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A STUDY OF DEGRADATION OF PLATES FOR NICKEL-CADMIUM SPACECRAFT CELLS

Interim Report

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ABSTRACT

The relative merits of coining and not coining of sintered nickel-oxide and cadmium plates was investigated. A survey of the industry including cell manufactures and users was made and results summarized. Sample plate materials from most commercial cell suppliers were obtained and characterized for properties that may correlate with the tendency toward physical disintegration during handling and over long periods of time in the cell. Special test methods were developed to obtain comparative data in a short time.

A wide range of physical properties and coining thicknesses was observed, resulting in a range of responses. The stronger, less brittle materials resisted loss of sinter better than weaker materials whether or not coined. Coining improved handling and resistance to electrochemical cycling in all materials tested. An apparent exception was found to be due to improper coining of a tapered edge.

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1. INTRODUCTION

1.1 BACKGROUND

The current state of the art of sealed spacecraft nickel-cadmium cell manufacturing and quality control technology permits cells to be made that are essentially free from many of the defects and life-limiting characteristics that plagued earlier cells. As a result, the ultimate life of modern cells is becoming more and more limited only by the life of the electrodes in the cells. Electrodes can fail by a variety of mechanisms — some subtle and difficult to detect, and others more simple and obvious. An example of the former is migration and passivation of active material within the pore structure of the plates. An example of the latter is detachment of pieces of sinter followed by shorting between positive and negative electrodes.

The present study is concerned with all forms of plate degradation that result in degradation of cell performance. Initially, the emphasis is being placed on the more obvious forms involving relatively gross changes of structure such as blisters and swelling of plate material. The first phase of the study is directed at the role of coining and related edge-treatments in controlling degradation of the edges of plates.

1.2 INTRODUCTION TO COINING OF PLATES

The function of coining is to compress the sinter structure, thereby making it more dense and reducing or eliminating porosity in coined areas. Increasing the density increases physical strength and rigidity; decreasing porosity reduces the amount of active materials that are deposited in the pores during impregnation and, hence, reduces the electrochemical activity of coined areas. Thus, coining reduces the capacity available from a given total area (and weight) of plate material, hence, reduces the energy density of a cell.

The capacity per unit area of coined areas is not zero, however, because some activity remains near the surface of these areas and at the cut edges. The amount of residual activity depends on the thickness reduction produced. Minimum activity is not achieved until thickness reduction approaches 50 percent.

Since the average width of the coined border is usually constant, and independent of the size of the plate, the percentage of total plate area, and hence capacity, affected by coining increases as cell size (and plate size) decreases. The area percentage is not insignificant, i. e. , for an average border width of 1. 5 mm, which is typical, coined area amounts to 7 percent of total plate area in a 20 Ah cell and 11 percent in a 6 Ah cell. Therefore, there is a trade-off between the potential benefits of coining and the energy density obtained from a cell. This study should provide information with which cell users and suppliers may make design decisions in this area.

The origin of coining of sintered plates is obscure. Some say that coining was done originally to control the cut ends of wires in screen grid plates. Others suggest that coining first was done to provide a thinner, more solid area to which a tab could be welded to plates cut from the interior of master plaques. This may explain why Falk and Salkind[1] state that "There is no need for coining plaques --- provided with perforated steel sheet grids, since the tabs are welded directly to the (uncoated) steel sheet at the edges ---."

Currently used production methods for sintered plates for nickel-cadmium and other cells require cutting of plate material at various stages in the processes. When plates are cut, the cut edges do not behave in the same manner as the rest of the plate surface. The edges may be rough, substrate metal becomes exposed, and the sinter structure is weaker and more susceptible to damage than at areas not having an edge. Damage may occur during handling for cell assembly or from the effect of use in a completed cell.

Crumbling edges from the manufacturers point of view are a nuisance during handling and may produce shorting of the cell during assembly, thus leading to scrapping of cells before delivery. From the users point of view crumbling can lead to internal shorting of the cell after a period of use, at any time before or after launch of a spacecraft or other critical

[1] S. Falk and A. Salkind, "Alkaline Storage Batteries," John Wiley and Sons, Inc., p. 124.

mission startup. Inasmuch as the spacing between positive and negative plates in most spacecraft cells today is less than 0.01 inch, any conductive particle of this dimension or larger, if it penetrates the separator layer, can produce a short and fail the cell.

Recognizing these potential problems, the cell manufacturer may do one or both of two things: coin certain edges, and/or coat one or more edges with plastic cement. Coining compacts the sinter and, when done properly, is capable of reducing crumbling of edges during cell assembly and later under cycling conditions. Coating with cement is done to improve handling during cell manufacturing. Cementing also may prolong the life of cut edges in the cell to some extent.

A review of the cell failure information from ground tests where adequate post-mortem analysis has been carried out reveals a low incidence of shorting failures attributable to loose particles. On the other hand, the preponderance of cells tested have had coined and/or edge-coated plates, and in many cases of shorts, the cause of the short was not found. In many cases of shorted cells, the location of shorting has been the area just under the tabs, as indicated by a burned or melted appearance of the separator. A contributing cause of this occurrence may be loosening of inadequately coined sinter material assisted by the flexing of the plates at the base of the tab during cell assembly.

Failed cells removed and torn down from synchronous orbit testing (after 4 or more years of testing at NAD, Crane) have shown blistering and loss of sinter along the tops of the positive plates. As these cells behaved normally when new, the fact of their condition at tear-down indicates that slow, cumulative deterioration of the sinter structure can occur which is a potential failure mechanism.

Recently, certain cell manufacturers have suggested that coining is unnecessary and therefore need not be done. They contend that when the uncoined edges of the plates are properly compressed by the separator layers in a cell, degradation of the uncoined edges will be controlled. No proof of this contention is offered, that is applicable to cells operating several years and longer. On the other hand, positive plates removed by TRW from a 50 Ah cell that was only about 1 year old showed considerable loss of sinter along uncoined edges. These results suggest that a number of variables are involved that are not presently recognized. To maximize cell reliability for very long term missions these variables need to be identified so that they may be controlled. The present study is directed at that end.

1.3 GLOSSARY

Because terminology in this field is not standardized, certain terms are defined here as they will be used in this report.

- **Sintered Plaque** — Sintered plaque, herein referred to simply as "plaque," is the form of sintered nickle type plates just after sintering and prior to impregnation.
- **Impregnation** — Impregnation is the process of depositing electrochemically active materials in the pores of the plaque.
- **Plate** — The term "plate" will be used to refer to impregnated plates, and usually those that have been subjected to several formation cycles. The term "plate materials," however, may include both plaque and plate.
- **Coining** — Coining, as related to battery plates, is the process whereby the thickness of the plaque is reduced in a narrow border around the outside edge of the plate. On sintered-type plates, this process is normally done prior to deposition of active materials in the pores (impregnation).
- **Thickness Reduction** — For the purpose of this report thickness reduction is expressed as a percent of the uncoined thickness; that is, if $\bar{t}_1$ = average original uncoined thickness, and $\bar{t}_2$ = average thickness in coined areas, then

  Thickness Reduction (T.R.) =

$$\frac{100\ (\bar{t}_1 - \bar{t}_2)}{\bar{t}_1} \text{ percent}$$

- Edge Coating

Edge-coating involves applying a film of material to the border and edges of plates. This film is usually deposited in the form of a solution of a solid in a volatile solvent. This process is also referred to as "cementing" or "doping."

2. SCOPE AND OBJECTIVES

The study of degradation of sintered plates reported in this document is designed to identify and evaluate the potential problems of edge effects and related mechanical aspects of plates, and to define directions toward solutions, but not to perform in-depth studies in any one area. The work will investigate mechanical and electrochemical modes of degradation of plates, including a determination of the relative merits of coining versus no coining or other methods of treatment of the edges of plates for nickel-cadmium cells, in order to recommend improvements in manufacturing methods leading to increasing cell reliability for long life applications.

3. SUMMARY STATEMENT OF WORK

The following task statements summarize the work to be performed under this subcontract:

- Gather and summarize the present state of the art of plate edge finishing in terms of methods used by cell manufacturers and experience with plates both in handling and under electrical operating conditions.
- Examine possible edge finishing methods other than those currently in use, indicating their potential relative advantages, why they are not being used, and the possible impact of implementation.
- Obtain plate materials, including unimpregnated sintered plaque (when available), and plates made to existing manufacturers specifications, from a number of commercial suppliers of sealed cells. Determine relevant physical and chemical properties of these materials.
- Determine susceptibility of the edges of the plates to mechanical damage from processing and handling.
- Evaluate accelerated methods for electrochemical testing of plates and select a method for rapid comparative testing of plates. Perform accelerated electrochemical testing on plates with different edge treatments. Correlate results with physical and chemical properties.
- Perform vibration and shock testing of plates in simulated and laboratory cell configurations, and evaluate edge damage and potential shorting problems under spacecraft qualification test environment conditions.

- Using the results from tasks above and other available information, examine the mechanism of disruption of the sinter structure and of the development of plate-to-plate shorts resulting from the breakdown of the sinter structure.

- Investigate electrochemical degradation of the plates under normal cycle conditions by performance of electrical testing of re-usable cells; inspection and chemical and physical analyses of plates from these cells; and analysis of data to correlate results of materials analysis with electrical performance.

4. PARTICIPATING ORGANIZATIONS

Early in the program a number of U. S. cell manufacturers potentially capable of producing hermetically sealed cells were contacted to see if they wished to participate in this work by supplying sample materials and exchanging information. Of those contacted, the following indicated they were willing to participate:

- General Electric, Battery Products Section
- Gulton Battery Company
- Heliotek Division of Textron
- Marathon Battery Company
- Tyco Laboratories.

Plate materials were ordered and have been received from each of the above companies. Two different types of materials were supplied by Gulton Battery Company: one made by SAFT (France) and one made by Gulton in the U. S. These two types were tested separately.

5. INDUSTRY SURVEY

5.1 GENERAL

A survey was made of both cell manufacturers and users of sealed nickel-cadmium cells to determine the current state of the art of plate edge treatment and experience with edge effects. Personal visits were made to General Electric, Gulton, and Heliotek. The other participating manufacturers and a number of government agencies and industrial users of cells for spacecraft applications were contacted by telephone.

The subject was covered from two points of view: 1) relating to cell manufacturing per se, and 2) relating to cell reliability for long-life applications. The findings of the survey are presented below in a factual manner under these two point-of-view headings.

5.2 CELL MANUFACTURING ASPECTS

5.2.1 Methods of Coining

Two methods for coining are currently in use: 1) stamping with dies using power press machinery; and 2) pressing one edge at a time, usually on a hand press. The former method coins the entire perimeter in one stroke and is used where plate material is made and impregnated in continuous strip form, and hence, where cutting follows impregnation. The latter method is used where the plaque is cut prior to impregnation. Coining of master plaques or individual plate-size plaques is done in a few cases by die-stamping also using a single-stroke press.

5.2.2 Utility of Coining for Cell Manufacturing

There were considerable differences of opinion from one cell manufacturer to another on the effectiveness and need for coining plates from the point of view of manufacturing. Those making plates with a screen grid said coining was essential, primarily as a means of controlling the sharp ends of the screen wires produced by cutting, as well as to provide a base for welding the tabs, as mentioned above. Those manufacturers making plates with perforated sheet grids and slurry coating were divided. Some felt that coining was necessary to facilitate plate making and cell assembly; others felt that coining was not important for manufacturing. Investigation revealed that these differences of opinion were associated with differences in details of processing plate material and/or differences in the design and properties of the materials.

The aspects of the process that may affect the value of coining during cell manufacturing include:

a) Whether or not plates are cut (from master plaques or strip) before being impregnated;

b) The amount of thickness reduction produced in coined areas;

c) Whether or not automatic machinery is used for cutting (blanking) plates;

d) Storage and handling practices after cutting and during cell assembly;

e) Whether or not edge-coating is used;

f) The compression, electrolyte concentration, and charge/discharge parameters used during open-cell formation cycling; and

g) Requirements for inspection of plates after formation cycling.

Additional comments of the above points are discussed in the following paragraphs.

Although sintered plaque prior to impregnation is much more ductile than after impregnation, cutting of uncompressed plaque usually results in cracking along the cut edge. This form of cracking is considered by manufacturers as normal and acceptable for further processing.

When cut plaque with such cracks is impregnated, the active material absorbed acts as a cement, filling in cracks and binding loose particles together along the edges. This results in fairly strong, relatively smooth edges that handle well during cell assembly (unless cut again after impregnation). However, such edges on positive plates are prone to damage due to electrochemical cycling.

The thickness reduction observed on coined plaque from six different sources varied over the range from 10 to 61 percent (see Table 2, Section 6). Because the properties and behavior of coined borders are expected to vary accordingly, a major source of differences in experience and opinion is apparent.

Automatic die-cutting machinery is much less tolerant of weak and/or brittle sinter structure than is hand-cutting. Worn or dirty die cutting edges can turn out large quantities of ragged edges in a short time, thus applying pressure to use such material rather than absorb the cost or schedule impact of making new plate.

Cracked edges can be put into cells without further loosening and/or loss of sintered material if the plates are handled gently at all times and stored without plate-to-plate contact. The imposition of the necessary controls would greatly complicate present processing methods, however.

Edge-coating is able to compensate for a certain amount of edge damage. Manufacturers using edge-coating were less concerned with the need for coining than those that did not edge coat. Such coating appears to be only a temporary "fix," however, as discussed below.

Instability of plate edges, primarily on positive plates, is accentuated by the formation cycling process as practiced by most cell manufacturers, in spite of the few cycles involved. Plates that may appear to have flawless edges prior to formation often show swollen and flaking edges afterward. Well coined edges (T. R. >30 percent) show a much lower incidence of such damage.

Many manufacturers trim plates to final size by cutting off longer plates. This cutting therefore must take place across mostly noncoined plate material. Because positive plates are significantly more brittle after formation than before, such trimming to size after formation causes more edge damage than when the trimming is done before formation.

Normal practice for inspection of plates after formation (plus washing and drying) involves a rapid visual inspection by the unaided eye. Most suppliers agreed that this method can only detect, reliably, effects and flaws having dimensions of 0. 5 mm (0. 02 inch) or more, and all felt that, if required, inspection sufficient to detect edge defect down to 0. 1 mm (0. 005 inch) in size would require magnification, and rejection of plates having defects larger than about 0. 1 mm would result in a very low yield under existing practices.

The second area of variables that appears to be responsible for differences in attitude toward coining among suppliers includes the design and properties of the plate materials per se. Those aspects that appear to have the largest effect include:

a) Strength of the sinter, both before and after impregnation.

b) Strength of adhesion of sinter to the grid.

c) Level of loading of active material in the impregnated plate, i. e. , the weight of active materials per unit volume.

Sinter material, whether or not impregnated, that is strong without being brittle and is strongly adherent to the grid may undergo multiple cracking along edges and elsewhere without loss of particles during plate handling and cell assembly. This is considered desirable by manufacturers, especially when radiography (X-ray photography) is used to inspect the cell for acceptance. In spite of this, there is a large range of brittleness and adherence in available plate materials, as will be shown later.

The higher the loading level, the more problems are seen with damage on lightly coined and uncoined edges of positive plates, primarily after formation cycling. This is as expected in view of the postulated mechanism wherein positive material increases in specific volume when charged, thus stressing the sinter structure. It would be expected also that this factor would interact with sinter strength, with the stronger sinter being able to tolerate higher loading without disruption. It will be shown later that this is the case.

In summary, from the cell manufacturing point of view, there was no consensus that coining was necessary. However, there was agreement that coining is desirable in that is improves handling qualities and increase the yield of defect-free edges throughout the cell manufacturing process.

5.2.3 Edge Coating

Again from the point of view of cell manufacturing, there was general agreement that edge-coating improved handling qualities of plates, and hence was desirable. Some suppliers coat only positive plate edges; others coat only cut, uncoined edges but not coined edges. Some coat edges before formation; others do not coat until after formation. It appeared that coating was done where the particular product and process required it for convenience of manufacturing.

The material used for edge-coating is usually a solution of polystyrene in a volatile organic solvent. This solution is brushed or rolled on, and the result, after drying in air, is a film of polystyrene. One supplier felt that this material left something to be desired in that the deposited film was not flexible enough to do the best job. As a result, when plates with coated edges were cycled in formation, the coating often cracks and becomes loosened, allowing the underlying edge surfaces to become active and disrupted.

5.2.4 <u>Other Edge-Finishing Methods</u>

Manufacturers were asked if they were familiar with or had used any methods of finishing edges of plates other than those now in use. The question was focused on plates rather than plaque, and particularly on the problem of protecting the cut, uncoined edge produced by trimming plates to final size. Mechanical aspects will be presented first, followed by coating aspects.

First, all those asked felt that no further mechanical treatment is necessary if the edge can be coined prior to cutting except possibly smoothing of the edge with a fine file or other abrasive tool. Such smoothing is done by all manufacturers as standard procedure.

For noncoined edges, it was inquired whether sandblasting, shot-peening, or grinding could or had been used to level or round off and possibly strengthen the raw sintered edge. All manufacturers claimed to be unaware of the use of these methods and felt that any treatment of an uncoined edge that did not significantly increase the sinter density and reduce porosity would not be useful. Furthermore, the type of operations suggested were considered to be slow and costly. Some felt that when noncoined edges were coated (with polystyrene), no other treatment was necessary.

Other edge-coating materials and processes were suggested for comment. These included epoxy resin solutions, other thermosetting resin formulations, and dipping in hot melts. Generally, there was little comment on these as alternatives to polystyrene solution, other than an indication that little or nothing had been done to investigate other materials. One supplier felt that most of these would be too brittle and hence would offer no improvement over polystyrene. This supplier mentioned that certain flexible rubber-based compounds had been used as edge coating in the past, but his company had not investigated the use of formulas other than polystyrene solutions. Other manufacturers appeared to be satisfied with polystyrene and claimed they were not familiar with the properties or relative merits of other coating materials.

5.3 RELIABILITY ASPECTS OF EDGE TREATMENTS

Reliability, as used here, is the probability that a cell will operate properly and deliver a large fraction of its initial power output on discharge throughout its mission. Emphasis is given to missions involving a relatively few number of cycles (less than 1000) and on utilization of energy densities of the order of 10 watt-hours per pound of cell weight for peak discharges throughout the mission. Thus high maximum depth of discharge (greater than 70 percent) is called for.

Information obtained from cell manufacturers on the effect of coining on cell reliability (as defined above) was meager. Manufacturers claimed that they were not aware of any data linking destruction of plate edges with cell failures. One supplier indicated that he had seen edge-coating remain on edges of plates that had experienced many thousands of cycles, but he admitted that the coating was also cracked and/or missing in many areas.

Cell users contacted generally had little data from tear-down analysis following extended electrical testing, and even less relative to comparing coined with uncoined plate edges under similar conditions. They all felt that coined edges are to be preferred. The original version of the NASA Interim Model Specification for High Reliability Nickel-Cadmium Spacecraft Cells (Specification No. S-716-P-23 dated 30 April 1969) called for coining of all plate (Paragraph 2.1.1.1.9), and this appeared to be the basis for the opinion of several users.

NASA Goddard Space Flight Center has found some disruption of positive plates in cells torn down after a number of years of life testing at NAD Crane. For example, plates from a 6 Ah cell which failed on test "Sync 6" (0°C, 80 percent DOD max) showed blistering near the top edge. The actual cause of this condition and the specific cause of the cell failure have not been established to the knowledge of the writer. The cell was manufactured by General Electric and the plates were coined.

Users were divided in opinions on the efficacy of edge coating for reliability. Some felt that edge coating was desirable during cell assembly but had no knowledge that the coating contributed to cell life. Others felt that such coating was better than nothing to strengthen the edges.

6. IN-HOUSE PROGRAM

6.1 GENERAL

The work performed in-house to date under this contract consisted of obtaining plate materials from various sources; characterizing these materials in terms of physical and chemical properties; performing testing to determine relative tendency for coined and noncoined edges to experience damage resulting from physical handling and electrochemical cycling; and making some preliminary correlations between measured properties and the tendency toward structural damage.

6.2 PLATE MATERIALS EXAMINED

New plate materials were received and/or available from all participating manufacturers. These materials are as shown in Table 1. Unless otherwise noted, all plaques listed were made by the wet slurry process on a perforated sheet grid.

Table 1. Plate Materials for Investigation

Manufacturer	Positive Plaque	Positive Plate	Negative Plaque	Negative Plate
General Electric	X	X	X	X
Gulton Battery Co.	X	X	X	X
Gulton - SAFT		X		X
Heliotek	X	X (Note 1)	(Note 2)	X (Note 1)
Marathon Battery Co.	X (Note 3)	X		X
Tyco Laboratories	X (Note 4)	X (Note 1)		X (Note 1)

Note 1. Electrochemical precipitation process used for impregnation.
Note 2. Negative plaque same as positive plaque for plates tested.
Note 3. Plaque made by dry powder process on nickel screen.
Note 4. Nickel screen grid used.

The materials labelled "Gulton Battery Company" are made in this country. Those labelled "Gulton - SAFT" are made by SAFT in France and used by Gulton in their standard spacecraft cell line.

Note that two representatives of the electrochemical impregnation process have been included. This was done purposely to obtain comparative data even though this new process is not yet in full-scale production.

6.3 CHARACTERIZATION OF PLATE MATERIALS

Plate materials are to be characterized by determination of certain physical and chemical properties; by photography under optical magnification and under the Scanning Electron Microscope (SEM); and by certain special tests.

6.3.1 Physical/Chemical Properties

The following properties will be determined for each material:

Plaque (Unimpregnated)	Plate
• Thickness	• Thickness
• Density	• Density
• Void Fraction	• Void Fraction
• Resistivity	• Resistivity
• Sinter Strength	• Sinter Strength
	• Active Material Content

6.3.1.1 Test Methods

The methods used for these determinations are described briefly below. A more complete description of the methods will appear in the Final Report.

- Thickness and Density

  Samples of plate are measured at five points with a micrometer and the average calculated. The dimensions parallel to the plane of the plates are measured and used to calculate the sample area. The sample is weighed and the density calculated as the weight divided by the product of average thickness and area.

- Void Fraction

The interconnected void volume is determined from the weight of water imbibed under vacuum by a sample of plate material. The ratio of void volume to total apparent sample volume as determined for the density determination is the void fraction.

- Resistivity

A Kelvin Bridge technique is used, involving a four-point contactor with separate current and potential leads. A diagram of the contactor is shown in Figure 1, and a photograph of a prototype device is shown in Figure 2. Regulated direct current at 1 ampere is used for measurement, supplied by a regulated power supply and a 10 ohm 100-watt resistor as a ballast load. Samples are cut to 7 cm (2.75 inch) wide by 10 cm (4 inch) or more long, and the contactor is placed along the center line. The voltage between the two potential contacts is read and is proportional to resistivity.

- Sinter Strength

The four point bend method is not used in this study because, in the opinion of the writer, it is not reliable, particularly for plate using sheet metal grids and on finished and cycled plates. Instead, a surface penetration resistance method is used. In this method, which is admittedly exploratory, the force required to compress an area approximately 1 cm^2 to a reduction in thickness of 0.25 mm (0.01 inch) is used as a measure of sinter strength.

- Active Material Content

Total nickel active material of positive plates and total cadmium active material of negative plates are determined by extracting samples with Muspratt solution and measurement of nickel or cadmium in solution by atomic absorption.

6.3.1.2 Results To Date

Characterization is still in progress at this date. Some results of tests completed are given below:

- Thickness

Thicknesses of both coined and uncoined areas of plate materials on hand were measured as indicated above. The results, together with the calculated thickness reductions, are shown in Table 2. In this table and henceforth throughout this report code letters are used to designate specific suppliers of materials for which data is given. It is felt that this approach will facilitate the presentation and discussion of the data.

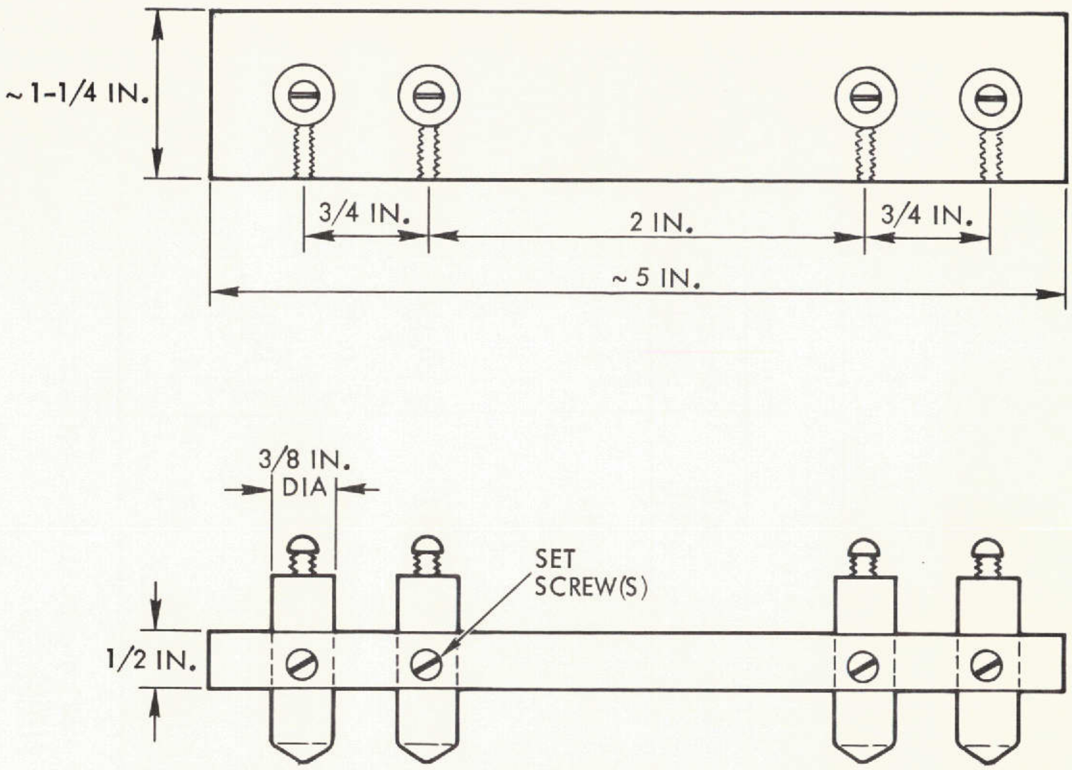

Figure 1. Four Point Contactor Dimensions

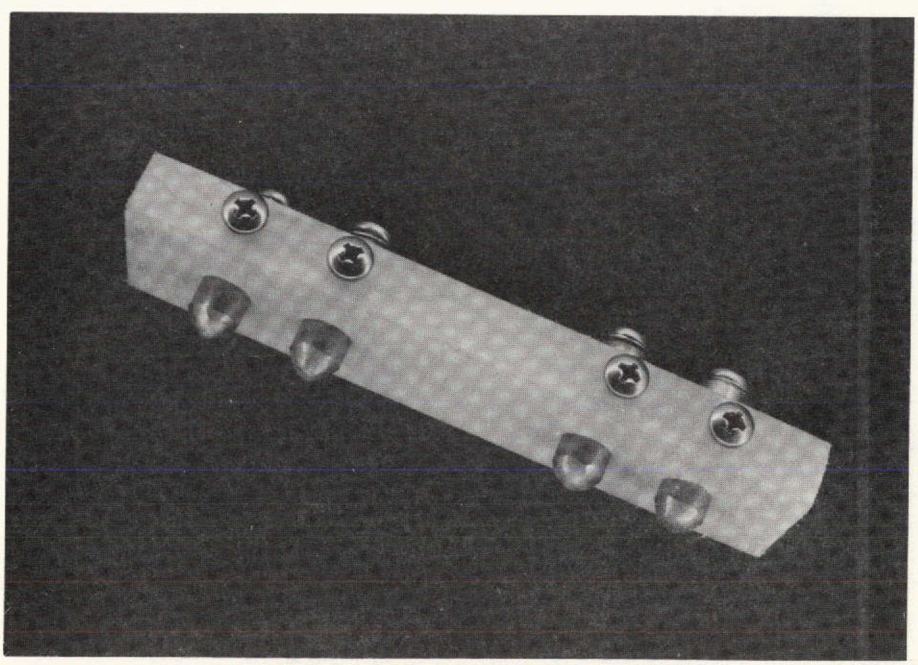

Figure 2. Four Point Contactor
(TRW Photo No. 104826-73)

Table 2. Thickness and Thickness Reductions by Coining

Supplier Code	"Positive" Plaque (Note 1)			Positive Plate			Negative Plate		
	Uncoined (mm)	Coined (mm)	T.R. (%)	Uncoined (mm)	Coined (mm)	T.R. (%)	Uncoined (mm)	Coined (mm)	T.R. (%)
A	--	--	32 (est)	0.85	0.56	34	0.90	0.50	44
B	0.68	0.62	10	0.69	0.62	10	0.79	0.66	17
C	0.88	0.51	42	0.88	0.46	47	0.86	0.43	50
D	--	--	50 (est)	0.77	0.35	55	0.79	0.29	63
E	0.69	0.29	58	0.64	0.26	59	0.94	0.35	63
F	0.75	0.29	61	0.71	0.25	65	0.85	0.54	37

Note 1: "Positive" Plaque is that plaque used for making positive plates. Only "positive" plaque was tested at this time.

- Density and Void Fraction

  Data for density and void fraction for materials analyzed to date from four of the suppliers are shown in Table 3.

- Resistivity

  The resistances as measured by the four-point contactor described above are given in Table 4 for most of the available materials in the as-required condition. Assuming that the current flux was uniform throughout the cross-section of the material between the potential contacts, the equivalent resistivities may be calculated from these resistances by use of the relation

$$\rho = 0.137\, t \cdot r$$

  where

  ρ = resistivity, in milliohm - cm

  t = thickness (of uncoined area), in mm (from Table 2)

  r = resistance, in milliohm (from Table 2).

- Sinter Strength and Active Material Content

  Data for sinter strength and active material content will be presented in the Final Report.

6.3.2 Photographic Characterization

The coined borders of plaques and plates from different suppliers tend to look similar to the unaided eye. However, there are in fact a wide range of variations in the materials received. These differences become apparent under 4 - 10X magnification. Photographs of typical coined borders and edges of plaque and plate materials are shown in Figures 3 through 10. Note that when the thickness reduction is 30 percent or greater the coined border is obvious, but when the thickness reduction is only 10 percent (as with Type B, Figure 7 and 8), the presence of a coined border is hard to see in places even under magnification.

Table 3. Density and Void Fraction Data for Uncoined Plate Materials

Supplier Code	Positive Plaque		Positive Plate		Negative Plate	
	Density (g/cm^3)	Void Fraction (%)	Density (g/cm^3)	Void Fraction (%)	Density (g/cm^3)	Void Fraction (%)
A	--	--	3.64	28.8	3.94	31.6
B	2.12	67.5	3.80	24.2	4.00	33.7
C	2.08	73.2	3.55	35.2	3.88	35.7
D	2.07	67.4	3.25	39.0	3.15	51.2
E	1.77	75.1	3.63	27.4	3.81	30.3
F	1.95	75.0	3.11	39.0	3.31	45.7

Table 4. Relative Resistances of Plate Materials (All Values in Milliohms)

Supplier Code	Positive Plaque	Positive Plate	Negative Plate
A	---	1.06	1.05
B	1.10 - 1.25	1.55 - 1.80	1.25 - 1.35
C	0.75	1.03	0.85
D	1.08	1.10	0.95 - 1.15
E	2.02	2.10	1.43
F	2.15	2.20	2.40

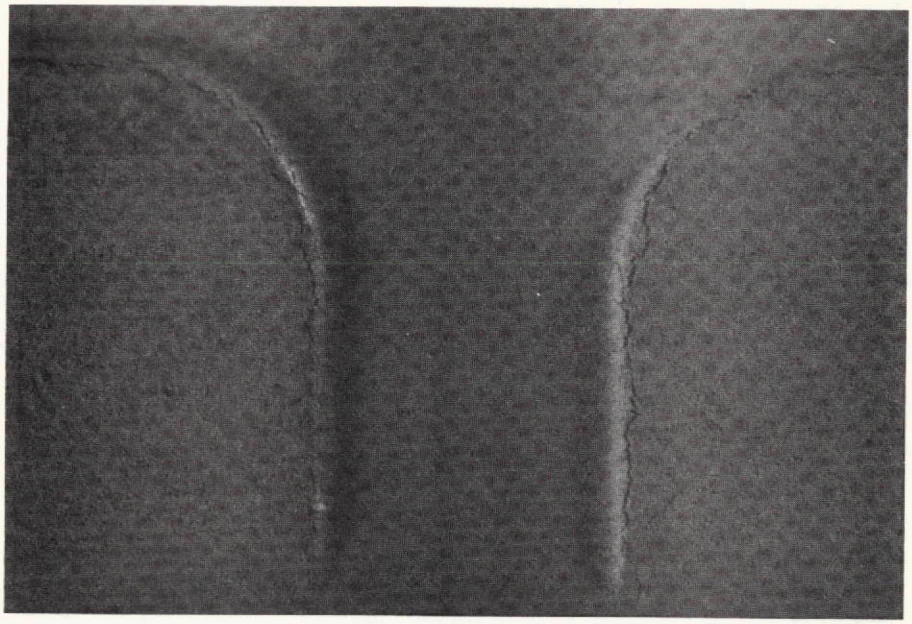

Figure 3. Supplier C Plaque Coined Corners Prior to Cutting. Note Typical Cracking Along the Shoulders (10X). (TRW Photo No. 104687-73)

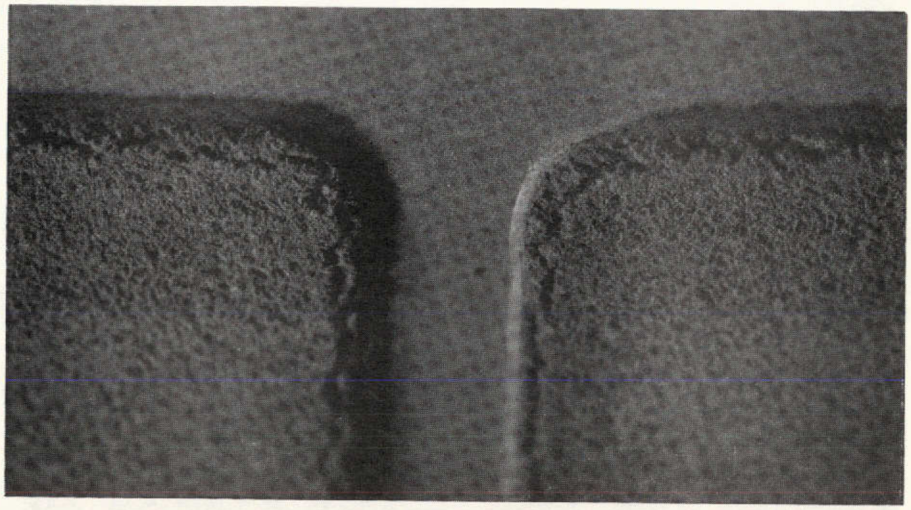

Figure 4. Supplier E Plaque Coined Corners Prior to Cutting Note Cracking Along the Shoulders. (TRW Photo No. 104275-73)

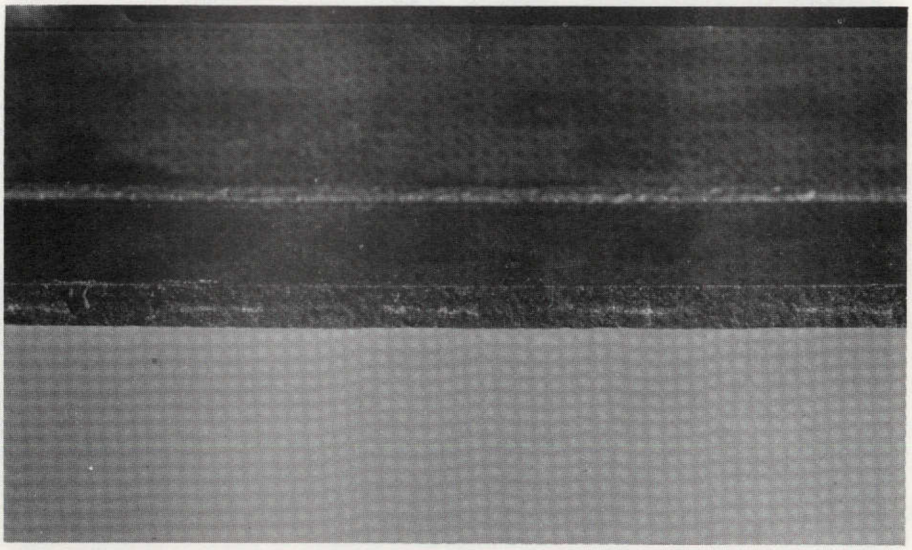

Figure 5. Supplier A Positive Plate Coined and Die-Cut Straight Edge (6X) (TRW Photo No. 104819-73)

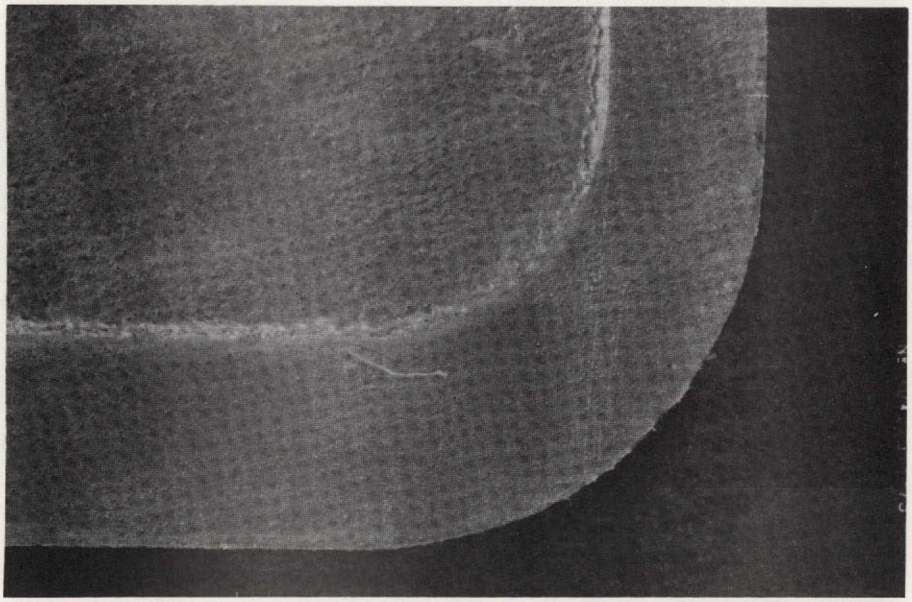

Figure 6. Supplier A Positive Plate Coined and Die-Cut Corner (10X) (TRW Photo No. 104273-73)

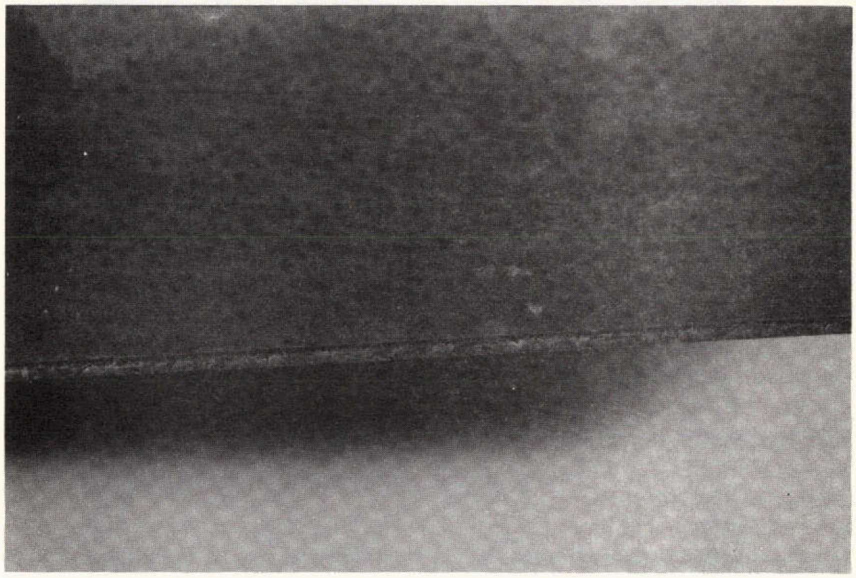

Figure 7. Supplier B Positive Plate Coined and Die-Cut Straight Edge (10X) (TRW Photo No. 104803-73)

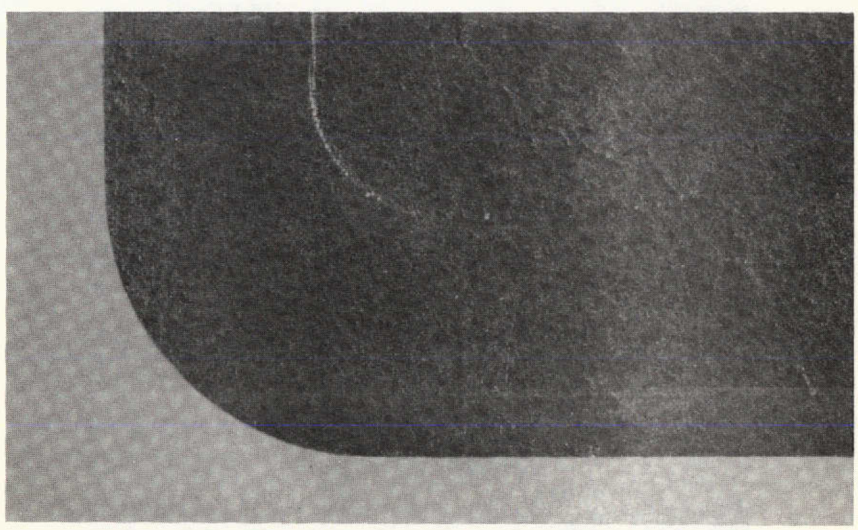

Figure 8. Supplier B Positive Plate Coined and Die-Cut Corner (TRW Photo No. 104685-73)

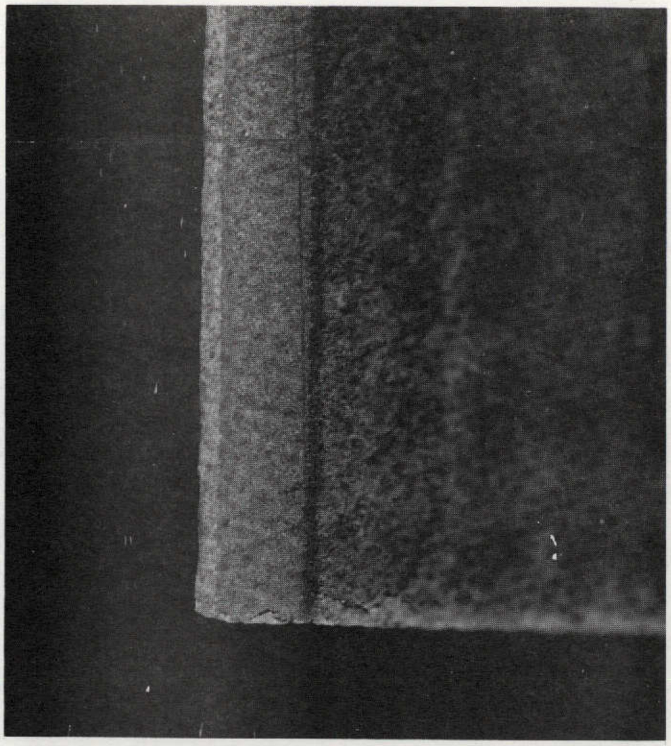

Figure 9. Supplier D Positive Plate Sheared and Coined Edge (5X) (TRW Photo No. 104278-73)

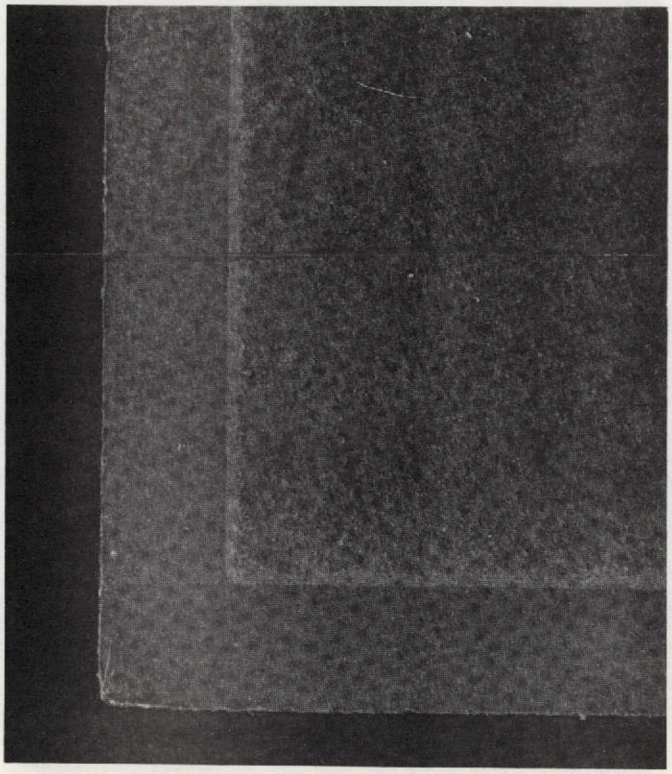

Figure 10. Supplier D Positive Plate Sheared and Coined Corner (10X) (TRW Photo No. 104274-73)

Scanning electron microscope photographs of plate materials will be presented in the Final Report.

6.4 SPECIAL MECHANICAL TESTING

6.4.1 Cutting Tests

Because the question of the effect of cutting of plaque and plates with and without coining is an important one in this study, plate materials were cut using a die-cutter and the cut edges examined. It was noted that, using the sharpest tool available in a model-shop, only occasionally could either plaque or plate be cut without producing appreciable cracking along the cut. Figures 11 through 14 show some examples of the results. It is apparent from the photographs that generally the cracking along cuts was much more controlled in coined areas than in uncoined areas.

6.4.2 Tests Brittleness and Adherence of Sinter

Various test methods were explored to characterize the brittleness of the sinter and its degree of adherence to the grid. In one such test, plates were bent in the "long" direction (i. e., in the direction parallel to the direction of the tab) by grasping the ends and bending until the ends were at an angle of 150° (30° out of the original plane), then bending to the same extent in the opposite direction. The results are summarized as follows:

Supplier Code	Positive Plate	Negative Plate
A	Cracked at one point near middle	Cracked at several points
B	Several hair-line cracks, uniformly spaced	No visible cracking
C	No visible cracks	Several hair-line cracks
D	Cracked at one point	Cracked at one point
E	No visible cracks	Cracked at several points
F	Cracked at several points	Cracked at one point

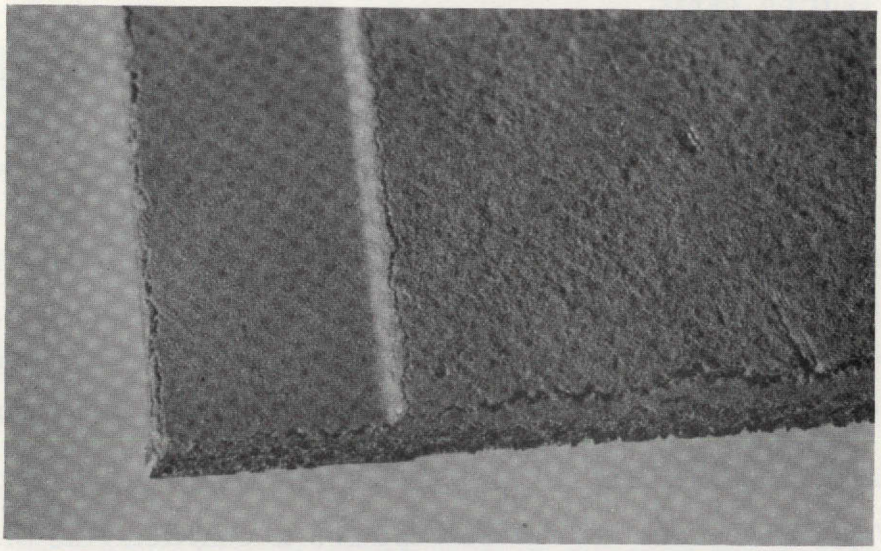

Figure 11. Supplier C Plaque Cut at TRW With and Without Coining (10X) (TRW Photo No. 104691-73)

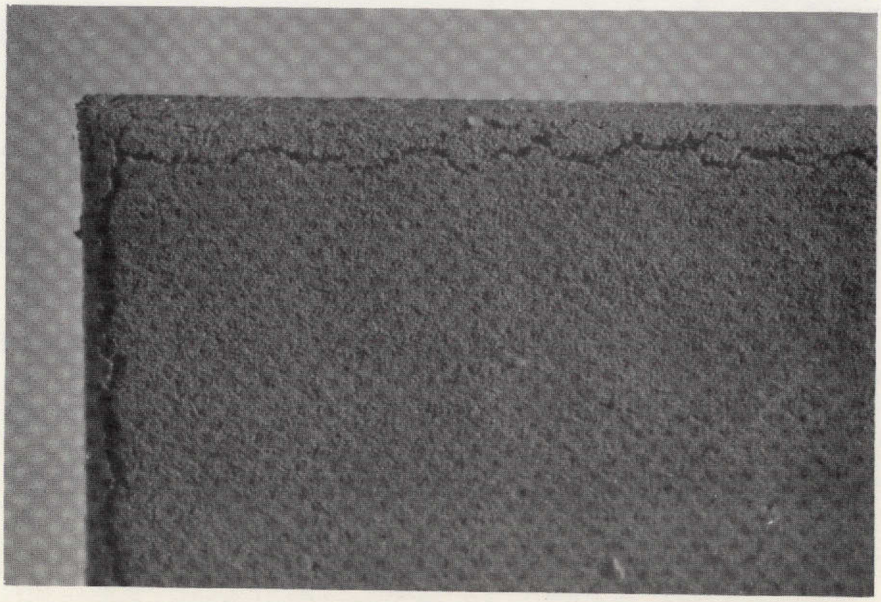

Figure 12. Supplier D Plaque Cut at TRW Without Coining (TRW Photo No. 104690-73)

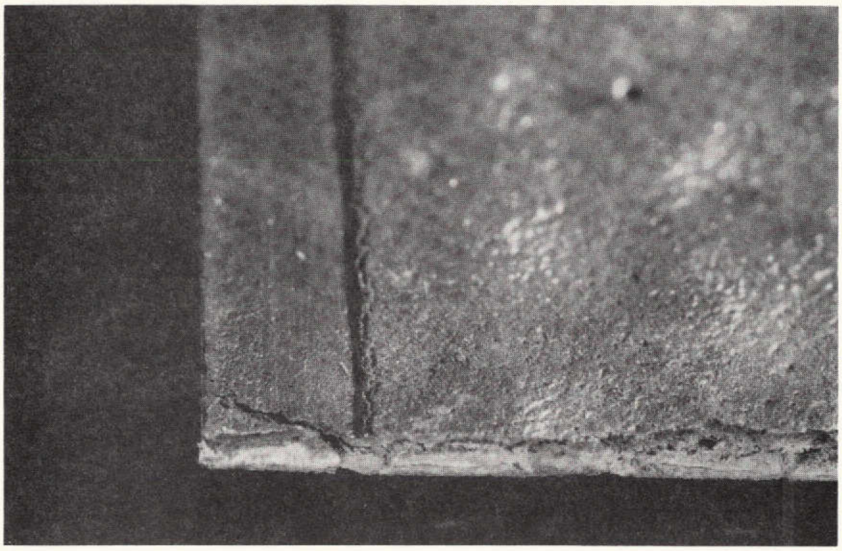

Figure 13. Supplier A Positive Plate Sheared through Coined and Uncoined Areas (8X) (TRW Photo No. 104281-73)

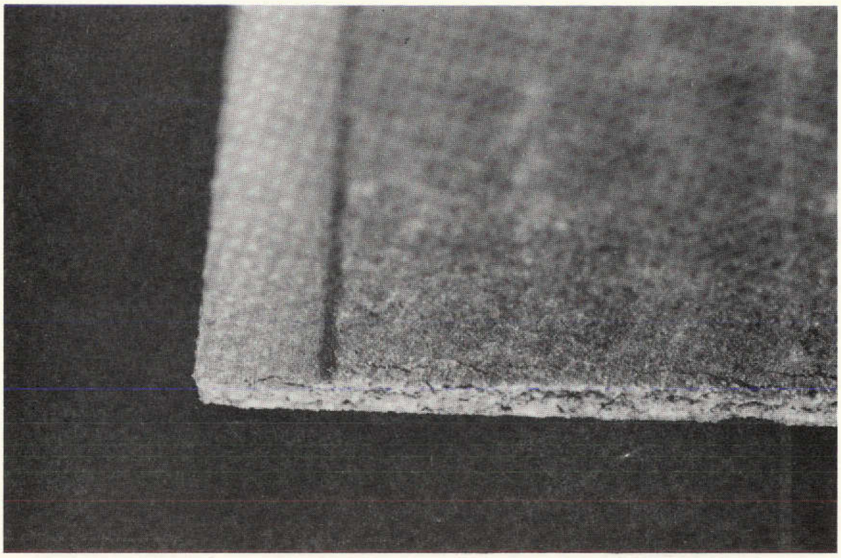

Figure 14. Supplier D Positive Plate Sheared through Coined and Uncoined Areas (TRW Photo No. 104277-73)

In most cases these cracks were easily visible particularly when the plate was flexed slightly. There was appreciably more loss of sinter at non-coined edges at these cracks than at coined edges.

Other tests involved dropping plates with coined and uncoined edges onto the bench-top from a height of 1 foot, and tapping plates on their edges on the bench-top, in a manner that might be expected during cell production. Qualitatively, the coined edges suffered less damage than uncoined edges. These tests have given variable results to date. Further work is planned in this area to refine test techniques and methods of evaluating the results.

Another test method that gave more repeatable results and that gives a good still-photographic representation was developed. In its present form it is performed as follows: A strip 1 cm wide and 7 cm or more long is cut from the plate. This strip is then "bent" around a polished, 3/8 inch diameter stainless steel rod at intervals determined by the hole pattern in the grid. When a length of about 40 cm has been so bent, the strip is pulled back and forth against the rod many times until it appears that the condition of the piece has stabilized, i. e., no further cracking or loss of material (if any) is occurring. The resulting specimen is then inspected and photographed under 2 - 3X magnification.

Photographs of typical specimens of positive plate material from Suppliers A, B, C, and D, after being subjected to the test involving wrapping around a 3/8 inch rod, are shown in Figures 15 through 18, respectively. This test is severe, but it also reveals distinct differences between materials from different sources.

6.5 ACCELERATED ELECTROCHEMICAL TESTING

Two forms of accelerated electrochemical tests are to be performed in this study. One is to be a rapid, comparative test applicable to individual plate samples; the other is to involve a real cell configuration where the plates are constrained by contact with separator layers under normal compression. Only the first type of test has been performed to date.

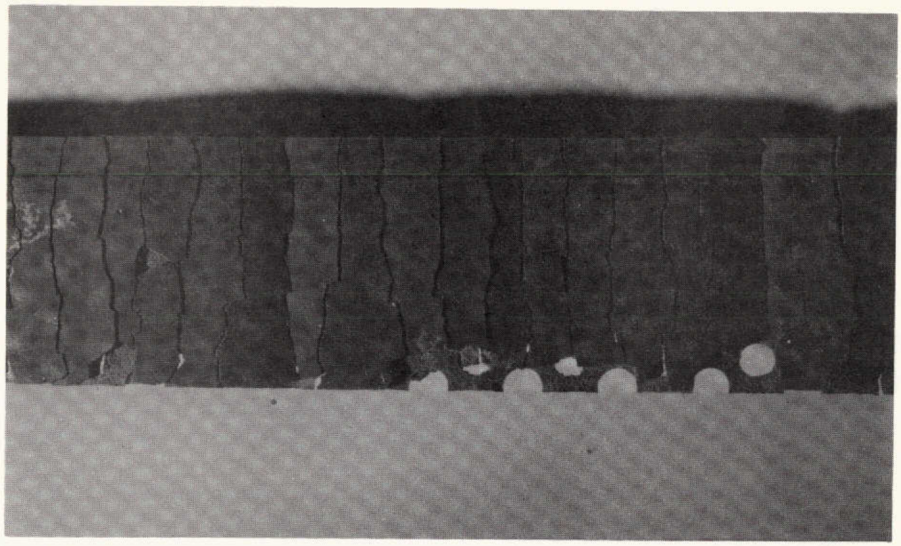

Figure 15. Supplier A Positive Plate After Bend Testing (TRW Photo No. 104684-73)

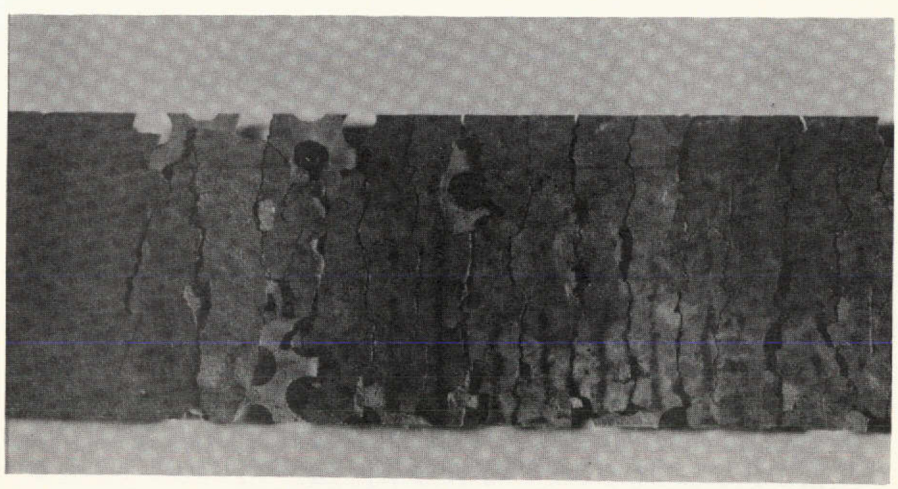

Figure 16. Supplier B Positive Plate After Bend Testing (TRW Photo No. 104825-73)

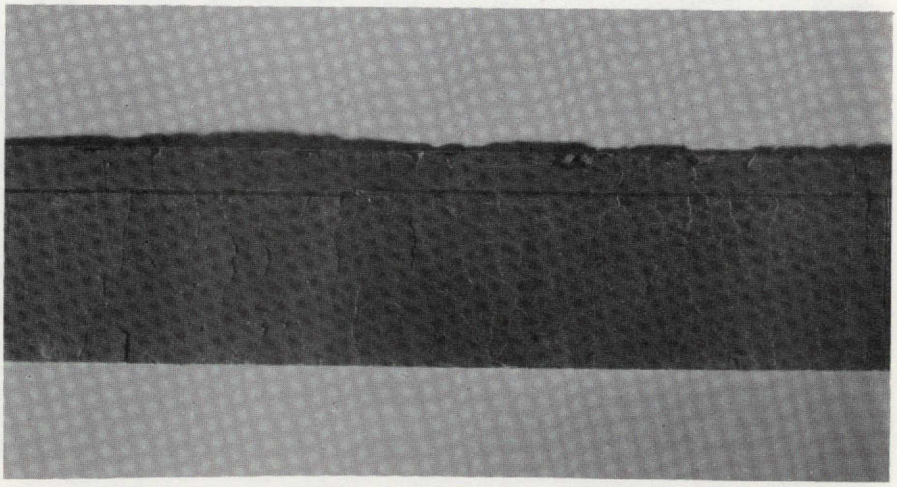

Figure 17. Supplier C Positive Plate After Bend Testing (TRW Photo No. 104682-73)

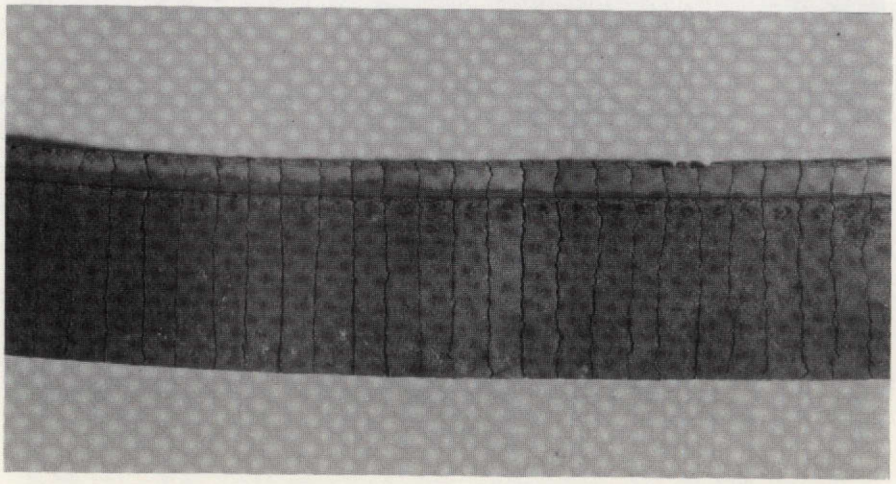

Figure 18. Supplier D Positive Plate After Bend Testing (TRW Photo No. 104824-73)

6.5.1 Test Method Development

Because the comparative test is not standardized and had not been performed under controlled conditions previously in this laboratory, some development was necessary. In starting up, several points known to the trade but not described in the open literature were considered, namely:

1) Sintered plates operated without constraint by separators tend to swell and disintegrate more rapidly than those under constraint.

2) Repeated cycling increases the rate of swelling relative to simple continuous overcharge or overdischarge.

3) Higher KOH concentrations in the electrolyte cause more swelling of positive plates than lower concentrations.

4) Rapid gas evolution contributes to disintegration of plates.

The objectives of the development work were to arrive at a procedure that would produce the desired result in a 24-hour period, with the desired result being the generation of damage effects at uncoated edges that are clearly visible (at least under 5X magnification) and that there be a range of severity from coined to uncoined edges and from one supplier's material to another.

Exploratory tests were conducted with plates suspended freely in a large excess of electrolyte. Concentrations of KOH of 25, 34, and 40 percent were tried. Electrical procedures tried included a) charging only for 24 hours at current densities of 0.5, 1.0, and 2.0 A per dm^2 of positive plate area (corresponding to approximately the 0.2C, 0.4C, and 0.8C rates based on currently available positive plate loadings); and b) cycling for 24 hours at 1.0 and 2.0 A per dm^2 with charge and discharge times such that plates are fully overcharged and overdischarged, and hence gassing freely at the end of each half-cycle. All tests were run at room temperature (22° to $24^{\circ}C$).

The results of this preliminary work were as follows: no appreciable damage occurred after 24 hours continuous charging at 0.5 A/dm^2 at any of the KOH concentrations used on any of the available plate materials.

Slight damage occurred at 1.0 A/dm^2, with little additional difference at 2.0 A/dm^2. Cycling at 1.0 A/dm^2 (4 cycles in 24 hours) gave significantly more results in 34 and 40 percent KOH, while cycling at 2.0 A/dm^2 (8 cycles in 24 hours) gave more damage on some materials, with the most consistent results in 40 percent KOH. Limited resources did not permit complete optimization of test conditions. As the range of results under the last set of conditions mentioned appeared satisfactory, this set was selected for further testing of materials.

6.5.2 Test Methodology

Because the positive (nickel-oxide electrode) plates are more fragile and subject to more disruption due to volume changes in the pores, most test effort was directed at positive plates. In most of accelerated comparative testing done, therefore, the "cell" was made using positive plates for both "positive" and "negative" electrodes, thus doubling the utilization of both time and equipment for positive plate materials. Current was driven through these "cells" using a regulated power supply. Timing and switch gear was used with the power supply to provide automatic cycling.

The plates were contained during testing in lucite cells made for this purpose. The inside dimensions of these cells are 3 x 1 x 7 inch high. This cell was adequate to hold a number of commercially available, aerospace-type flat plates, many of which are 2.75-inch wide and up to 6-inch long over the sintered area.

When plates that were coined on all edges were to be tested for noncoined edge effects, the coined border was sheared off along one side. Also, pieces of nickel sheet 0.005-inch thick, 0.5-inch wide, and 4-inch long were spot-welded to the tabs of the as-received plates to facilitate electrical connections in the test cell.

When unformed plate materials, or those for which the degree of formation was either uncertain or not adequate, were to be tested by high-rate cycling, such materials were first cycled at 1.0 A per dm^2 in 25 percent KOH for 3 to 4 cycles. Plates showing significant damage from this cycling were so noted and not tested further.

The voltage of the test cell was recorded using an Esterline-Angus strip chart recorder. When two positive (or two negative) plates were used, the recorder was set for center zero and 5 volts full scale (±2.5 volts from center zero), as the cell voltage ranged from +1.8 volts to -1.8 volts. For individual electrode potential measurements, a low resistance Hg/HgO/KOH reference electrode was used with the open tip immersed in the electrolyte in the test cell.

For convenience of reference, the test procedure as presently carried out is summarized by the steps listed in Table 5.

Table 5. Accelerated Comparative Cycle Test Procedure

1. Inspect plate under 5X magnification for initial defects.
2. Shear off one coined border (if desired).
3. Inspect sheared edge under 5X magnification.
4. Spot-weld auxiliary tab.
5. Weigh plate assembly.
6. Perform preliminary cycling (if desired)
    a) Immerse and soak in 25 percent KOH solution
    b) Cycle: current 1.0 A per dm^2, 3 hours in each direction; 3 to 4 cycles.
7. Remove and inspect under 5X magnification.
8. Immerse in 40 percent KOH solution.
9. Cycle: current 2.0 A per dm^2, 1.5 hours in each direction; 8 cycles (24 hours).
10. Remove and inspect under 5X magnification.
11. Wash and dry.
12. Reweigh and calculate loss of weight.

Note that the specimen is inspected before washing and drying. This is because it was observed that the character and visibility of many of the effects were altered considerably by drying, if not merely by rinsing the affected areas with water.

6.5.3 Results of Testing to Date

Only plates from Suppliers A and B have been subjected to accelerated cycle testing to date, and only plates with noncoated edges have been tested. Some typical results are illustrated in Figures 19 through 23.

No appreciable change was observed to occur on the coined edges on the sides and bottom of positive plates from Supplier A (thickness reduction = 34 percent). Thus the appearance of these edges was essentially the same as shown in Figure 19. The uncoined sheared edge appeared unchanged to the unaided eye; however, when magnified, some swelling of the cut face and many small pits were evident, as shown in Figure 19.

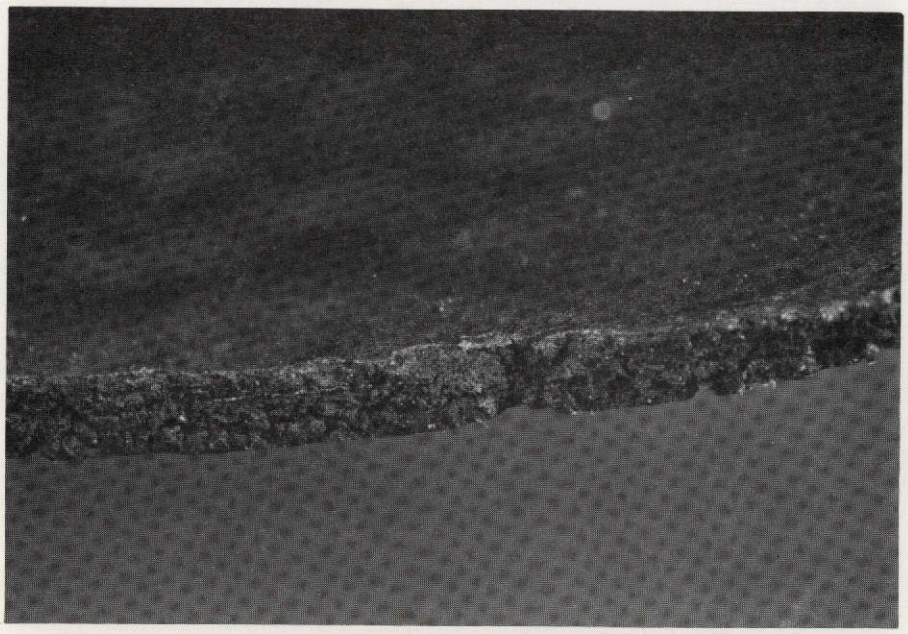

Figure 19. Supplier A Positive Plate Uncoined Cut Edge at Side of Plate, after Cycle Testing (10X) (TRW Photo No. 104821-73)

Another example of the type of effect produced by the accelerated cycle test is shown in Figures 20 and 21. This plate was made without coining and was not cut again before testing. Note the relatively extensive loss of sinter at the edges.

Of particular interest is the photograph of Figure 22, which shows the coined top edge of a positive plate from Supplier A. Note that the sinter closest to the edge has been extensively disrupted. An investigation to determine the reason why this edge behaved so differently than the other coined edges was then conducted. A number of new plates of this same type were examined closely under the microscope. It was soon evident that the border at the top on many of the plates was different in that the "coined" area was tapered at the outside edge, whereas the coined borders on the other three sides were not tapered. On some top edges the taper was only slight. Examination of the tab area, shown in plan view in Figure 23, shows that these plates are die-cut to remove a thin line of sinter at the very edge of the originally sintered area of the strip as manufactured. A profile of the border between sintered and non-sintered areas (as on the tab) is shown (Supplier A) in Figure 24. This photo was taken looking at the edge of the plate at the tab side. The taper at the border is clearly shown. The same type of specimen from Supplier B, Figure 25, shows an even longer taper. Both of these plates were "coined" on all sides and the top.

Cross-sections taken through the top cut edge on two different positive plates (Supplier A) in the as-received condition are shown in Figure 26 and 27. Figure 26 shows no appreciable taper near the edge, while Figure 27 shows part of the original taper remaining at the edge. Note in Figure 26 that the coining indentation occurred on one side of the plate only.

It appears therefore, that an uncompressed, tapered-thickness border of sinter will remain when the plate is coined too near the edge of the sintered area of the strip, and/or the reduction in thickness is too slight. These two variables interact, as illustrated in the drawing of

Figure 20. Supplier B Positive Plate Uncoined Corner, Before Testing (10X) (TRW Photo No. 104822-73)

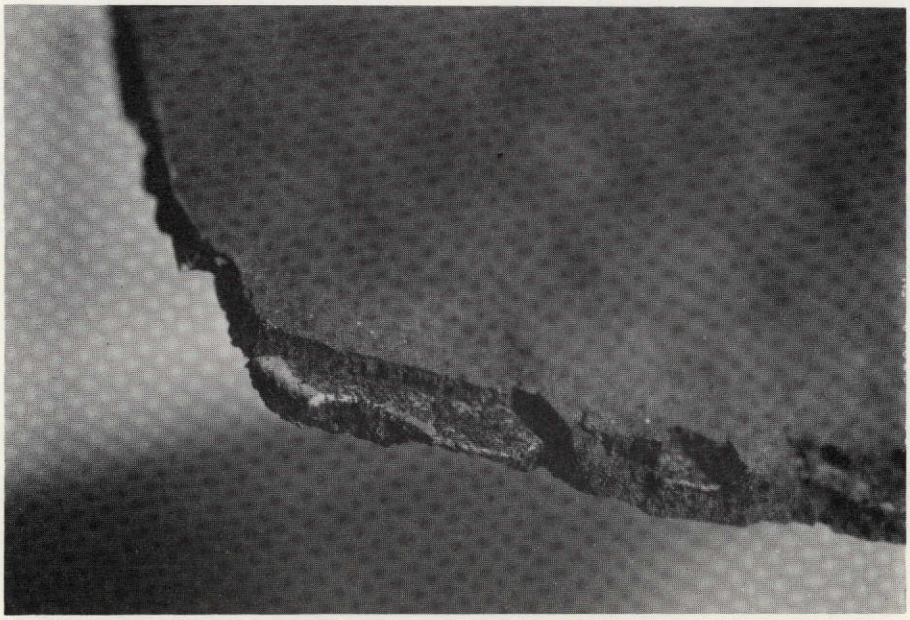

Figure 21. Supplier B Positive Plate Uncoined Corner after Cycle Testing, Showing Loss of Sinter Material (TRW Photo No. 104823-73)

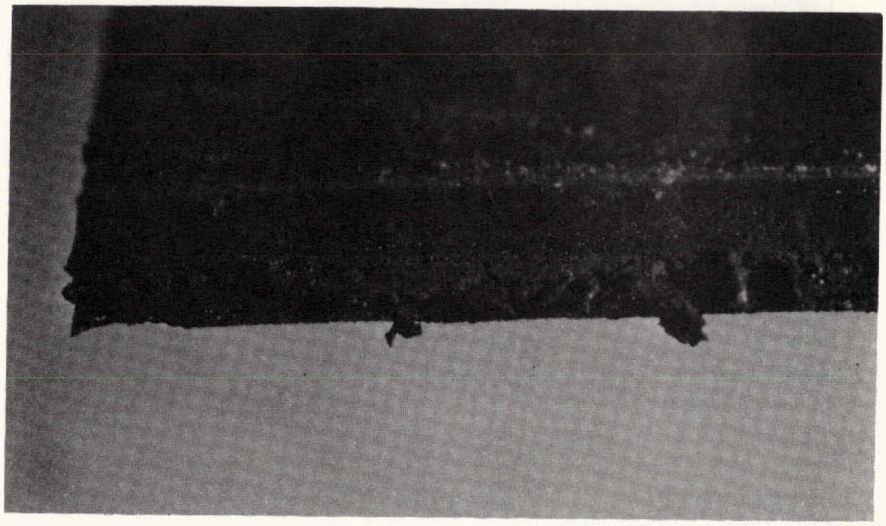

Figure 22. Supplier A Positive Plate Coined Edge at Top, after Cycle Testing, (8X) (TRW Photo No. 104820-73)

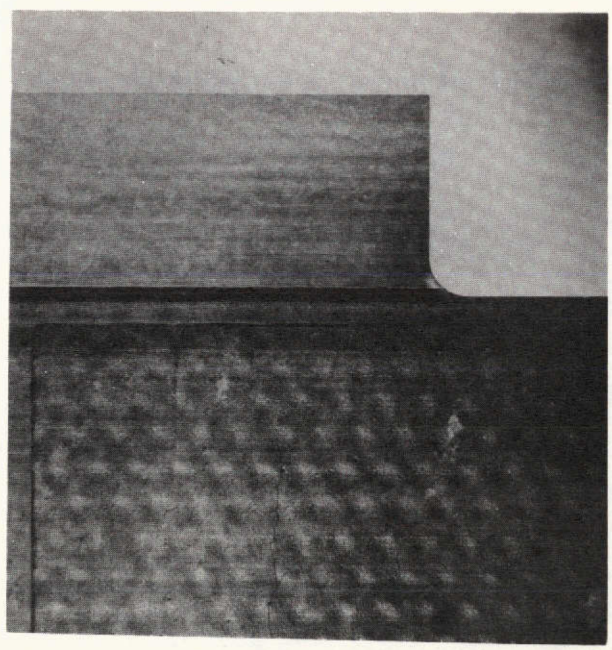

Figure 23. Supplier A Positive Plate Tab and Surrounding Area Showing Borderline of Sintered Portion (2X) (TRW Photo No. 104803-73)

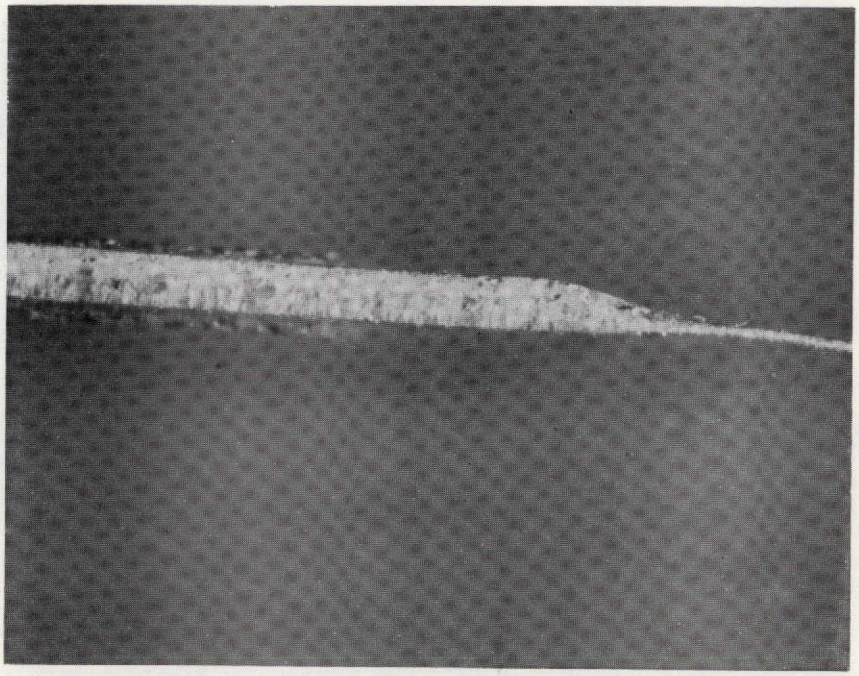

Figure 24. Supplier A Profile of Sinter to Non-Sintered Border on Tab (10X)

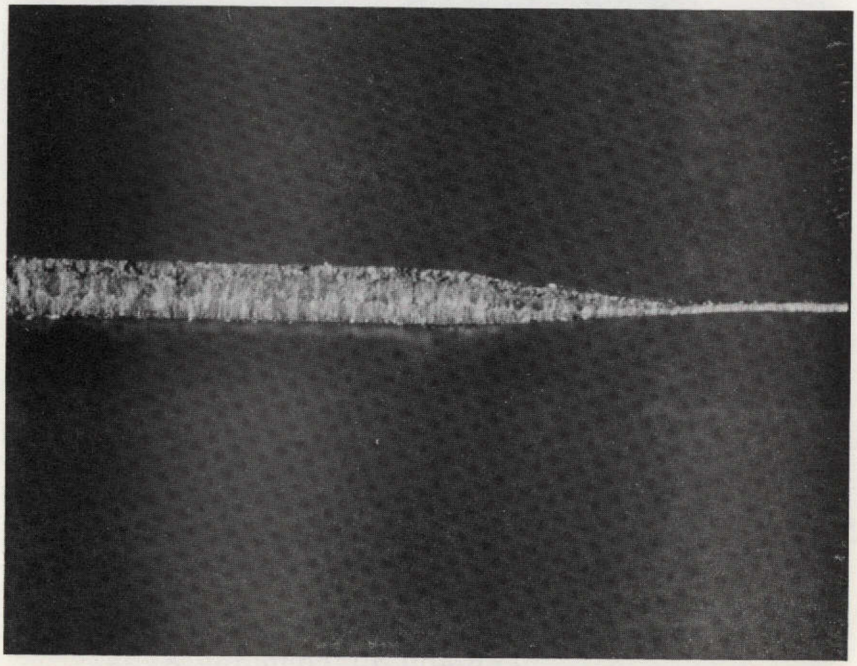

Figure 25. Supplier B Profile of Sinter to Non-Sintered Border on Tab (10X)

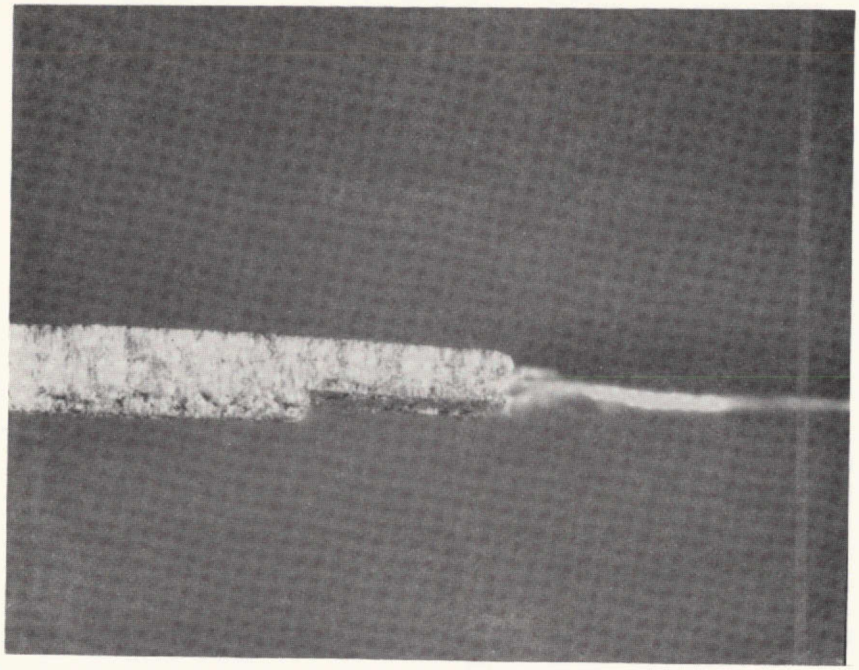

Figure 26. Supplier A Profile of Trimmed Coined Top Edge, Showing Taper Removed (10X)

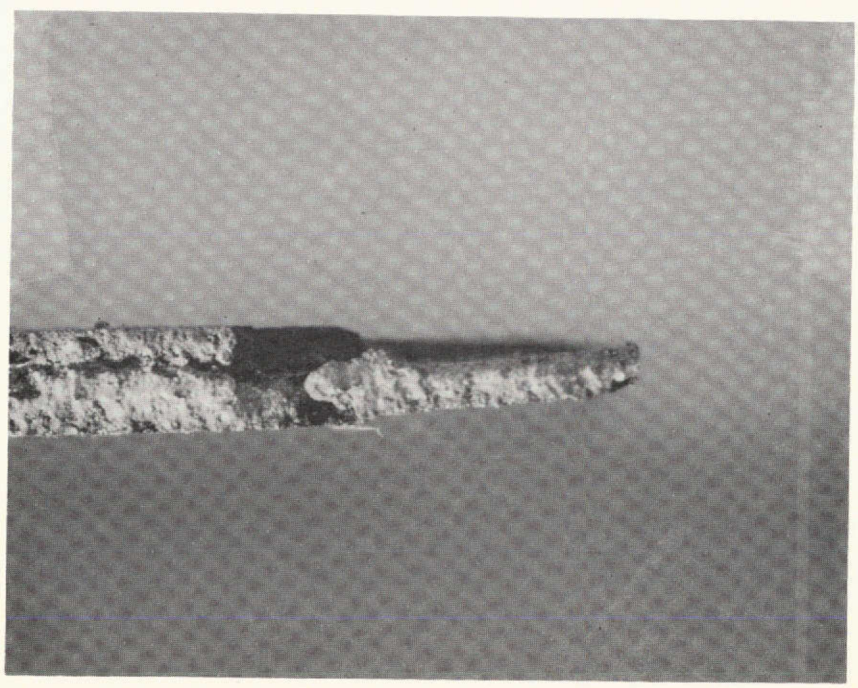

Figure 27. Supplier A Profile of Trimmed Coined Top Edge Showing Same Taper Remaining (10X)

Figure 28. In this discussion, it is assumed that the dimension W_c from the inside limit of the coined border to the die-cut edge is always the same.

The type of coining and cutting that leaves a taper on the outside edge is shown as Coin A and Cut A. That portion of the original sinter edge taper between point P (at the cut) and point Q (where the compression actually ends) thus remains uncompressed and hence in the same condition of strength and porosity as the uncoined sinter in the body of the plate.

Two other possible cases, either of which would eliminate the uncompressed outer rim, are shown in Figure 28. Coin B with Cut B brings the coined area entirely inside the original taper, and hence no taper is left outside of point R. Thickness reduction, $(t_o - t_1)/t_o$, remains the same as for Coin A. Coin C and Cut C requires a greater thickness reduction $(t_o - t_2)/t_o$ and brings the properly compressed area out to point P. Hence the Cut C at point P leaves no uncompressed taper. Coin B would require that the plate pattern be moved slightly toward the center of the sintered strip from current practice. This would result in a corresponding extension of the sintered area up the tab on the cut plate. An example of this type of cutting at the top of the plate is shown in Figure 29.

6.5.4 Status of Testing Program

Only a small fraction of the comparative accelerated testing of unsupported plates planned has been completed at this time. Results of the remaining testing and final conclusions will be presented in the Final Report.

6.6 TESTING IN REAL CELL CONFIGURATIONS

6.6.1 Vibration and Shock Testing

The plan calls for subjecting plates with coined and uncoined edges, in packs with separators under normal compression, to vibration and shock spectra such as those expected on the Viking Orbiter 75 launch vehicle. This task is as yet in the analysis stage. Results will be presented in the Final Report.

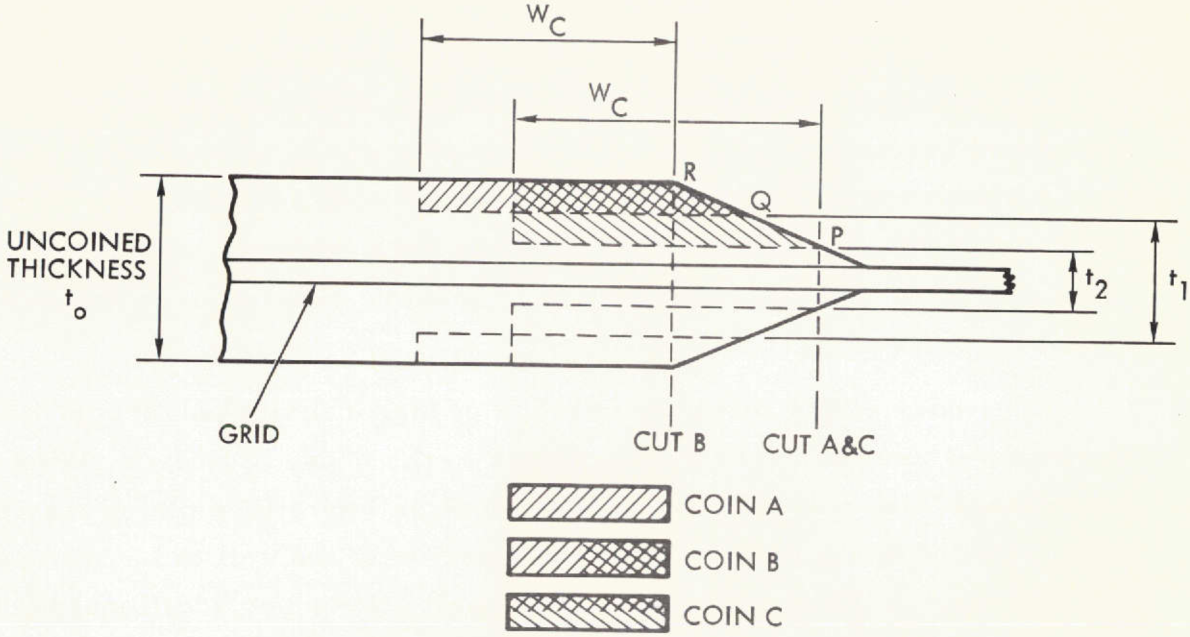

Figure 28. Coining and Cutting Interaction with Tapered Sinter Border

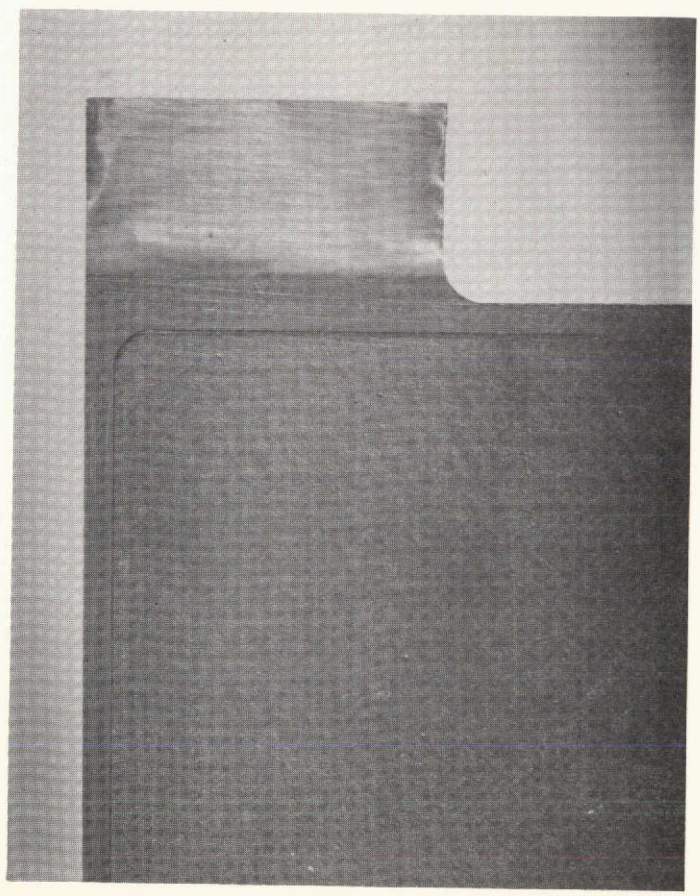

Figure 29. Plate Tab Area, Showing Wide Coining and Trim to Remove All Taper at the Top Cut Edge (TRW Photo No. 104798-73)

6.6.2 Electrochemical Testing

Cells will be assembled at TRW using reusable cell cases designed and fabricated by TRW for this type of work. A photograph of one such completed assembly equipped with a pressure gauge is shown in Figure 30. A view with the face plates removed is shown in Figure 31. The integrity of this cell and the seal used has been demonstrated by prior testing of 36 of these assemblies for 18 months with frequent pressure cycles up to 75 psig.

The body of the cell case is made of high-density polypropylene. O-rings are used to seal the face plates to the body. The face plates are interchangeable, and either stainless steel or lucite face plates may be used. Use of lucite plates allows the interior of the cell to be seen easily at all times. A gland on the side of the cell allows two additional leads to be introduced for auxiliary electrodes or thermo-sensors.

Cells will be assembled using the plates supplied by participating companies. Nylon separator material, Pellon Type 2505, will be used. Results of these tests will be presented in the Final Report.

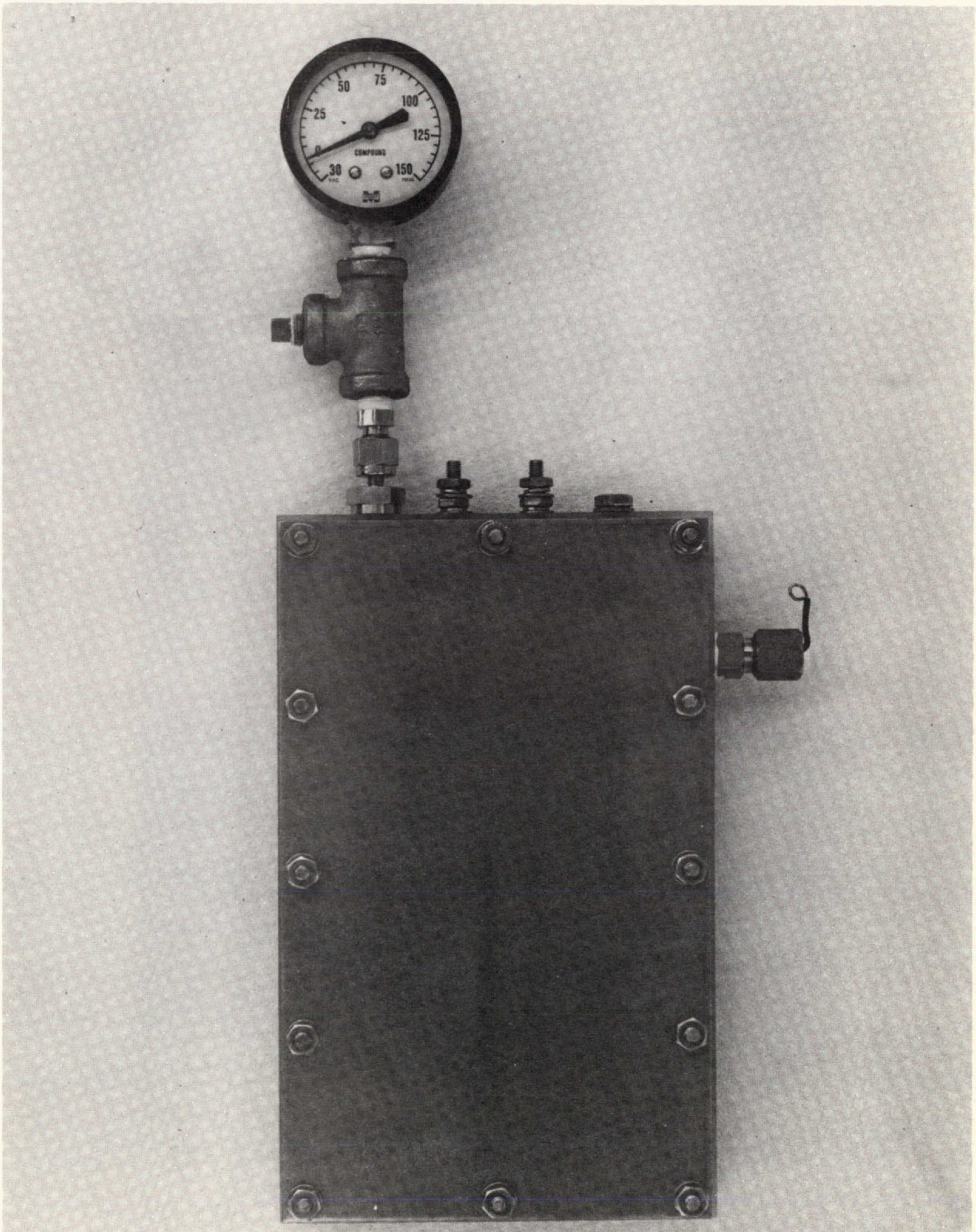

Figure 30. Re-Usable Cell Assembly Fitted with Stainless Steel Face Plates (TRW Photo No. 88261-71)

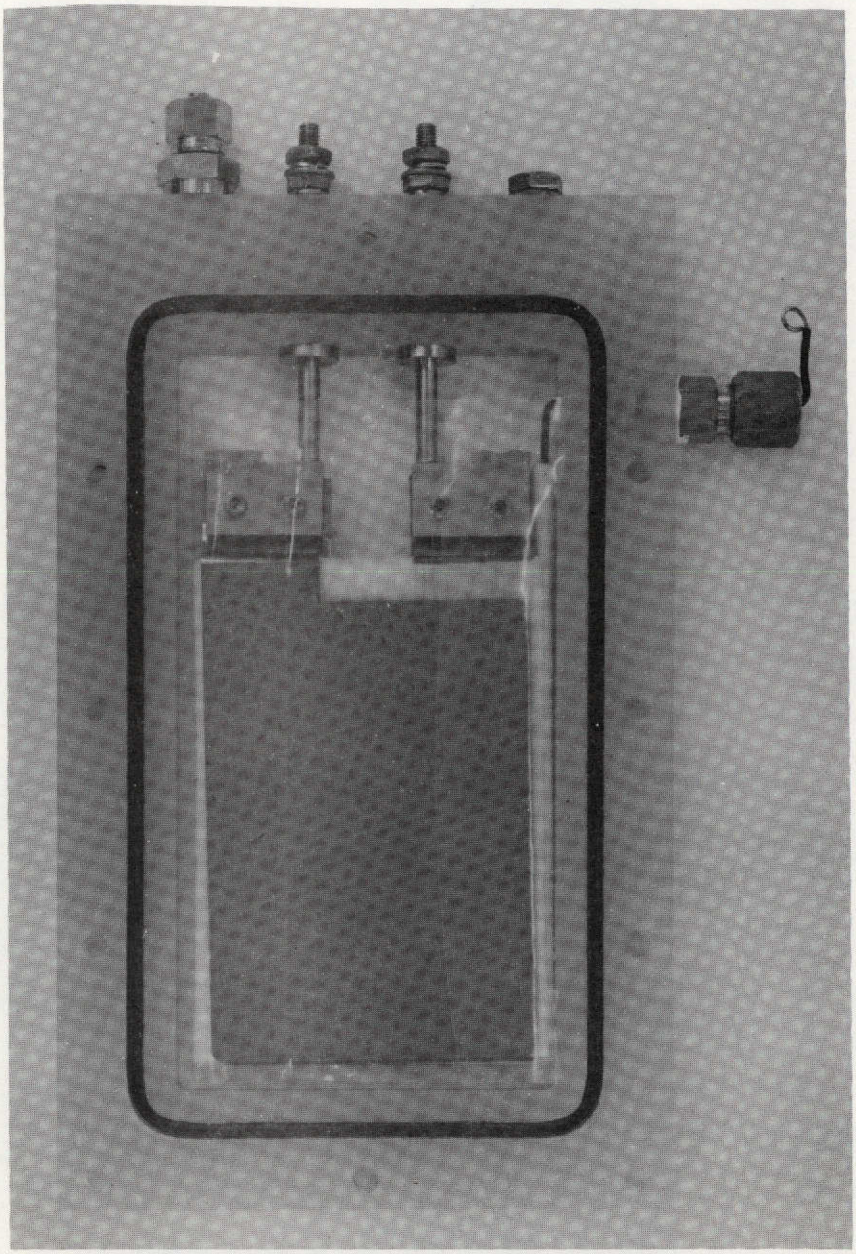

Figure 31. Re-usable Cell with Faceplates Removed (TRW Photo No. 88256-71)

7. DISCUSSION

The rather wide range of brittleness and adhesion of uncompressed sinter to the grid, and of thickness reduction in coined areas, evident in the plate materials tested appear to explain the range of opinions on the merits of coining revealed by the survey mode. For example, Table 2 shows that the thickness reductions in coined areas on positive plates ranged from 10 percent to 65 percent. With reference to Table 3, it seems clear that a reduction in thickness of 10 percent in a plaque having an initial density of 2.12 g/cm^3 (resulting in a calculated coined density of 2.36 g/cm^3) will produce an increase in strength in coined areas which is much less than that resulting from a thickness reduction of 65 percent in a plaque with an initial density of 1.95 g/cm^3 (calculated coined density of 4.34 g/cm^3).

On the other hand, there are some disadvantages to compressing too much. The problem to the cell manufacturer is that the force required, and hence the size and power of the equipment required for coining at practical production speeds, increases rapidly as thickness reduction increases. From the reliability standpoint, one must consider that the shoulder between coined and uncoined sinter becomes more highly stressed as thickness reduction increases, and the degree of cracking along the shoulder increases. The seriousness of this cracking (prior to impregnation) is not known at this time. It does appear that the stress is a function of the sharpness of the transition between the two thicknesses, and hence stress and cracking could be reduced by providing a more gradual transition from uncompressed to compressed material.

There does not appear to be a correlation between experience with edge problems and plate thickness or density of the uncoined materials. The range of these variables is quite small, however, as shown in Tables 2 and 3.

The void fractions of plates from Suppliers D and F are significantly greater than those of plates from the other suppliers. These two types were electrochemically impregnated, whereas the others were immersion impregnated. As these materials are newly developed, there was no experience data available on plate degradation. Testing on this program has not begun on these plates.

Relative resistance values varied over a range of three to one for materials from different sources, as shown in Table 4. It has been shown[1] that the sinter porosity of unimpregnated plaque is directly proportional to resistivity for a single type of plaque. The data in Tables 3 and 4 do not indicate that this proportionality applies when plaques from different sources are included in the sample, although the material from Supplier C is the only one out of line. This material has a resistivity that is only 50 percent of that predicted by the trend of the other materials tested. The reader is reminded that the void fractions shown are based on total plate apparent volume, and hence are not "sinter-only" values as are sometimes used in the laterature.

The resistance data in Table 4 show that the resistance of the positive plate material increases in going through impregnation and preliminary formation, with the least increase being 2 percent (Supplier D) and the greatest being over 50 percent (Supplier B). Since this increase is due primarily to corrosion of the sinter, and such corrosion reduces sinter strength, greater resistance changes are expected to correlate with low physical strength and low resistance to decomposition under cycle conditions. The tests planned should verify this prediction.

The various tests for brittleness of the sinter and adhesion of sinter to the grid performed to date tend to show that plate materials made with all-nickel grids are superior in these respects to those made with nickel-plated steel grids. This is particularly the case for adhesion. Although the same degree of cracking of the sinter per se may have occurred in each case for some plates under severe bending conditions, little appreciable detachment occurred from all-nickel grids relative to that which occurred from plated steel. This difference is illustrated by Figures 3 through 6. It has been suggested that the bond between the plating and the mild steel may be breaking in the latter materials; this interface does not exist in the former type.

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[1] Final Report: "Development of Uniform and Predicted Battery Materials for Nickel-Cadmium Aerospace Cells," Contract No. NAS5-11561 (1972), pp. 17-27.

It thus appears from the results obtained to date that the program and test methods described herein will demonstrate significant differences between plate materials having different properties, and between coined and noncoined edges, with respect to ease of handling during plate and cell manufacturing and extent of decomposition in a working cell. This information should prove helpful in specifying and producing cells having higher reliability for critical applications.

8. TENTATIVE CONCLUSIONS

Based on the results of work done to date, the following tentative conclusions are submitted subject to confirmation by tests and analysis remaining:

1) Although coining may not be essential to the physical integrity of sintered plates, it does provide greater resistance to damage during plate manufacturing, handling, and cell assembly.

2) Coining is needed more on positive plates than on negative plates due to the more brittle nature of positive plates.

3) The degree of benefit to be expected from coining depends largely on how brittle and weakly adherent the sinter structure is. These two characteristics tend to vary together.

4) Cracking of sinter along cut edges can be greatly reduced by proper coining and cutting only in coined areas. This is particularly important when plates are die-cut by automatic machinery. Although it is less true for hand-cutting, tools must be kept sharp, clean, and finely adjusted to prevent cracking when uncoined plate is cut.

5) The problem of edge damage produced by cutting is minimized when plates are cut to final size before impregnation, and not cut thereafter. Such a procedure does not lend itself to volume production, however.

6) Electrochemical cycling produces disruption of uncompressed positive plate material under the same conditions adequately coined material remains intact.

7) A reduction in thickness of at least 33 percent of the uncompressed value appears necessary for adequate protection against mechanical and electrochemical damage of positive plates.

8) If plaque is coined too close to the edge of the coated (sintered) portion of the strip, compression of the outer edge at the top of the plate may not be sufficient to prevent subsequent edge damage due to cycling.

9) "Edge-coating" or "cementing" can be beneficial if done properly. Presently used formulas are of questionable value, as they are too brittle, not sufficiently adherent, and hence not long lasting in the internal cell environment.