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FORMING ALUMINUM FOR SOLAR ENERGY CONCENTRATORS

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QUARTERLY PROGRESS REPORT NO. 2 FOR THE PERIOD 1 JANUARY THROUGH 31 MARCH 1964

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1.0 SUMMARY OF WORK

Fourteen aluminum electroforming runs were completed during the second quarter. The immediate purpose of these was: a) the refinement of the parametric and design data described in the previous quarterly report; b) preparation of the contractually required test specimens; c) check-out of the full process as designed for the scaled-up (30" mirror) equipment; as well as d) to gain more operating experience.

The basic soundness of the process was visibly demonstrated by the successful forming of a 5" aluminum paraboloidal mirror on first try, in our laboratory cell, using conditions closely approximating those anticipated in the scaled-up equipment. Subsequently, a 7-inch aluminum parabolic mirror and two $2\frac{1}{2}$ " hemispherical mirrors were electroformed. Electroformed nickel masters were used in each case. The excellent surface replication and lack of visible distortion not already present on the masters are evident in the photographs contained within this report. The separation of the electroforms from the masters proceeded smoothly, without applying mechanical force.

Physical property measurements of electroformed aluminum specimens were completed and appear within this report. The mechanical property data was applied to the structural equations given in the previous report.

To date, approximately 44% of the initial aluminum content of the laboratory plating solution has been deposited out. This percentage represents sufficient aluminum to electroform over thirty 30-inch mirrors from the large size plating bath. Close control and strict bath maintenance made this extended plating life possible. These procedures have been made part of the equipment design and operating procedures.

All equipment and appurtenances required to electroform the 30-inch mirrors were defined, work statement and specifications were prepared and sent to three potential vendors for quotation on its fabrication. Bart Manufacturing Company was selected to fabricate the 30-inch mirror electroforming equipment from our design and with our close direction. Detailed blueprints are nearly completed. The materials of construction, valves, piping, components, etc., have already been ordered to avoid delay.

Component support structures have been designed, and drawings were issued. A sample epoxy spincasting was made to re-test this process equipment.

All program efforts are on schedule and within their allotted budget.

2.0 MAJOR PROBLEMS

No major problems affecting the anticipated schedule, cost or results are foreseen at this time. A minor problem is the lack of suitable procedure for the removal of the thin silver film (parting layer) from the aluminum. Though this film is highly reflective and spotless, it is likely to tarnish in time. Commonly used silver removers are not applicable, because they affect the underlying aluminum.

3.0 WORK CONTEMPLATED FOR THE NEXT MONTH

During the coming report period, the spincast support structure will be built. All detailed drawings for the aluminum electroforming equipment will be completed, and its fabrication will begin. Additional runs are planned in the laboratory cell to determine the bath exhaustion point and further refine the techniques employed.

4.0 TECHNICAL STATUS

4.1 Preparation of the Aluminum Plating Solution

Aluminum plating solutions were parepared by the procedure reported by Beach and Faust (Ref 1) - the addition of aluminum chloride and the hydride to diethyl ether. The practical advantages of this procedure, as compared to the addition of ether to AlCl₃ and LiAlH₄, described previously (Ref 2), are the improved cooling and agitation characteristics of a solution vs. a slurry. Also by controlling the AlCl₃ additions to the bath, this highly exothermic reaction (heat of solution 580 calories per gram) is more easily kept under full control. Data, observations and handling techniques developed by the laboratory preparation of this solution will be useful in preparing the 200-gallon plating bath for electroforming the two 30-inch mirrors.

4.2 Effective Life of the Aluminum Plating Solution

The 3.5 liter plating solution, prepared prior to 1 January, was used to electroform the mechanical and optical test specimens contractually required during this report period. Subsequently, an additional 2 liters of plating solution was prepared separately and combined with the above bath. This 5.5 liter (approx 1½ gallon) plating solution was used to electroform some prototype geometries and for runs to further refine some of the process parameters. At this point, 46.7% of the initial aluminum content of the plating solution had been deposited, and the aluminum deposit is still low stressed and ductile. It should be remembered that the first 1.6 liter plating bath yielded highly stressed, unsuitable electroforms after 22.2% of the initial aluminum content had been removed. The extended plating life of the present solution is attributed to the close control and maintenance of the electrolyte during these runs. These techniques have been incorporated into the 30-inch electroforming equipment design and operating specifications.

A time photograph of the electrode potential (Fig 1), measured on an oscilloscope, taken after power interruption of this last run, shows that the plating solution moisture content is still sufficiently low (seen by the instantaneous depolarization of the electrodes). The excellent results confirm the validity of this procedure. Additional runs are planned to measure the plating bath life under present maintenance techniques.

4.3 Regeneration of the Aluminum Plating Solution

The initial plating bath, after five weeks of intermittent use, showed signs of exhaustion, as described previously (Ref 2). In the course of a study aimed at developing bath maintenance and control techniques, this plating solution was restored by the addition of $0.1 \text{M} \text{ LiAlH}_4$, after which the aluminum deposits resumed their previous low stress-ductile characteristics. Oscilloscope photographs, taken at current interruption, show the depolarization time before and after the hydride addition (Figures 2A and 2B, respectively). The depolarization time was restored to zero, illustrating the merits of this procedure for bath control.

This initial plating solution was subsequently sealed and returned to storage, and the runs proceeded with the new, larger volume bath.

4.4 Deposited Aluminum Electroforms

4.4.1 Flat Plates

Flat aluminum plates were electroformed over electroformed nickel tools which had been plated on static-poured GE epoxy (spincast formulation). Flat plates produced ranged in size from 2 square inches to 24 square inches, and in thickness from several thousandths to 0.029-inch. The plating solution was contained either within a circular glass tank or two different-sized rectangular glass tanks during these runs. The electroformed plates lacked visible distortion, were highly reflective and exhibited excellent replication of the master surface.

4.4.2 5-Inch Parabolic Mirror

All aluminum specimens electroformed to this point were flat plates. An electroformed nickel master was secured to attempt electroforming a parabolic aluminum mirror by bringing it through the entire planned process (pretreatments, residence times, geometry, etc.). The data from this run was regarded as an essential feature of our laboratory study. The result (a 5-inch parabolic mirror) was highly pleasing, and is presented pictorially in Figures 3A and 3B.

The surface replication was excellent. The reflected images (i.e. florescent lights, camera, ceiling beams, etc.) indicate a distortion-free surface resulting from the process. A conforming anode was not used, therefore, the throwing power appears surprisingly high (uniform thickness!). A conforming anode will be used for the large mirrors. After about a month's storage in a desk drawer, the silvered surface shows some tarnish.



4.4.3 2½-Inch Diameter Hemispherical Mirrors

A $2\frac{1}{2}$ -inch diameter hemispherical electroformed nickel master was used to electroform two $2\frac{1}{2}$ -inch diameter mirrors. The results were comparable to that for the 5-inch parabolic mirror and is presented pictorially in Figures 4A and 4B. The reflected images indicate a very good replication.

In Figure 4A, the left mirror was made without periodic reverse cycling, therefore, increased nodulation occurs on the back surface. On one of these mirrors the silver was removed by gentle mechanical polishing (impregnated cotton), but this ruined the specularity.

4.4.4 7-Inch Parabolic Mirror

The $1\frac{1}{2}$ -gallon plating solution was brought up to 2 gallons to accommodate a 7-inch male electroformed copper master, coated with nickel. The quality of the nickel surface was rather poor with some scratches, nicks, pits, and show-thru of copper leak visible; however the geometry was acceptable. The master was secured at no cost to the program from Bart Manufacturing's scrap pieces. Despite these shortcomings, the scale-up data gained justified this run. These defects are less noticeable on a convex surface and are not well visible in the photograph (Fig 5B).

The results were, once more, highly pleasing, although faithful replication of the defects marred the aesthetic appeal. The 7-inch parabolic mirror is shown pictorially in Fig. 5A. The reflected images (florescent lights, camera, ceiling beams, the authors of this report, stripes in tie, miscellaneous objects) indicate surface free from visible distortions. A conforming anode was not used, but the uniformity of deposit was good.

The thickness of the deposit exceeded 0.030 inch.

One important lesson learned from all these mirror runs was that the larger nodules on the rear surface show through to the front. Rotation, agitation and conforming anodes will prevent the formation of nodules on the 30" mirrors.

4.5 Materials Compatibility

In an effort to reduce fabrication costs in the 30-inch electroforming equipment, the compatibility of selected standard materials was investigated. High cost of glass-lined equipment recommended this study. The first phase was reported previously (Ref. 2, 4). The results of this concluding study appear in Table 1.

The data cited in Table 1, in conjunction with previous data (Ref 2), enabled the materials of construction specifications to be drawn up.

5.0 PHYSICAL PROPERTIES OF ELECTROFORMED ALUMINUM

Electroformed aluminum sheets were deposited from the plating bath on an electroformed nickel substrate under conditions which will be employed in fabricating the two 30-inch mirrors. All the test specimens were machined from these sheets, as described below.

5.1 Mechanical Properties

5.1.1 Data from Stress-Strain Curves

5.1.1.1 Test Specimens

Four standard tensile specimens, with a reduced cross-section of 0.406 inch x 0.026 inch, were machined from 0.028 inch electroformed aluminum plate. One specimen was used to determine the proper stress and strain magnification scales. Data from the remaining three are reported below.

5.1.1.2 Test Equipment and Procedure

Stress-strain curves (Ref 3) were obtained from tests conducted on a Floor Model TT Instron Testing Machine, using an F-cell for load pick-up. The cell was adjusted to 200 pounds full chart reading. Strain was measured with a Tinius-Olsen S-3 extensometer with a one-inch gauge length, adjusted to produce a 125x magnification. The total elongation was measured over one-inch gauge marks with a vernier calipers. The strain rate of each test was approximately 0.05 inch per inch per minute at the start of the test.

5.1.1.3 Test Results

Data reduction of the stress-strain curves yielded the following average values: Modulus of Elasticity - 7.98×10^6 psi; Tensile Yield Strength @ 2% Offset - 7810 psi; Ultimate Tensile Strength - 11050 psi; and Elongation - 26%. All mechanical property data are tabulated and compared with handbook values of 2% aluminum in Table 2. A typical tensile stress vs. elongation curve for electroformed aluminum appears in Figure 6. The actual Instron run data has been replotted for one specimen to provide a reproducible copy and appears in Figure 7. The data, as run on the original chart paper, is on file at RSD and is available on request.

5.1.2 Thermal Expansion

5.1.2.1 <u>Test Specimens</u>

Four $3\frac{1}{4}$ -inch long, $\frac{1}{2}$ -inch wide specimens were machined from 0.28-inch electroformed aluminum plate. One specimen was used for set-up, therefore, data for the remaining three specimens are reported.

5.1.2.2 Test Equipment and Procedure

The more common dilatometer method of measuring thermal expansion was impractical for the soft, thin, 0.026-inch electroformed aluminum specimens. Therefore, an alternate method was devised. A model KE-2 Keuffel and Esser Theodolite was mounted at a known distance from the specimen to measure the angle subtended by a 3-inch gauge length of the electroformed aluminum specimen. The change in gauge length was calculated from the measured change in the subtended angle which resulted from the temperature variation. The accuracy of the theodolite is within one-half second of arc.

The test specimens were taped to an aluminum block to prevent buckling. However, the adhesive side of the tape was covered where it could exert any restraint on the specimen. The specimens were placed in a Missimer High-Low Temperature Chamber and sighted during test through a 10-inch x 10-inch viewing port.

The selected temperature range of $-70^{\circ}F$ to $+200^{\circ}F$ was divided into three increments: $-70^{\circ}F$ to $0^{\circ}F$; $0^{\circ}F$ to $+100^{\circ}F$; and $+100^{\circ}F$ to $+200^{\circ}F$.

Measurements were made 30 minutes after the specimen temperature had stabilized for each temperature cited above. A shielded thermocouple mounted on the gauge length surface of the specimen monitored the temperature during these tests.

5.1.2.3 Test Results

An average value of 13.6 in/in/^OF over the -70^OF to +200^OF temperature range was measured for the linear coefficient of thermal expansion of electroformed aluminum. These data are tabulated in Table 2.

5.1.3 Density

5.1.3.1 <u>Test Specimens</u>

Two specimens were deemed sufficient because of the routine and simple character of the density determination.

5.1.3.2 <u>Test Equipment and Procedures</u>

The density of the electroformed aluminum specimens was determined by the standard ASTM method. All weighings were done with an analytical balance accurate to within 0.1 milligram. The specimen volume was determined by measuring the water displacement weight of the sample.

5.1.3.3 Test Results

Values of 97.4 and 98.9 percent of theoretical density were measured for the two electroformed aluminum specimens. These data appear in Table 2.

5.2 Optical Properties

5.2.1 Specular Reflectivity

5.2.1.1 Test Specimens

Flat aluminum plates, approximately 2 3/8-inch x 2 3/8-inch x 0.016-inch thick, were electroformed over an electroformed nickel tool. Deposition of the aluminum was by our optimized operating conditions which are part of our large equipment design. One electroformed aluminum specimen was mechanically polished. This electroform, a second specimen in the as-removed condition, and the nickel master from which these replications were made, were sent to Libbey-Owens-Ford for vacuum coating with aluminum and silicon monoxide, successively. These specimens were required to re-test previous samples, which were vacuum coated with an excessive (pink) thickness of SiO in-house. The specular reflectivity data of the latter specimens have been reported previously (Ref 4) and will not appear in this report.

5.2.1.2 <u>Test Equipment and Procedures</u>

The specular reflectivity of the three repeat optical test specimens is measured at $100^{\circ}F$ over the spectrum of 0.3 to 7.0 microns. A Beckman Model DKIL Reflectometer is used to determine the specular reflectivity in the 0.3 to 2.7 micron spectral range. For the 2.0 to 7.0 micron spectral range, the optical test specimen is measured in a Perkin-Elmer Model 205 Reflectometer. Reflectivity of an aluminum mirror standard is simultaneously determined to enable calculation of absolute values for each specimen.

5.2.1.3 Test Results

The specular reflectivity of the electroformed aluminum specimens and nickel mirror appears in Table 3. These data indicate that mechanical polishing of the optical surface prior to vacuum coating markedly reduced the specular reflectivity in the 0.3 to 1.5 micron wave length range. At the low wave lengths (0.3 to 0.4 microns) the reflectivity of the non-polished electroformed aluminum specimen was 14-16% lower than the nickel master. For the remaining spectral range, the reflectivity for electroformed aluminum was comparable to that of the nickel master.

Liberty Mirror indicated that the silver film is not a desirable substrate for their coating process.

6.0 <u>ANALYSIS OF RESULTS</u>

6.1 Storage of the Plating Solution

Anticipating that the plating solution will have to be prepared 1-2 weeks prior to actual deposition and stored while various components and systems are checked out, one prepared solution was sealed and stored in the laboratory for one

month prior to use. Aluminum electroforms, which were deposited from this stored plating bath, did not differ from specimens deposited from freshly-prepared solutions. Therefore, the anticipated 1-2 weeks bath storage time should present no problems.

6.2 Adhesion of the Electroform to the Cathode

Successful electroforming requires that the deposit exhibit sufficient adhesion to prevent premature parting during deposition, and still permit easy separation at the conclusion of the run so that the electroform need not be pried off and thus distorted. A breakthrough was achieved by developing a simple treatment procedure, which consists of an oleic acid-isopropyl alcohol solution immersion of the cathode substrate prior to deposition. This procedure effectively controls the adhesion of the electroform to the cathode and can be varied from poor to inseparable by utilizing different concentrations of oleic acid. This contrasts the complex, costly techniques found in the literature. Details have been described in Reference 2.

In a further development, this procedure was somewhat modified to make it more reproducible and applicable to the 30-inch mirror electroforms. This modification consists of applying to the substrate a chemically-reduced silver film (approx. 4 millionths of an inch thick) for easy parting and, subsequently, an oleic acid solution dip formulated for maximum adhesion. The parabolic and hemispherical mirrors were prepared in this manner. The drawback of this method is the adherence of the silver film to the electroformed aluminum; however, it permits distortion-free separation of the soft aluminum deposit, which is quite significant when considering the problems generally encountered in pulling off the much more rugged nickel electroforms from their masters.

6.3 Current Density

The character of deposit for a range of current density (0 to 140 asf) was investigated utilizing General Electric's proprietary Schmidt Stress Cell. The deposit from one run permits evaluation of the entire current density range cited above. A relatively wide current density range (15 to 35 asf) was found to be suitable for our requirements. Deposition of the 30-inch mirror will be at 20 asf.

6.4 Current Reversal

The smooth deposits which can be achieved with current reversal have been reported by Couch and Brenner (Ref 6). Our investigation came to the same conclusion. A 294 seconds plating - 6-seconds deplating time cycle produced excellent results for our process conditions. Short deplating cycles were not effective. The 2% (6 seconds) deplating cycle proved optimum in preventing nodular growths, while not greatly reducing the overall plating efficiency.

The effect of not using current reversal in electroforming "thick" deposits can be seen pictorially in Figures 4A and B. A special current reverser was designed and assembled for the laboratory runs, and this same unit will be used in the large scale equipment.

6.5 Deposition Temperature

The plating current heated the plating solution during deposition. It was found that the character of the aluminum deposit was not particularly temperature dependent. However, cooling of the plating bath is desirable to reduce ether volatilization losses and was, therefore, included in the equipment design.

6.6 Electrode Spacing

An electrode spacing of $2\frac{1}{2}$ inches was established for electroforming the 30-inch mirror. The plating solution resistivity, current distribution, agitation, minimum volume of solution, etc., were taken into account in deriving this separation. The parabolic and hemispherical mirrors were electroformed with this spacing.

6.7 Agitation of the Plating Solution

Mechanical agitation was observed to stir up anode sediment, making filtration more difficult and resulting in a roughened deposit. However, agitation, particularly during deposition of a large size electroform, is mandatory. Therefore, the filter return line to the large plating tank will be located so that the filtered solution will impinge on the male nickel master.

A slow rotational motion of the male nickel master will provide additional agitation and serve to make the deposited thickness more uniform. This has been included in the equipment design.

6.8 Filtration

Filtration is mandatory during the deposition of the aluminum mirrors, as indicated by comparative runs in our cell. This contradicts the conclusions of Conner and Brenner (Ref 5). With all other parameters held constant, failure to filter the plating solution during deposition results in a rough nodular deposit when the aluminum deposit exceeds 0.008 to 0.010-inch without current reversal, or exceeds 0.012 to 0.014-inch with current reversal. With repeated filtration during deposition, the number and size of nodules sharply decreased when the proper current reversal cycle was employed, even at deposit thickness in excess of 0.30 inch. Continuous filtration will be used in the large equipment.

During a recent visit to Metal Hydrides Incorporated, Beverly, Mass., a self-built filtration unit, identical in principle to our own home-made laboratory filter and our scaled-up filter unit design, was observed. Metal Hydrides filters an ethereal solution similar to our plating bath without difficulty. Although no modifications in our original design were necessary, it was encouraging to know that the scaled-up filtration unit would not provide any difficulties, and that others have arrived at the same conclusions and design as we did.

6.9 Thickness

To date, our aluminum electroforms have been deposited to thicknesses of 0.029-inch for a flat plate and in excess of 0.030-inch for a 7-inch parabolic mirror. Brenner (Ref 5) recommended the addition of methyl borate to the plating solution to attain such thick deposits; however, bath additives, like most bath contamination, increase the stress levels in the aluminum deposits, and we will not use any.

6.10 Plating Bath Control

A small secondary plating cell, independent of the 30-inch mirror, has been included in the design and will be utilized for periodic stress checks during the deposition period.

6.11 Simulated Power Failure

In electroforming the nickel master for a $9\frac{1}{2}$ -foot nickel mirror, a power failure occurred during deposition. The interruption of current is particularly critical in aluminum electroforming, and it invariably causes laminations in the deposit. Power interruptions of varied duration were evaluated in conjunction with several corrective procedures. Long power shut-downs and removal of the electroform from the bath proved particularly detrimental. To prevent such mishaps, an auxiliary power source is now incorporated into the design, which will maintain a small negative potential on the master in case of power failure. This technique was demonstrated in a run, when the current density was reduced to a trickle for 8 hours. The deposition run was later resumed, and the resultant electroform was devoid of laminations.

6.12 Cell Voltage Balance

The polarization voltage, the plating solution conductance, etc., were determined as parts of an overall cell voltage balance (Ref 2) to enable the determination of electrode spacing, cell geometry, and the selection of a suitable power supply. The values found did not significantly differ from those in the literature.

6.13 <u>Cell Volume</u>

To date, our initial plating solution volume of $1\frac{1}{2}$ liters has been scaled-up to $7\frac{1}{2}$ liters (approx 2 gallons). The electroforming operating conditions have remained predictable during the cell volume and specimen size scale-ups.

6.14 Mechanical Properties of Electroformed Aluminum

The laboratory test data from the mechanical property tests were applied to the structural analysis for the electroformed aluminum mirror. The calculations indicate that the stress and deflection are so small in the mirror under a 10g thrust load, that an even thinner mirror skin than was estimated originally would be structurally sufficient. The calculations appear in the appendix of the report.

7.0 DEFINITION OF THE 30-INCH MIRROR ELECTROFORMING EQUIPMENT

A flow diagram, showing all equipment and process line requirements for electroforming the 30-inch mirror, was prepared. This was based on lab results, and considering all available information (experimentally re-checked, where doubtful) in the published literature, and after discussions with vendors and people experienced in this or like processes. A schematic of the electroforming equipment appears in Figure 8.

7.1 Requirements from Vendor

The vendor's responsibility is the fabrication and installation (at a site provided by the vendor) of the aluminum electroforming pilot plant equipment and appurtenances, shown in Figure 8, with the additions described below. Two carbon steel tanks, to be utilized for the pre-treatment of the male nickel master, are required. Required utilities (water, drains, electrical connections, etc.) were specified. A leak-tight specification (an initial 2.5 psi pressure should not drop below 2.0 psi in 30 minutes) was established. A time schedule was specified (not to exceed 3½ months for fabrication and checkout of the equipment). Manpower will be supplied by the vendor for the duration of the aluminum electroforming operation (2 mirrors). This manpower will be operating under GE's direction.

7.2 Selected Vendor

A work statement with equipment specifications was sent to three vendors requesting a quotation for the fabrication and installation of the 30-inch mirror electroforming equipment as designed by RSD.

A quotation was requested from Battelle Memorial Institute, Columbus, Ohio, even prior to the start of this program. Battelle, which has engaged laboratory investigation of this process (aimed at cladding metals), declined to bid citing the high risk involved. The Advanced Technologies Laboratory of the General Electric Company, Schenectady, New York, had submitted a bid a year ago, but rescinded it because of an increased work load from outside agencies (primarily NASA). (A number of other plating and electroforming companies declined to bid, because they did not consider aluminum electroforming feasible.) Bart Manufacturing Co. and General Technologies Corp. submitted bids, and Bart was the low bidder.

Bart Manufacturing, which has no aluminum electroforming experience, has considerable experience in general electroforming. Bart has a good engineering department, and their own equipment fabrication facility, in addition to a considerable pool of useful equipment from which they can draw, if necessary. Since Bart will be executing RSD's design and work under RSD's direction, their lack of direct aluminum electroforming experience is not objectionable. Therefore, Bart Manufacturing was selected on a technical and cost basis.

7.3 Process Description

Figure 8, a schematic of the aluminum electroforming equipment, should be referred to for the discussion of the planned operation.

7.3.1 Preparation of Plating Solution

Anhydrous diethyl ether will be pumped by pressurized dry nitrogen into the argon-purged solution storage tank. Additions of solids are made from the glove box while a gas-driven stirrer agitates the solution. Cooling coils control the solution temperature during the exothermic reaction of solution. The solution will be recirculated into the storage tank through the filter before being pumped to the plating tank.

A condenser in the gas vent line was inserted to minimize ether volatilization losses. A pressure relief valve maintains the proper tank pressure. A rupture disc (silver foil) was included in a vent by-pass line as an emergency precaution.

7.3.2 Start-Up of the Plating Cell

The conforming aluminum anode, properly shrouded with glass cloth to retain the anode slime and minimize contamination of the plating solution, will be mounted in the empty plating tank. The solution return line from the filter passes through a centrally located hole in the anode. The plating tank will be purged with argon prior to start-up. The plating solution can now be pumped through the filtration system from the solution storage tank into the plating tank. The filtration system to the plating tank will be thus checked out before deposition. A plating tank cover will seal the tank, and a positive pressure will be maintained therein.

The glove box will then be unclamped and removed so that the male nickel master (cathode) can be attached to the hoist mechanism within the glove box. The entire assembly (male master within the glove box) is then placed in position over the plating tank, clamped down and purged thoroughly with argon. Sparkproof electrical connection is made to the cathode and anode, and the male nickel master is quickly lowered into the plating solution while the plating current is on. Current density is then adjusted to 20 asf, and the plating solution will start circulation through the filter unit.

The operation of the filtration apparatus has been described in previous progress reports. In this design, a pressure relief valve will be utilized to reduce the internal filter pressure. A vacuum pump was used during laboratory operation, but was difficult to control because of the boiling of ether in the vacuum cycle. The modification has proven better workable by test.

7.3.3 Operating Conditions During Deposition

The following plating parameters will be used during the aluminum electroforming operation:

Anode-cathode Parallel, at a distance of $2\frac{1}{2}$ ", the anode

to remain stationary.

Solution Aluminum chloride (3.4M), lithium aluminum

hydride (0.4M) in diethyl ether.

Temperature 20-30°C (heating by the plating current)

Filtration 50 gal/hr, continuous.

Agitation Provided by filtration and cathode rotation.

Cell Voltage 10-16 volts

Cell Current 110 to 160 amps

Direction, control tests and additions to be provided by RSD.

Agitation will be provided by the filtering system, which will impinge the solution on the cathode through a hole at the center of the anode.

In addition, a slow rotational motion will be imparted to the male master to provide additional agitation and obtain a more uniform thickness in the 30-inch aluminum electroform.

Periodic current reversal will be utilized. The planned cycle will be: Plating - 494 sec.; Deplating - 6 sec. RSD will supply the current reverser unit. Removal of a filter unit from the process line for cleaning includes a blow-off line, which will be used to purge the unit. Additions during operation will be made through the solution preparation tank.

7.3.4 Termination of the Deposition Run

At the conclusion of the deposition period, the plating current and filter operation will be halted. The male master with the adhering 30-inch mirror will be hoisted up into the glove box and permitted to drain into the plating tank. The plating tank cover is now replaced, and the solution is stored therein. (In an emergency situation, rapid removal of the plating solution from the plating tank can be made to either the storage tank or outside the plant (i.e., dumped into a deep ditch). Return to the storage tank could also be made through the filtration unit.)

The glove box assembly is now unclamped and hoisted away from the closed plating tank. The male master with aluminum electroform will be removed and set down near a water drain. The assembly will be washed with a heavy stream of water to react the adhering plating solution. After separation of the first 30-inch aluminum mirror, the male master surface will be prepared for electroforming the second mirror. Run start-up procedures will be as indicated previously.

7.3.5 Plating Electrical System

Current interruptions during deposition will cause laminations in the aluminum electroform. To protect the system against inadvertent current interruptions because of power failure, sluggish contactors, etc., an auxiliary power system will be installed. This consists of a DC battery connected to the electrodes. A pair of diodes prevent the primary plating power supply from discharging the battery. A series resistor limits the battery discharge to 2 amperes. In the absence of plating current, the battery automatically and instantaneously supplies a low current (2 amperes) to maintain the plating surface cathodic and prevent laminations from occurring when the primary current is restored. This technique was evaluated in the laboratory cell, and the resulting electroform was not laminated, proving the advisability if including this device in our design.

A schematic of the plating electrical systems appears in Figure 9.

7.4 RSD Responsibilities

RSD will maintain over-all charge and responsibility for the electro-chemical aspects and for the operation of the equipment. Design consultation will be furnished by RSD throughout the entire fabrication period. During the electroforming operation, all control tests and required additions (if any) to the plating solution will be handled by RSD.

In addition, RSD will provide the current reversal unit to be connected to the plating power supply, the necessary plating solution chemicals and additions for same, the electroformed nickel master, anodes, anode bags, electrode connections, surface treatment chemicals and solutions, as required.

Any changes or modifications by the vendor can be made only with the prior consent of RSD.

7.5 Present Status

RSD personnel, working closely with the vendor, Bart Manufacturing, have outlined in detail all aspects of the equipment design. Detailed blueprints are nearly completed; the materials of construction and component items have already been ordered.

8.0 MALE NICKEL MASTER

RSD proposed using one of several existing 30-inch male nickel masters, which would be loaned to this program. These male masters had rim angles higher (70-75°) than specified in the contract. After discussions with NASA-Langley technical representatives, it was concluded that continuation of the original plan would be more desirable.

8.1 Spincast Support Structure

The support structure for the spincast mold is fully described in Figure 10 (GE Drawing 243R306). All component parts have been ordered, and the structure will be fabricated during the coming month, on schedule.

8.2 Epoxy Spincasting

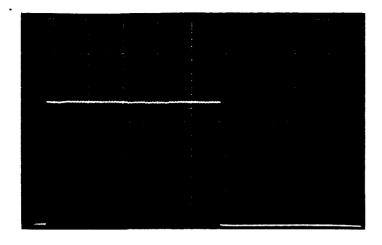
The spincast equipment was checked out in preparation for the 30-inch epoxy spincasting. A sample spincast mold, 10 inches diameter, was made. No defects, except for one visible bubble, were noticed. Refurbishing of the epoxy metering and mixing equipment has been started. The system has been entirely disassembled and cleaned.

8.3 <u>Male Master Support Structure</u>

The support structure for the male nickel master is fully described in Figures 11A, B and C (GE Drawing SKS 0250). Component parts have been ordered. The procedure for attachment to the male master is described in the attached work statement. This grown-in peripheral support was chosen because our solution would attack the plastic of full back-up structures used in previous nickel-mirror programs.

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- 2. Schmidt, FJ, "Forming Aluminum for Solar Energy Concentrators"; Quarterly Progress Report No. 1 (1 Oct 31 Dec 1963).
- 3. Smith, DE, "Tensile and Thermal Expansion Properties of Electrodeposited Aluminum"; PIR No. 8383-099, 23 March 1964.
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- Conner, JH and Brenner, A, "Electrodeposition of Metals from Organic Solutions"; J. Electrochem Soc, 103, p 657-662 (1956).
- Couch, DE and Brenner, A, "A Hydride Bath for the Electrodeposition of Aluminum"; J. Electrochem Soc, 99, p 234-244 (1952).





Time photograph of oscilloscope voltage measurement at the end of the 7-inch parabolic mirror electroforming run. At this point, 44.2% of the initial aluminum content of the plating bath had been depleted. Power interruption dropped the voltage to zero instantaneously.

Vertical scale: 2 volts/major div. Horizontal scale: 1/5 second/major div.

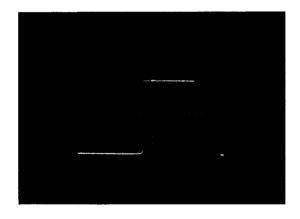


FIGURE 2A

Time photograph of oscilloscope voltage measurement at the interruption of a psuedo-deposition run in the "exhausted" initial plating bath. Vertical scale: 2 volts/major division. Horizontal scale: 1 second/major division. Power interruption dropped voltage to 0.6 volt (polarization voltage). NOTE: Voltage did not return to zero within the 4½ seconds recorded in the photograph.

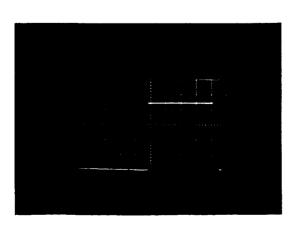


FIGURE 2B

Time photograph of oscilloscope voltage measurement at the interruption of a psuedo-deposition run after 0.1M LiAlH₄ was added to the initial plating bath shown above. Vertical scale: 2 volts/major div. Horizontal scale: 1 second/major div. Power interruption dropped voltage to zero. Deposits from this bath were again low stressed and ductile.

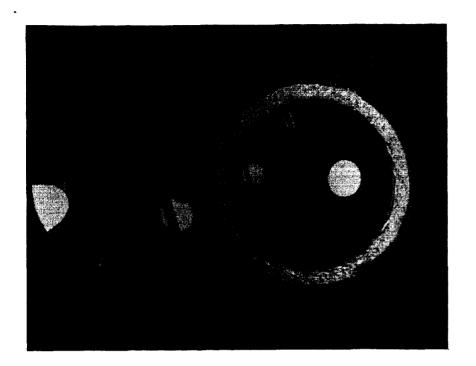


FIGURE 3A

First electroformed aluminum parabolic mirror (5-inch) on left; nickel master from which mirror was replicated on right. Note the undistorted reflections in the mirror (i.e. camera, florescent lights, flat rod held in hand, fingers, etc). Mirror surface is as removed from nickel substrate - unwashed, unpolished, etc.

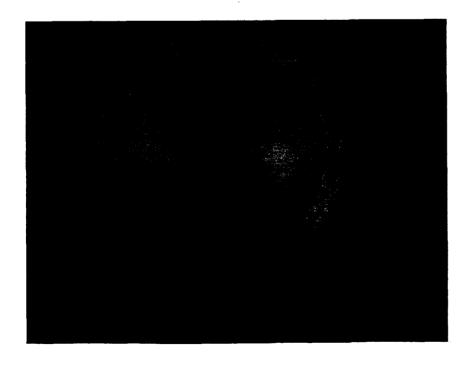


FIGURE 3B

Five-inch electroformed aluminum mirror (see Fig. 3A) prior to removal from nickel substrate. Note the smooth deposit and lack of nodular growths over the mirror portion of the deposit. Edge growths ("trees") are relatively minor as compared to nickel or copper electroforming.

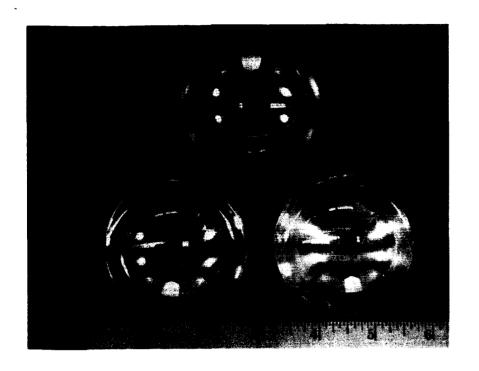


FIGURE 4A.-

First electroformed aluminum hemispherical mirrors (2½ inch dia). Bottom left mirror is as removed from electroformed nickel master above. Bottom right mirror was mechanically polished. Note undistorted images (i.e. camera, florescent lights, ceiling poles and beams, etc). Haze lines about incandescent lamps appear in mirror and master, demonstrating excellent replication.

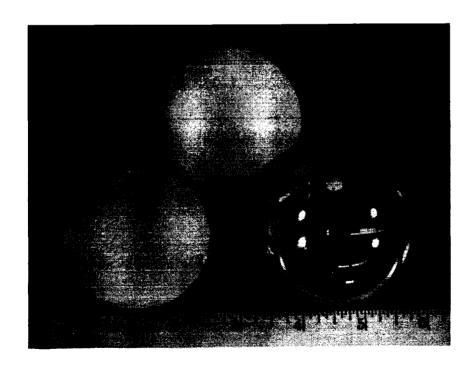


FIGURE 4B.

Back surface of the 2 hemispherical mirrors. Note the lack of nodular growths on the deposited surface. Electroformed nickel master is also shown.

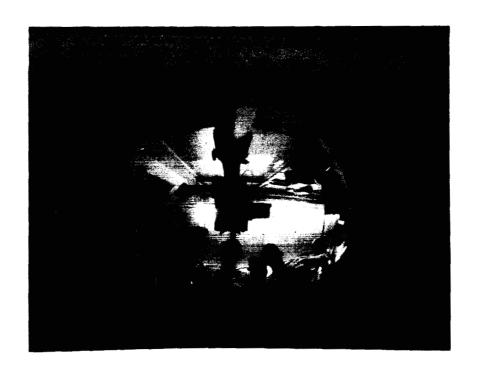


FIGURE 5A - 7-inch electroformed aluminum parabolic mirror replicated from nickel master shown in Figure 5B.

The aluminum mirror was electroformed using design parameters of the 30-inch mirror. Note the undistorted reflections in the mirror (i.e. camera, florescent lights, ceiling poles and beams, image of Dr. F. Schmidt and I. Hess, facial detail, stripes of tie, etc.). Mirror surface is as removed from nickel substrate.

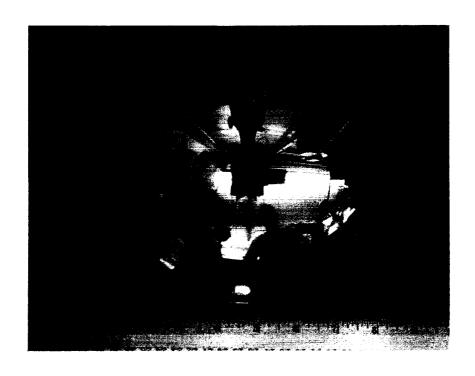


FIGURE 5B - Male Nickel Master - Used to electroform the 7-inch aluminum parabolic mirror shown in Figure 5A. Certain imperfections in the master are visible at 10 o'clock-position of this photograph.

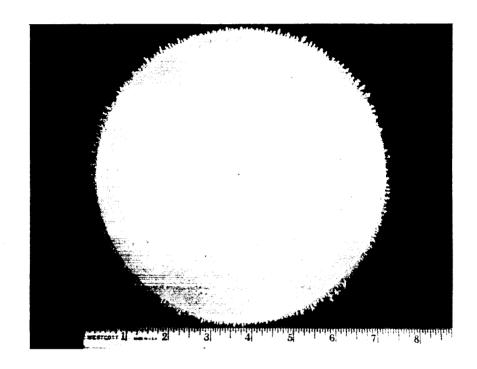
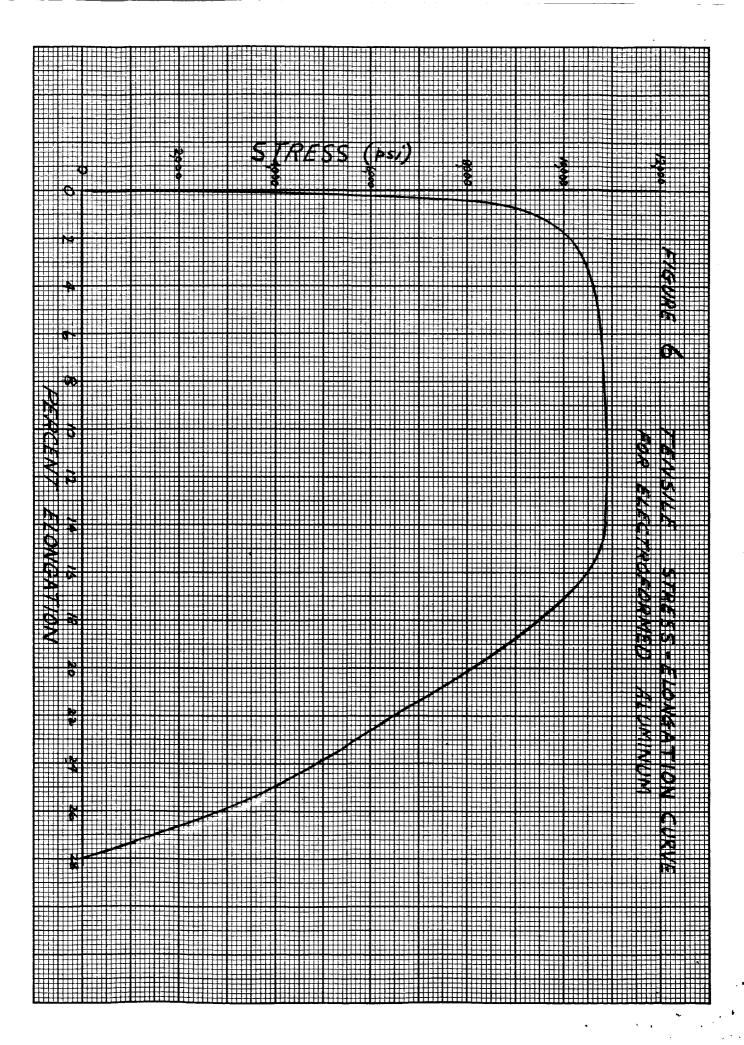
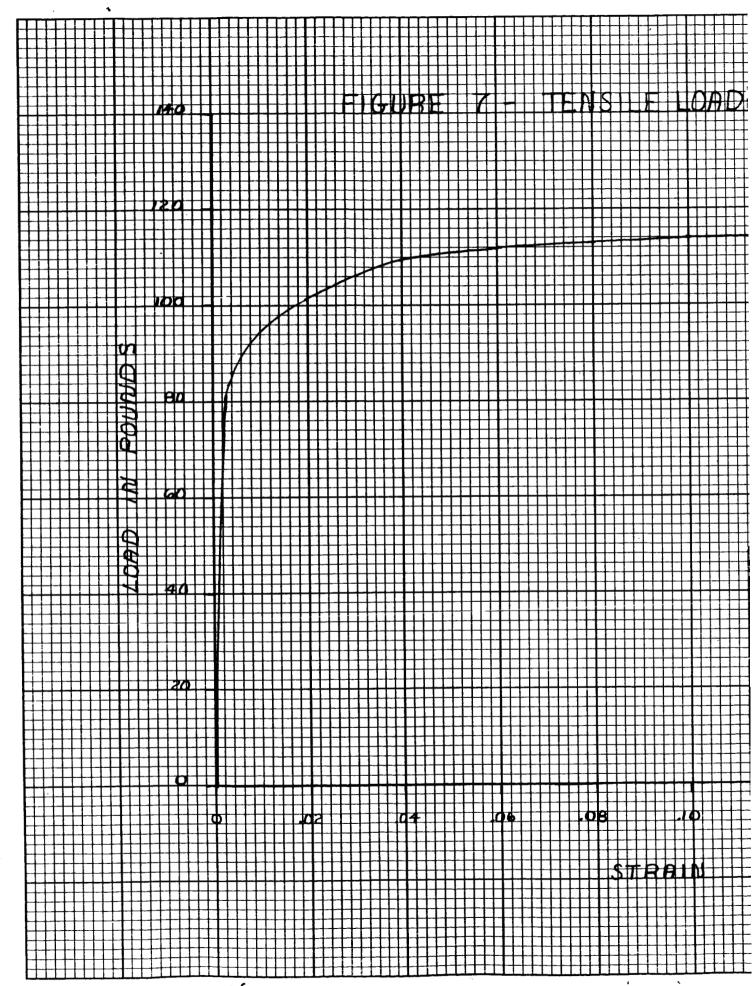


FIGURE 5C - Back surface of the 7-inch electroformed aluminum parabolic mirror prior to removal from nickel master.

Note the small edge growths ("trees") despite the 0.030-inch thick deposit.





Fi9 1

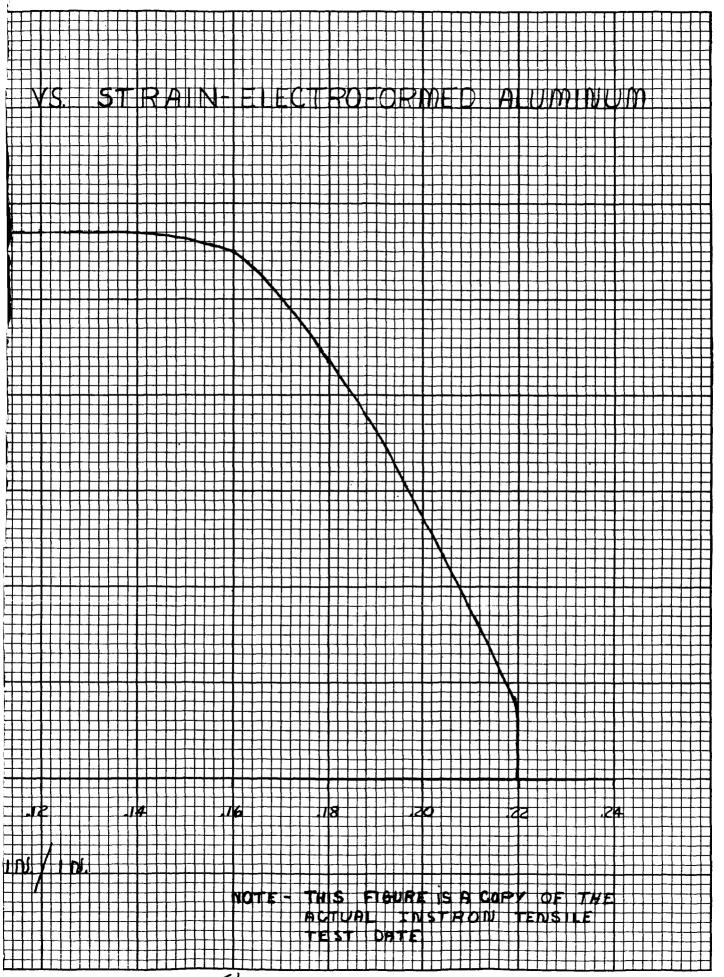


Fig 2

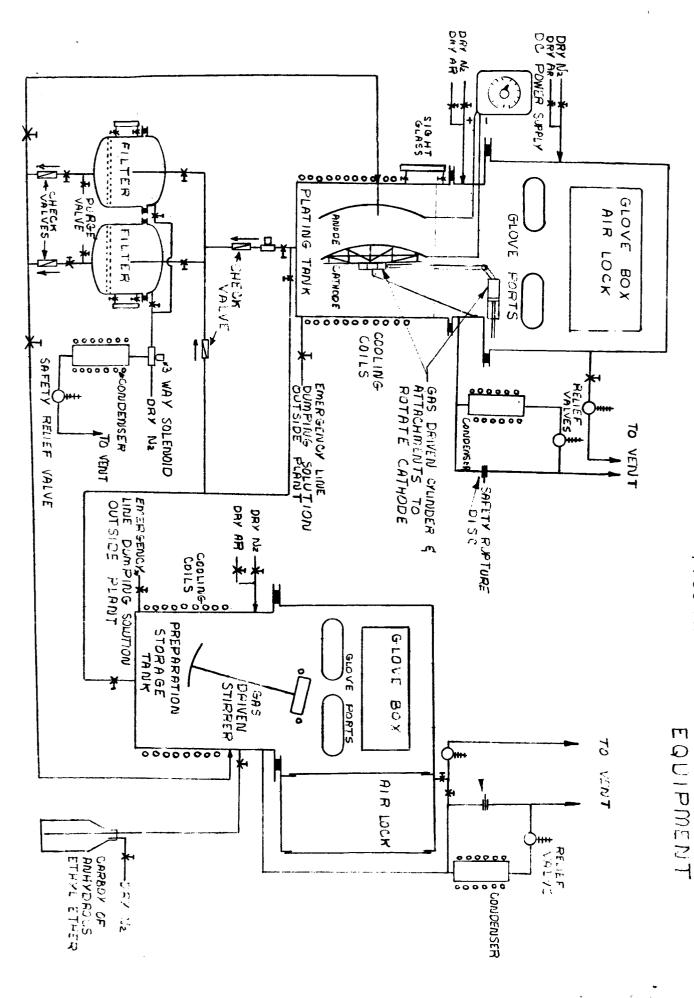
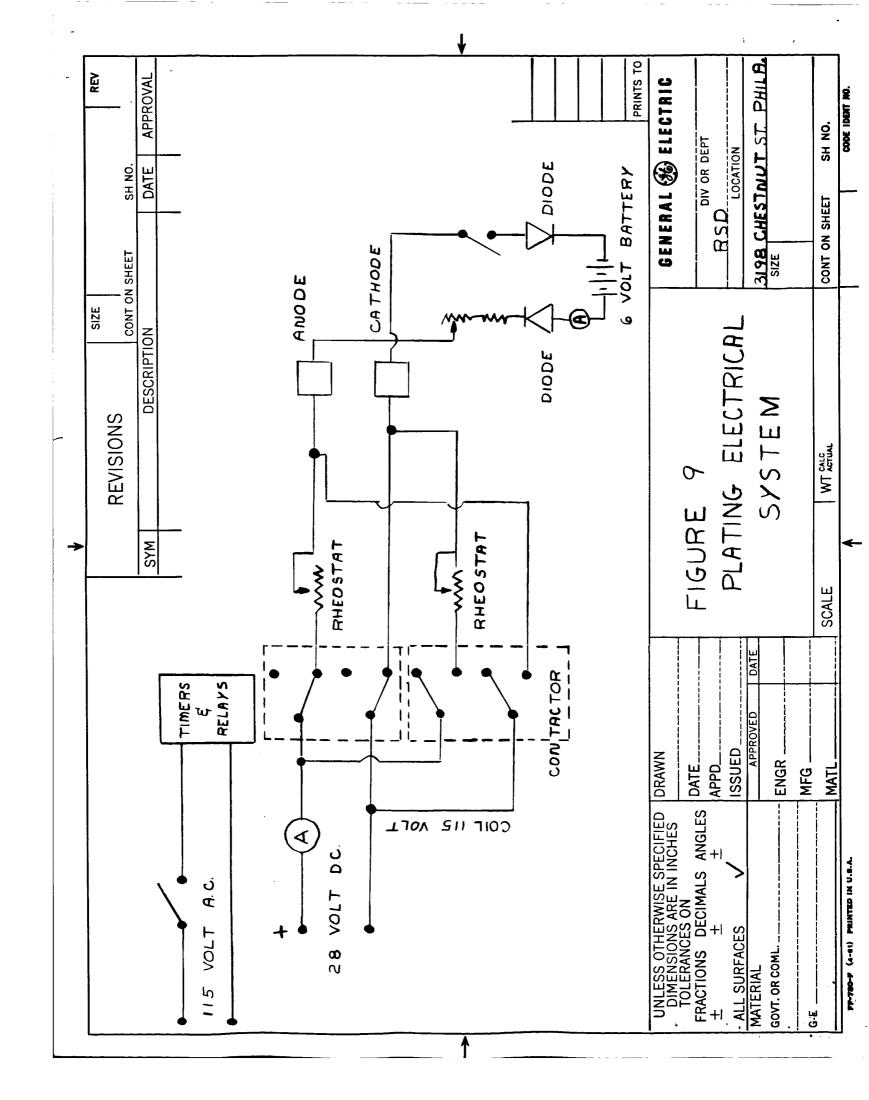


FIGURE 8 ALUMINUM ELECTROFORMING



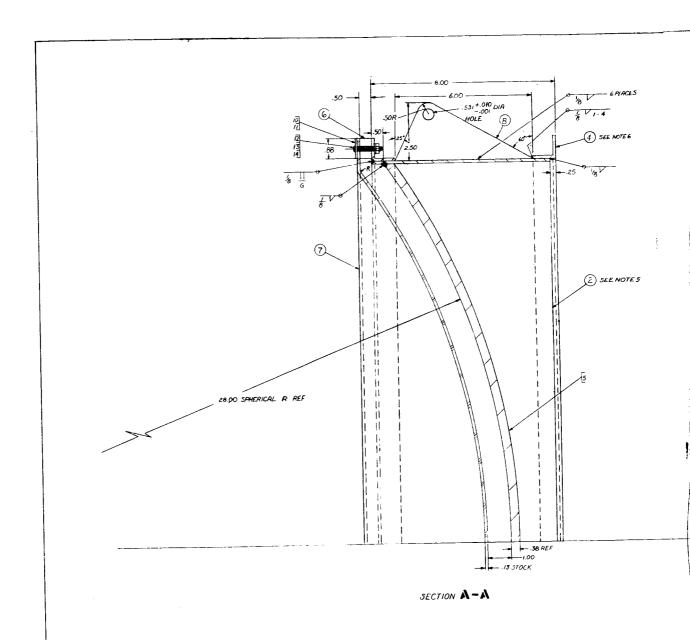
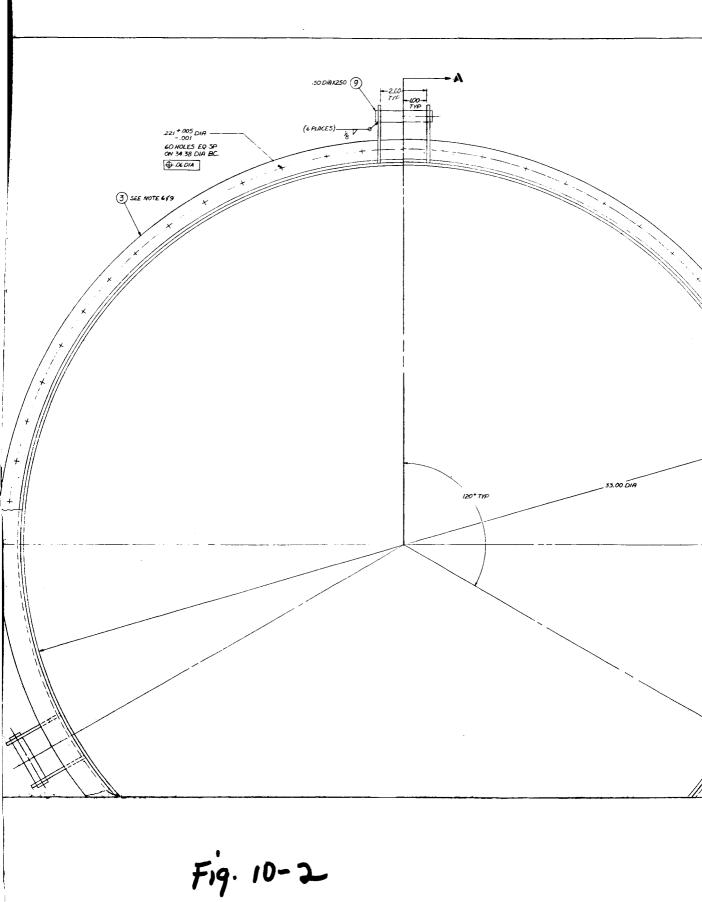
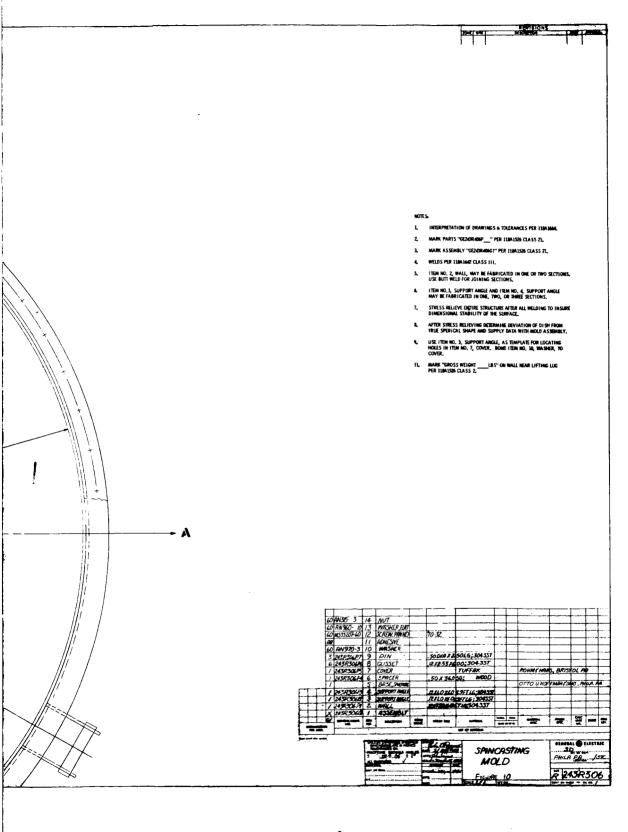
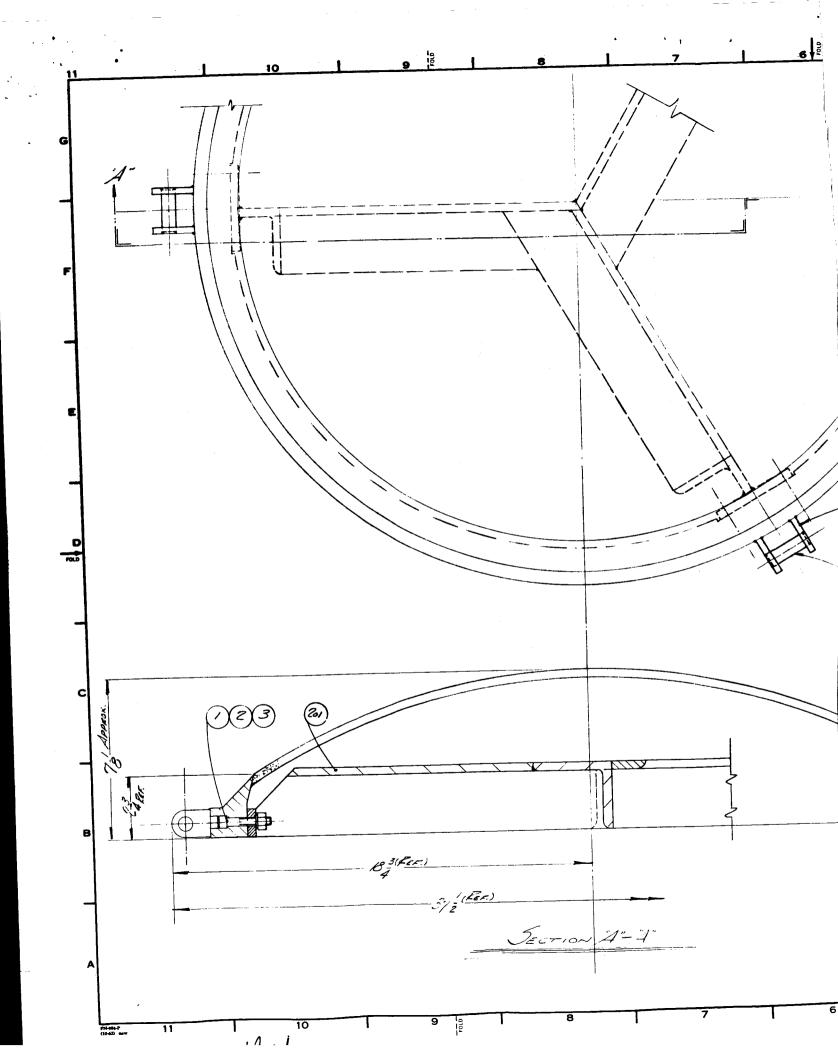
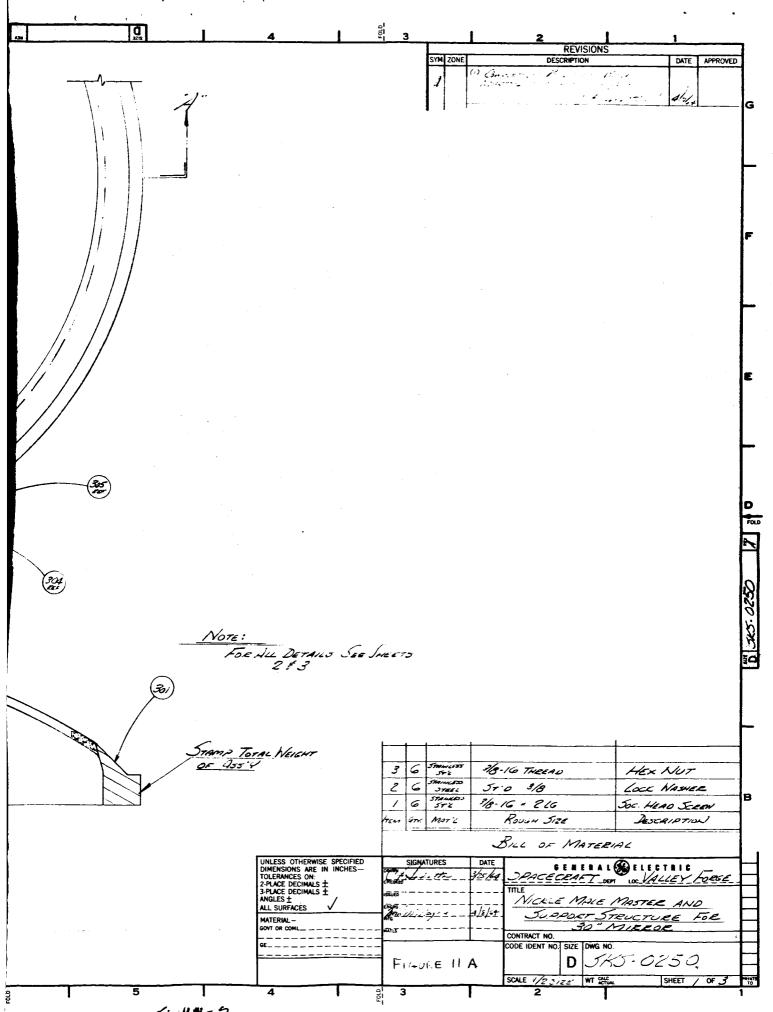


Fig 10-1

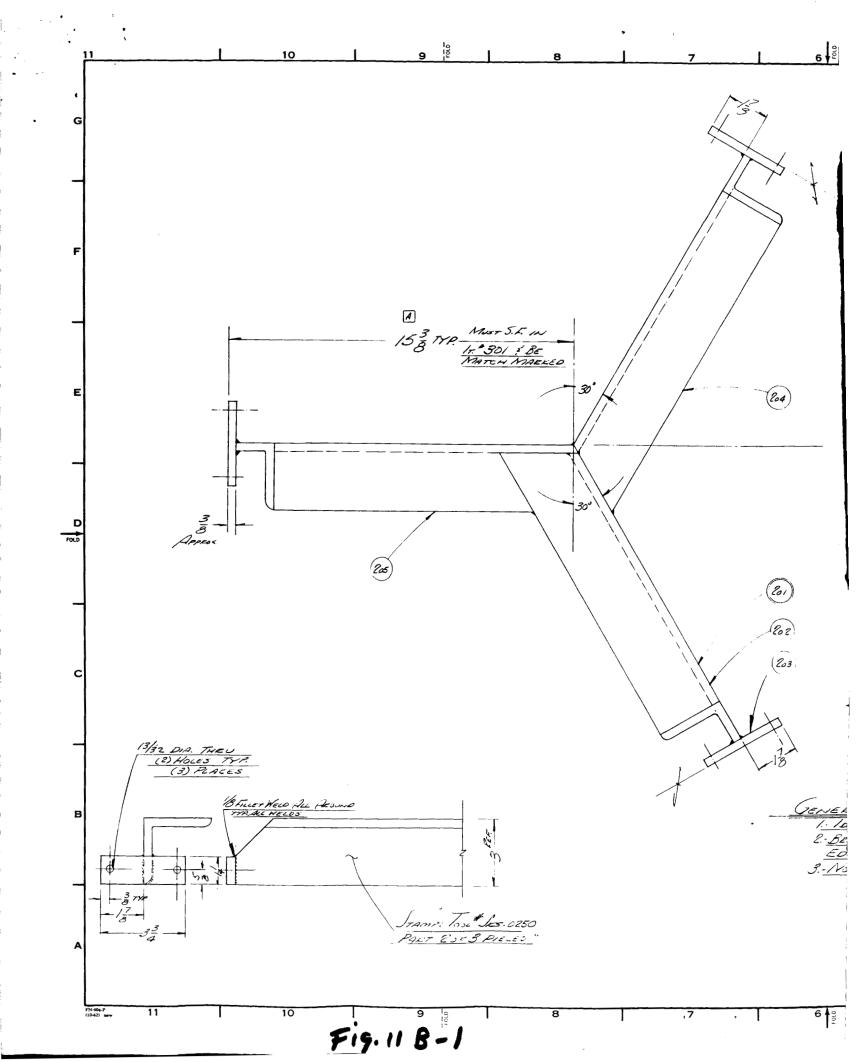


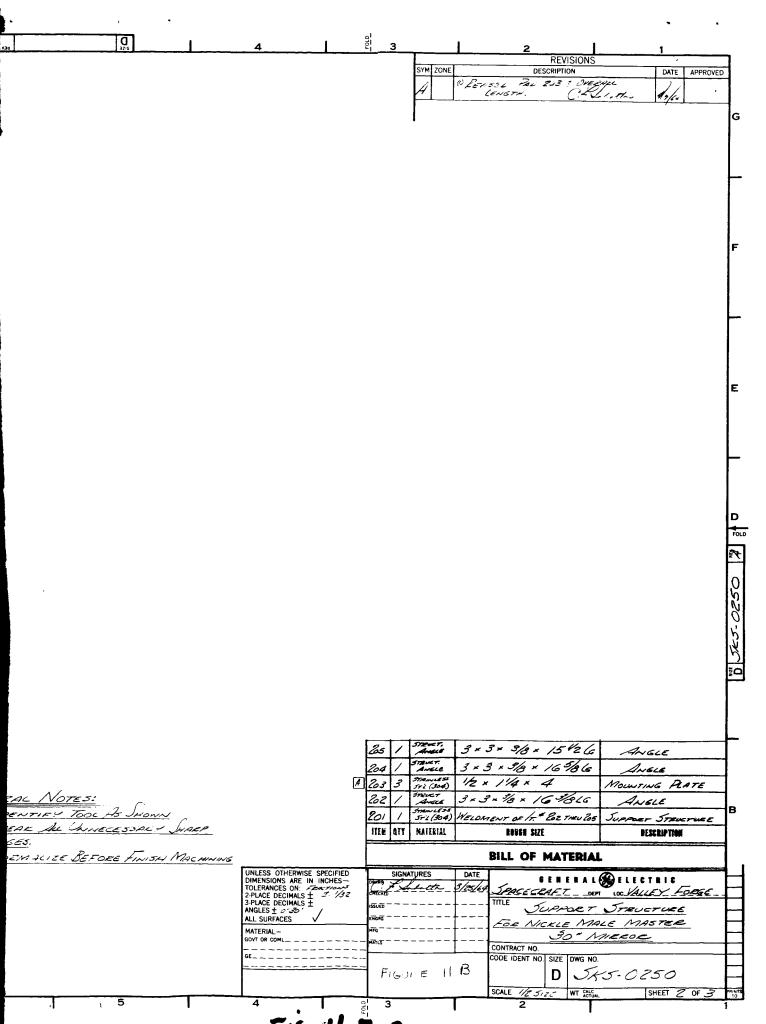


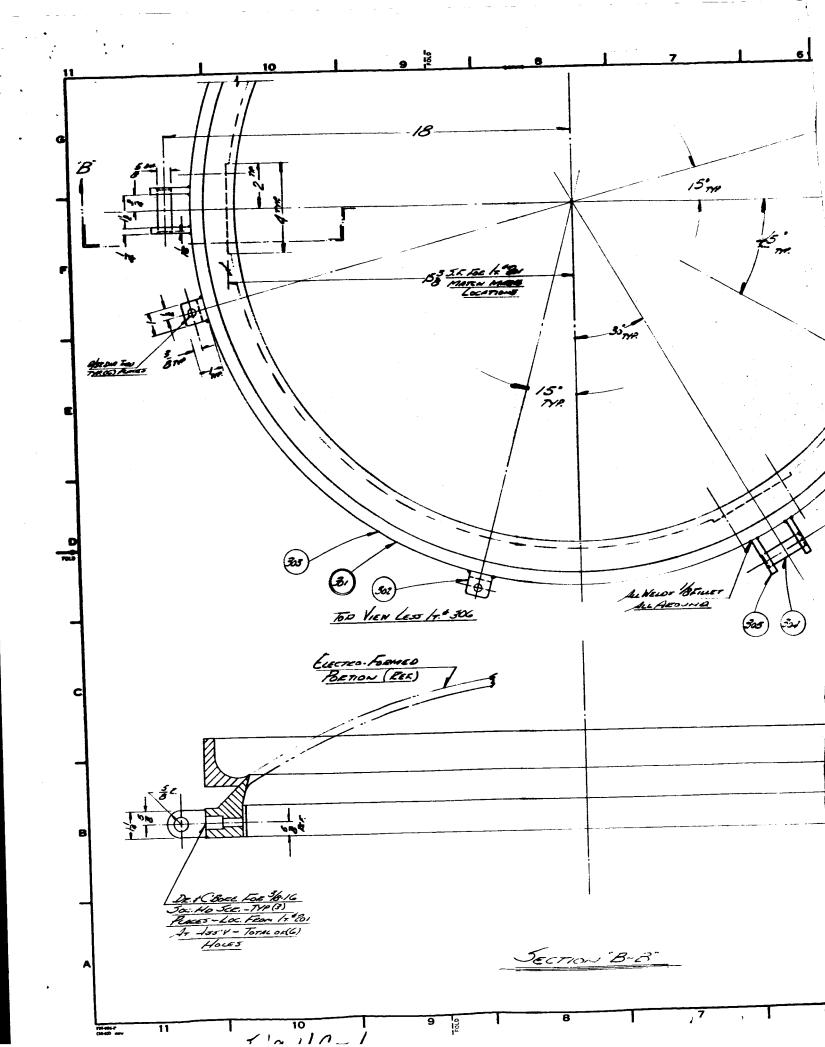




FigIN-2







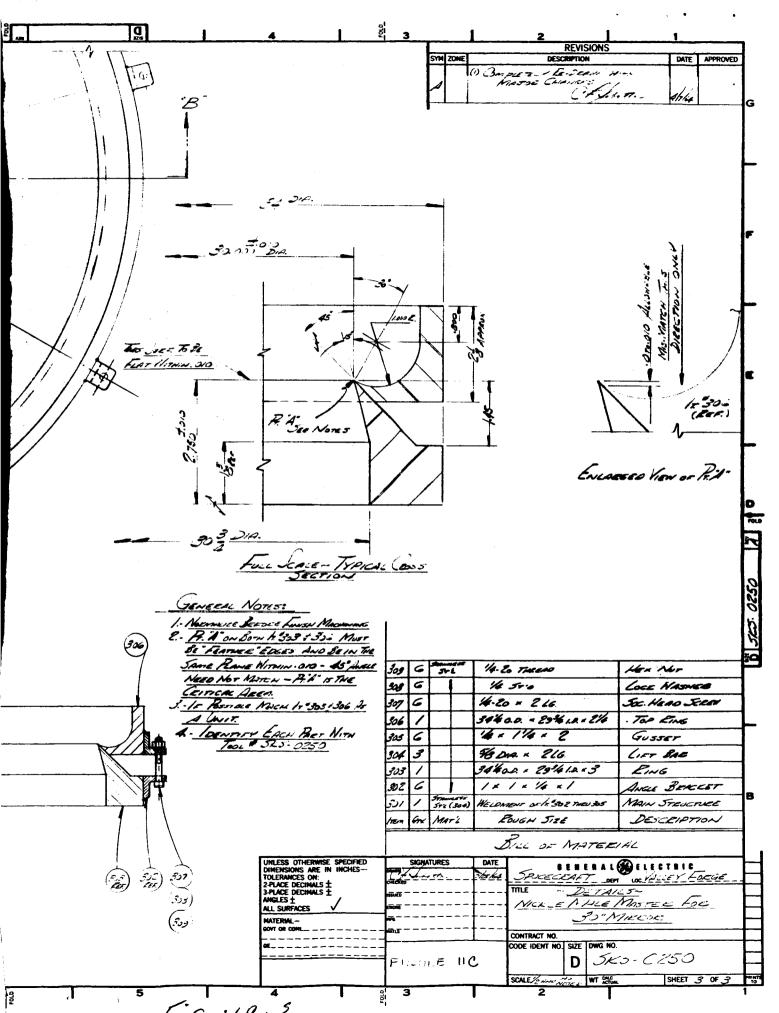


Fig 110-2

TABLE 1 ADDITIONAL RESULTS OF MATERIALS COMPATIBILITY TESTING

Weighed specimens were immersed in sealed bottles containing the hydride plating solution for a duration of 18 days. After this initial exposure, the specimens were removed, weighed and then returned to the sealed bath for an additional 18 days, after which they were again weighed. All specimens extended above the liquid level during the exposure.

<u>Material</u>	1st 18-day period Weight Change	2nd 18-day period Weight Change	<u>Remarks</u>
PVC*	- 0.025%	-0.07%	Excellent appearance
Lined fiberglass*	-0.12 %	-0.18%	Excellent appearance
Teflon tubing	+0.27%	+0.30%	Excellent appearance
Polyethylene tubing	+2.6%	+2.55%	Slight discoloration
Synthesine coating (fired, 400°F - 20 min	-0.11% n)	-0.21%	Excellent appearance
Mild steel**	-0.02%**		Slight rust in localized areas**

^{*} Specimens supplied by a plating vendor.

Weighed specimens were immersed in sealed bottles containing the hydride plating solution for a duration of 34 days. The specimens were removed, weighed and the results tabulated below.

<u>Material</u>	Weight Change	Remarks
Araldite 6020 Teta loaded with Mg0, Silica Microballoons	+0.094%	Excellent appearance
Araldite 6020 Teta heavily loaded with Silica Micrabaloons	+0.39%	Excellent appearance
Araldite 6020 Teta heavily loaded with Al203	-0.0081%	Excellent appearance
Araldite 6020 Teta	+0.076%	Excellent appearance
RSD Series 124 Epoxy	+0.83%	Excellent appearance
Product Research Corp.	-	Portion submerged in
Proprietary information		liquid dissolved

^{**} Only one 18-day exposure to the plating bath.

TABLE 2

MECHANICAL PROPERTIES OF ELECTROFORMED ALUMINUM

TENSILE PROPERTIES

Specimen Number	Modules of Elasticity (psi)	Ultimate Tensile Str. (psi)	Tensile Yield Str. @ 2% Offset (psi)	Elongation (%)
1	7.83 x 10 ⁶	10,850	7,880	24
2	8.17×10^6	11,290	(a)	28
3	7.95×10^6	11,000	7,740	(b)
Average	7.98×10^{6}	11,050	7,810	26
2S Aluminum	10.0×10^6	13,000	5,000	3 5

LINEAR COEFFICIENT OF THERMAL EXPANSION

Specimen Number	1st Run	No.2nd Run Aretziika	Averages
1	$13.2 \times 10^{-6} \text{ in/in/oF}$	14.6 x 10 ⁻⁶ in/in/ ^o F	13.9 x 10 ⁻⁶ in/in/oF
2	13.8	12.9	13.4
3	14.1	12.7	13.4
Average		<u> </u>	13.6
2S Aluminum	-	-	$(12.2 - 13.1)10^{-6}(C)$

DENSITY

Specimen Number

1	2.64 g/cc (97.4% of theoretical	density)
2	2.68 g/cc (98.9% of theoretical	density)
Average	2 66 g/cc (08 2% of theoretical	

2S Aluminum 2.71 g/cc -

- (a) Erractic extensometer readings in yield area
- (b) Failed through radius area before full elongation was reached
- (c) Alcoa Alunimum Handbook cites 12.2 x 10^{-6} in/in/°F from -58° to +68°F and 13.1×10^{-6} in/in/°F from 68° to 212°F.

TABLE 3
SPECULAR REFLECTIVITY OF ELECTROFORMED ALUMINUM

All specimens were vacuum coated with aluminum and with silicon monoxide at Libbey-Owens-Ford.

	Electroform	ed Aluminum	Nickel Master
Wave Length, microns	As Deposited	Polished Surface	
0.3*	71.1	31.4	85.8
0.4	71.2	45.7	87 . 7
0.5	82.6	56.9	89.0
0.6	84.8	64.2	88.3
0.9	87.2	75.1	88.0
1.2	93.7	86.5	94.1
1.5	95.6	90.9	96.8
1.8	95.8	93.1	96.7
2.1	96.5	94.3	96.7
2.4	96.3	95.6	96.8
2.7	96.0	96.4	97.4
2**	95.3	94.3	96.2
3	95.3	96.0	97.0
4	95.9	95.9	96.6
5	96.2	96.7	97.4
6	95.8	97.2	97.7
7	96.0	96.4	97.8

^{*} Reflectivity determined with Beckman-Model DK1L Reflectometer for spectral range of 0.3 to 2.7 microns.

^{**} Reflectivity determined with Perkin-Elmer Model 205 Reflectometer for spectral range of 2-7 microns.

STRUCTURAL ANALYSIS

The analysis reported upon in the Appendix to the First Quarterly Progress Report was programmed for an IBM 7094 digital computer. Slope rotations, meridional and circumferential stresses were calculated for aluminum paraboloidal collectors of 30-inch diameter and 60 degrees rim angle having thicknesses of 0.010, 0.020, 0.030 and 0.040 inch. The collectors were assumed fully fixed at their outer peripheries, i.e. no displacements or slope rotations were permitted. This corresponds to the practical case in which a support torus is connected to the outer periphery of a collector with the torus being much stiffer than the collector shell.

The results are presented in Tables A1 and A2 and in Figure A1. As is apparent from Table A1 and Figure A1, the slope rotations are extremely small, and they occur over the area adjacent to the support torus only. The thinner the collector membrane, the smaller the affected area, but the larger the maximum slope rotation. A thickness of .020 or .030 in. appears best from Figure A1, with the maximum slope rotation not exceeding .12 minutes, i.e. 7 seconds of arc. If the collector is made thicker, the maximum slope rotation is slightly reduced, but the area of slope deviation is considerably enlarged. If the collector is made thinner, the maximum slope rotation rises rapidly, and the area of slope rotation is decreased. Either of these changes probably results in a decreased geometric collector efficiency.

Table A2 shows that the stresses under a 10g axial load condition are very small. The maximum stresses are practically independent of the collector thickness; differences showed up in the third significant figure only. The maximum meridional stress occurs at the outer rim, and is equal to 32 psi. The maximum circumferential stress equals 14 psi and occurs close to the outer rim; it moves inward with increasing collector thickness.

The independence of the maximum stresses from the collector thickness can be explained as follows. The membrane stress is obviously independent of the thickness because weight, load and resisting area all vary linearly with the thickness. The bending deformations increase with decreasing thickness (see Figure Al), but smaller moments only are required to cause these increased deformations as the thickness decreases. Similarly, the section modulus becomes smaller as the thickness decreases. The sum of these effects is an insignificantly small change in maximum bending stress over the thickness range investigated.

The expression for A'', given on the last page of the Appendix to the First Quarterly Progress Report, is in error. The correct expression should be as follows:

$$A_2'' = \frac{E \int_0^{3/2} \frac{9 * (\theta_1)}{\sqrt{R_0} \sqrt{4 + (1 - \mu^2)}} - A_2'$$

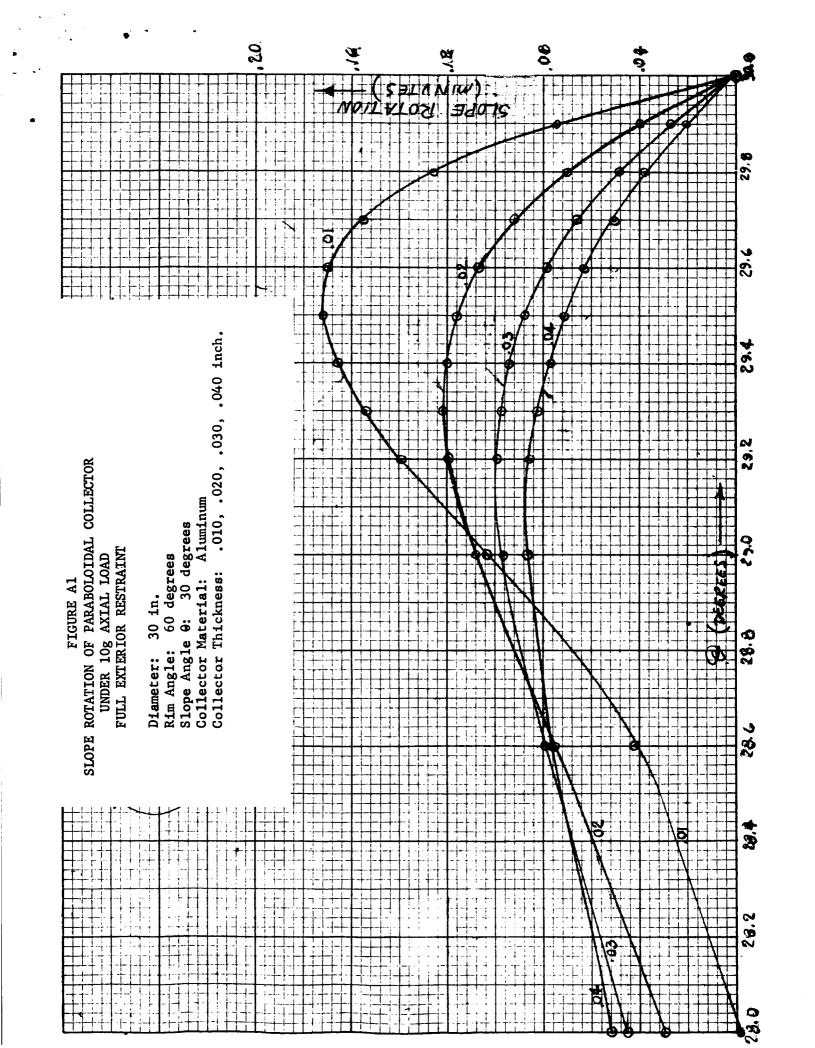


TABLE A-1 SLOPE ROTATION OF PARABOLOIDAL COLLECTOR UNDER 10g AXIAL LOAD FULL EXTERIOR RESTRAINT

Diameter: 30 in Rim Angle: 60 degrees

Angle 9		ON (MINUTES OF	ARC)	
(degrees)	.010	ICKNESS (IN.)	.030	.040
30.0	.000	.000	.000	.000
29.9	.075	.040	.027	.021
29.8	.125	.070	.049	.038
29.7	.155	.092	.066	.051
29.6	.170	.107	.079	.063
29.5	.172	.116	.088	.071
29.4	.166	.121	.094	.077
29.3	.154	.122	.098	.082
29.2	.139	.119	.099	.085
29.0	.103	. 108	.097	.086
28.6	.042	.075	.079	.077
28.0	001	.029	.045	.052
27.0	005	003	.007	.016
25.0	000	002	004	004
20.0	001	001	001	001
10.0	001	001	001	001

 $R_0 = 26.0 \text{ in.}$ $\theta_1 = 30 \text{ degrees}$

= 10

Material = aluminum

TABLE A2

MAXIMUM STRESSES IN PARABOLOIDAL COLLECTOR UNDER 10g AXIAL LOAD FULL EXTERIOR RESTRAINT

Diameter: 30 inch. Rim Angle: 60 degrees

Collector Thickness (in.)	.010	.020	.030	.040
Maximum Meridional Stress (psi)	32	32	32	32
Location (θ, degrees)	30	30	30	30
Maximum Circumferential Stress (psi)	14	14	14	14
Location (0, degrees)	28	27	25	25

NOTE: The stresses indicated are the maximum total stresses (membrane plus bending) at the concave surface of an aluminum collector.

WORK STATEMENT FOR ELECTROFORMING OF NICKEL MALE MASTER

The vendor will electroform a nominally 3/8 inch thick, 30 inch diameter, nickel master over the spincast epoxy mold, using the best up-to-date practices. The following steps will be used, except when variations are approved in advance by the RSD Project Engineer. RSD's representative will be present during the entire sequence and jointly with the vendor's representative will agree on and direct all process details. Final responsibility will rest with RSD's Project Engineer.

- a. The spincast epoxy assembly will be prepared for the electroforming sequence by protecting all areas which should not be plated with masking compounds.
- b. The working surface of the epoxy mold will be cleaned by spraying with mild alkaline cleaning and detergent solutions at room temperature. This cleaning step will be continued until a breakfree water film is observed over the entire working surface when distilled water rinse is sprinkled over the working surface.
- c. The surface will be sensitized using a slightly acidic filtered stannous chloride sensitizing solution not over three hours old.
 - d. The sensitizing step will be followed by a sprinkled distilled water rinse.
- e. The working surface will now be silverized by chemical reduction of silver from an ammoniacal silverizing solution. The ammoniacal silver and the reducer solutions (temperature not exceed $70^{\circ}F$) will be sprayed simultaneously over the working surface, and this spraying will continue until the silver deposit will become uniformly bright over the entire working surface.

The distilled water spray will be maintained over the surface during steps (a) through (e), to maintain the breakfree water film condition once established, but the amount of such spray will be decreased during the sensitizing and silverizing steps. The temperature of neither the master nor any of the solutions shall exceed 75°F during the steps (a) through (e).

- f. The assembly will be transferred without delay into a 100° F nickel electroforming bath, and the electrolysis will start as soon as it is possible to establish correct positioning of the electrodes and contacts. The initially low CD will be gradually raised to full working value as specified below.
- g. The nickel anode assembly will be constructed in such a manner as to provide a highly uniform current density distribution over the cathode.
 - h. The electrolyte will consist of a low stress Ni-sulfamate solution.

As soon after immersion of the mold assembly as equilibrium conditions are established, the following indicative parameters will be maintained.

Temperature: 120°F - 1° Current Density: 20 asf

Agitation: Impinging solution

Filtration: Continuous

Electrolytic Purification: Continuous

Controls: 1. Essentially continuous monitoring of bath composition and pH.

Continuous monitoring of temperature, current and voltage.
 Daily check of stresses using a Schmidt-cell.

4. Surface tension.

- i. The spincast mold to be furnished by General Electric is fully described on GE Dwg. 243R306. All metal parts on this structure are fabricated from 300 series stainless steel, and will, therefore, not contaminate the bath.
- j. Also furnished by General Electric will be a stiffening ring structure which is to be attached to the electroform by electroforming during the process. This ring is described on GE Dwg. SKS 0250. All necessary cleaning and clamping of this ring to the spincast is to be performed by the vendor. GE will monitor the attaching of the GEfurnished stiffeners which are mounted to this ring prior to removing the electroform from the spincast. These stiffeners are also described on GE Dwg. SKS 0250.

A guide ring, described on GE Dwg, SKS 0250, will be furnished by GE and is to be assembled to the male master support structure under the supervision of GE engineering. This ring contains its own clamps so that no external clamping is required.

k. At the end of the nickel electroforming process, when the proper deposit thickness is obtained, the assembly will be removed from the plating tank, and the bath residues will be rinsed off with de-ionized water.

Separation of the electroform will be effected by alternate expansion and contraction of the electroform, obtained by alternate hot and cold rinses and will be assisted by forcing 5-10 psi compressed air between the electroform and mold and by mechanical forces applied to the attachments.