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LARGE RADIOISOTOPE HEAT SOURCE
CAPSULE PROGRAM
Topical Report No. 4
T-111 Pressure Vessel Development
and Fabrication

AEC Research and Development Report

ATOMICS INTERNATIONAL

A DIVISION OF NORTH AMERICAN AVIATION, INC.

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Topical Report No. 4
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By
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P. B. Ferry

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ABSTRACT

The Large Radioisotope Heat Source Capsule (LRHSC) Program is developing technology for high temperature (2000°F) long life (5 years or longer) heat sources which can be used in the space environment. The LRHSC program is specifically concerned with the development of a tantalum alloy fuel capsule. Data are to be obtained under conditions of biaxial creep, impact and burial, and included strain, compatibility and thermal maps. This Topical Report presents the developmental results for the critical process of deep drawing the tantalum alloy T-111. Since this process represents a "first" for such extreme l/d ratios with the T-111 alloy, close documentation of the process has been conducted.

This report contains the details of tooling development and checkout, and the history of problems and solutions encountered during the development effort. At this point, the process can be considered "state-of-the-art".

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I. INTRODUCTION

A. PURPOSE OF EFFORT

Atomics International, a Division of North American Rockwell Corporation under contract with the Atomic Energy Commission*, has developed the fabrication processes required to deep draw a T-111 (Ta-2Hf-8W) pressure vessel. This development effort was part of the Large Radioisotope Heat Source Capsule (LRHSC) Program which was established to develop the technology of a radioisotope fuel capsule capable of operating in a space environment at a surface temperature of about 2000°F for five years or more.

The objectives of the present phase of the LRHSC Program are the preliminary design, fabrication, test and evaluation of a test capsule whose design takes into account the operating requirements of a typical heat source. The work includes process, fabrication and test activities. The process development and fabrication include the work required to define an acceptable manufacturing specification for the reference design capsule and includes manufacturing the test items required for the program. The test program involves various coupons and capsules with evaluation of chemical compatibility, thermal cycling and adherence of coatings, capsule creep, capsule impact and capsule earth burial. In addition to indicating the suitability of the reference capsule for ground test, the data generated in this study will be used to validate presently accepted design approaches and define design margins that would actually exist for high temperature capsules intended for flight systems.

Figure 1 shows the design of the test capsule. The fuel would be contained in the cylindrical annulus between the inner and outer Ta-10W fuel liners. The welds in the inner liner are intermittent to permit the helium to diffuse into the central void volume. The fuel liner assembly is inserted into a T-111 (Ta-8W-2Hf) pressure vessel which is the major structural member in the

* Atomics International Contract No. AT(29-2)-2338)

7-05-245-9

Figure 1. LRHS Capsule Configuration

design. To protect the T-111 material from oxidation at elevated temperature, the capsule is clad with Pt-10Rh. To prevent interdiffusion of the noble and refractory metals, there is an alumina diffusion barrier. An iron titanate emissivity coating is applied to the outer surface of the platinum alloy.

This report describes the development and fabrication of the T-111 pressure vessel which is shown in Figure 2. This vessel consists of a deep drawn T-111 structural shell with an OD of 1.675 inches, a length of 6.500 inches, and a 0.127 inch wall. The second part is a coined T-111 end cap also with a 0.127 inch wall. The end cap has been developed with a T-111 boss for joining to a Ta-10W pressure feed through tube which is required for pressurizing the vessel during biaxial creep testing.

B. PROCESS SELECTION

At the beginning of the program a survey of refractory metal alloy developers was made to evaluate the state of the technology for fabrication of second generation tantalum base alloys. The processes commonly used for the alloys were compressive in nature, i. e. forging, rolling, extrusion, swaging, etc. However, tensile fabrication techniques were being used for drawing of hollows for tubing. Spinning of Ta-10W had also been sufficiently developed for inclusion as a candidate.

Of the processes surveyed three were selected for screening evaluation:

- 1) Spinning of Plate
- 2) Back Extrusion of Bar
- 3) Deep Drawing of Plate

The program schedule required that the semi-stock be specified, ordered, fabricated, inspected, received, and the fabrication process developed in six months. Therefore an early decision on the fabrication process was required.

Figure 2. T-111 Pressure Vessel Design

On the basis of the requirements for deep drawing or spinning, specifications for plate were prepared and negotiated, and an order was placed for 0.170" thick stock.

Tooling was fabricated for both spinning and deep drawing using copper blanks of the required shape for tooling checkout. Upon receipt of material a T-111 plate was fabricated by each of the two processes to determine feasibility. The initial effort on spinning indicated that substantial modification and strengthening of the tooling would be required in order to fabricate the part by this technique. The part resulting from the initial attempt at spinning is shown in Figure 3. The result of the first deep drawing was a cup which, while heavily galled, did establish feasibility of the process. This first deep drawn part is shown in Figure 4.

During approximately the same period of time the Metals Division of Norton Company, Newton, Massachusetts, and Torrance Extrusion Corporation, Banning, California, experimented with back extrusion of T-111 bar for the fabrication of tube hollows. The result was a blind cup very close to that required for the program. However, due to the nature of the forming operation, the leading (open) section of the shell received very little mechanical working. The structure of the leading end was essentially that of the starting billet. There was, therefore, a grain size transition between the leading end and the remainder of the shell. It was concluded that this transition could cause a poor response to heat treatment, and that the properties of the unworked region might be inferior to the remainder of the shell. It does appear, however, that this would be an excellent process for forming ironing blanks for a two stage fabrication process - blank formation by back extrusion followed by ironing to form the final part. Semi-stock could be formed by forging and bar rolling and could well be cheaper than plate. Some problems still do remain to be worked out relative to die wear in the extrusion process.

Figure 3. T-111 Plate after Initial Spinning attempt

Figure 4. Cross Section of First T-111 Deep Drawn Part

At this point in the program the three fabrication processes were evaluated with reference to the total program requirements. Based on demonstrated feasibility and uniformity of material work, deep drawing was selected as the reference fabrication process. Effort was then concentrated on refining the drawing parameters.

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II. MATERIALS

A. SEMI-STOCK FABRICATION

There were two basic requirements for the T-111 semi-stock to be fabricated on this program. First, 650 lbs. were required to be from one single heat of material. Secondly, the stock had to be amenable to severe triaxial deformation.

The first requirement was a restraint imposed by the application of the finished parts. A large portion of the pressure vessels fabricated from this stock are to be the test specimens in a biaxial creep program. The intent of that program is to obtain creep relationships between these vessels for creep model verification. Therefore heat variations would only tend to confound the experimental data. This requirement eliminated a possible consolidation practice of melting small ingots for processing to sheet. This did not necessarily impose a problem on any of the potential suppliers; it did however, impose a production practice which would be designed for maximum yield. It is here that the two basic requirements of maximum yield and amenability to triaxial deformation had to be weighed by the fabricator. That is to say one schedule may give maximum yield but not the desired fabrication properties. However, this did not appear to impose a feasibility problem on the four alloy fabricators contacted.

The second requirement was not easily specified. In that the alloy had not been fabricated into this shape previously, it was difficult to define the condition which would be most amenable to severe blind drawing and ironing, or to spinning. The properties specified were mechanical isotropy, grain size and allowable grain size variation, maximum interstitial contents, internal soundness and surface imperfections. These specifications are detailed in Topical Report No. 1⁽¹⁾.

(1) "Large Radioisotope Heat Source Capsule Program - Topical Report No. 1 Capsule Materials Specifications," NAA-SR-12635, January 1968.

While the specification is a general plate and sheet specification it was written specifically for this fabrication effort. Prior to the procurement of material the fabrication program was reviewed with each of the potential suppliers. Additional requirements which are only implied in the product specification were discussed. These were in the form of processing methods. For example, in order to achieve the mechanical isotropy the material should be cross fabricated, and to achieve the grain size the material should have a nominal 75% cold work prior to the final heat treatment.

The material produced for the program was fabricated in the following sequence. The conditioned 6-3/4 inch diameter ingot was hot forged to sheet bar, squared cold to minimize edge losses during subsequent rolling, and process annealed. The sheet bar was sectioned and cold cross rolled to slab. Following a process anneal the slab was straight rolled to finished plate.

B. PREPARATION OF DRAW BLANKS

The T-111 draw blanks used in this development effort were cut from 10" x 10" x 0.170" plate. In the early development of this process, a 10" diameter blank was used to establish the feasibility of "Cold Drawing" of T-111. As this development effort continued, the blank size was gradually reduced with the final blank size being 6-1/2" diameter. All T-111 shells presently being produced for the LRHSC Program are from 6-1/2" diameter blanks.

C. DIE AND INSERT MATERIALS AND DESIGN

It was originally thought that the draw rings could be fabricated from 4130 alloy steel. However, severe galling of the T-111 was experienced when the steel rings were used and it was necessary to go to a self-lubricating material for the draw rings. Aluminum-bronze was chosen for this self-lubricating material.

One steel draw ring and seven aluminum-bronze draw rings are now used in the fabrication sequence of the T-111 shell. The punches used to

push the T-111 through the dies are made from hardened 4130 alloy steel and are polished to a No. 8 micro-finish. In order to maintain concentricity of the ID to OD during forming and to minimize the amount of "Earing" during drawing, a 4130 alloy steel concentricity fixture is employed during the third through the eighth draws. Figures 5 through 12 show the eight stage tooling used to produce the T-111 shell.

The tooling used to form the T-111 end cap is made from 4130 alloy steel and is shown in Figure 13. Forming of the end cap is accomplished in this single stage die.

D. PRESS DESIGN

The hydraulic press used in the forming of the shell and end cap is shown in Figure 14. This press has a 150 ton load capability with simultaneous operation of the table used for positioning and holding of the concentricity fixture during drawing.

Figure 5. Die No. 0 - 4130 Alloy Steel Die
and Punch

Figure 6. Die No. 1 - Al-Bronze Draw Ring
with Steel Punch

Figure 7. Die No. 1a - Al-Bronze Draw Ring with Steel Punch and Concentricity Fixture

Figure 8. Die No. 2 - Al-Bronze Draw Ring with Steel Punch and Concentricity Fixture

Figure 9. Die No. 2a - Al-Bronze Draw Ring with Steel Punch and Concentricity Fixture

Figure 10. Die No. 3 - Al-Bronze Draw Ring with Steel Punch and Concentricity Fixture

Figure 11. Die No. 4 - Al-Bronze Draw Ring with Steel Punch and Concentricity Fixture

Figure 12. Die No. 5 - Al-Bronze Draw Ring with Steel Punch and Concentricity Fixture

Figure 13. T-111 Structural End Cap Tooling

Figure 14. 150 Ton Hydraulic Press Used in Drawing
of T-111 Structural Shell and T-111 Structural
End Caps

III. FABRICATION PROCEDURE

A. T-111 STRUCTURAL SHELL

1. Process Development

Successful fabrication of the first T-111 shell was accomplished on blank S/N 014. Initial tool designs and fabrication techniques were modified extensively to produce this first successful shell. Table 1 tabulates the development history of this fabrication process. The detailed development is presented below.

S/N 001

This 10 inch diameter blank was drawn through the first stage and stalled the press during the second draw. The part was then annealed at 2700°F for 1 hour at a pressure of less than 1×10^{-5} torr. After annealing, the second draw was completed without any problems. The part was then drawn through the third and fourth stages without annealing. The part cracked on the fourth draw.

S/N 002

This 8 inch diameter blank was drawn through the first three stages without annealing. The part was then annealed for 1 hour at 2700°F in vacuum and was subsequently drawn through the fourth and fifth (final) stages. Examination of the part revealed superficial galling.

S/N 003 and 004

Two 5-3/4 inch diameter blanks were drawn through the first stage die successfully. Both parts failed during the second draw. S/N 003 cocked in the drawing process while S/N 004 failed at the end when the part locked in the draw ring. Figure 15 shows S/N 003 after drawing through the second die. Figure 16 shows S/N 004 which pulled apart at the open end during the second draw.

BLANK

S/N	Initial Blank Size	Lubricant Used	Die Material	Concentricity fixture Used	Dies Used								Annealed After X Draw	Anneal Temp.	Anneal Time	Results Obtained (Insert Stage Number)					
					0	1	1a	2	2a	3	4	5				Outside Surface Galling	Inside Surface Galling	Press Stalled	Axial Cracks at Top	Axial Cracks at Bottom	Circum. Cracks at Top
001	10"	Paste	4130 Steel	No		X		X		X			None	None	None						
002	8-1/2"	Paste	4130	No		X		X		X	X	X	None	None	None	1, 2, 3, 4, 5	1, 2, 3, 4, 5	3, 4, 5			
003	5-3/4"	Paste	4130	No		X		X					None	None	None			2		2	
004	5-3/4"	Paste	4130	No		X		X					None	None	None					2	
005	5-3/4"	STP	4130	No		X		X		X			2	2700°F	1 Hr.	3					
006	5-3/4"	Graphite & STP Paste	4130	No		X		X		X			2	2700°F	1 Hr.						
007	5-3/4"		4 & 5 Al Bronze	No		X		X	X	X	X	X	3	2700°F	1 Hr.	3	5				
008	5-3/4"	Paste	4 & 5 Al	No		X		X	X	X	X	X	3	2700°F	1 Hr.	3	5				
009	5-3/4"	Paste	Al Bronze	No		X	X	X	X	X	X	X	3	2550°F	1 Hr.				4	4	
010	5-3/4"	Paste	Al Bronze	No		X	X	X	X	X	X	X	3	2550°F	1 Hr.				4	4	
011	5-3/4"	Paste	Al Bronze	No		X	X	X	X	X	X	X	3	2550°F	1 Hr.				4	4	
012	5-3/4"	Paste	Al Bronze	No		X	X	X	X	X	X	X	3	2550°F	1 Hr.				4	4	
013	6-1/2"	Paste	Al Bronze	Yes	X	X	X	X	X	X	X	X	3	2700°F	1 Hr.		5		3		
014	6-1/2"	Paste	Al Bronze	Yes	X	X	X	X	X	X	X	X	3	2700°F	1 Hr.						

Circum. Cracks at Bottom	Surface Checking	Bottom Punched Out	Other (Explain)	Press Loading (Tons)							Part Utilization			
				0	1	1a	2	2a	3	4		5		
4			Part pulled apart near top during 4										Develop.	
													Develop.	
		2	Part cocked during 2 draw		115		120						Develop.	
			Pulled apart during 2 draw		115		120						Develop.	
		3			115		120		110				Develop.	
		3			115		120		110				Develop.	
					115		120	115	110	75	75		Develop.	
					115		120	115	110	75	75		Develop.	
		A	Uncleaned prior to anneal, also too short		115	110	105	75	75	70	60		Develop.	
		A	"		115	110	105	75	75	70	60		Develop.	
		A	"		115	110	105	75	75	70	60		Develop.	
		A	"		115	110	105	75	75	70	60		Develop.	
			Used Concentricity Fixture		60	115	110	105	75	75	70	60		Develop.
			Used Concentricity Fixture		60	115	110	105	75	75	50	60		Develop.

Table 1. T-111 Structural Shell
Fabrication History

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Figure 15. T-111 Shell S/N 003
Cocked During Second Draw Through
Steel Die

Figure 16. T-111 Shell
S/N 004 Pulled Apart at
the Top After the Draw

S/N 005 through 008

Four T-111 plates were deep drawn through the first and second stages and the parts annealed at 2700°F for 1 hour in a vacuum of 1×10^{-6} torr. After annealing the parts were drawn through the third and fourth stages. During drawing of S/N 005 and S/N 006 through the third stage die the parts locked in the die, resulting in the punch being pushed through the nose end of the part. S/N 007 and 008 were drawn through the third, fourth, and fifth stage dies successfully. However, severe galling of the parts occurred at stage 3 on the OD and stage 5 on the ID. Figure 17 shows S/N 005 with the bottom punched out during the third stage draw.

Investigation of S/N 005 and 006 failures indicated that the material reduction was too great resulting in a forming force that failed the parts in tension. Two intermediate dies (1a and 2a) were added to reduce the amount of material being moved per draw in order to reduce the forming force on the part.

S/N 009 through 012

To eliminate the galling experienced in deep drawing of the T-111 structural shell, new draw rings were fabricated from aluminum bronze. With the draw rings softer than the T-111, the hypothesis was that any galling or wear should damage the draw ring and not the part.

Based on this evaluation four 5-3/4 inch diameter T-111 blanks were drawn through the single steel and seven aluminum-bronze dies to form four T-111 structural shells. Annealing of parts between draws was determined by hardness readings measured after each draw. Table 2 shows the fabrication sequence used for these four parts.

Immediately after annealing and drawing through die No. 4, all four shells showed longitudinal surface tears and checking on the outside diameter of the parts. After drawing through the final sizing die, inspection of the shells indicated there were four problems to be solved. These were surface tears, shell length, internal galling and concentricity.

Figure 17. T-111 Shell S/N 005 with Bottom Punched out During Third Stage Draw After Annealing at 2700°F/1 Hour

TABLE 2

T-111 SHELL FABRICATION SEQUENCE

<u>Die Draw</u>	<u>Ra</u>	<u>Annealed</u>	<u>Temperature</u>	<u>Die Material</u>
1	63	No	---	Steel
1a	65	No	---	Al Bronze
2	66	No	---	Al Bronze
2a	67	No	---	Al Bronze
3	68	Yes	2700°F/1 Hr.	Al Bronze
4	66	No	---	Al Bronze
5	--	No	---	Al Bronze

Preliminary analysis indicated that the surface tears were due to aluminum bronze from the dies being deposited on the T-111 and not being completely removed before annealing. To avoid this problem, NaOH and nitric acid cleaning after each draw was used on all subsequent parts up to S/N 014.

The four shells were under size in length due to "earing" which required trimming of the parts at the open end. The blank diameter was increased from 5-3/4" to 6-1/2" for trim allowance required by this earing effect.

Examination of the galling on the ID of the shells indicated that the draw punch needed polishing and must be removed in such a manner as to eliminate any cocking of the punch as it is withdrawn from the shell. A removal fixture was designed to assure alignment of the punch and shell during the operation.

Due to uneven load distribution on the shell during final sizing, the ID and OD were eccentric approximately 0.050". To correct this condition a guide and locating fixture was designed to support the shell during drawing and final sizing.

One of the first T-111 shells drawn through the aluminum bronze dies is shown in Figure 18. The shell is short in length and has many small surface checks. Figure 19 shows the design of the concentricity fixture utilized during the remaining draws to maintain the ID to OD concentricity of the shell to 0.005" total indicator reading (TIR). The punch diameter "A" is concentric to the draw ring diameter "B" by mechanics of tool alignment. Diameter "C" on the T-111 part is round by the mechanics of drawing around the punch on the previous draw. Therefore, by machining the ring, diameter "X" is concentric to diameter "Y" which, when utilized in the drawing process as shown in Figure 20, aligns diameter "C" so it is concentric with diameter "A" within 0.005" TIR. All shells fabricated utilizing this concentricity fixture were drawn with the ID's concentric to the OD's within this 0.005" allowable.

Figure 18. T-111 Shell Drawn from 5-3/4" diameter Blank. Part was contaminated during Annealing Resulting in Surface Cracks Appearing in the Drawing Sequence Immediately following Annealing at 2700°F per One Hour

Figure 19. Concentricity Fixture Design Capable of Maintaining .005 TIR Concentricity of Shell ID to OD.

Concentricity Fixture

T-111 Shell

Al-Bronze Die

Figure 20. T-111 Shell Ready to be Deep Drawn Through Die Number 7.

S/N 013

This 6-1/2" diameter blank was the first shell drawn through the fabrication sequence utilizing the concentricity fixture. After annealing at 2700°F for 1 hour following draw No. 3, two longitudinal surface cracks were exposed approximately 3/4" from the open end of the part. These appeared to be gathering defects. The end 3/4" was removed from the open end and the part drawn through the fourth and fifth dies. During the fourth draw two small surface tears developed approximately 1/8" from the tangent point of the closed end. The part was then drawn through the final die to see if any new tears developed. No new tears were visible. However, the two existing tears opened up. Investigation indicated that the table load holding the concentricity guide fixture was too large, resulting in a "torquing" of the material and thus causing the gathering defects.

S/N 014

This 6-1/2" diameter blank was drawn through all dies through the No. 3 die and held from further fabrication pending metallographic review of the annealing cycle.

After careful examination of both the T-111 structure drawn through the first six of eight dies and the annealing time/temperature cycle being employed after the sixth draw, the T-111 shell was annealed at 2700°F for 1 hour and successfully drawn through the final two stages of the fabrication process. Throughout this fabrication sequence, chemical etching of the T-111 was used after each draw as an inspection technique to examine the part for surface defects. Figure 21 shows this T-111 structure after passing through the eight stage deep drawing process. The shell is 1.674" in diameter and 8.000" long. Six and one-half inches is required for the test capsule structural shell while the last 1-1/2" can be used in the before and after welds required when the T-111 shell is electron beam welded to the T-111 end cap.

Figure 21. T-111 Structure Shell

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This was the first shell successfully fabricated and was designated for use on Creep Capsule B-1a, the first creep capsule to be delivered to test. The fabrication sequence for the T-111 plate as it passed through the forming sequence is shown in Table 3.

2. Metallurgical Analysis

A number of problems occurred during the development of the deep drawing process. The phenomenological aspects of the problems and solutions are detailed in the previous section. This section deals with the analytical metallurgy associated with the solution of these problems.

a. Tensile Rupture of the Blank

After carefully analyzing those parts which failed by punching out the ends of the drawing blank, in a number of different drawing stages, it was concluded that the excessive loads required to deep draw the blank were associated in all cases with a breakdown of the lubricant and contact of the blank with the hardened steel die. This breakdown caused a cold welding of the part to the die resulting in superficial tearing of both the die and blank. This phenomena is observed in the early development of new drawing processes and is commonly referred to as galling. One approach to the solution of this problem is the selection of a lubricant which will withstand the high compressive stresses required by the process.

Some of the high compressive stress lubricants investigated included stearates, STP (a commercial extreme pressure lubricant, one of the better intermediate temperature films) and additions of solid lubricants such as hexagonal carbon and molybdenum disulfide mechanically mixed with the basic carriers. While the experiments indicated that a satisfactory lubricant could be mixed, it was concluded that a more positive solution would be to use a softer die material. This concept is used in many industrial wear applications.

PART NO N73420003-01
 SERIAL NO 014

T-III DEEP DRAW FORMING
SEQUENCE

DATE 2/14/68

T-III BLANK

DRAW #0

LOAD 600 Tons

REMOVED -

DRAW #1

LOAD 1150

REMOVED .13

DRAW #1A

LOAD 1100

REMOVED .075

DRAW #2

LOAD 1050

REMOVED .095

HOLD DOWN LOADS ON DRAWS
 # 1A, #2, #2A, #3, #4 & # 5 = 90 lbs.

DRAW #2A

LOAD 75.0

REMOVED .075

DRAW #3

LOAD 75.0

REMOVED .070

DRAW #4

LOAD 50.0

REMOVED -

DRAW #5

(FINAL)
 LOAD 70.0

REMOVED .125

Table 3. T-III Fabrication Configuration During Eight Draw Sequences which Produced the First T-III Structural Shell

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Aluminum bronze was selected as a die insert material. To reduce compressive loads the size of the reduction at each step was reduced by the introduction of intermediate dies when the blank and, therefore the load on the part, was large. This solution eliminated the tearing and galling problems.

b. Crazing of the Blank Surface

After solution of the galling/end-punch-out problem, the drawing process appeared in control. Four blanks were committed for tooling checkout. After drawing the blanks through the 0, 1, 1a, 2, 2a, and 3 dies, the parts were heat treated (1 hr. at 2700°F) to recover the alloy's fabricability. (It is noted that this alloy had previously never been shaped to such an extreme blind L/D as through the No. 3 die.) Upon subsequent reduction, all four parts exhibited multiple surface cracks which in the glass industry would be termed crazing. This condition is shown in Figure 22. Examination of the blanks (now 3" dia. x 5" long) indicated that the cracking occurred whenever the bronze dies had been in contact with the alloy prior to the heat treatment. The die wash apparently had not been adequately cleansed from the part surface prior to heat treatment. If it alloyed with the part during the heat treatment and embrittled its surface, the die wash could be the source of the crazing. To test this hypothesis, the next blank was cleansed in caustic followed by a nitric acid soak between each reduction. This procedure was designed to remove all of the bronze die wash but not require special handling facilities.

Results of a test of a blank going through this process proved conclusively that this procedure eliminated the crazing problem.

c. Gathering and Nose Deformation

The part used to prove out the die wash postulate exhibited two additional phenomena, either of which would render the part unacceptable for creep testing. A series of discontinuities (three or four observed) in the open end of the cup occurred and two cracks at the nose were observed.

(Approx. 8X)

Figure 22. Multiple Cracks on the surface of the T-111 Shell drawn through bronze dies, heat treated and redrawn. Cracks appeared on the redraw

The discontinuities near the mouth of the shell were machined out with a single point tool. The maximum indicated depth was 0.050" of the 0.215" gathered wall which left the wall very close to the initial thickness of the plate blank (0.160"). It would appear that these defects were a direct wall gathering defect - one which is very common in the early stages of developing a deep drawing process. Upon detailed examination of the process, it was noted that the concentricity fixture was compressively loading the blank during the ironing operations. While no detailed examination of this practice has been performed, parts have since been drawn through the exact process which produced the gathering defects but without the load on the concentricity fixture. None show evidence of the gathering defects.

d. Nose Cracks

The surface of the part which suffered nose cracks was examined. The nose cracks apparently were associated with the deformation line of the No. 4 die, the first after the heat treatment. There was no previous evidence as to why this phenomena should occur. Since the part cracked on the first draw after the heat treatment (1 hr. at 2700°F), the heat treatment was suspect. In any of the early stages of drawing (up to die No. 3) the side walls of the cup received severe deformation while the nose or blind end received virtually none. It was expected that different areas of the part would react differently to heat treatment due to this work variation. A very critical area was at the radius of the nose of the part which had received a small amount of deformation prior to the heat treatment. This is the area that is first deformed after the recrystallization treatment. If there were a variation in recrystallization, grain size or precipitation across the wall or around the radius, poor response to the heat treatment could well account for the subsequent cracks in this area.

A scrap part which had been previously drawn through dies 1, 2, and 3 was sectioned for a heat treating study. While the fabrication sequence of this part was not absolutely identical with the present process, it was sufficiently close to warrant evaluation. To bracket the 1 hr. at 2700°F, samples were treated for 1 hr. at 2500, 2600, 2700, and 3000°F.

The as-fabricated part showed conclusively that there was very little work in the blind end of the cup as indicated in Figure 23. The grains are relatively equiaxed and uniform across the wall in the flat nose region, but into the radius there is a transition across the wall evident in the same figure. The structure on the outer surface is relatively highly worked as evidenced by the elongated grains, with a transition to the relatively equiaxed grains at the inner region of the cup. In the side wall there is a relatively uniform region of deformed grains, completely across the wall. The continuous nature of the grain boundaries indicates that the part could possibly be further deformed lightly

Figure 23. Grain structure through wall of T-111 cup after six stages of deep drawing as fabricated prior to anneal.

BLANK

without rupture. An example of the structure further deformed is shown in Figure 24, typical of the side wall of a part fabricated to the finished shape (through die No. 5).

The structure of the heat treating study part (dies 1, 2, and 3) after 1 hr. at 2500°F is shown in Figure 25. Except for partial recrystallization in some of the higher worked regions, there appears to be very little change from the as-fabricated condition. While recovery has occurred, sufficient recrystallization for further fabrication has not.

The same lack of recrystallization is true for the 1 hr. at 2600°F. There is still not enough recrystallization for the sizable deformation during the ironing that occurs in the No. 4 and 5 dies.

The sample heated for 1 hr. at 3000°F is shown in Figure 26 and exhibits what could be considered a poor response to the heat treatment. On the flat section of the blind dome the grains are completely equiaxed and there is some evidence of partial grain growth in the central regions of the plane. In the wall

Figure 24. Microstructure of T-111 Shell After Drawing through Die Number 5. (100 X)

BLANK

GRAIN STRUCTURE THROUGH WALL OF T-111 CUP AFTER SIX STAGES OF DEEP DRAWING WITH ANNEAL AT 2500°F FOR ONE HOUR

Figure 25. Grain structure through wall of T-111 Cup after six stages of deep drawing with anneal at 2500°F for 1 Hr.

BLANK

GRAIN STRUCTURE THROUGH WALL OF T-111 CUP AFTER SIX STAGES OF DEEP DRAWING WITH ANNEAL AT 3000°F FOR ONE HOUR

Figure 26. Grain structure through wall of T-111 Cup after six stages of deep drawing with anneal at 3000°F for 1 Hr.

BLANK

of the cup complete recrystallization has occurred and some partial grain growth. While in this region there is evidence of preferred growth in bands, this does not occur further up the wall. In the radius there has been complete recrystallization and major grain growth on the outer surface.

The location of the section in the figure was selected to show the transition across the wall. In the mid radius, the grain growth is complete across the wall, and the transition to the smaller grains in the side wall is not nearly as steep as in this section. The location of the section shown with the transition is approximately where the next die will deform first. This type of transition could well render the part incapable of further fabrication without cracking.

The 1 Hr. at 2700°F sample is shown in Figure 27. There is complete recrystallization throughout the part and the grains are relatively homogeneous throughout. There is evidence of grain growth in the radius, however, the variation cannot be considered severe.

On the basis of this study it is concluded that 1 Hr. at 2700°F is the optimum heat treatment for the part and other causes for the nose cracks had to be sought. Since this study was completed, six cups have been drawn without evidence of the nose crack. The source of the crack could well have been an inhomogeneity in the starting blank.

B. T-111 STRUCTURAL END CAPS

Four T-111 blanks for structural end caps were drawn through the first stage die, annealed for 1 Hr. at 2700°F and sized to the final configuration of the end cap.

During the second stage die operation the boss on the end cap failed to form. This was due to bridging of the punch load to the female die so that force was unavailable to form the boss. Four new blanks were prepared with the material for the boss protruding from the blank (see Figure 28). These four blanks were drawn through the final die and inspected. Two problems developed:

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GRAIN STRUCTURE THROUGH WALL OF T-111 CUP AFTER SIX STAGES OF DEEP DRAWING WITH ANNEAL AT 2700°F FOR ONE HOUR

Figure 27. Grain structure through wall of T-111 Cup after six stages of deep drawing with anneal at 2700°F for 1 Hour.

BLANK

Figure 28. T-111 Structural End Cap Blank

1) Due to the small boss to plate radius (0.010 R), the material separated at this point resulting in a 0.010" - 0.015" deep crack.

2) Due to the blank configuration coupled with the high loading of the press (approximately 70 ton), the T-111 material thinned at the nose.

Evaluation of the blank design and the forming process resulted in the following modifications:

1) A full radius was provided at the boss-to-plate transition to eliminate the cracking of the material at the boss during forming.

2) To eliminate the thinning at the end of the cap, tapering of the blank plus forming at a reduced load was employed. The constant load was set at 30 ton. The blank size is shown in Figure 29.

Three T-111 blanks were prepared for drawing and were formed to the "structural end cap with the feed-through" configuration. A boss formed without cracking on these parts but it was shorter than desired for welding.

A metallographic analysis of sectioned T-111 end caps was conducted in the as-received and heat treated conditions. This effort was directed at determining what degree of work had been done in the vicinity of the boss and how this structure responds to heat treatment. Inspection of the wall thickness indicated that the 0.140" dimension of Figure 29 should be increased to 0.146 and the 0.120 dimension decreased to 0.118 to provide the 0.127 constant wall thickness. Figure 30 shows the wall measurements taken on the T-111 end caps formed from the blank design shown in Figure 29.

Ten new end cap blanks were prepared to the new blank design, satisfactorily formed at the 30 ton constant forming force, cleaned and annealed at 3000°F for 1 hour, and delivered to weld development. These end caps had the feed through attachment boss lengthened by T-111 "TIG" weld build-up to provide a satisfactory weld joint for the EB welding of the Ta-10W pressure feed-through tube to the T-111 boss.

Figure 29. Revised T-111 Structural Cap Blank Design

Figure 30. End Cap T-111 Structural Cap Wall
Variations After Forming

C. T-111 PRESSURE VESSEL ASSEMBLY

The T-111 structural shell S/N 014 was machined to final dimensions to provide a constant wall thickness of $.1240 \pm 0.0005$. The shell was then final heat treated 1 hour @ 3000°F. The final 1-1/2 inch of this shell was removed and machined to provide the T-111 shell "Before" and "After" weld material with the T-111 end caps.

The Ta-10W pressure feed-through tube was welded to the T-111 end cap and the cap final machined to provide proper fitup for welding with the T-111 shell. After welding of the pressure feed-through to the end cap, helium leak check and dye check inspections were completed satisfactorily on the assembly.

The T-111 shell (S/N 014) was helium leak checked, ultrasonically and dimensionally inspected, and the final OD, ID, wall thickness and length were recorded. Weld development of the T-111 shell to cap was completed and the T-111 shell was EB welded to the T-111 cap. The capsule was then helium leak checked and ultrasonically inspected. The feed-through flange was then brazed to the Ta-10W tube. Following all assembly operations the capsule was proof tested successfully at 10,000 psi.

The "Before" and "After" weld samples for the EB weld of the T-111 shell to T-111 end cap are shown in Figure 31. The T-111 structural shell and the T-111 end cap with the Ta-10W tube welded in place are shown prior to final EB welding in Figure 32. The capsule after welding is shown in Figure 33.

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Figure 31. T-111 Structural Shell "Before" and "After" Weld Samples

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Figure 32. T-111 Structural Shell and End Cap with RF Tube

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Figure 33. T-111 Structural Shell and End Cap with RF Tube

IV. CONCLUSIONS

The above described development program has demonstrated conclusively that fabrication of T-111 alloy (Ta-2Hf-8W) for pressure vessel use can be accomplished by the deep drawing/ironing process. The reproducibility of the process is considered adequate for a process in an early stage of development. Although the discussion of the development of this process was confined to the successful development of the first T-111 structural shell on blank S/N 014, continuation of the development of this process has resulted in the satisfactory fabrication of six additional T-111 structural shells.

It is also concluded that definition of draw ring material, die design, annealing time and temperature, and press loading is a critical part of the process development.

Although a development program to provide a satisfactory lubricant could allow the use of steel dies, it appears more economical at this time to fabricate dies from self-lubricating material such as Al-Bronze. However, this does require a new "last stage" die and moving up of the dies from stage to stage as the dies wear. Approximately 6 to 8 parts can be pushed through a single die before it becomes excessively worn.