active material for plaque fibers is self defeating.

Thermal nickel active material is loaded into the plaque structure using a roll pasting procedure. To insure uniform distribution and reproducible loadings for a given plaque thickness, the paste must remain as a single fluid phase during the rolling process. Deflocculant must be added to the aqueous paste to prevent curdling. Small plate tests indicate that the additive does not significantly affect the performance of the active material at these small concentrations.

A limited number of full size plates (345 cm^2) have been constructed and tested in order to meet the volumetric and gravimetric goals required for the finished cell. Outputs between 20 and 23 Ah have been achieved at the 3 hour discharge rate. These plates have shown stable performance over 50 cycles tested, before experiencing accidental cell voltage reversal due to a tester malfunction.

Thickness measurements with cycling time have been obtained for nickel electrodes made with thermally prepared active material. These results show about 10 percent swelling occurred, mostly during the first 50 test cycles. The results for full size plates confirm those found in small plate runs and indicate that the swelling problem usually found for different types of nickel electrodes can be controlled using this process.

2.1.2 Iron Electrode

Iron electrode development for the iron-nickel battery has centered on two specific technologies, a briquet process and a composite process. Both of these electrode types were developed at Westinghouse over the past few years and have shown a high degree of performance and reliability in actual battery systems. Specifically, the iron-nickel battery design requires an iron electrode 345 cm^2 in active area and ~1 mm thick having a cell performance of 26.5 Ah at the three hour discharge rate. The ultimate target is to obtain 0.27 Ah/g of total iron electrode, all on the upper voltage discharge plateau.

2.1.2.1 Iron Plaque Preparation

The current collector for the iron active material is a porous structure, presently formed from a combination of steel fibers and an expanded steel sheet. The single plaque is sintered, trimmed and a tab seam welded. Since the expanded metal at present is only 22.9 cm wide, plaques for single plates are being fabricated. An advantage of this composite grid structure is that the expanded metal appears to provide some stiffness in handling during the seam welding operation, during which the bus-tab is attached to the plaque.

2.1.2.2 Briquet-type Iron Electrode

The briquet approach is based on a process in which the active iron oxide power is reduced, die-pressed between expanded metal screens, and then sintered into a finished plate.

Full size briquet electrodes were tested in three plate cells versus nickel electrodes of the EPP type. They were of two different formulations, representing the state-of-the-art technology. The reduced rouge iron oxide powder used for pressing was either the fraction passed through a 60 mesh screen or through a 400 mesh screen. Both the fine and coarse fractions were mixed with pore former, prior to processing. Electrodes prepared, using the 400 mesh powder, also included an additional chemical treatment step. All of the briquet electrodes were sintered in hydrogen after pressing. Similar outputs for all of the briquet types during the first 50 cycles (20-22 Ah at the C/3 discharge rate) indicate that the particle size and chemistry changes used in processing did not significantly affect performance.

2.1.2.3 The Composite-type Iron Electrode

For the composite type iron electrode, the active material consists of rouge iron oxide (Fisher Scientific Company) and additives. This material is incorporated as an aqueous paste into a steel wool grid, containing a steel expanded metal center. As mentioned previously, the electrode is reduced prior to sizing. Full-size electrodes produced in this manner consistently produce 23-25 Ah outputs at the C/3 discharge rate.

Some differences are apparent in the peformance characteristics of these two types of electrodes, particularly at higher drain rates. Small plate (~6.5 cm^2) qualification runs on both types indicate that the briquet-type exhibits poorer performance at higher drain rates. The average voltage on discharge for a composite iron electrode is about 900 mv versus an Hg/HgO reference while the briquet average is about 70 mv lower. Again, in these small plate tests at the C rate, the composite type suffered a 20 mv loss in average discharge voltage while the briquet dropped nearly 120 mv. The excellent power characteristic demonstrated by the small composite electrode is attributed to the fiber metal-exmet combination conducting grid system which both carries the current and allows firm attachment of a connecting tab.

Research is now underway to replace the Fisher Red Iron Oxide in the composite process with a less expensive, mass produced iron oxide. Candidate materials were chosen on the basis of particle size and method of manufacture, relative to the Fisher material. Experiments with certain Columbian Carbon Company calcined red oxides yield results in small plate tests (~6.5 cm^2) which are comparable to the Fisher material, at approximately 1/3 the projected materials coat. Some problems in fabrication have been incurred in scale-up to full size but these problems are expected to be resolved in the near future.

All full size cells manufactured to date contain composite-type iron electrodes. Performance in full cells parallels almost exactly that which was found in small plate (6.5 cm^2) qualification runs and three plate test cells. The basic process steps in the production of the full size electrode are the loading ot the grid with iron oxide, the reduction of the oxide in-situ, and the pressing of the composite to the requisite thickness. The control over the amount and uniformity of the loading is based on the inherent expansive structure of the steel wool grid and upon the use of a suitable paste of the oxide. By means of hard rolls the loaded grid is then sized to 2.2 mm to remove the excess paste, so that the controlled volume of the retained paste is uniformly loaded within the voids of the grid.

Following the pasting step and prior to the reduction step, a drying operation is required to remove most or all of the paste water. With the water removed a rather open structure results whch offers easy access for the hydrogen gas during the in-situ reducing step.

A continuous belt hydrogen atmosphere furnace with a 20 foot long hot zone (BTU Engineering WP-25) is used for the reducing step.

The overall dimensions of the electrode are not affected by the reduction step. In pressing to the final thickness, the electrode with the requisite 68 g loading is readily pressed to 1 mm with no distortion to the other two dimensions.

2.1.3 Cell Components

Development work on cell components includes: cell and inter-cell connectors, separator(s), cell case and cover, and cell auxiliaries that deal with automatic water feeding and/or electrolyte recirculating systems.

2.1.3.1 Separator Development

The separator development work involves two tasks:

1) Selection of a suitable separator system that can be used in evaluating the initial prototype iron-nickel test cells.
2) Selection and evaluation of new separator materials.

Most of the effort to date has been concentrated on the first task with contacts with vendors, primarily Kimberly-Clark, with regard to task 2. A sample of permanently-wetted, non-woven polypropylene has been received from the RAI Corporation, but has not yet been tested.

The effort in task 1 has been directed toward initiating a quality control program on "as received" separator mateiral, with emphasis on electricl resistivity. To develop a data base, resistances were measured on materials purchased in the past for other battery programs, as well as for those purchased for this development contract.*

The tests performed were directed toward determining lot-to-lot variations, within-lot variations and resistivity levels of various materials. Results obtained to date are summarized in Table 2.1. These results indicate that the non-woven materials show reasonable within-lot variations but can have large lot-to-lot variations. These variations will be discussed wtih the vendor to determine actions to correct the problem. The microporous separator material (Celgard) has lot-to-lot and within-lot variations which are about equal. All the resistance obtained, however, are well under the manufacturers maximum specification of 15 milliohm-in^2.

2.1.3.2 Connector Development

The overall objective of this subtask is to develop a reliable connector that can be readily manufactured. The viability of new connector assembly technique(s) are being evaluated in terms of electrode resistance, reliability, manufacturability, and resistance to attack by caustic. Intercell connections and module terminal connection concepts are also being evaluated in the program.

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*The resistance tester used was of the type described in Falk & Salkind, <u>Alkaline Storage Batteries</u>, John Wiley and Son, 1969, pp. 257-262.

TABLE 2.1

SEPARATORS AND THEIR RESISTANCES

Material I.D. No.	Lot No.	Sample No.	Material Type	Average Resistance milliohm-in^2
ABSORBER MATERIALS:				
Kendall M1583	4382	1	Non-Woven Polypropylene	41
Kendall M1583	4382	2	Non-Woven Polypropylene	45
Kendall M1583	4382	3	Non-Woven Polypropylene	45
Kendall M1583	3840	1	Non-Woven Polypropylene	24
Kendall M1583	3840	2	Non-Woven Polypropylene	18
Kendall M1583	3840	3	Non-Woven Polypropylene	19
Kendall M1583	2754	1	Non-Woven Polypropylene	18
Kendall M1583	2754	2	Non-Woven Polypropylene	22
Kendall XM1253	0448	1	Non-Woven Polypropylene	19
Kendall XM1253	9365	1	Non-Woven Polypropylene	10
Kendall SM263.4	6745	1	Non-Woven Polypropylene	13
Pellon 2506K	9847	1	Non-Woven Nylon	6
Pellon 2506K	9847	2	Non-Woven Nylon	6
BARRIER MATERIALS:				
Celgard 1401	10814	1	Microporous Polypropylene	6
Celgard 1401	10814	2	Microporous Polypropylene	10
Celgard 1401	10815	1	Microporous Polypropylene	10
Celgard 1401	5768	1	Microporous Polypropylene	6

Several design concepts have been considered for connecting and terminating the plates in a single, free-standing cell. In the present cell design, a comb-type strap with a terminal exits from the top of the cell (see Figure 2.4). A grommet, located on the terminal inside the cell, provides a mechanical seal for the cell. A mold to form these grommets from rubber has been designed and fabricated for terminals of either steel or clad copper.

The steel terminal for the present test cell has approximatley seven times the cross-sectional area of a terminal made of copper for equivalent electrical resistance. Preliminary design calculations for the terminal indicated that a 0.95 cm diameter would be satisfactory (i.e., lower losses) for current carrying at the C rate (250A). The equivalent steel terminal diameter would be 2.2 cm. This component is, in fact, larger in diameter because of the flange required to support the grommet used to seal the cell.

Copper terminals and collector combs have been nickel plated and corrosion testing has been initiated in the standard lithiated, potassium hydroxide electrolyte, used in the cell. Some of these plated components had some pitting in the surface of the nickel coating. However, macro examination did not show evidence of exposed copper at the base of the pit. These samples, after exposure for 40 hours as a positive electrode in a cell at the gassing potential, showed no evidence of corrosion in any area of the terminal.

Electrical measurements have been made on the copper terminals and the steel terminals to determine their resistance. The steel terminals at the grommet section of 5.4 cm diameter has a resistance of 2.9×10^{-6} ohm/cm. This agrees with the calculated value for a steel having a resistivity of 10×10^{-6} ohm-cm. The copper terminal at 2.4 cm diameter is equivalent in resistance, 3.2×10^{-6} ohm/cm.

2.1.3.3 Cell Case and Cover

Development work in this area is twofold:

1) The design and construction of prototype size 3 plate cell containers for nickel and iron plate evaluations.

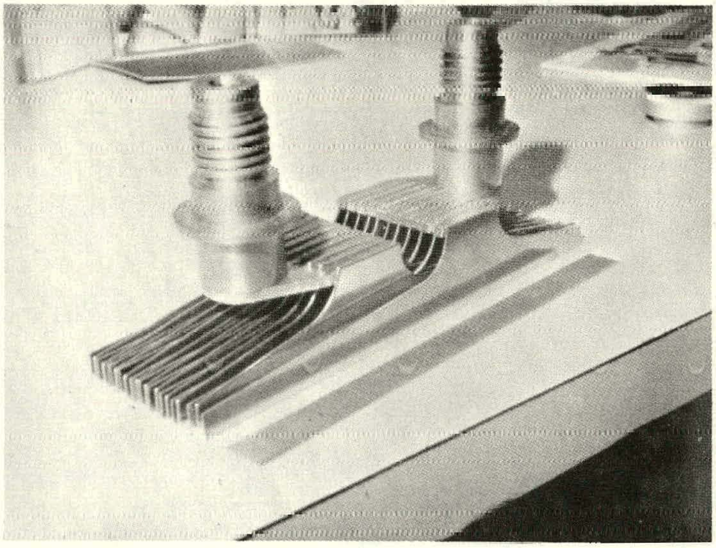

Fig. 2.4—Appearance of a full size iron-nickel cell post-comb assembly, attached to plate bus-tabs to show fabricated appearance

2) The design of prototype size iron-nickel cell containers and covers, the selection of vendors, and the procurement of the cell containers and covers.

The design concept for the 3-plate cell container has been completed. Three-plate cell test cases were designed and fabricated from plexiglas (as are present full-cell test cell cases). These have been used for all tests thus far.

Design of the final prototype, full-size single cell cases and covers has also been completed, with two case-to-cover sealing options; ultrasonic and induction versions. Based on present plans the induction adhesive bonding process will be used. The mold has been designed for the cell case and cover and the order has been placed. The case and cover will be of Noryl (polyphenylene oxide). We anticipate receipt of the first molded pieces in November.

2.1.3.4 Cell Auxiliaries

Development work on cell auxiliaries relate to electrolyte water makeup and, if needed, electrolyte recirculation. Emphasis will be on an automated, system capability.

The need for recirculation of electrolyte is being determined by cell thermal conditions, which are being established in some of the prototype size iron-nickel cells that are on test.

2.1.4 Cell Testing

The ultimate purpose of the testing program is two-fold:

1) Provide performance information for use by the manufacturer as feedback to determine operating characteristics, suggest modifications, and reinforce design.

2) Provide information of interest to a potential user, who has a need to know many performance, environmental and safety aspects of the system.

For Task 2, the System Development portion of the contract, testing concentrates on two areas. Prototype size iron and nickel plate tests are being performed to assist in evaluating design, materials, and process changes and determining their effect on improving performance. Also, full size iron-nickel cells are being tested, toward establishing the intermediate target goals (specified in Section 3.0) that must be met, prior to proceeding with the first battery build.

The testing is to provide, ultimately, information on the operational and performance characteristics in the following areas: 1) pre-test, 2) charging, 3) capacity, 4) power, 5) charge retention, 6) life and 7) environmental aspects.

2.1.4.1 Tester Facilities and Hardware

A preliminary test program plan was included in the Initial Iron-Nickel Battery Specification draft, submitted at the end of May. Only one minor change in a testing procedure has been made in that plan. Eleven experiments were included, the details of which will be submitted with the final battery specification. The titles of those experiments and the curves which will result are listed in Table 2.2.

The physical apparatus to perform these tests are either in existence (see Figures 2.5 and 2.6) or being set up.* Cells are being cycled on a continual basis and at maximum frequency. Modifications include: 1) replacement of power supplies, 2) control repairs to the tester, 3) rewiring, 4) recorder set-ups, 5) cell station rebuilding, 6) self-extinguishing material substitution, 7) installation of a safety spill detection system, 8) installation of an integrating system, 9) installation of an auto-watering system, 10) installation of a cell reversal protection system, and 11) meter replacements.

The last two items were recently added when a terminal voltage meter malfunctioned and caused a severe reversal of the 3 plate cells to occur at the beginning of August.

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*The auto-cycle system has been the major concern thus far, as it is the primary instrument in data collection.

Table 2.2 Preliminary Test Program Plan

Experiment #	Title	Plots	
1	Capacity vs. Ah input	i	Ah efficiency @ C/3 vs. input
	& Wh input	ii	Wh efficiency @ C/3 vs. input
2	Capacity vs. charge rate	1	Charge acceptance
3	Capacity vs. discharge rate	i	Ah efficiency vs. rate
		ii	Wh efficiency vs. rate
		iii	% rated Ah vs. rate
		iv	% rated Wh vs. rate
		v	discharge curves vs. rate
4	Internal Resistance vs. state	i	Ri vs. charge
	Internal Resistance vs. rates	ii	Ri vs. discharge
		iii	delivered kW vs. (discharge, rate)
		iv	potential vs. (rate, state
		v	power vs. (state)
5	Open Circuit Losses	i	Capacity vs. O.C. @ 0°C
		ii	Capacity vs. O.C. @ 25°C
		iii	Capacity vs. O.C. @ 50°C
6	Capacity vs. Ambient Temperature	i	Ah vs. T
		ii	Wh vs. T
7	Temperature Rise	i	°C/hr vs. I_C & state
		ii	°C/hr vs. I_D & state
8	Capacity vs. (Cycle Temperature, and Depth of Discharge	i	life @ 0°C vs. depth
		ii	life @ 25°C vs. depth
		iii	life @ 50°C vs. depth
		iv	capacity @ 25°C
9	Gassing vs. (state, rate)	i	C/3 charge
		ii	C/6 charge
		iii	C/3 discharge
		iv	composition
10	Electrolyte Displacement vs. state	i	C/3 charge
		ii	C/6 charge
		iii	C/3 discharge
11	Short Circuit Current - SCC	i	SCC vs. duration

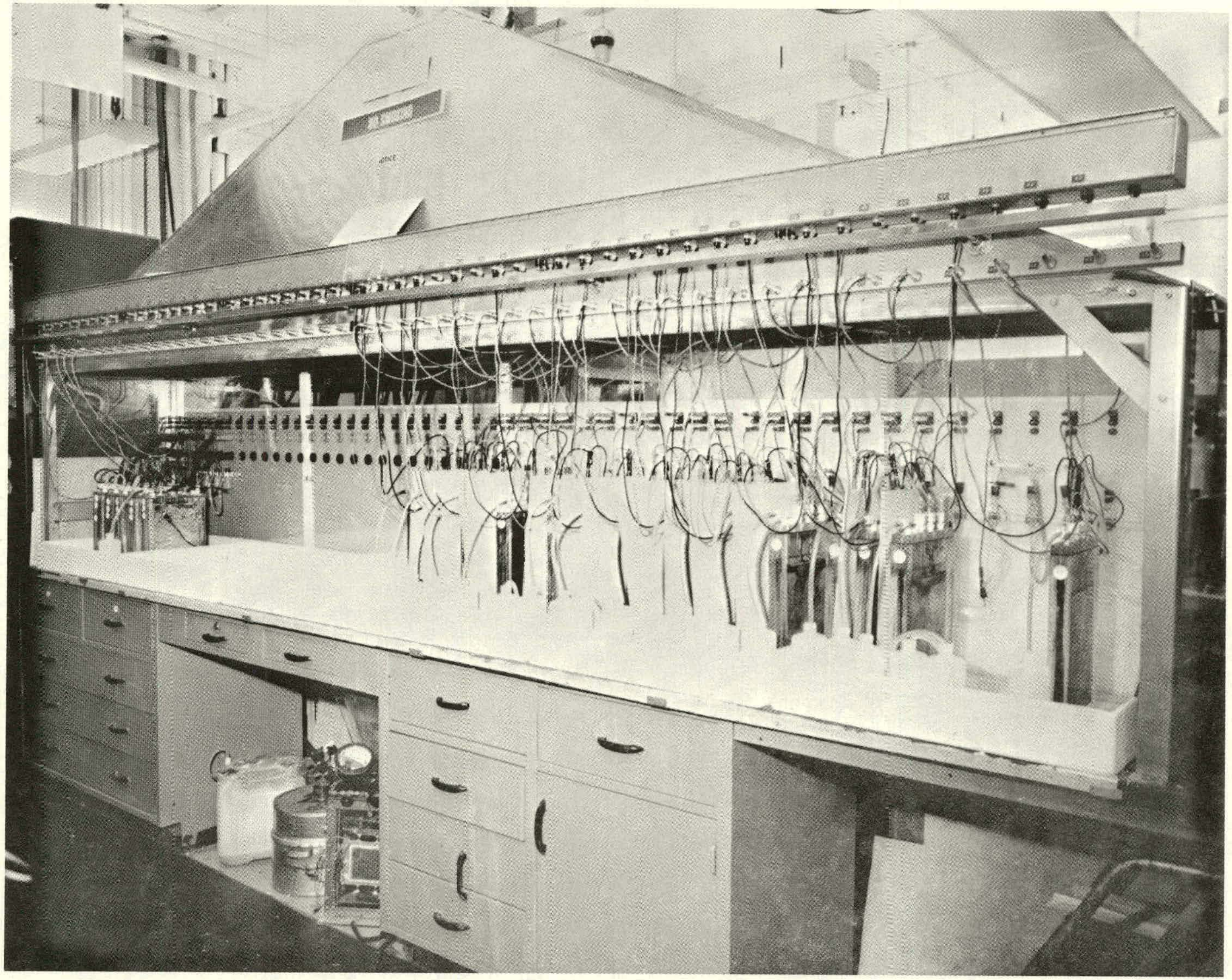


Fig. 2.5 — Ⓦ Auto-cycle tester, showing 3 plate and full size cell tests

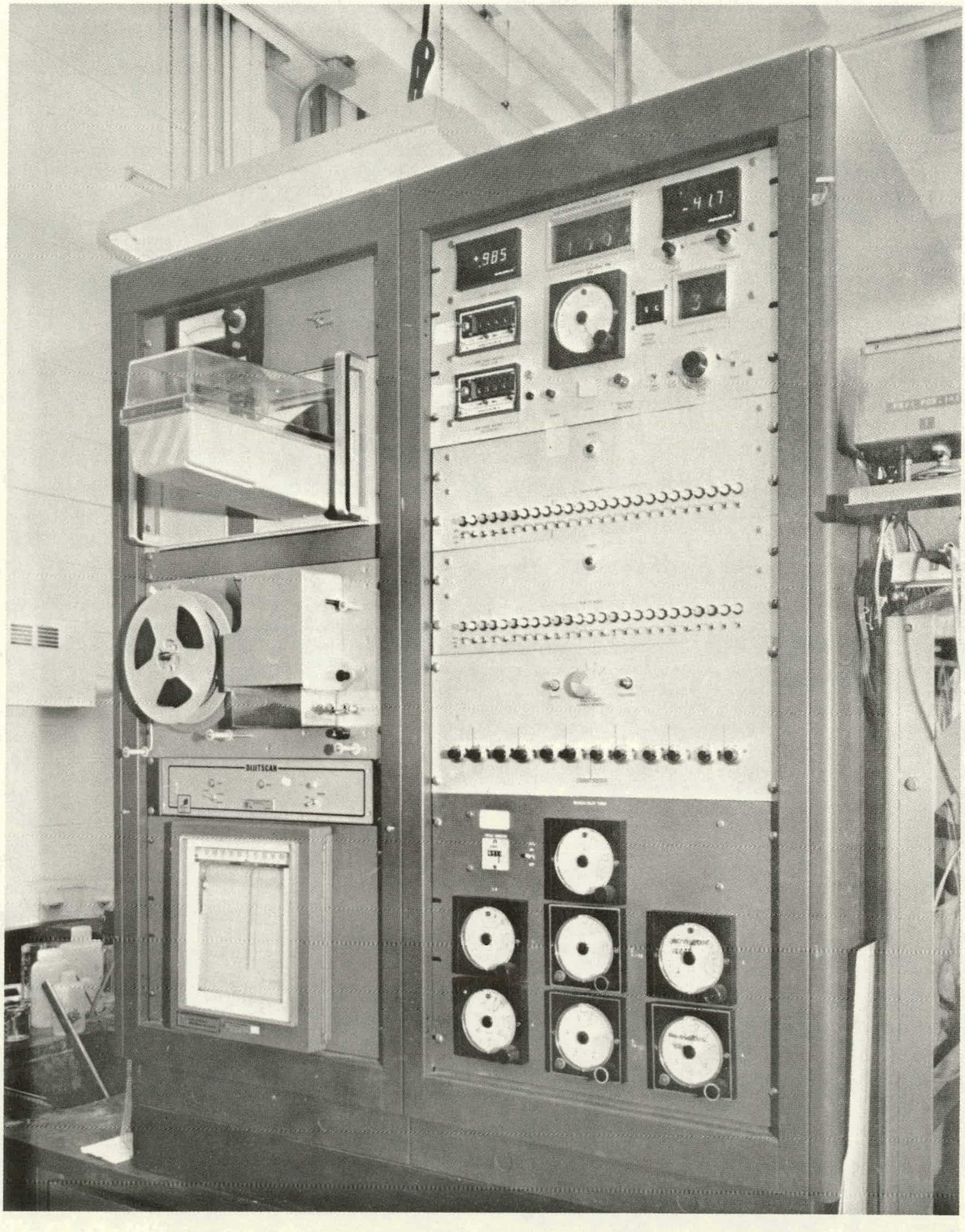


Fig. 2.6 — Ⓦ Auto-cycle tester, showing present control and data logging equipment

The temperature protection system, has been designed and necessary hardware is on hand. It is hoped that this can be installed at about the same time as the temperature baths. Power testing apparatus are already in existence and will likely require only slight modifications. Short circuit current analysis instruments are also in hand. Gassing and electrolyte displacement measurement equipment will require moderate fixturing. The gas analysis instrumentation is ready for use.

To date cell data reduction has occurred manually, but in the last few months, computer data collection and reduction software has been under modification. Two of three programs are complete and have been tested with sample data. Hardware for cycler-to-computer data transition has been identified by one source and a second is now making a study. A system may include computer interfacing, graphic display media, and a graphic terminal, or the possible use of a minicomputer. The primary objective here is to eliminate the paper tape stage of the data transition,and add CRT capability for improved accuracy and interpretation. The former hardware has been identified by one source. A second source is presently studying the latter. Until either can be implemented, paper tape data transition will allow the use of updated programs, as they become available.

2.1.5 Pilot Plant Operation

Planning is underway to prepare the iron-nickel battery pilot plant for the manufacture of the first prototype battery, commencing later this year.

3.0 TECHNICAL PERFORMANCE

The technical performance goals are shown in Table 2.3. The immediate goals specify attainment of 50 Wh/Kg and 80 Wh/l cell performance within C/2 to C/4 discharge rate. Final goals are 60 Wh/Kg and 100 Wh/l. To establish our status of development, all tests (3 plate and full-size cell) on the program are routinely performed at C/3 charge rate for 4 hours (33% excess coulombic charge) and at C/3 discharge rate, to 0.970 V cutoff on 3 plate cells and to 1.00 V cutoff on full-size cells.

Test results will be described that relate to plaque evaluation, iron and nickel plate results and full-size iron-nickel cell results.

Seven full size cells have been constructed to date, in containers of the proper volumetric constraints, as per our design specifications. However, the cells have containers made of plexiglas, with oversized terminals and are, consequently, overweight. This situation will prevail until the mold has been obtained (estimate-late September), so that actual cases and covers can be prepared and final design terminals for that cover have been machined. In the meantime, all cell gravimetric data is based on estimated cell weight, by taring out excess weight components and adding in final design components and adjusted electrolyte weight.

3.1 THREE-PLATE TEST CELLS

Three-plate test cells are used to evaluate both the iron and nickel electrode performances, to establish their state of development and to assist in the preparation of electrodes used in fabricating stackups for full-size prototype cell tests. Some of these results are summarized in Table 2.5.

3.1.1 EPP Nickel Tests

Tables 2.5 and 2.6 summarize 3-plate cell test results on EPP and thermal nickel electrodes. The best of the EPP electrodes have delivered

Table 2.3. Ⓦ Iron-Nickel Battery Technical Performance Goals and Present Status

	Characteristics		Contract Goals (Set Dec. 1977)	Present Demonstration*(Date August 1978)[e]	Contractor Forecast[d] (for Feb. 1979) Performance
1.	Battery Capacity (KW-hr) (100% rated)		25	21-25	20
2.	Battery Dimension (m H x m W x m L	m H m W m L	.28 .38 2.21	.28 .38 2.21	.28 .38 2.21
3.	Weight (kg)		417	411-445	417
4.	Volumetric Energy[a] (W-h/L)		100	89-106	80
5.	Specific Energy[a] (W-h/kg)		60	48-58	50
6.	Specific Power (W/kg)				
	Peak Battery - with 17% Voltage drop for 30 sec.		100	-	100
	Sustained @ C/3		20	-	20
7.	Duty Cycle				
	Charge (h)		4-8	C/3 for 4h	6
	Discharge (h)		2-4	3	3
8.	Lifetime				
	Deep Discharges[b]		2000	-	-
9.	Price/Energy[c] ($/KWh)		<60	-	-
10.	Energy Efficiency (%)		>60	-	>50

[a] C/3 rate discharge; 4-8 h charge

[b] 80% depth of discharge based on rated capacity

[c] price delivered to auto manufacturer for production ≈ 10000/y

[d] First prototype.

[e] Extrapolated from test available cell data.

TABLE 2.5

THREE-PLATE CELL TEST RESULTS ON IRON AND NICKEL ELECTRODES
(CHARGE, GENERALLY C/3 RATE FOR 4 HOURS; DISCHARGE, C/3 RATE TO .970 V ON CELL)

				CAPACITY VS. CYCLE														Utilization		Best (After cycle six) Data:			
																						Utilization	
PLATE	ACTIVE	ELECTRODE	THICKNESS	1	2	3	4	5	6	*-2	*-1	*	*	*+1	*+2	*+3	Latest Cycle	Latest Cycle	Last Active	Last Electrode	Best Capacity	Best Cycle	Best Active	Best Electrode
No.	g	g	mm	Ah	Ah	Ah	Ah	Ah	Ah	Ah	Ah	Ah	cycle#	Ah	Ah	Ah	Ah	#	Ah/g	Ah/g	Ah	#	Ah/g	Ah/g
2B	66.9	97.6	0.98	22.8	20.3	13.1	19.3	20.3	20.3	16.6	17.0	16.6	191	11.6	11.2	10.2	12.6	212	0.19	0.13	22.4	35	0.33	0.23
3B	67.4	98.5	0.99	21.3	23.1	22.8	22.6	22.6	22.5	16.6	17.1	16.9	187	13.7	14.2	13.4	16.4	208	0.24	0.17	23.8	15	0.35	0.24
39C	66.3	117.	0.99	21.6	22.0	20.7	19.9	23.0	21.5	23.1	24.0	23.5	175	21.0	20.6	20.3	21.2	196	0.32	0.18	25.6	52	0.39	0.22
106C	70.4	119	1.02	32.7	27.9	25.3	24.4	25.6	25.0	21.8	22.0	22.4	142	20.0	21.6	20.8	18.9	163	0.27	0.16	25.7	13	0.37	0.22
125C	74.8	127	1.02	27.6	22.9	22.3	21.6	21.4	21.9	20.8	21.6	21.7	95	20.7	21.4	19.7	19.5	116	0.26	0.15	24.5	17,22	0.33	0.19
59E			2.5	20.9	21.2	22.6	20.7	22.C	23.1	20.4	20.4	20.0	195	18.4	20.2	17.6	18.5	211**			23.9	68		
61E			2.3	14.4	19.2	22.5	23.1	22.4	23.1	17.9	17.9	18.0	173	14.9		13.2	13.4	194			23.1	7		
138E			3.2	21.2	21.4	21.5	20.1	18.9	20.1	20.3	21.0	21.0	130	14.4	13.8	11.4	15.6	151			22.0	100,101		
139E			3.1	20.9	21.1	20.7	19.3	18.5	19.4	19.6	19.7	19.8	130	12.9	12.2	10.6	13.4	151			21.6	47,64		
156E			3.4	21.1	22.4	22.5	20.7	19.1	20.4	22.9	23.2	23.6	130	14.9	19.4	15.5	18.7	151			24.3	99		
157E			3.7	24.5	25.0	25.2	23.9	21.6	24.0	23.1	23.2	23.1	130	15.3	17.9	14.3	16.4	151			24.4	17		
186E			2.6	18.5	19.5	19.2	17.6	17.5	18.5	20.3	20.1	20.3	130	16.0	14.2	12.4	16.7	151			20.9	101		
187E			2.9	22.7	23.1	23.1	22.0	20.8	22.5	19.4	19.7	19.7	130	14.5	13.9	12.5	14.7	151			22.7	8		
5T	125	211	2.3	8.9	12.0	14.1	16.9	17.9	20.9	20.4	21.1	20.9	48	14.0	14.8	12.7	13.7	69	0.11	0.06	23.6	19	0.19	0.11
6T	106	193	2.0	14.9	16.8	16.9	16.8	16.8	17.4	19.7	20.1	20.0	49	13.1	11.7	10.7	17.7	70	0.17	0.09	20.4	36	0.19	0.11
7T	119	194	2.1	9.0	8.7	6.5	8.3	8.3	7.7	6.8	7.1	7.1	13	10.4	11.0	10.3	8.3	34	0.07	0.04	11.0	15	0.09	0.06

NOTES: Target thicknesses - Nickel Electrode = 2.5 mm
Iron Electrode = 1.0 mm

B = briquet-type iron electrode
C = composite-type iron electrode
E = EPP nickel electrode
T = thermal nickel electrode

Immediately after *cycle , cells were charged normally,
discharged 5 hours,
charged normally,
discharged 34 hours in reverse polarity by power supply.
at cycle *+1, cells were charged C/2 for 5 hours,
discharged normally
at cycle *+2, charged C/2 for 4 hours,
discharged normally (C/3)
at cycle *+3, charged normally (C/3, 4 hours)

**Off test

TABLE 2.6

SPECIFIC NICKEL ELECTRODE TEST RESULTS

Cell	Description	Initial Capacity[c] Ah	Initial Capacity[c] Wh	Rates Disch.	Rates Charge	Initial Eff,[c] % Ah	Initial Eff,[c] % Wh	Life Characteristics Days[d]	Life Characteristics Cycles[d]	% Decline in[e] Capacity	% Decline in[e] Energy	% Decline in[e] Ah Eff.	% Decline in[e] Wh Eff.	Remarks
51E	lithiated	22.3		C/3	C/6	67			25	23				
59E		21.9			C/3	66			>225	9/20[f]				
138E		21.2				64			150	1/25				
139E		20.9				63			152	5/34				
156E	ph 5.0 3.5 mm	21.1				64			152	-12/11				(minus sign denotes increase)
157E		24.5				74			152	6/32				
186E	ph 2.0 2.5 mm	18.5				56			152	-10/10				
187E		22.7				68			152	13/38				
6T		17.6				53			>84	-14/-6				
9T		17.5				53			>5	N/A				
10T		20.6				62			>5	N/A				

NOTES:

a. Special feature, components, weight (kg) and volume (l)

b. C/3 discharge, 4 to 8 hour charge

c. Based on at least 5 consecutive cycles

d. "Greater than" symbols denote continuing operation

e. Percent decline from initial

f. Where two values occur, the second is ~20 cycles after a 35 hour reversal, the former is the cycle immediately preceding the cell reversal.

23.1 and 23.6 Ah at cycle 130.* However, these plates are over thickness, at 3.4 to 3.7 mm (target: 2.5 mm). 20 Ah plates have been obtained at up to 195* cycles at target thickness. The EPP plates do demonstrate stability. About 22 Ah plates are needed in an iron-nickel cell, displaying 1.15 V and weighing 5.0 kg to attain the initial goal of 50 Wh/kg in the specified cell volume. Recent improvements in the experimental line show capacity increasing to 24.0 Ah in formation discharge at a plate thickness of just under 2.5 mm.

3.1.2 Thermal Nickel Tests

A full-size electrode, 2.3 mm in thickness, made from an early batch of thermal active material, displayed 21 Ah capacity at nearly 50 test cycles. This plate, meeting the initial performance requirements for nickel electrodes, had shown stable performance since cycle 6. Similar stability in capacity is shown in an even thinner nickel electrode (2.0 mm and 20 Ah capacity).

3.1.3 Briquet Iron Tests

Testing of two briquet iron electrodes has been conducted up to ~190 cycles, with initial capacities of 20.3 to 22.5 Ah (Table 2.7). However, they decreased in capacity to 17 Ah during this testing interval.** Further effort toward optimization of the briquet iron electrode has been deferred, due to the encouraging results obtained to date in the composite-type of iron electrode, as well as due to the need for a suitable pressing die of the right design and geometry to fabricate briquet-type irons.

3.1.4 Composite Iron Tests

Composite iron electrodes have initially delivered 22.0 to 25.2 Ah (average) initially (Table 2.7), to 21.7 to 23.5 Ah to 100 to 175

---

*An auto cycle tester malfunction caused voltage reversal of the 3-plate cells at this cycle level and forced termination of these tests.

**An auto cycle tester malfunction caused voltage reversal of the 3-plate cells at this capacity level and forced termination of these tests.

TABLE 2.7

SPECIFIC IRON ELECTRODE TEST RESULTS

Cell Description[a]	Initial Capacity[c]				Initial Eff.[c] %		Life Characteristics						Remarks
									% Decline in[e]				
	Ah	Wh	Disch.	Charge	Ah	Wh	Days[d]	Cycles[d]	Capacity	Energy	Ah Eff.	Wh Eff.	
1B	22.5		C/3	C/3	68			86	16				
2B	20.3		↓	↓	61			213	18/37[f]				
3B	22.5		↓	↓	68			>214	25/29				
39C	23.5		↓	↓	71			>210	0/13				
40C	23.6		↓	↓	71			68	1				
54C	23.4		↓	↓	70			68	7				
62C	22.0		↓	C/6	67			17	8				
106C	25.2		↓	C/3	76			164	11/26				

NOTES:
- a. Special feature, components, weight (kg) and volume (l)
- b. C/3 discharge, 4 to 8 hour charge
- c. Based on at least 5 consecutive cycles
- d. "Greater than" symbols denote continuing operation
- e. Percent decline from initial
- f. Where two values occur, the second is ~20 cycles after a 35 hour reversal, the former in the cycle immediately preceding the cell reversal.

cycles* (see Table 2.5). Stability in capacity (0 to 11 percent degradation) has been noted in these samples (Table 2.7).

3.1.5 Summary of Three-Plate Test Results

With respect to individual electrode gravimetric performance goals, the composite iron electrodes are approaching target performance, (.40 Ah/g active and .27 Ah/g electrode), at .39 Ah/g active and .22 Ah/g electrode, based on best capacity. EPP plates are wet and cannot be accurately estimated in this regard. However, as projected results in full-size cells shown in Section 3.3, overall cell performance, using EPP nickel and composite iron electrodes in the specified cell volumetric constraint, are meeting and exceeding the initial goal of 50 Wh/kg at C/3 drain rate. Tables 2.6 and 2.7 show capacity variation for a number of 3-plate cell tests on iron and nickel electrodes. The better composite irons exhibit 0 to ~10 percent capacity loss in up to 200 cycles, before cell voltage reversal, due to the test malfunction occurrence. Briquet iron electrodes decreased 18 to 25 percent in ~200 test cycles.

Nickel EPP electrodes (see Table 2.6) indicated ~10 percent loss to ~10 percent gain over initial capacity in ~150 to 200 test cycles.

3.1.6 Effect of Prolonged Cell Voltage Reversal in Iron and Nickel Plate Performance

Figures 2.7 and 2.8 show the effect on capacity of the accidental 35-hour voltage reversal in the iron and nickel 3- plate, power supply driven test cells. With respect to the iron electrode, composite irons decreased 10 to 16 percent and briquet-type electrodes decreased 5 to 25 percent in capacity, when measured 20 cycles after the reversal. EPP nickel electrodes decayed 12 to 29 percent and a thermal nickel electrode decayed 37 percent over this same interval. Surprisingly, the plates (both iron and nickel) did not suffer irreparable damage under these extreme reversal conditions. This behavior suggests that the Westinghouse iron-nickel cells may be able to sustain deep discharge, as cells in a battery pack, without catastrophic effect to voltage and capacity of the battery in subsequent cycles.

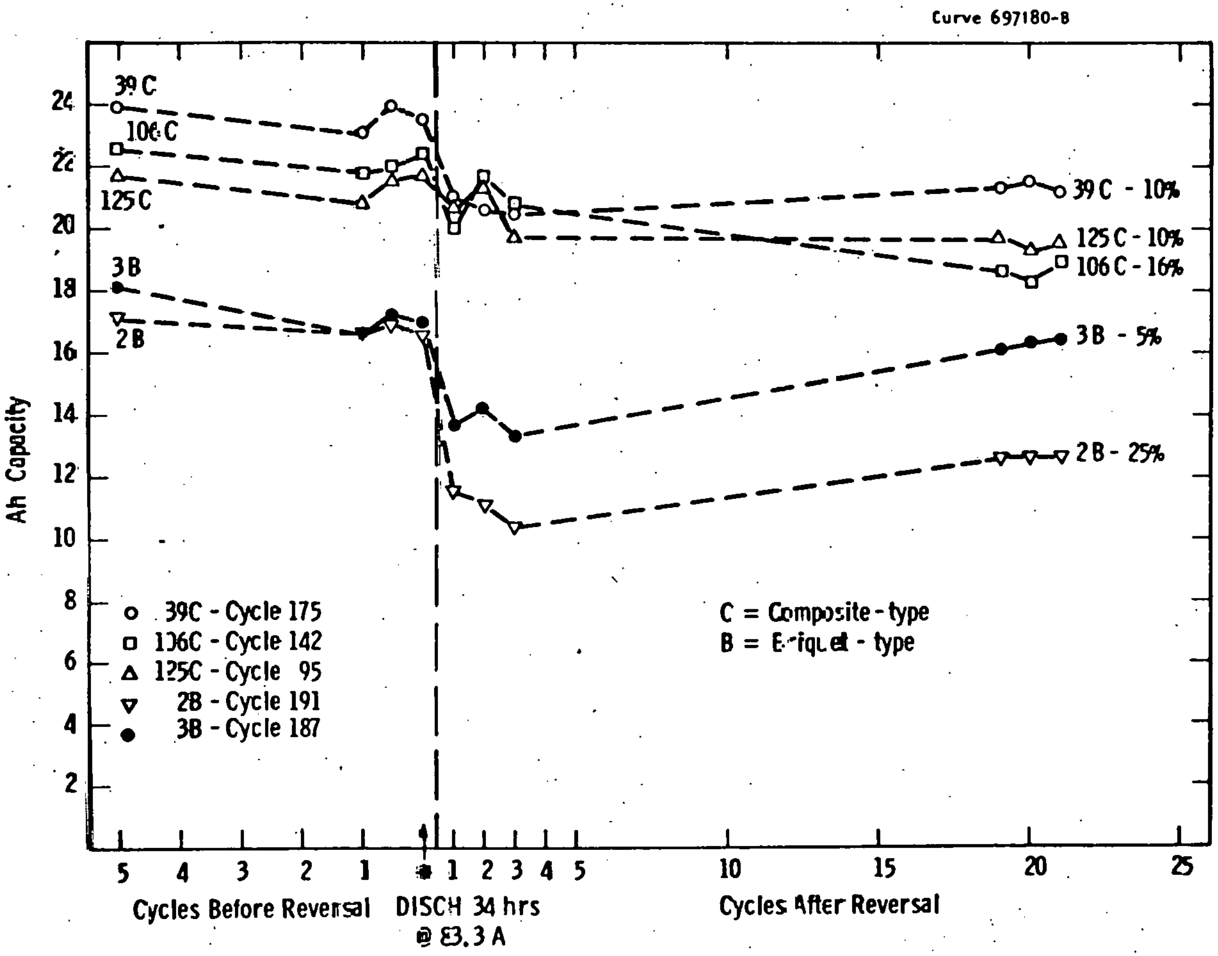


Fig. 2.7 – Iron electrode deep discharge effect

Curve 697179-B

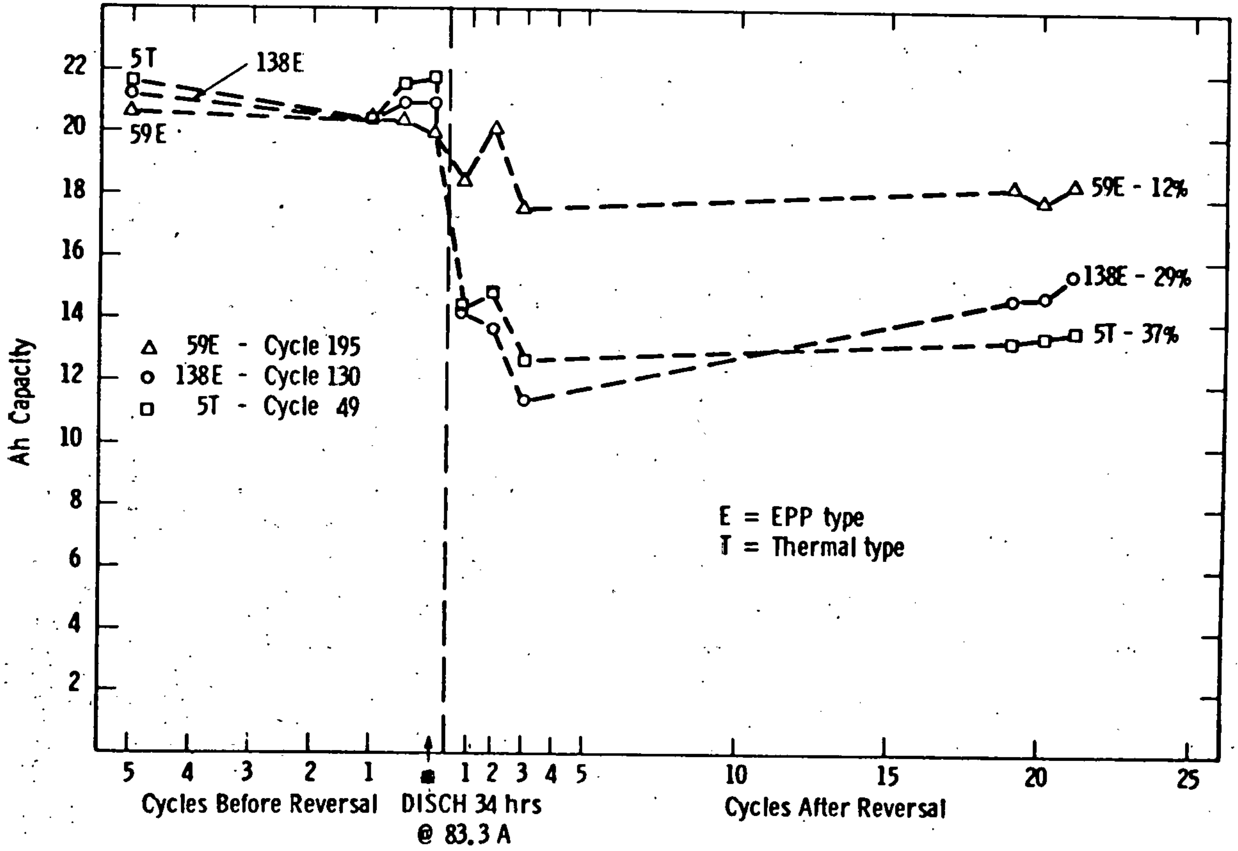


Fig. 2.8 – Nickel electrode deep discharge effect

3.2 FULL-SIZE CELL TEST RESULTS

Figure 2.9 shows the appearance of the full-size test cell used to evaluate Westinghouse non-nickel cells in the program. The cell contains, per the specification, 10 nickel plates and 11 iron plates in the target volume specified for the cell. The cell has provision for a plate reference electrode, bulk electrolyte temperature (thermometer or thermocouple) electrolyte maintenance and gas venting.

Tables 2.8 and 2.9 present the results of testing seven prototype cells to date.

Couloumbic efficiencies have not been optimized in the test results reported in these tables, since the cells were cycled by a standard procedure to obtain performance as a function of cycling.

The makeup and results obtained to date on these cells are now described. Some of the cells were fabricated to gain experience toward otpimizing cell stack-up assembly procedures and do not necessarily represent attempts at maximizing gravimetric performance. Unless noted, all cells were constructed using polypropylene absorber/Celgard barrier combination separators.

<u>Cell 001CE.</u> Here, composite iron electrodes and EPP nickel electrodes (from the mini-line) were used to assemble the stack-up. The nickel had delivered 23 Ah in formation discharge. However, they were 3.4 mm average thickness and had to be sized to 2.5 mm, prior to cell assembly. The cell delivered 219 Ah, initially (to 1.0 V cutoff) and 211 Ah at cycle 2. At cycle 12, the cell delivered 251 Wh at C/3 based on an average discharge voltage of 1.16 V. The projected weight for this cell was 5.1 kg (based on all allowances for the final case, cover, terminals and electrolyte) so that the cell delivered an estimated 49 Wh/kg at C/3 during cycle 12.

Nickel electrode swelling apparently caused a drop-off in capacity with further cycling, to 150 Ah. However, when the stack-up constraint was removed and the cell was further tested,

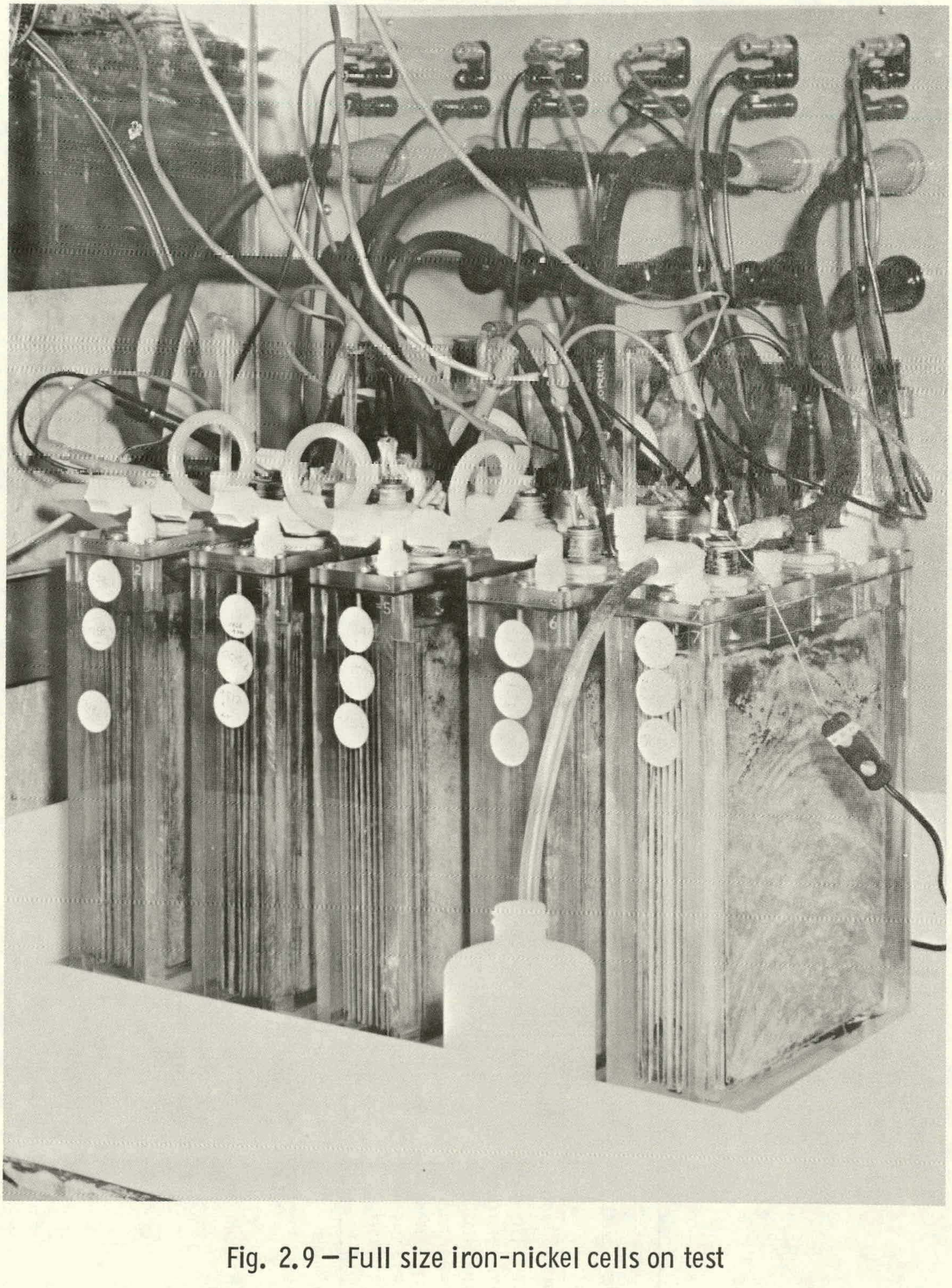


Fig. 2.9 — Full size iron-nickel cells on test

TABLE 2.8

FULL-SIZE WESTINGHOUSE IRON-NICKEL CELL TEST RESULTS
(CHARGE, GENERALLY C/3 FOR 4 HOURS: DISCHARGE, C/3 TO 1.0 V)

CELL DATA				CAPACITY VS. CYCLE												BEST (AFTER 6 CYCLE DATA)						
														Utilization		Utilization						
Cell	Active	Electrode	Thickness	1	2	3	4	5	6	N-2	N-1	N	N Cycle	Last Active	Last Electrode	Capacity	Cycle	Best Active	Best Electrode	Projected Cell Weight	Best Wh	Best Wh/kg (cycle)
No.	g	g	mm	Ah	Ah	Ah	Ah	Ah	Ah	Ah	Ah	Ah	#	Ah/g	Ah/g	Ah	No.	Ah/g	Ah/g			
	Fe/Ni*	Fe/Ni*	Fe/Ni																			
001CE	835/-	1338/2532		219	211	185	180	190	198	194	194	191	24			216	11	/	/	5.0	251	50.0
002CE	798/1094	1351/1930	1.0/2.2	175	165	159	154	154	157	204	202	199	28	0.25/0.18	0.15/0.10	219	20	0.27/0.20	0.16/0.11	4.9	254	52.0(20)
003CE	712/1345	1252/2213	0.99/2.2	222	233	243	212	230	243	225	220	218	17	0.31/0.16	0.17/0.10	247	7	0.35/0.18	0.20/0.11	5.0	284	57.0(7)
004CE	763/1479	1323/2340	1.0/2.5	207	217	217	210	204	206	208	210	207	12	0.27/0.14	0.16/0.09	210	8,11	0.28/0.14	0.16/0.09	5.2	247	48.0(8,11)
005CE	751/1271	1302/2131	1.04/2.5	202	209	210	217	215	218		216	214	8	0.28/0.17	0.16/0.10	216	7	0.29/0.17	0.17/0.10	5.0	280	50.0(7)
006CE	792/1127	1349/1895	1.04/2.7	230	250	251	245						4	0.31/0.22	0.18/0.13	-	-	-	-	5.1	299	58.0(6)
007CE	718/	1273/	/	215	231+											-	-	-	-	5.3	-	-

NOTES: *Wet weight

+Cell internal resistance = 0.67 mΩ @ 45 minutes into discharge this run

TABLE 2.9

SPECIFIC FULL-SIZE IRON-NICKEL CELL TEST RESULTS
(CHARGE, GENERALLY C/3 FOR 4 HOURS: DISCHARGE, C/3 TO 1.00 V CUTOFF)

Cell Description[a]	Est. Wt. (kg)	Initial Capacity[c]		Rates, h		Initial Eff.[c] %	Life Characteristics					Remarks
								% Decline in[e]				
		Ah	Wh	Disch.	Charge	Ah	Latest Cycles[d]	Capacity	Energy	Ah Eff.	Wh Eff.	
001CE	5.0	197	227	C/3	C/3	( )	24	3				Off Test
002CE	4.9	160	184	↓	↓	64	>34	-32				Minus sign = increase
003CE	5.0	248	280	↓	↓	74	>23	8				
004CE	5.2	211	243	↓	↓	63	>18	-3				
005CE	5.0	211	243	↓	↓	62	>14	2				
006CE	5.1	252	290	↓	↓	75	>10	-3				
007CE	5.3	214	257	↓	↓	64	>7	N/A				

NOTES:
- a. Special feature, components, weight (kg) and volume (1)
- b. C/3 discharge, 4 to 8 hour charge
- c. Based on at least 5 consecutive cycles
- d. "Greater than" symbols denote continuing operation
- e. Percent decline from initial
- f. Generally, based on average voltage of 1.15 (Cells 001-006CE), 1.20 (Cell 007CE)

the capacity improved to 194 Ah. Testing was terminated at 24 test cycles in this cell.

Post-mortem evalaution of the nickel plates from cell 001CE confirmed that they had swelled, as predicted, measuring an average increase of 0.9 mm for the 10 plates.

Cell 002CE. This cell was constructed from nickels of 2.16 mm thickness and composite-type iron electrodes. The nickel delivered 20 Ah in formation discharge. Initially, the cell delivered 175 Ah capacity and this increased to 219 Ah at the C/3 rate at cycle 20. With an average voltage of 1.16 V, the cell delivered 254 Wh on this cycle. With a projected cell weight of 4.9 kg, 002CE has demonstrated 52 Wh/kg at C/3 (50 Wh/kg, target for 2/79). Cell 002CE is still on test.

Cell 003CE. This cell was constructed, using EPP nickel plates that delivered 24 Ah in formation discharge. The nickel plates averaged 2.46 mm in thickness. The cell has composite-type iron electrodes. Initially, the cell delivered 243 Ah at cycle 3. At cycle 7, the cell delivered 247 Ah for 284 Wh at 5.0 Kg, a projected 57 Wh/kg at C/3, which exceeds the 2/79 target goal of 50 Wh/kg on that cycle. Testing of this cycle is continuing, with 224 Ah at cycle 19.

Cell 004CE. This cell was constructed using EPP nickel pltaes that delivered 23 Ah in formation discharge. The nickel plates averaged 2.60 mm in thickness. The cell has composite-type iron electrodes. The cell, still on test, has delivered 217 Ah during cycle 3 at C/3. This corresponds for that cycle, to 249 Wh at 5.2 Kg, or 48 Wh/kg for this cell. The cell is delivering 218 Ah at cycle 14 and testing is continuing.

Cell 005CE. This cell was fabricated with EPP nickel plates that delivered 22.5 Ah on formation discharge. The nickel plates averaged 2.50 mm in thickness. The cell, having composite iron electrodes, delivered 210 Ah at C/3 on its third test cycle. This corresponds to 242 Wh at 5.0 Kg, or 48 Wh/kg for this cell, which is still on test. The cell is at 229 Ah at cycle 10 and testing is continuing.

Cell 006CE. This cell was constructed using EPP nickels that delivered 22.5 Ah on formation discharge at 2.6 mm in thickness. At cycle 3 the cell delivered 251 Ah, corresponding to 289 Wh at 5.1 Kg or 57 Wh/kg for this cell. At cycle 13 the cell is delivering 232 Ah with testing continuing.

Cell 007CE. This cell was constructed using EPP nickel that delivered 24.0 Ah on formation discharge at 2.8 mm in thickness. The cell was constructed using a low resistance nylon separator material to determine a lower limit for the cell resistance. The cell delivered 231 Ah on its second cycle, 266 Wh at 5.3 Kg, or 50 Wh/kg with testing continuing.

3.2.1 Summary of Present Full-Size Cell States

Initial results have indicated the feasibility of attaining 48 to 58 Wh/kg in full-size iron-nickel cells, using EPP nickel and composite iron electrodes. Cyclic life testing is continuing on these cells. Life will be highly dependent on minimizing growth of the EPP nickel electrode on cycling. Present EPP plates are being prepared at target finished thickness (2.5 mm) and this factor should help enhance cyclic life.

The separator seems to be the major contributor to the total cell resistance and work on improving the separator system continues. With cell resistance reduced to the 0.6 to 0.8 milliohm level, as in the cell containing Pellon nylon, high-power characteristics will be attainable.

Table 2.10 summarizes the "best data" obtained to date on a given cycle on the full size test cells, with respect to projected Wh/Kg (due to not having final molded case/cover and terminals). Best projected Wh/Kg for the seven cells range from 48 to 58 Wh/Kg at the C/3 discharge rate to 1.0 V cell cutoff. These best data show promise of meeting the final contract goal of 60 Wh/Kg on the final cell.

Table 2.10

Summary Of Best Gravimetric Energy Density Results Obtained To Date On Full-Size Prototype Cells (at C/3 discharge rate)

Cell	Best Ah	Cycle	Wh	Best Projected Wh/Kg
001CE	219	12	251	49
002CE	219	20	254	52
003CE	247	7	284	57
004CE	218	14	247	48
005CE	229	7	248	50
006CE	259	6	299	58
007CE	231	2	266	50

Figures 2.10 and 2.11 show end of charge (C/3 for 4h) and discharge voltage characteristics of cells 002CE (Cycle 12) and 003CE (Cycle 7). These cells contained EPP nickel electrodes and composite-type iron electrodes. In Figure 2.10 cell 002CE delivered 202 Ah (232 Wh) at an average voltage of 1.15 V. Here the iron electrodes (24 Ah capacity plates) maintained a stable upper plateau (decrease of 80 mv in the discharge). In Figure 2.11 cell 003CE delivered 247 Ah (284 Wh) at an average voltage of 1.14 V. Because the iron electrodes were only ~24 Ah/plate in capacity and the two end iron plates in the stackup are not completely utilized, the iron capacity on the upper plateau was being taxed in this discharge, as evidenced by the dip in the iron plate voltage in its discharge curve. This dramatizes the necessity of improving and attaining the iron plate capacity to the 26.5 Ah minimum design limit in future iron nickel cells of ~250 Ah capacity.

Curve 697164-A

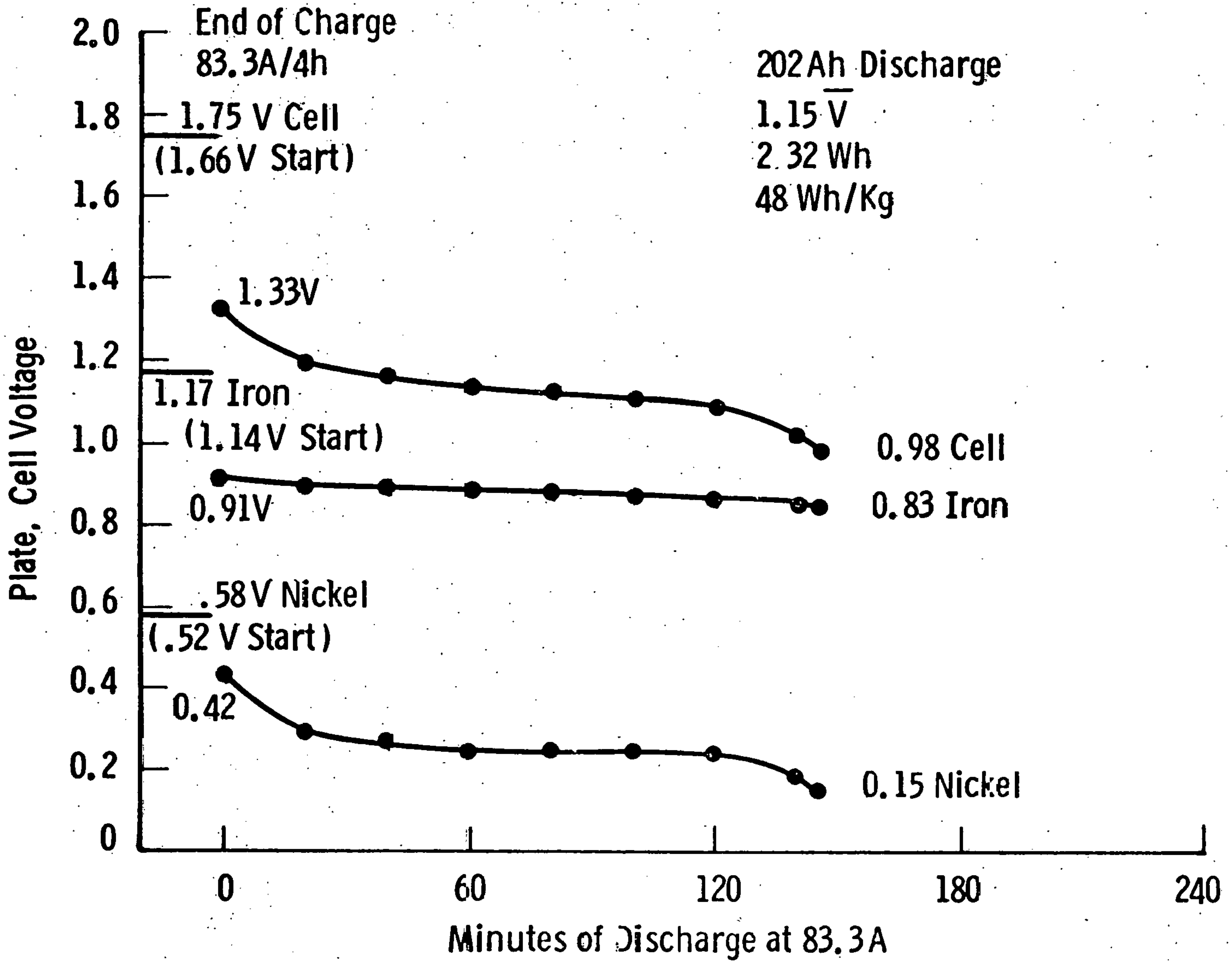


Fig. 2.10 — Discharge characteristics of Cell 002CE, Cycle 12

Curve 697163-A

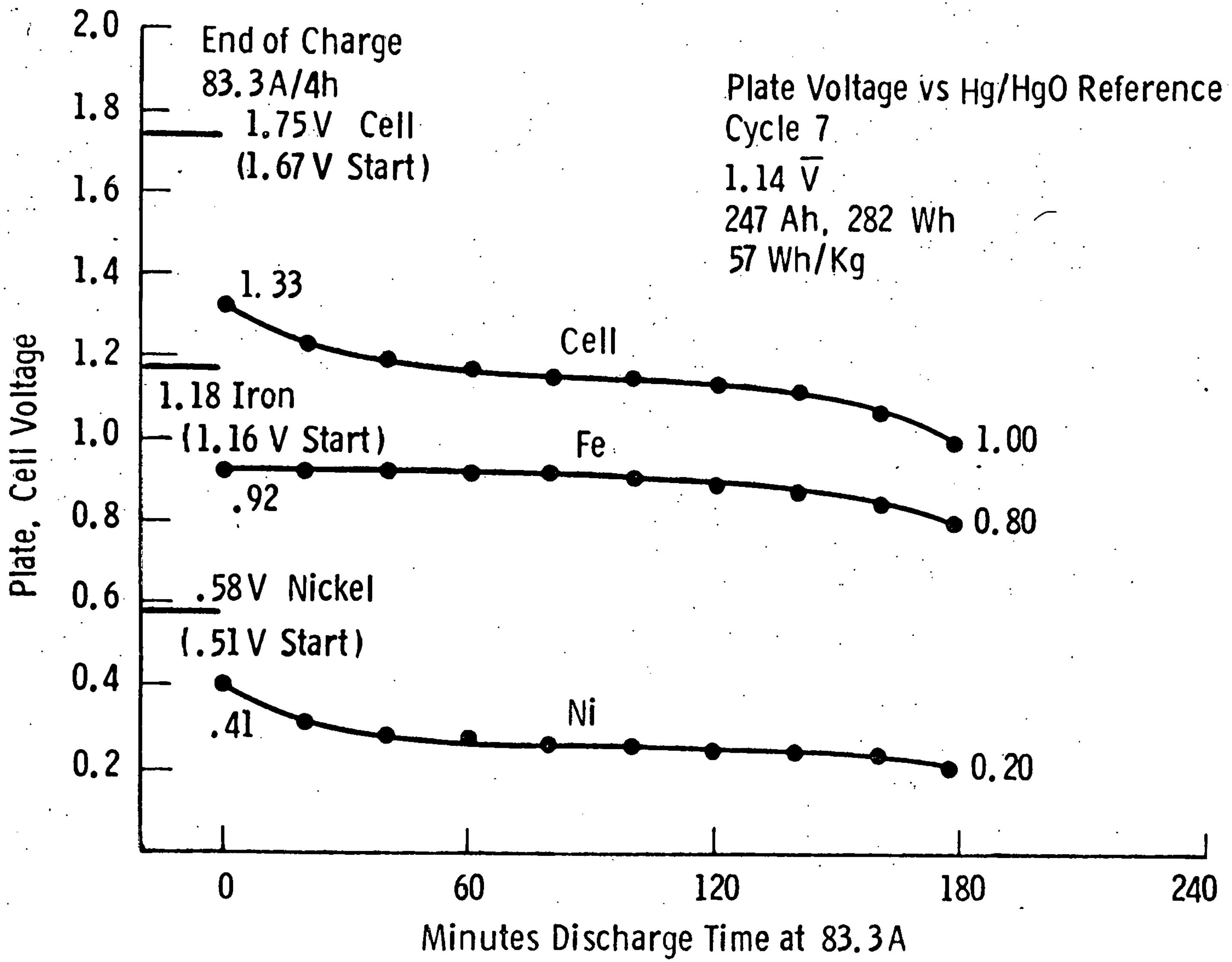


Fig. 2.11 – Discharge characteristics of Cell 003CE, Cycle 7

4.0 MILESTONE PLAN AND MANAGEMENT REPORT

The Milestone Plan and Management Report is shown in Figure 5.1. No significant variances have occurred during the last fiscal year.

FORM DOE 535
(10/77)

U.S. DEPARTMENT OF ENERGY
MILESTONE PLAN AND MANAGEMENT REPORT

Fig. 4.1

PAGE 1 OF 2

FORM APPROVED OMB NO.

1. Contract Identification: Research, Development and Demonstration of a Nickel Iron Battery for Electric Vehicle Prop.

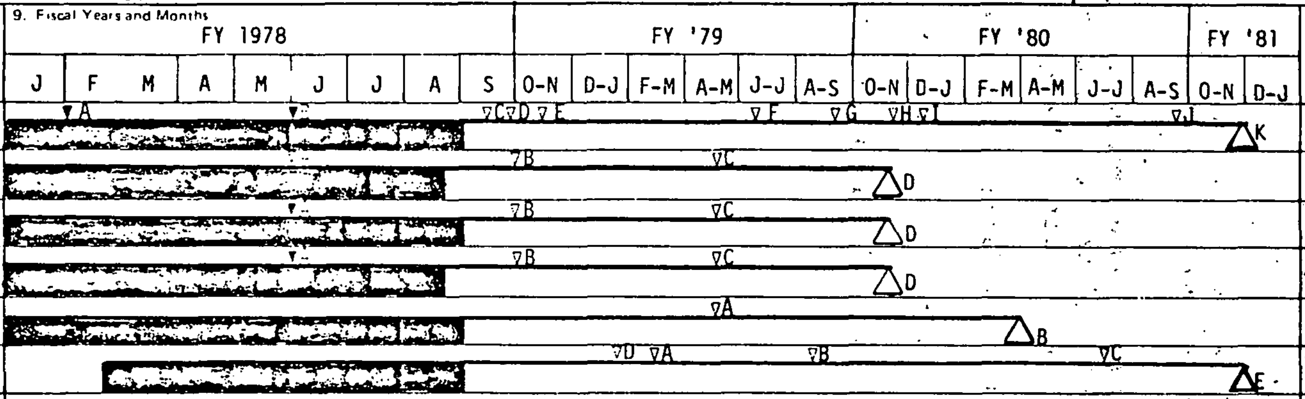
2. Reporting Period: Aug. 1, 1978 through Aug. 31, 1978

3. Contract Number: 31-109-38-4141

4. Contractor (name, address)
Westinghouse Electric Corporation
Research and Development Center
1310 Beulah Road
Pittsburgh, Pennsylvania 15235

5. Contract Start Date: 1 - December - 77

6. Contract Completion Date: 30 - November - 80



7. Identification Number	8. Reporting Category (e.g., contract line item or work breakdown structure element)	10. Percent Complete
3.1	Program Management	24
2.1	Nickel Electrode Dev.	28
2.2	Iron Electrode Dev.	30
2.3	Cell Hardware/Auxiliaries	28
2.4	Cell Testing	30
2.5	Pilot Plant Operations	51

11. Remarks: Program Management is also responsible for: a) Recycling Study, b) Environmental Impact Study, c) Safety Analysis Reports to be issued at each design review.

12. Signature of Contractor's Project Manager and Date: R. Rosey R. Rosey

13. Signature of Government Technical Representative and Date: R. Elliott

5.0 REFERENCES

1. C. C. Hardman, U.S. Patent 3,600,227, Method of Impregnating Flexible Metallic Battery Plaques, August 17, 1971.

2. E. Hausler, "Electrochemical Impregnation of Porous Sintered Nickel Grids according to the Kandler Procss," proceedings of the Power Sources Symposium, Brighton, England, September 1966, pp. 287-295.

3. J. F. Jackovitz and E. A. Pantier, U.S. Patent 3,941,614, March 2, 1976.

4. J. F. Jackovitz and E. A. Pantier, U.S. Patent 4,016,091, April 5, 1977.

5. J. F. Jackovitz and E. A. Pantier, U.S. Patent 4,029,132, June 14, 1977.

6.0 ACKNOWLEDGEMENTS

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Fiber Grid Structures	F. J. Sabados
Nickel Plate EPP Process	J. H. Dugan and J. F. Buchholz
Thermal Nickel Plate	S. L. Karaffa and E. A. Pantier
Test Cell Assembly	T. M. Valko
Testing Apparatus and Testing	L. W. Martz
Iron Electrodes	S. L. Karaffa and E. A. Pantier

Also, L. A. Doggrell assisted in the test apparatus setup and evaluation of separators considered an used to date in this program.

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