

POSITIVE ELECTRODE FABRICATION FOR

BI-POLAR NI-H<sub>2</sub> CELLS\*

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\* Portions of this paper are based on work performed at Eagle-Picher Industries, Inc. under contract to NASA Lewis Research Center.

## INTRODUCTION

Bi-Polar nickel hydrogen design studies (ref. 1) have indicated a potential improvement in energy density and specific energy if positive electrodes could be manufactured in configurations of twice or more of the present "normal" thickness. Such electrodes would have to achieve similar active material loading, utilization, and cycle life to conventional 0.8mm I.P.U. nickel hydrogen electrodes. Eagle-Picher's experience with D.O.E. near term nickel iron batteries (ref. 2) indicated that 2.0mm or greater thickness electrodes could be manufactured by the wet slurry process (ref. 3), loaded by electrochemical impregnation processes, and achieve useful capacities and cycle lives.

Both the dry sinter and wet slurry plaque processes are presently producing 0.8mm thin positive electrodes for nickel hydrogen flight programs. The slurry process is known to be capable of producing thick electrodes, but the dry sinter process used in many space applications had not yet demonstrated thick electrode capability.

The EPI/USAF electrochemical impregnation process (ref. 4) is utilized for flight quality thin electrodes (ref. 5) and was thought to be adaptable to impregnation of thicker electrodes. The reported high utilization, and limited swelling with electrochemical impregnation processes were seen as desirable qualities to achieve for thick electrodes. In addition a large data base incorporating formation, electrochemical characterization tests, accelerated high rate tests, and cell tests, exists for electrochemically loaded thin electrodes, that offers a good comparison to the thick electrode data generated in this test.

## ELECTRODE PRODUCTION

### FLOW CHART (fig. 1)

Nickel powder and wire plus binders and solvents for the slurry process are combined and sintered at high temperatures. Subsequently the nickel plaque is characterized for bend strength, porosity, thickness, and weight. Impregnation lots of forty plaque are selected through a weight sorting technique and process tabs are attached. The electrochemical impregnation system processes three lots at a time and individual lots may be independently controlled in terms of time, current density, etc. Following a short rinse in D.I. H<sub>2</sub>O the plaque undergo a five and one half cycle formation procedure followed by scrubbing, rinsing, drying, and weight pickup calculation. Plaque lots are returned to the formation tanks for twenty-one cycles of electrochemical characterization testing which continues

the active material formation and allows a plaque capacity determination. Following a second scrubbing, rinsing, and drying operation random plaques are selected to undergo an accelerated, high rate (10C) stress test consisting of two hundred cycles followed by five capacity measurement cycles.

#### SINTER PRODUCTION

Sintering process data is shown in figure 2. The dry sinter process utilizing I.N.C.O. 287 powder produced 1.03 mm thickness, 84.4% porosity and bend strength of 521.6 P.S.I. All properties are well within the tolerances set for this program. Plaque size is 11 x 14.5 in, and 150 plaque were produced for this program. The wet slurry process which incorporates I.N.C.O. 255 powder produced 1.52 mm thickness, 84.4% porosity, and 847 P.S.I. bend strength. Slurry plaque size is 11.25 x 12 in. and 160 plaque were manufactured.

#### ELECTROCHEMICAL IMPREGNATION

The EPI/USAF impregnation process (fig. 3) utilizes a mixed solvent consisting of 45% ethanol and balance water. Metal salt content is analyzed by Atomic Absorption prior to each run and adjusted to the range of 1.6 to 1.8 molar  $\text{Ni}(\text{NO}_3)_2$  and 0.12 to 0.18 molar  $\text{Co}(\text{NO}_3)_2$ . pH is continuously monitored and held between 2.8 and 3.2 by the automatic addition of dilute nitric acid. Impregnation bath temperature is maintained between 70° and 80°C, near the solution boiling point. Current, voltage, and time of impregnation are microprocessor controlled functions. Three different current regimes and variable times between 140 and 320 minutes were employed to achieve a target loading level of 1.60 to 1.70 grams per cubic centimeter of plaque void. Plaque are cathodically protected during all phases of the impregnation procedure to assure minimal corrosion of the nickel sinter. Immediately following impregnation plaques are rinsed in D.I. water to remove bulk nitrates.

#### FORMATION

The formation procedure (fig. 4) employs five and one half cycles of charge/discharge starting with discharge. Formations are carried out in 20% KOH electrolyte against nickel sheet counterelectrodes. Current densities are calculated on the basis of surface area at 0.45 amps/in<sup>2</sup> through cycle 4, 0.10 amps/in<sup>2</sup> for cycle 5, and 0.07 amps/in<sup>2</sup> for the last half cycle. Currents were run for the larger size 1.03mm plaque which gave the higher rates shown for the smaller 1.52mm slurry plaque. Time of each half cycle is twenty minutes. Plaque are subsequently rinsed and scrubbed, dried under nitrogen, and weighed.

## ELECTROCHEMICAL CHARACTERIZATION TEST

E.C.T. (fig 5) is again carried out in 20% KOH with nickel counterelectrodes. The test consists of twenty complete formation cycles at the nominal capacity rate. Charge is 120% of capacity and the plaques are discharged to -0.2 volts vs. a Hg/HgO reference electrode. The final charge is sixteen hours at a C/10 rate followed by discharge to -1.0 volts vs. Hg/HgO. The C rate for E.C.T. was a calculated average from the formation loading data for the three lots subjected to this test. Following E.C.T. plaques are rinsed, scrubbed, and dried. A final thickness and weight for each plaque are also taken. Capacities for each lot are calculated based on the time of the final discharge.

## IMPREGNATION RESULTS

The results of six different impregnated lots are shown in table 1. Data is presented on lot sinter characteristics followed by results after either formation, E.C.T., or both. Impregnation efficiency has been calculated in terms of ampere hours loaded divided by ampere hours input to the impregnation tanks.

$$\frac{\text{AH loaded}}{\text{AH Impreg. Input}} \times 100 = \text{Loading Efficiency}$$

Lot nos. 7023, 7024, and 7027 were loaded with the same current/voltage regime with impregnation time varied to achieve different loading levels or compensate for increased void volumes. Lot nos. 7025 and 7026 were loaded at reduced current densities, but at a similar number of total ampere hours to the above lots. Lot no. 7028 was loaded with an Eagle-Picher proprietary current/voltage regime with the same total ampere hour input as lot no. 7026. All loading levels were 1.60g/cm<sup>3</sup> void or greater. Thickness change after impregnation, formation, and E.C.T. was calculated on the basis of nine points per plaque averages. Weight loss during E.C.T. is shown for the three lots that went through this procedure.

## STRESS TEST

Two plaques per lot were selected at random for the EPI/HAC accelerated stress test. From each plaque two test electrodes were taken as well as samples for chemical analysis and active material distribution testing. Stress test electrodes were sized on the basis of their parent

plaque loading to give a theoretical capacity of 1.70 ampere hours per electrode. The stress test (fig. 6) incorporates a two cycle hot formation step to prepare electrodes for the test. Hot formation is carried out at 70°C in 20% KOH at the 5C rate. After formation, test electrodes are characterized for weight, thickness, and visual appearance. The actual stress test takes place in flooded, excess, 31% KOH against nickel counterelectrodes. An initial electrode capacity cycle is run consisting of 5C charge for 12 minutes followed by C/2 charge for 1 hour or 150% charge. Discharge is at the capacity rate to -1.0 volts vs. nickel sheet. The 200 stress cycles are 10C rate for 12 minutes of charge or 200% charge, and discharge at 10C for 8 minutes with the reverse voltage clamped to -1.0 volts vs. Ni. by individual cell bypass diodes. After stress, electrodes are subjected to five capacity cycles identical to the initial capacity above. When stressed electrodes are removed from the test fixture, they are examined for visual appearance then scrubbed, rinsed, and dried. Dried electrodes are characterized for weight loss, swelling, and other anomalies.

## STRESS TEST RESULTS

### ELECTRODE BASIS

Table 2 gives the test results for the twenty-four individual electrodes from six lots. Prestress and post stress capacities are indicated as well as individual weight and thickness changes. Electrode capacity increase during stress is given as a percentage.

### LOT BASIS

In table 3 stress tests results have been compiled as lot averages. Utilization has been calculated using 1.70 ampere hours as the theoretical capacity.

## DISCUSSION

Individual plaque lots of the same type exhibited remarkably uniform sinter characteristics, indeed all lots are quite similar in terms of porosity, and differ mainly in void volume and thickness. These plaque similarities accommodate relatively straightforward comparison of impregnation results. Lower current density impregnation appears to increase void volume loading in both types of plaque, while the Eagle-Picher proprietary current/voltage regime gave the highest loading results. Thickness change through impregnation, formation, and E.C.T. is quite small and with plaque visual appearance and weight change is indicative of little or no surface loading

or plating of active material. Impregnation efficiencies are all quite similar, but interestingly are maintained at the higher void volume loadings.

Stress test results indicate low capacities prior to test especially on the thicker more highly loaded electrodes. The three dry sinter lots seem to show some benefit from the extended E.C.T. formation that two of them received, however, this correlation is not clear for the slurry plaque lots. Post stress utilization for all dry sinter lots is uniform and slightly high for that capacity regime, slurry lots demonstrated dramatic capacity improvement through stress although their utilization is still below expected values. Thickness increase follows a general trend upward with increased loadings and in conjunction with respective void loadings may indicate some support for the contentions of McHenry (ref 6) concerning practical limits of loading.

#### CONTINUING INVESTIGATIONS

Eagle-Picher and Hughes Research Laboratories are pursuing thick electrode development and testing under the sponsorship of NASA Lewis Research Center. 0.86mm thick dry sinter plaque will serve as a baseline for this program which will look at the feasibility of producing 2.0mm thick dry sinter plaque with and without a supporting wire grid. Plaque characterization and electrochemical impregnation experimentation will take place as well as stress testing, electrode chemical tests, and cell testing. Reports of this work will be presented as it progresses.

#### SUMMARY

We have demonstrated a capability for producing 1.0mm dry sinter and 1.5mm slurry sinter structures on production processes and equipment. The EPI/USAF electrochemical impregnation process proved capable of loading thick electrodes to 1.60 g/cm<sup>3</sup> void or higher. Sample electrodes incurred 200 high rate stress cycles without significant structural or performance degradation. Formation regimes developed for thin plaque are not optimal for thick plaque and further investigation into electrode capacity build-up would seem warranted. Thick electrode technology development is continuing under other programs and shows promise of fulfilling the projected energy density and specific energy improvements.

## REFERENCES

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Table 1  
Impregnation/Formation Results

Lot Number	Original Thickness	Porosity	Void Volume	Plaque Loading	Void Loading	Thickness Change	E.C.T. Wt. Loss	Impregnation Efficiency
7023	0.0402 in.	84.2%	85.2 cm <sup>3</sup>	1.38.3 g	1.62 g/cm <sup>3</sup>	0.0005 in.	---	30.7%
7024	0.0404 in.	84.1%	85.4 cm <sup>3</sup>	138.3 g	1.62 g/cm <sup>3</sup>	---	- 1.59	28.7%
7025	0.0405 in.	83.9%	85.5 cm <sup>3</sup>	147.4 g	1.72 g/cm <sup>3</sup>	0.0009 in.	+ 0.65	29.8%
7026	0.0582 in.	84.1%	105.6 cm <sup>3</sup>	187.5 g	1.77 g/cm <sup>3</sup>	---	- 4.08	31.1%
7027	0.0588 in.	83.9%	106.4 cm <sup>3</sup>	170.7 g	1.60 g/cm <sup>3</sup>	0.0015 in.	---	29.1%
7028	0.0597 in.	83.9%	108.0 cm <sup>3</sup>	195.6 g	1.81 g/cm <sup>3</sup>	0.0027 in.	---	32.2%



Table 2  
Stress Test Results: Electrode Basis

Lot/Electrode #	Pre-Stress Capacity	Weight Change	Thickness Change	Post-Stress Capacity	Capacity Increase
7023-36 A	1.42 AH	-0.11 g	+0.0011 in.	2.04 AH	144%
7023-18 A	1.19 AH	-0.086 g	+0.0017 in.	1.87 AH	157%
7023-36 B	1.36 AH	-0.277 g	+0.0021 in.	1.87 AH	138%
7023-18 B	1.36 AH	-0.099 g	+0.0021 in.	1.885 AH	139%
7024-66 B	1.56 AH	-0.107 g	+0.0002 in.	1.93 AH	124%
7024-66 A	1.50 AH	-0.069 g	+0.0003 in.	1.81 AH	121%
7024-49 B	1.62 AH	-0.067 g	+0.0004 in.	2.07 AH	128%
7024-49 A	1.53 AH	-0.072 g	+0.0002 in.	1.96 AH	128%
7025-113 A	1.50 AH	-0.031 g	+0.0016 in.	1.64 AH	109%
7025-93 A	1.50 AH	-0.070 g	+0.0004 in.	1.96 AH	131%
7025-113 B	1.56 AH	-0.101 g	+0.0010 in.	1.93 AH	124%
7025-93 B	1.45 AH	+0.011 g	+0.0010 in.	1.93 AH	133%
7026-15 B	1.16 AH	+0.031 g	+0.0029 in.	1.615 AH	139%
7026-34 B	0.96 AH	-0.117 g	+0.0034 in.	1.47 AH	153%
7026-15 A	0.79 AH	-0.042 g	+0.0037 in.	1.605 AH	203%
7026-34 A	0.65 AH	-0.188 g	+0.0047 in.	1.655 AH	255%
7027-44 A	0.99 AH	-0.216 g	+0.0025 in.	1.59 AH	161%
7027-44 B	0.99 AH	-0.137 g	+0.0018 in.	1.64 AH	166%
7027-68 B	0.62 AH	-0.418 g	+0.0078 in.	1.345 AH	217%
7027-68 A	1.02 AH	-0.181 g	+0.0046 in.	1.81 AH	177%
7028-86 A	0.96 AH	-0.402 g	+0.0097 in.	1.575 AH	164%
7028-114 B	0.57 AH	-0.360 g	+0.0045 in.	1.47 AH	258%
7028-114 A	0.71 AH	-0.393 g	+0.0072 in.	1.67 AH	235%
7028-86 B	0.57 AH	-0.375 g	+0.0060 in.	1.30 AH	228%

Table 3  
Stress Test Results: Lot Basis

Lot Number	Wt. Change	Thickness Change	Pre-Stress Capacity	Pre-Stress Utilization	Post-Stress Capacity	Post-Stress Utilization	Capacity Increase
7023	0.143 g	0.0018 in.	133 AH	78%	1.92 AH	113%	123%
7024	0.079 g	0.0003 in.	1.55	91%	1.94 AH	114%	129%
7025	0.048 g	0.0010 in.	1.50	88%	1.87 AH	111%	125%
7026	0.079 g	0.0037 in.	0.89	52%	1.59 AH	93%	178%
7027	0.238 g	0.0042 in.	0.91	53%	1.60 AH	94%	177%
7028	0.383 g	0.0069 in.	0.70	41%	1.50 AH	88%	214%

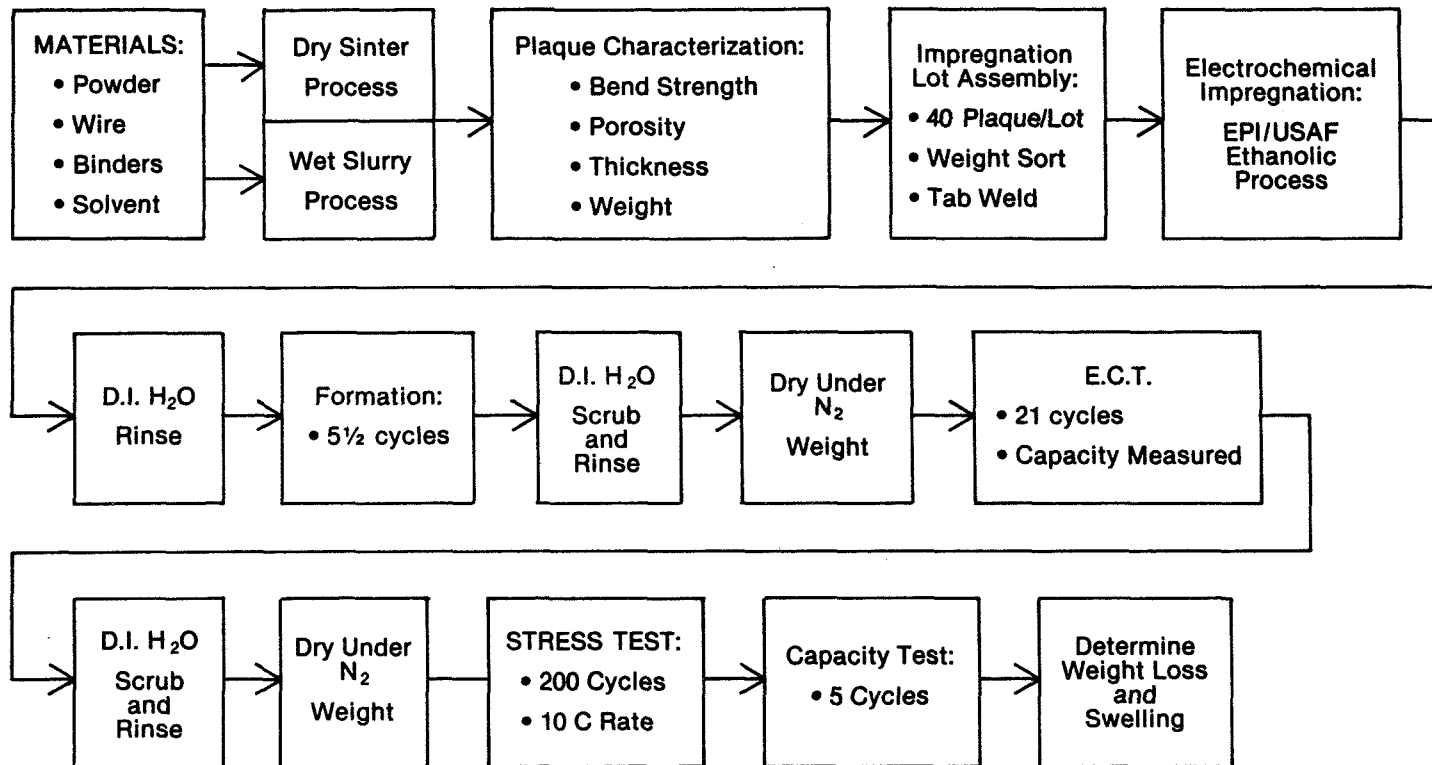


Figure 1. Electrode production flow chart.

**Dry Sinter Process**

**I.N.C.O. 287 Powder**

**Thickness: .04 ± .002 In.**

**Actual .0406 In.**

**Sinter Porosity: Nominal 82-87%**

**Actual Porosity: 84.41%**

**Bend Strength: 521.6 P.S.I.**

**Plaque Size: 11 × 14.5 In.**

**Substrate: Nickel Wire**

**Lot Size: 150 Ea.**

**Wet Slurry Process**

**I.N.C.O. 255 Powder**

**Thickness: .060 ± .003 In.**

**Actual .0619 In.**

**Sinter Porosity: Nominal 82-87%**

**Actual Porosity: 84.35%**

**Bend Strength: 847 P.S.I.**

**Plaque Size: 11.25 × 12 In.**

**Substrate: Nickel Wire**

**Lot Size: 160 Ea.**

Figure 2. Sinter production.

- Concentration: 1.6 to 1.8 M  $\text{Ni}(\text{NO}_3)_2$ , 0.12 to 0.18 M  $\text{Co}(\text{NO}_3)_2$
- Solvent: Ethanol 45% Balance  $\text{H}_2\text{O}$
- pH: 2.8 to 3.2
- Temperature: 70° to 80° C
- Current Density: 3 different regimes
- Target Loading Level: 1.60 to 1.70 g/cm<sup>3</sup> void
- Time: Variable 140 to 310 minutes

Figure 3. E.P.I./USAF electrochemical impregnation.

- 5½ cycles beginning with discharge • Nickel Counterelectrodes
- 20% KOH

Cycle #	-/+	Amps/in. <sup>2</sup> .040 Plaque	Rate .040 Plaque	Amps/in. <sup>2</sup> .060 Plaque	Rate .060 Plaque	Time: Min.
1	-	0.45	1.8 C	0.53	1.35 C	20
	+	0.45	1.8 C	0.53	1.35 C	20
2	-	0.45	1.8 C	0.53	1.35 C	20
	+	0.45	1.8 C	0.53	1.35 C	20
3	-	0.45	1.8 C	0.53	1.35 C	20
	+	0.45	1.8 C	0.53	1.35 C	20
4	-	0.45	1.8 C	0.53	1.35 C	20
	+	0.45	1.8 C	0.53	1.35 C	20
5	-	0.10	C/2.5	0.12	C/3.5	20
	+	0.10	C/2.5	0.12	C/3.5	20
6	-	0.07	C/3.6	0.08	C/4.8	20

Figure 4. Formation procedure.

- 20 cycles at C Rate:  
120% Charge Discharge to  $-0.2\text{V}$  vs Hg/HgO
- 16 Hr. Charge at C/10 rate
- Discharge at C/2 rate to  $-1.0\text{V}$  vs Hg/HgO: Capacity Calculated
- 20% KOH
- D.I. H<sub>2</sub>O Rinse and Scrub
- Final Weight and Thickness

Figure 5. Electrochemical characterization test.

- Hot Formation: 5C Rate, 70°C, 20% KOH, 2 Cycles
- Pre-Stress: Weight, Thickness, and Visual Appearance Characterization
- Initial Capacity: Charge 5C for 12 min. C/2 for 1 hr.  
Discharge C rate to – 1.0V vs Ni sheet
- 200 Cycles: Charge 10C for 12 min.  
Discharge 10C for 8 min. Diode Clamped to – 1.0V
- Ending Capacity: 5 Cycles Same as Initial Capacity
- Visual Appearance
- Scrub, Rinse, and Dry
- Weight Loss and Swelling

Figure 6. E.P.I./H.A.C. electrode stress test.



- Thick Electrodes offer Improved Energy Density and Specific Energy
- Thick Electrode Technology well established by E.P.I. for NI-FE Traction Batteries
- Both Dry Sinter and Wet Slurry Sintered Plaque processes Producing Flight Quality Thin Electrodes
- Automated, Ethanolic, Consumable Anode, Electrochemical Impregnation Process should be Adaptable to Thick Plaque Loading
- Formation, Electrochemical Characterization Test, and Stress Test offer Comparison to Large Data Base of Thin Electrodes

Figure 7. E.P.I./NASA-LeRC bi-polar positive electrodes.

- 0.034 In. Thick Dry Sinter Plaque with wire
- 0.080 In. Thick Dry Sinter Plaque with wire
- 0.080 In. Thick Dry Sinter Plaque without wire
- Plaque Characterization: Weight, Thickness, Porosity, Bend Strength, Pore Size
- Electrochemical Impregnation: Various Regimes to achieve  $1.60 \pm .05 \text{ g/cm}^3$  void
- Formation/E.C.T.
- Stress Test
- Electrode Chemical Tests
- Cell Testing

Figure 8. E.P.I./Hughes Research/NASA-LeRC Electrode Program.

- **Demonstrated Capability of Producing 0.040 in. Dry Sinter, and 0.060 in. Slurry Plaque.**
- **EPI/USAF Impregnation Facility Flexible enough to load thick plaques to at least the 1.60 to 1.70 g/cm<sup>3</sup> void level.**
- **Sample Electrodes Subjected to Severe High Rate Test without Significant Degradation.**
- **Thick Electrode Capacity Buildup Through Formation and Stress Cycles Should be Investigated Further**
- **Thick Electrode Technology Undergoing Further Development with The Assistance of NASA, and Hughes Research Laboratories.**

Figure 9. Summary.

- Q. Hendee, Telesat Canada: Were these with dry powder or was it the slurry process?
- A. Edgar, Eagle Picher Company: We used both. Most of the work has been done really with dry powder plaque although some of the later data you saw on the last sheet were with slurry process. Principally, I think for the aerospace type electrodes we would be looking to dry sooner at the moment.
- Q. Hendee, Telesat Canada: Okay, any schedules?
- A. Edgar, Eagle Picher Company: As I said we are following up the production equipment essentially right now for full-scale type production and then we are going to go into an evaluation phase on those electrodes and we are hoping to talk more about it this year and talk you guys into it a little bit.
- Q. Hendee, Telesat Canada: Well when would you come out and say hey, Ed I've got something new for you?
- A. Edgar, Eagle Picher Company: We are hoping for this year Ed. Really.
- Q. Lim, Hughes Research Lab: You said the voltage against the reference electrode there is a kind of indication for the end of the loading. Is the loading after the voltage against reference an indication of surface loading?
- A. Edgar, Eagle Picher Company: Yes, it seems typically to be surface loading if you stop the impregnation at the point of where you reach about - .65 volts you have a pretty good surface plaque that is fairly uniform appearance and if you continue beyond that point even under the same regime you get a considerable amount of thickening on the surface you get a very dark deposit of cadmium.
- Q. Lim, Hughes Research Lab: The second question is the weight loss during the initial cycling is that related to a cadmium solubility in the KOH. If so, it should be related to the volume of KOH you used if you are using those conditions.
- A. Edgar, Eagle Picher Company: I think the weight loss is principally cadmium that is loosely adhered to the surface of these plaques either loaded on the surface or in pores that are very close to the surface that the electrodes have typically been in a large excess of KOH and subsequent cycling of course is done in smaller amounts but I'm not sure that I really follow your question about the solubility of KOH.

- Q. Maurer, AT&T Bell Labs: Have you measured the percent utilization or have you done a chemical analysis of the plates? One of the problems with comparing weight gains in several different processes is the possibility of a difference in nickel corrosion in various cases so you get a lot of nickel hydroxide precipitated you will get an artificially high weight gain.
- A. Edgar, Eagle Picher Company: Right.
- Q. Maurer, AT&T Bell Labs: One of the problems I was worried about is in the current reversal technique? We would be making a plaque more susceptible corrosion.
- A. Edgar, Eagle Picher Company: Yeah we are quite aware of that problem and I agree with you. We have done some chemical analysis of the electrodes that hasn't been shown here. We intend to talk about that in the future as well as the utilization. We've experienced utilizations that reported in the literature by yourselves at Bell Labs and Dr. Pickett. The corrosion problem we considered early on to be a significant problem to worry about especially with the current reversal techniques. So we followed that in a couple of ways. One is by the traditional chemical analysis of the plates and utilization testing which indicated in fact that the sintered materials did corrode quite a bit and we got some loaded with nickel hydroxide and that showed up in the chemical analysis. You can also see it building up in your impregnation solution which is a real clear cut way of looking at it. That's one of the big driving reasons we went to the dry sinter plack and we experienced a significant reduction in corrosion by doing that and we intend to continue with that especially since we are using the current reversal techniques as you said.
- Q. Ritterman, Comsat: You showed a viewgraph of impregnation time versus voltage against the counter reference at three different current densities and if memory serves me properly you had a significant increase in the impregnation time and evidently in the loading level when you went from .2 to .16. Did you do that for a whole bunch of plaques or is that data for one plaque?
- A. Edgar, Eagle Picher Company: Those were data points for single runs but they are indicative of numerous runs that we made and essentially what we are doing there Paul is monitoring the impregnation with the potential and turning it off as the potential rose above a certain level.
- Q. Ritterman, Comsat: It looks like you would get maybe two or three times as much impregnation going to .16. I gather you went below .16 in current densities.

- A. Edgar, Eagle Picher Company: Yes.
- Q. Ritterman, Comsat: Did you continue to get this increasing impregnation time or was there some sort of limit on the lower current density?
- A. Edgar, Eagle Picher Company: Yeah, we did seem to encounter that you could in fact run for tremendously long periods of time without reaching the higher potentials but you didn't seem to get the benefit in terms of added loading.