LARGE RADIOISOTOPE HEAT SOURCE
CAPSULE PROGRAM
TOPICAL REPORT NO. 12
TRANSIT CAPSULE PROCESS METALLURGY

AEC Research and Development Report

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LARGE RADIOISOTOPE HEAT SOURCE
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TOPICAL REPORT NO. 12
TRANSIT CAPSULE PROCESS METALLURGY

BY
ROBERT G. BRENGLE

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CONTRACT: AT(29-2)-2338
ISSUED: JULY 3, 1970

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AI-AEC-12967
ABSTRACT

T-111 (Ta-8W-2Hf), Ta-10W and Pt-20Rh piece parts have been fabricated for the Transit and Pioneer flight qualification programs. This report presents the metallurgical characteristics of the piece parts by using photomicrographs of various areas. Even though some process variations took place during the program the photomicrographs from the various capsules show that very little difference occurs in the final microstructure.
I. INTRODUCTION

The Large Radioisotope Heat Source Capsule Program has developed the technology for a tantalum alloy refractory metal capsule designed to operate for 5 years at 2000°F in a space environment. The LRHSC program developed material specifications, forming and assembly processes and also compatibility information about the capsule components.

Early in GFY 1970 the decision was made to apply this technology to the heat source for the Transit Radioisotope thermoelectric generator (RTG). The program was directed to determine the feasibility of producing tantalum alloy piece parts to the Transit design. One of the piece parts, the Ta-10W fuel liner, had not previously been deep drawn but because of the forming experience with T-111 the development of this process was completed successfully. LRHSC Task 8 was a feasibility experiment to determine if the process could be adapted to different capsule designs. Task 8 developed the initial forming, welding and assembly procedures for the Transit capsule and impact tested five (5) capsules of the Transit design (1). After the feasibility of the processes and materials had been established, the pre-production or Task 9 effort began. This task upgraded the Task 8 processes and finalized the tooling and specifications. It also produced the first five (5) sets of weld development hardware for MRC. Following Task 9 the production contract began producing hardware for the Flight Qualification Test Program.

The production program has now produced over 100 sets of piece parts. The purpose of this topical report is to document the metallurgical structure characteristics of the Transit piece parts.

(1) LRHSC Quarterly Progress Report No. 10, p. 49-109
II. SAMPLE SELECTION AND PREPARATION

The metallurgical samples used for this report were obtained by sectioning actual capsules after the desired configuration had been obtained. In some cases the capsules were sectioned before heat treatment so that several heat treatments could be evaluated with a single capsule piece part. All sectioning was accomplished by band sawing since abrasive wheel cutting tends to produce surface cracks in the cut face.

The samples that were to be used for metallographic examination were mounted in a suitable mounting compound and prepared using standard polishing techniques. The metallographic samples were etched with solutions of the compositions of Table 1.

TABLE I
ETCHING AGENTS

<table>
<thead>
<tr>
<th>Material</th>
<th>Etching Agent</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>T-111</td>
<td>40 H₂SO₄</td>
<td></td>
</tr>
<tr>
<td></td>
<td>20 H₂O</td>
<td></td>
</tr>
<tr>
<td></td>
<td>20 HF</td>
<td></td>
</tr>
<tr>
<td></td>
<td>10 H₂O₂</td>
<td></td>
</tr>
<tr>
<td>Ta-10W</td>
<td>75 HNO₃</td>
<td></td>
</tr>
<tr>
<td></td>
<td>25 HF</td>
<td></td>
</tr>
<tr>
<td>Pt-20Rh</td>
<td>20% HCl saturated with NaCl</td>
<td>Electrolytic etch 5.5 volts AC ≥ 1.5 min, platinum electrodes</td>
</tr>
</tbody>
</table>
III. RESULTS

A. LINER BODY (Ta-10W)

The liner body is deep drawn from a 0.060 x 6-1/4 inch diameter plate of Ta-10W in six (6) stages. Tables 2 and 3 provide the general process characteristics of the part. The part is annealed after the first three draws to recover formability and after the final draw to remove forming stresses and produce a fully recrystallized component for service. Figures 1 and 2 show the as-fabricated microstructure just prior to the final anneal. The photos show the material is severely cold worked in the sidewalls of the part (Figure 7) but as one proceeds around the hemisphere towards the nose, the amount of cold work is less obvious. The cold work in the nose area was limited to bending rather than ironing which is prominent in the sidewall. Figure 3 shows the microstructure after annealing for one hour at 2600 °F in a vacuum of 10^-5 Torr. Figure 3-1 shows views of the sidewall that may be compared to Figure 1. The material has been recrystallized at 2600 °F to a uniform ductile structure, which is readily weldable and on the LRHSC Impact Program has been shown to withstand severe hairpin bends.

B. LINER CAP (Ta-10W)

The process flow sheet, Table 4, gives the general process characteristics for the piece part. The liner cap is formed by a single stage coining operation using a die set that consists of a punch, a female die and blank hold-down fixture. The material used is 0.035-in. thick Ta-10W die blanks. After the part is formed, it is cleaned and annealed for one hour at 2600 °F. Figures 4 and 5 show the microstructure of the liner cap after the final anneal. The microstructure consists of basically equiaxed grains and some residual flow lines. These flow lines are in almost all formed products especially the rolled sheet starting stock (Figure 6), and can only be seen when viewing perpendicular to the forming plane as is the case in all piece part photos presented. The liner cap forming and annealing processes have been consistent throughout the Transit and Pioneer Development and Production Programs.

(2) AI Specification Numbers ST0611NA008 and ST0610NA0010

AI-AEC-12967
TABLE 2
MANUFACTURING FLOW CHART FOR THE BODY LINER

<table>
<thead>
<tr>
<th>Step</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Issue Blanks in Serialized Containers</td>
</tr>
<tr>
<td>2.</td>
<td>Visual Inspection of Blanks</td>
</tr>
<tr>
<td>4.</td>
<td>Draw &amp; Color Body</td>
</tr>
<tr>
<td>6.</td>
<td>Draw &amp; Visual inspection</td>
</tr>
<tr>
<td>7.</td>
<td>Setup &amp; Start to Cont. Sta.</td>
</tr>
<tr>
<td>9.</td>
<td>Draw &amp; Final Inspection</td>
</tr>
</tbody>
</table>

Note: The flow chart includes various steps and processes involved in the manufacturing of the body liner, with specific operations indicated at each step. The chart is detailed and includes machine setups, inspections, and other procedural steps.
<table>
<thead>
<tr>
<th>CLEARANCE</th>
<th>PERCENT OF REDUCTION OF AREA</th>
<th>DIAMETER REDUCTION</th>
<th>WALL THICKNESS</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>.786</td>
<td>13%</td>
<td>.875</td>
</tr>
<tr>
<td>2</td>
<td>.100</td>
<td>25%</td>
<td>1.37</td>
</tr>
<tr>
<td>3</td>
<td>.060</td>
<td>19%</td>
<td>.795</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>ANNEAL FOR ONE HOUR AT 2600°F</td>
</tr>
<tr>
<td>4</td>
<td>.060</td>
<td>20%</td>
<td>.715</td>
</tr>
<tr>
<td>5</td>
<td>.060</td>
<td>9%</td>
<td>.250</td>
</tr>
<tr>
<td>6</td>
<td>.038</td>
<td>10%</td>
<td>.255</td>
</tr>
<tr>
<td>7</td>
<td>.036</td>
<td>≈0</td>
<td>≈0</td>
</tr>
</tbody>
</table>

ANNEAL FOR ONE HOUR AT 2600°F
Figure 1. Liner Body
Before Final Anneal
(Ta-10W)

History:
3 Draws
Intermediate Annealed
for 1 hr. at 2600 °F
3 Draws
As fabricated condition
AI-AEC-12967
Figure 2. Liner Body Before Final Anneal (Ta-10W)

History:
3 Draws
Intermediate Annealed for 1 hr at 2600°F
3 Draws
As fabricated condition

AI-AEC-12967
Figure 3. Liner Body After Final Anneal, S/N 037, Lot No. 3 (Ta-10W)

History:

a. 3 Draws
b. Intermediate anneal 1 Hr. at 2600 °F
c. 3 Draws
d. Final annealed for 1 Hr. at 2600 °F

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### TABLE 4.
MANUFACTURING FLOW CHART FOR CLAD AND LINER CAP

#### CLAD CAP

<table>
<thead>
<tr>
<th>Step</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>M2C Material</td>
<td>Control STA 0/1BL</td>
</tr>
<tr>
<td>Issue Blanks inSerialized Containers</td>
<td>Issue Tooling Req.</td>
</tr>
<tr>
<td>Verify Mat. Val. Req.</td>
<td>COIN OPER</td>
</tr>
<tr>
<td>Punch Die To Final Size</td>
<td>Punch Die To Final Size</td>
</tr>
<tr>
<td>Insert Die</td>
<td>Insert Die</td>
</tr>
<tr>
<td>Punch Pom-Jf-oct, MfcTLCen Tfai^o in^o -/^^</td>
<td>Final Set Up Return Tools To Cont. Sta.</td>
</tr>
</tbody>
</table>

#### LINER CAP

<table>
<thead>
<tr>
<th>Step</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>MRC Material</td>
<td>Control STA 0/1BL</td>
</tr>
<tr>
<td>Issue Blanks inSerialized Containers</td>
<td>Issue Tooling Req.</td>
</tr>
<tr>
<td>Verify Mat. Val. Req.</td>
<td>COIN OPER</td>
</tr>
<tr>
<td>Punch Die To Final Size</td>
<td>Punch Die To Final Size</td>
</tr>
<tr>
<td>Insert Die</td>
<td>Insert Die</td>
</tr>
<tr>
<td>Punch Pom-Jf-oct, MfcTLCen Tfai^o in^o -/^^</td>
<td>Final Set Up Return Tools To Cont. Sta.</td>
</tr>
</tbody>
</table>

---

**Legend:**
- **M2C Material:** Clad Cap
- **MRC Material:** Liner Cap
- **COIN OPER:** Coin Operator
- **Visual Insp.:** Visual Inspector
- **Final Clean Identity:** Final Clean Inspector
- **Package For Del. Hld In Cont. Sta.:** Package For Delivery Hold in Cont. Sta.
Figure 4. Liner Cap (Ta-10W)
S/N 046, Lot No. 4
History:
a. Coined
b. Annealed for 1 hr at 2600°F
Figure 5. Liner Cap (Ta-10W)
S/N 046, Lot No. 4
History:
a. Coined
b. Annealed for 1 hr at 2600°F
a. Perpendicular to Rolling Plane  
b. Parallel to Rolling Plane

Figure 6. MRC T-111 Plate, 0.170 x 6.5-Inches As-Received HT 9446B
C. STRENGTH MEMBER BODY (T-111)

The strength member body (SMB) is produced by deep drawing 0.170 in. thick \times 6.5 \text{ in. diameter} T-111 (Ta-8W-2Hf) plate. The process characteristics are given in Tables 5 and 6. Approximate configurations of the part in various four stages is given in Tables 7 and 8. The process consists of seven deep drawing and ironing operations, a coining operation and two anneals. Microstructures of the Task 8 development and Transit production as-received material heats are shown in Figures 6 and 8 respectively.

During Task 8 the SMB parts were given a 2700 °F in-process anneal and a 3000 °F final anneal. It was felt from a metallurgical standpoint the in-process full recrystallization could potentially cause problems. This could tend to produce a variation in grain size because of the different responses to two heat treatments in the various areas of the part. At the end of Task 8 a part was drawn with no intermediate anneal, given a final anneal and machined. The part was apparently good, however, at this time it was not possible to inspect the ID and OD radii.

The Task 9 Manufacturing Production Order (MPO) was written on the basis of the previous part and no intermediate anneal was specified. A new heat of material was introduced during Task 9 (NRG Ht 9446 B, Figure 6). Observation of the in-process parts formed with this material showed a texturing effect on the inside surface of the part that had not been observed with previous heats. The shape of the texture was obviously related to the rolling direction of the plate. The finished part exhibited surface cracks on the ID and excessive ovality. It was concluded that some processing variation in this fabrication of the heat was the apparent cause, and efforts were directed to solve this problem by varying the drawing process to make use of the available material. It was found that the material heat (NRG Ht 9446B) had been given a somewhat lower final anneal than usual so a plate was reannealed at AI for one hour at 3000 °F (Figure 7) in hopes of producing the desired forming characteristics. This effort was not successful for reducing ovality because the material was not formable and cracked after the third draw. A mechanical straightening operation for rounding the open end of the piece parts was proposed and adopted. A change

(3) LRHSC Topical Report No. 4, T-111 Pressure Vessel Development and Fabrication, p. 42-51
TABLE 5
MANUFACTURING FLOW CHART FOR STRENGTH MEMBER BODY
# TABLE 6.

## STRENGTH MEMBER BODY

<table>
<thead>
<tr>
<th>DRAW RING TO PUNCH CLEARANCES</th>
<th>PERCENT REDUCTION OF AREA</th>
<th>DIAMETER REDUCTION</th>
<th>WALL AFTER DRAW</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 .786</td>
<td>13%</td>
<td>.875</td>
<td>≈ .170</td>
</tr>
<tr>
<td>2 .200</td>
<td>21%</td>
<td>1.17</td>
<td>.170 - .160</td>
</tr>
<tr>
<td>3 .200</td>
<td>16%</td>
<td>.715</td>
<td>.170 - .155</td>
</tr>
<tr>
<td>4 .200</td>
<td>18%</td>
<td>.695</td>
<td>.170 - .150</td>
</tr>
<tr>
<td>5 .150</td>
<td>12%</td>
<td>.370</td>
<td>.155 - .135</td>
</tr>
</tbody>
</table>

**ANNEAL FOR ONE HOUR AT 2700°F (RECRYSTALLIZATION)**

<p>| | | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>6 .104</td>
<td>9% Dia.</td>
<td>.243</td>
<td>.107</td>
</tr>
<tr>
<td></td>
<td>30% Wall</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>COIN NOSE</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8 .101</td>
<td>&lt;&lt;1%</td>
<td></td>
<td>.107</td>
</tr>
</tbody>
</table>

**ANNEAL FOR ONE HOUR AT 3000°F**
TABLE 8.
FITUP OF 5TH DRAW PART
WITH FINAL PUNCH

FINAL PUNCH

PART FROM 5TH DRAW

WALL AROUND SPHERICAL END
VARIES FROM .142-.163
AFTER 5TH DRAW AND COIN

AI-AEC-12967
22
Figure 7. T-111 HT 9446B After Annealing at 3000°F
Figure 8. LRHSC Task 8
Material T-111
in punch design was also incorporated into the process at this time which subsequently eliminated the internal cracking.

Figures 9, 10, and 11 show the results of the 2700 °F in-process anneal that was performed after the fifth draw on most of the piece parts. The photomicrographs show a uniform, small grain size in the sidewall with somewhat larger grains at the transition zone (Figures 10-4, -5, -6). The nose of the SMB has not recrystallized because of the small amount of cold work in that area. After the part has been given an intermediate anneal, it is redrawn to the final configuration. Figures 12, 13, and 14 show a SMB just before it is final annealed. Figure 12-1, -2, show the sidewalls that have received the most deformation. The grains are very distorted and appear fibrous. The nose areas (Figure 12-9, -10, -11) also have grain distortion but not to the extent of the sidewalls. This variation in deformation is inherent in the deep drawing process, and produces the so called "duplex" structure that consists of a mixture of large and small grains.

Figures 15, 16, and 17 show the condition of the Task 9 and the first production lot. These parts were processed without an intermediate anneal. The sidewalls have a uniform ASTM 6 average grain size. Traveling from the sidewall to the nose, areas of large grains are seen (Figure 16-4) and then the duplex structure begins (Figure 17-5, -6). The amount of large grains in the structure is reduced as the nose is approached where the structure is again equiaxed and of uniform grain size. All strength members show this characteristic behavior in the blind end although the areas occur at slightly different locations. Figures 18, 19, and 20 show a strength member configuration that was not shipped on the Transit program. The process in question substituted an intermediate one hour 2250 °F stress relief in place of the 2700 °F recrystallization in the interest of better structure. It was determined that concentricity and cylindricity could not be met with only an intermediate stress relief and the process returned to the 2700 °F in-process anneal. By this time it had been shown that the intermediate annealing temperature or the use of an intermediate anneal at all had very little effect on the final microstructure. Figures 21 and 22 show the structure variation in a part produced with the final parameters.
Figure 11. Strength Member Body
After 2700°F in Process Anneal
AI-AEC-12967

History
a. 5 Draws
b. Annealed for 1 hr at 2700°F
AI-AEC-12967 28
Figure 12. Strength Member Body Before Final Anneal (T-111) (Reduced to 70% of Original)

History:

a. 5 Draws
b. Anneal for 1 Hr. at 2700 °F
c. 2 Draws + Coin

As-Fabricated Condition
Figure 13. Strength Member Body Before Final Anneal (T-111) (Reduced to 75% of Original)

History:
- a. 5 Draws
- b. Anneal for 1 hr at 2700°F
- c. 2 Draws + Coin

As-Fabricated Condition

AI-AEC-12967 30
Figure 14. Strength Member Body
Before Final Anneal (T-111)

History:

a. 5 Draws
b. Anneal for 1 hr at 2700 °F
c. 2 Draws + Coin

As-Fabricated Condition
Figure 15. Strength Member Body (S/N 025 Lot 3), Final Annealed but with no in Process Anneal (T-111)

History:
a. 6 draws — coin
b. Final Annealed for 1 hr at 3000°F
Al-AEC-12967
32
Figure 16. Strength Member Body (S/N 025 Lot 3), Final Annealed but with no in Process Anneal (T-111)

History:

a. 6 draws — coin
b. Final Annealed for 1 hr at 3000°F

AI-AEC-12967
33
Figure 17. Strength Member Body (S/N 025 Lot 3)
Final Annealed but with no in Process Anneal (T-111)

History:
a. 6 draws - coin
b. Final Annealed for 1 hr at 3000 °F
AI-AEC-12967
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Figure 18. Strength Member Body
(T-111) S/N 031
Intermediate Stress Relief History:
- a. 5 Draws
- b. Stress Relieved 1 hr at 2250 °F
- c. 2 Draws + Coin
- d. Final Annealed for 1 hr at 3000 °F
Figure 19. Strength Member Body (T-111) S/N 031
Intermediate Stress Relief History:
   a. 5 Draws
   b. Stress Relieved 1 hr at 2250°F
   c. 2 Draws + Coin
   d. Final Annealed for 1 hr at 3000°F
Intermediate Stress Relief History:

a. 5 Draws
b. Stress Relieved 1 hr at 2250 °F
c. 2 Draws + Coin
d. Final Annealed for 1 hr at 3000 °F

Figure 20. Strength Member Body (T-111) S/N 031

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Figure 21. Strength Member Body
S/N 012, Lot 2 (T-111)
Final Process Condition History:
a. 5 Draws
b. Anneal for 1 hr at 2700°F
c. 2 Draws + Coin
d. Final Annealed for 1 hr at 3000°F
Figure 22. Strength Member Body
S/N 012, Lot 2 (T-111)
Final Process Condition History:
a. 5 Draws
b. Anneal for 1 hr at 2700°F
c. 2 Draws + Coin
d. Final Annealed for 1 hr at 3000°F

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In all cases the strength member bodies were given a final anneal for one hour at 3000 °F. This heat treatment is a solution treatment and is somewhat higher than that necessary to recrystallize the material. It can be seen from the photomicrographs in this section that there is little difference observable in the microstructure of the SMB.

D. STRENGTH MEMBER CAP (T-111)

The strength member cap has been produced by two methods, a single stage coining procedure and a more recent deep drawing method. In both cases the T-111 was given a final anneal at 3000 °F for one hour. The manufacturing flow sheet for the deep drawing method is given in Table 9. Figure 23 shows the microstructure of the coined cap. The weld preparation area has a uniform grain size and this structure continues for approximately 1.0 inch around the cap. As the top of the hemisphere is approached, the grains become larger for a short distance (Figure 23-2) and then duplex structure occurs and continues. The illustration in Figure 23 shows the area where the duplex structure can be seen. The deep drawn cap (not pictured) has more uniform grain size than the coined cap and less extensive area of duplex structure. If uniform grain size is the criterion then the deep drawn cap is better than the coined cap.

E. CLAD BODY (Pt-20Rh)

The clad body is formed in seven draws from 0.030 in. thick x 7.5 in. diameter Pt-20Rh sheet. The part is given an in-process anneal after the fourth draw and final anneal after the seventh draw. Both anneals are performed in air at 1800 °F for one hour. A photomicrograph of the as-received microstructure is given in Figure 24 and has an ASTM 5 average grain size. The reduction schedule and manufacturing flow charts are given in Tables 10 and 11.

The Pt-20Rh piece parts are annealed after four draws to recover the formability necessary for the final draws. Figure 25 shows a clad body cross section after the in-process anneal. Large grains (approximately ASTM 3-4) can be seen in the bend area (Figure 25-2). The grains in the sidewall and nose, however, are small and uniform. Figure 26 shows the final condition of
Figure 23. Strength Member Cap After Final Anneal, S/N 038 Lot 3

History:
1. Coined
2. Final Annealed for 1 hr at 3000°F

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Figure 24. Pt-20Rh, As Received
**TABLE 11.**
**CLAD BODY**

<table>
<thead>
<tr>
<th>CLEARANCE</th>
<th>PERCENT OF REDUCTION OF AREA</th>
<th>DIAMETER REDUCTION</th>
<th>WALL THICKNESS</th>
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<tr>
<td>1</td>
<td>.040</td>
<td>36%</td>
<td>2.5</td>
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<tr>
<td>2</td>
<td>.040</td>
<td>13%</td>
<td>.580</td>
</tr>
<tr>
<td>3</td>
<td>.035</td>
<td>13%</td>
<td>.510</td>
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<tr>
<td>4</td>
<td>.030</td>
<td>12%</td>
<td>.410</td>
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**ANNEAL FOR ONE HOUR AT 1800°F**

<p>| | | | |</p>
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<thead>
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<td>3%ΔD</td>
<td>.062</td>
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<tr>
<td></td>
<td></td>
<td>33%ΔW</td>
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</table>

**ANNEAL FOR ONE HOUR AT 1800°F**

<p>| | | | |</p>
<table>
<thead>
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<tbody>
<tr>
<td>7</td>
<td>.017</td>
<td>~0</td>
<td>~0</td>
</tr>
</tbody>
</table>

10% Wall Reduction
Figure 25.
Clad Body After in Process Anneal (Pt-20Rh)

History:
a. 4 Draws
b. Annealed for 1 hr at 1800°F
Figure 26. Clad Body After Final Anneal (Pt 20Rh)

History:

a. 4 Draws
b. Annealed for 1 hr at 1800°F
c. 2 Draws
d. Final Annealed 1 hr at 1800°F
a clad body after the final anneal. The grain size is uniform in the weld preparation area, however, it is rather large (ASTM 3-4). Figure 27 shows the transition area of the sidewall to the nose. The sidewall is the thin section that tapers into the thicker nose. It can be seen that the grains are again quite large, however, there is no duplex structure. This structure is apparently adequate and has withstood impact Task 8 processed with very similar parameters.

F. CLAD CAP (Pt-20Rh)

The clad cap is formed from 0.03 in. thick sheet in a single stage coining operation. The part is given a final anneal at 1800 °F for one hour and resultant microstructure is shown in Figure 28. The weld preparation area has a uniform equiaxed grain structure (ASTM 5).

The grains in the other areas of the cap are slightly larger, however, none are as large as those observed in the clad body. The forming and annealing processes for this part have been fixed through the entire production effort.
Transition Section of Clad Body After Final Anneal History:

1. 4 Draws
2. Annealed for 1 hr at 1800°F
3. 2 Draws
4. Final Annealed for 1 hr at 1800°F

Figure A1: Transition Section of Clad Body After Final Anneal.