SANDIA REPORT

SAND97-2609 • UC-713

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Printed October 1997

SAND--97-2609

Milliwatt Generator Heat Source Shelf-Life-Related Pressure-Burst Capsule Evaluations

Brian D. Ritchey, Kenneth H. Eckelmeyer, Alice C. Kilgo, Bonnie B. McKenzie

Prepared by

Sandia National Laboratories

Albuquerque, New Mexico 87185 and Livermore, California 94550

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Brian D. Ritchey
Energy Storage Device Testing and Evaluation Department
Kenneth H. Eckelmeyer, Alice C. Kilgo, and Bonnie B. McKenzie
Microstructural Characterization Department

Sandia National Laboratories
P.O. Box 5800
Albuquerque, New Mexico 87185-0613

Abstract

Three Milliwatt Generator Heat Source (MWGHS) shelf-life-related capsules were pressure-burst tested after thermal aging. Shelf-life capsules PB-08-03 (MC2893) and MPT-11-33 (MC3599) were tested at 1010°C and capsule PB-07-13 (MC2893) was tested at 1100°C. Subsequent to pressure-burst testing, each capsule was bubble-leak tested then metallographically examined. Post-mortem evaluation consisted of metallography, microhardness, scanning electron microscopy (SEM), and oxygen and nitrogen analysis. Capsules PB-08-03 and PB-07-13 failed by elevated temperature stress-rupture in the coarse-grained cap to body welds, as has been documented for previous capsules. Pressure-burst testing of capsule MPT-11-33 was terminated prior to failure after 739 hours on test at 1010°C, however, microscopic examination of the weld indicated that similar failure had begun to occur in the interior portion of the capsule to body weld. Evidence was obtained indicating that metallurgical changes occurred during the pressure-burst test performed at 1100°C. The metallurgical observations of a preferred site for elevated temperature deformation and fracture (the coarse-grained weld region) and structural instability (the property changes during testing at 1100°C) need to be considered in component lifetime prediction and modeling efforts.

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Contents

1.0 Shelf-Life-Related Pressure-Burst Capsule Evaluations.....	7
2.0 Pressure-Burst Test And Bubble-Leak Test Results.....	7
2.1 Pressure-Burst Test Information for MWGHS Capsule PB-08-03.....	7
2.2 Pressure-Burst Test Information for MWGHS Capsule MPT-11-33	7
2.3 Pressure-Burst Test Information for MWGHS Capsule PB-07-13.....	8
3.0 Metallographic and SEM Examination Results	9
3.1 Surface Characterization	9
3.1.1 Weld Bead On Inside of Capsules	9
3.1.2 Weld Bead On Outside of Capsules.....	9
3.1.3 Interior Surfaces of Cap, Side Wall, and Dome.....	10
3.2 Microstructural Characterization.....	11
3.2.1 Cap Microstructures.....	12
3.2.2 Dome Microstructures.....	13
3.2.3 Side-Wall Microstructures	13
3.2.4 Weld Microstructures and Crack Morphologies.....	14
3.3 Hardness Characterization	16
3.4 Chemical Characterization.....	23
4.0 Summary And Conclusions.....	23
4.1 Failure Mode	23
4.2 Metallurgical Variability and Changes During Testing.....	24
4.3 Differences in Welding Procedures	24
4.4 Differences in Oxygen Analysis Results from SNL/NM and Mound Laboratories.....	24
Appendix A. Pressure-Burst Test Post-Mortem Evaluation Data Sheet Capsule PB-08-03.....	25
Appendix B. Pressure-Burst Test Post-Mortem Evaluation Data Sheet Capsule MPT-11-33.....	26
Appendix C. Pressure-Burst Teat Post-Mortem Evaluation Data Sheet Capsule PB-07-13.....	27

Figures

1	Scanning electron micrograph showing intergranular cracking along weld metal-side wall interface on interior surface of PB-07-13. 30x.....	9
2	Higher magnification view of area in Figure 1. 200x.....	9
3	Scanning electron micrograph showing grain boundary sliding and possible leak sites near weld tail-out termination site on exterior surface of PB-08-03. 50x.....	10
4	Higher magnification view of area in Figure 3. 150x.....	10
5	Light macrograph of weld exterior tail-out region in PB-07-13. 17x.....	11
6	Light macrograph of weld exterior tail-out region in MPT-11-33. 17x.....	11
7	Scanning electron micrograph of interior surface of dome in MPT-11-33 showing some grain definition as a result of grain boundary sliding. 500x.....	11

9	Microstructure of cap material in MPT-11-33. 100x.....	12
10	Higher magnification view of cap microstructure in MPT-11-33 showing "grain boundary second phase." 500x.....	12
11	Cap microstructure near weld heat affected zone, right. 100x.	13
12	Cap microstructure in PB-07-13. 100x.	13
13	Dome microstructure in MPT-11-33. 100x.....	14
14	Higher magnification view of dome microstructure in MPT-11-33. 500x	14
15	Microstructure of side wall in MPT-11-33. 100x.	14
16	Microstructure of weld fusion zone and side wall heat affected zone in PB-08-03. 50x, interference contrast.	14
17	Banded substructure and intergranular cracking in weld fusion zone in tail-out region of PB-07-13. 80x.....	15
18	Banded substructure and intergranular cracking in weld fusion zone opposite tail-out region in PB-07-13. 80x.	15
19	Vickers microhardness trace across side wall of capsule PB-08-03.....	17
20	Vickers microhardness trace across dome of capsule PB-08-03.	17
21	Vickers microhardness trace across cap of capsule PB-08-03.....	18
22	Vickers microhardness trace across weld of capsule PB-08-03.	18
23	Vickers microhardness trace across side wall of capsule MPT-11-33.	19
24	Vickers microhardness trace across dome of capsule MPT-11-33.....	19
25	Vickers microhardness trace across cap of capsule MPT-11-33.	20
26	Vickers microhardness trace across weld of capsule MPT-11-33.....	20
27	Vickers microhardness trace across side wall of capsule PB-07-13.....	21
28	Vickers microhardness trace across dome of capsule PB-07-13.	21
29	Vickers microhardness trace across cap of capsule PB-07-13.....	22
30	Vickers microhardness trace across weld of capsule PB-07-13.	22

Tables

1	Means and Standard Deviations of Vickers Microhardnesses Measurements from Side Walls, Domes, Caps, and Welds*	16
2	Oxygen Analyses from Side Walls, Domes, Caps, and Welds (ppm)	23
3	Nitrogen Analyses from Side Walls, Domes, Caps, and Welds	23

1.0 Shelf-Life-Related Pressure-Burst Capsule Evaluations

Three MWGHS shelf-life-related capsules were pressure-burst tested at SNL/NM, B894/140E. The pressure-burst test consists of an individual capsule being placed in a fast-ramping, high vacuum furnace (tester) capable of detecting helium in minute quantities. The furnace is quickly and precisely raised to the test temperature (1010°C or 1100°C) and maintained until a vent of helium is detected from the capsule. Test capsules exceeding 200 hours test time are individually evaluated for merit to continue on test. When the furnace is cool, the capsule is removed in preparation for post-mortem examination and testing.

A summary of fabrication data, thermal aging time and temperature, test information, and metallographic and SEM examination results for each of the three capsules tested is recorded on the post-mortem evaluation data sheets (Appendices A, B, and C).

Information and discussions relative to pressure-burst testing and bubble-leak testing of the shelf-life capsules are outlined in Section 2.0 of this document. Section 3.0 outlines the metallographic examination results for each of the capsules tested.

2.0 Pressure-Burst Test And Bubble-Leak Test Results

2.1 Pressure-Burst Test Information for MWGHS Capsule PB-08-03

Capsule PB-08-03 was the first shelf-life-related pressure-burst test capsule tested at SNL/NM. The capsule was pressure-burst tested at 1010°C for a total of 535 hours. Analysis of test data indicated that after 535 hours on test, the tester detected helium at a sufficient quantity to end the test. Upon removal from the tester, the capsule was visually inspected at up to 40 power and a bubble-leak test was performed.

Results from the bubble-leak test, which has a low end detection sensitivity of 1×10^{-5} atm-cc-sec⁻¹ (helium), positively identified a leak site near the weld tail-out region of the capsule. Polaroid photographs were taken of the leak site at 10 power. Metallographic evaluations performed subsequent to the bubble-leak test confirmed the leak site in the weld tail-out region of the capsule. Reference Section 3.0 of this document for a thorough discussion of the

metallographic and SEM examination results for this capsule.

A summary of fabrication data, thermal aging time and temperature, test information, and metallographic and SEM examination results for capsule PB-08-03 is recorded on the post-mortem evaluation data sheet (Appendix A).

2.2 Pressure-Burst Test Information for MWGHS Capsule MPT-11-33

Capsule MPT-11-33 was pressure-burst tested for a total of 739 hours at 1010°C. Analysis of the test data indicates this pressure-burst test was characterized by several false end of test scenarios caused by B894 power surges (as a result of local lightning storms), chilled water supply pump switching, and off-gassing of helium from the tester ion pump. The tester performed as designed for each scenario and shut off the test.

Upon removal from the tester, the capsule was visually examined at up to 40 power and bubble-leak tested. Visual observation indicated the capsule had swelled in the dome region. No bubbles indicative of a leak site were observed as a result of the bubble-leak test with a low end detection sensitivity of 1×10^{-5} atm-cc-sec⁻¹ (helium).

A summary of fabrication data, thermal aging time and temperature, test information, and metallographic and SEM examination results for capsule MPT-11-33 is recorded on the post-mortem evaluation data sheet (Appendix B).

Metallographic evaluations performed subsequent to the bubble-leak test indicated no leak path was found that is consistent with the bubble-leak test results. Reference Section 3.0 of this document for a thorough discussion of the metallographic and SEM examination results for this capsule.

2.3 Pressure-Burst Test Information for MWGHS Capsule PB-07-13

Capsule PB-07-13 was pressure-burst tested for a total of 640 hours at 1100°C. After 16 hours at 1100°C, the tester detected helium at a sufficient quantity to end the test. The test data from this shutdown were very similar to data from previous test runs in which an air leak had occurred in the test system.

Results of a leak test performed on the tester indicated a probable air leak in the thermocouples. Two of the four thermocouples were damaged during the removal process as a result of

the sheaths swelling at high vacuum and temperature. The vacuum worthiness of the damaged thermocouples, therefore, could not be determined. The remaining two thermocouples were determined to be leak tight. The two damaged thermocouples were replaced with new thermocouples and the test system was determined to be leak tight. Because all test data and parameters were strongly indicative of an air leak, the capsule was not bubble-leak tested prior to test restart.

The test was restarted and continued for an additional 624 hours for a total test time of 640 hours. No helium leak indicators were detected by the tester during this additional 624-hour test period.

Upon removal from the tester, the capsule was visually examined at up to 40 power and bubble-leak tested. Results from the bubble-leak test, which has a low end detection sensitivity of 1×10^{-5} atm-cc-sec⁻¹ (helium), positively identified a leak site near the weld tail-out region of the capsule. Polaroid photographs were taken of the leak site at 10 power.

A summary of fabrication data, thermal aging time and temperature, test information, and metallographic and SEM examination results for capsule PB-07-13 is recorded on the post-mortem evaluation data sheet (Appendix C).

Metallographic evaluations performed subsequent to the bubble-leak test confirmed the leak site in the weld tail-out region of the capsule. Reference Section 3.0 of this document for a thorough discussion of the metallographic and SEM examination results for this capsule.

3.0 Metallographic and SEM Examination Results

3.1 Surface Characterization

Each capsule was longitudinally sectioned (cut in half parallel to the fill tube axis) to facilitate examination of the interior and exterior surfaces. The interior and exterior surfaces were characterized by a combination of light microscopy at magnifications of 60 power and lower, and scanning electron microscopy at magnifications of 30 power and higher. Particular attention was given to the weld regions.

3.1.1 Weld Bead On Inside of Capsules

Evidence of grain boundary sliding and intergranular fracture was observed along the weld metal-side wall interfaces in all three capsules. Examples are shown in Figures 1 and 2. This cracking occurred continuously around the entire perimeter of capsules PB-08-03 and PB-07-13, and the cracks were most widely opened in the regions of the weld tail-out. In capsule MPT-11-33, cracking was limited to 40 to 50% of the interface in the weld tail-out region, and only a few isolated cracks were observed 180

degrees from this region. The extent of grain boundary sliding mirrored the extent of cracking—maximum in the weld tail-out regions where extensive cracking was observed, and minimum in the portions of MPT-11-33 where less cracking was observed.

3.1.2 Weld Bead On Outside of Capsules

Evidence of grain boundary sliding was also apparent on the exterior surfaces of each weld, as shown in Figures 3 and 4, however, more occurred in capsules PB-08-03 and PB-07-13 than in MPT-11-33.

Scanning electron micrographs were taken of areas in the weld tail-out regions corresponding to the leak sites identified by bubble-testing of capsules PB-08-03 and PB-07-13, but it was difficult to conclusively identify a *unique* leak site in either capsule. Numerous grain boundaries on which a considerable amount of sliding had occurred were all possible leak sites. However, metallographic examination confirmed that

Figure 1. Scanning electron micrograph showing intergranular cracking along weld metal-side wall interface on interior surface of PB-07-13. (Also note definition of grain boundaries in weld metal resulting from grain boundary sliding.) 30x.

Figure 2. Higher magnification view of area in Figure 1. (Note evidence of grain boundary sliding in both weld metal and side wall heat affected zone.) 200x.

Figure 3. Scanning electron micrograph showing grain boundary sliding and possible leak sites near weld tail-out termination site on exterior surface of PB-08-03. 50x.

intergranular crack paths extended through the entire weld in the vicinity identified by the bubble-leak test. This is discussed in more detail in a following section. A possible leak site in PB-08-03 is shown in Figures 3 and 4. No weld defects were apparent in the tail-out/leak site areas of any of the capsules.

Additional characterization of the capsule weld surfaces, specifically markings corresponding to the weld pulses, revealed an obvious difference between MPT-11-33 and the other two capsules. The surface markings corresponding to the weld pulses were closer together by a factor of about 3 for capsule MPT-11-33, as shown in Figures 5 and 6. This indicates that the ratio of pulse rate to travel speed was considerably higher for MPT-11-33, i.e., the weld in this capsule was more nearly continuous than those in the other two capsules. It is not clear whether this difference in welding conditions was related to the fact that capsule MPT-11-33 was able to withstand 739 hours of testing at 1010°C without failing.

Figure 4. Higher magnification view of area in Figure 3. 150x.

3.1.3 Interior Surfaces of Cap, Side Wall, and Dome

No evidence of cracking was seen on any of the capsules except those associated with the welds. Some evidence of grain boundary sliding, but no cracking, was observed on the dome interiors of each capsule, as shown in Figure 7. This was most apparent near the center (base) of the dome, and decreased upon moving outward toward the side walls.

Little direct evidence of grain boundary sliding was seen on the capsule side-wall interiors except in MPT-11-33, where some sliding was observed near where the side wall blends into the dome. Significant differences were observed, however, between the side-wall surface appearances of the three capsules. The side-wall interiors of PB-08-03 and PB-07-13 were dominated by circumferential machining marks (see side-wall portions of Figures 1 and 2), with occasional evidence of ill-defined "surface rumpling" perpendicular to the machining marks, particularly near the top (weld). In capsule MPT-11-33, however, the circumferential machining marks were much less apparent and the "vertical rumpling" dominated the interior surface appearance, as shown in Figure 8. The

Figure 5. Light macrograph of weld exterior tail-out region in PB-07-13. (Note wide spacing of initial weld pulses, right, and weld tail-out region, left and center.) 17x.

Figure 6. Light macrograph of weld exterior tail-out region in MPT-11-33. (Note narrower spacing of initial weld pulses, right, and weld tail-out region, left and center.) 17x.

Figure 7. Scanning electron micrograph of interior surface of dome in MPT-11-33 showing some grain definition as a result of grain boundary sliding. (Small elliptical features are believed to be residue particles from cutting of capsule.) 500x.

origin of this "vertical rumpling" was not apparent.

No evidence of deformation or any other unusual features were seen on the insides of the caps.

Figure 8. Scanning electron micrograph of unusual side-wall interior surface of MPT-11-33. 200x.

3.2 Microstructural Characterization

Following optical and SEM surface characterization, each capsule was mounted and prepared for metallographic examination. The samples

were polished using standard metallographic procedures, then etched with a solution of 25% HF, 25% HNO₃, 25% H₂SO₄, 25% deionized water. The samples were then viewed and photographed at magnifications ranging from 20 power to 500 power.

NOTE: In reading this portion of the report, it is important to keep in mind that capsule PB-07-13 was pressure-burst tested at a higher temperature (1100°C) than capsules PB-08-03 and MPT-11-33, which were tested at 1010°C.

3.2.1 Cap Microstructures

The cap materials all consisted of heavily banded material. The banding implies that the original ingot material contained chemical inhomogeneties, and that these inhomogeneties were strung out in the plane of the plate during subsequent rolling operations.

All the cap materials exhibited fairly fine grains (ASTM 6-1/2 to 8). Capsule MPT-11-33 exhibited slightly coarser grains than the other two.

The grain boundaries in PB-08-03 and MPT-11-33 were easily revealed with only a limited amount of etching, as shown in Figure 9. Higher magnification examination, as in Figure 10, revealed that dark etching was not confined

to the grain boundaries, but extended out from the boundaries several micrometers into the grains. This implies that substantial variations exist in the vicinity of the boundaries, apparently resulting in the formation of second phases in these regions. Electron microscopy should be employed to better characterize these grain boundary phases.

The grain boundary etching effects diminish as the weld heat affected zone is approached. Within the heat affected zone, the grain boundaries appear sharp and free of obvious second phases, as shown in Figure 11. It appears that the grain boundary phases were resolutionized at the higher temperatures experienced by these regions during the welding process. The fact that the grain boundary phases were not present in the weld heat affected zones suggests that these phases (in the cap base metal) formed during processing of the material, not during aging at 210 or 410°C, nor during pressure-burst testing at 1010°C.

The grain boundaries in PB-07-13 were more difficult to etch and required additional etching time. While extensive banding remained apparent, there was markedly less second phase associated with these grain boundaries, as shown in Figure 12. It is possible that the grain boundary phases were resolutionized at the 1100°C

Figure 9. Microstructure of cap material in MPT-11-33. 100x.

Figure 10. Higher magnification view of cap microstructure in MPT-11-33 showing "grain boundary second phase." 500x

Figure 11. Cap microstructure near weld heat affected zone, right. (Note grain growth and absence of second phase at grain boundaries in heat affected zone.) 100x.

pressure-burst test temperature (90°C higher than that of PB-08-03 and MPT-11-33). This is also consistent with the observations of similar resolution in the weld heat affected zone regions of capsules PB-08-03 and MPT-11-33, described above.

3.2.2 Dome Microstructures

The dome materials are less banded than the cap materials, but contain a substantial number of stringers—apparent impurities strung out in the plane of the sheet during rolling operations.

All of the dome materials exhibited fine grains in the ASTM 8 to 8½ range.

The microstructures in the domes all appear to be relatively free of grain boundary phases, as shown in Figures 13 and 14. However, there was a tendency for the grain boundaries to etch more readily near the inside surface and be more resistant to etching near the outside surface—this was particularly evident in capsule PB-08-03 and least apparent in capsule PB-07-13.

3.2.3 Side-Wall Microstructures

The major portions of the side walls exhibit microstructures similar to and continuous with

Figure 12. Cap microstructure in PB-07-13. (Note absence of grain boundary second phase.) 100x.

those observed in the domes. All of the side walls exhibited grain sizes in the ASTM 7 to 8 range.

The microstructures in the side walls of capsules PB-08-03 and MPT-11-33 change as the weld and heat affected zones are approached. Near the domes the microstructures all appear to be relatively free of grain boundary phases. As the heat affected zones are approached, increasing amounts of grain boundary phases become apparent, as shown in Figure 15. These grain boundary phases appear similar to those observed throughout the cap materials in these capsules. In the portion of the heat affected zone immediately adjacent to the weld, where temperatures were sufficiently high to cause grain growth, these second phases appeared to be resolutionized, as was seen in the cap heat affected zones. It is possible that the original base metal in the side walls and domes of capsules PB-08-03 and MPT-11-33 were relatively free of grain boundary phases, but that these then formed during the welding process in regions that were heated to intermediate temperatures. In regions heated to higher temperatures, these phases were again resolutionized.

Figure 13. Dome microstructure in MPT-11-33. 100x

Capsule PB-07-13 differed in that minimal microstructural changes occurred approaching the weld heat affected zone. As in the cap of this sample, the grain boundaries were relatively free of second phases throughout—irrespective of proximity to the weld.

3.2.4 Weld Microstructures and Crack Morphologies

The predominant features of the weld regions in all three samples were large grains, grain

Figure 15. Microstructure of side wall in MPT-11-33. Grain boundary second phase increases as weld heat affected zone is approached (main portion of photo), then disappears in heat affected zone (top). 100x.

Figure 14. Higher magnification view of dome microstructure in MPT-11-33. 500x

boundaries free of second phases, and bands that denote the recession and rapid advance of the solidification front associated with consecutive weld pulses, as shown in Figure 16.

Grain sizes in the weld fusion zones were consistently very large. In all three capsules examined, fewer than 10 grains spanned the distance from the inside surface to the outside surface of the weld fusion zone. In some cases, single grains extended over half of this zone.

Figure 16. Microstructure of weld fusion zone and side wall heat affected zone in PB-08-03. Note large grains in both weld and heat affected zone, absence of second phases on grain boundaries, bands indicative of weld pulsing, and cracking along grain boundaries. 50x, interference contrast.

All three capsules exhibited intergranular cracks in the weld fusion zone. All of these cracks originated along the interior surfaces where the weld metal joined the side wall. Some of these cracks began in the fusion zone, while others began in the coarsest grained portion of the heat affected zone immediately next to the fusion zone. In all cases, these cracks then extended into the fusion zone toward the exterior surface. All of these cracks were 100% intergranular—proceeding exclusively along the boundaries of the very coarse grains.

In the two capsules that failed (leaked) during pressure-burst testing (PB-08-03 and PB-07-13), the intergranular cracks were found to extend continuously from the interior wall to the outside surface in the vicinity identified by the bubble test as the leak site. An example is shown in Figure 16. Both leaks occurred in the weld tail-out region, but the intergranular crack initiated in, and grew mostly in, the underlying *initial* weld material. Only the *final* portion of crack extension appeared to be through the material that was remelted during the tail-out pulses. Similar, but less extensive, intergranular cracking (extending 40 to 50% from the interior corner to the outside surface) was seen in the weld 180° from the leak site.

Intergranular cracking was also seen in the MPT-11-33 weld and adjacent heat affected zone, but these were confined to the innermost 25% of the wall.

Metallographically observable continuous bands marked the locations where rapid solidification occurred following each weld pulse. These can be seen in Figure 16, but are even more apparent in Figures 17 and 18. A cellular or dendritic substructure, whose characteristic spacing was at least 10 times finer than the grain size, was also observable within the fusion zones. This substructure was particularly evident in the bands. Considerable sample-to-sample differences in the etching response of this substructure were also observed. In PB-08-03, extensive etching was required to reveal the substructure. Even after such etching, the substructure was difficult to see except in the bands. MPT-11-33 also required extensive etching, but afterwards, the substructure was more uniformly evident throughout the weld. In PB-07-13, the bands etched very quickly and darkly, suggesting a significant difference in their composition and structure.

Figure 17. Banded substructure and intergranular cracking in weld fusion zone in tail-out region of PB-07-13. 80x.

Figure 18. Banded substructure and intergranular cracking in weld fusion zone opposite tail-out region in PB-07-13. 80x.

Microhardness measurements and electron probe microanalyses were conducted on PB-08-03 in an effort to determine the differences between the darker etching bands (which apparently corresponded to regions of slow solidification or melt-back during each weld pulse), and the lighter etching regions (corresponding to periods of rapid solidification between weld pulses). The bands exhibited considerably higher hardnesses, ~47 points on the Vickers scale. The electron microprobe measurements showed considerable microsegregation of hafnium (apparently to the dendrite cores) and tungsten (apparently to the interdendritic spaces) in the bands, but much less segregation in the regions of rapid solidification. The reasons for these differences are not yet clearly understood.

Similar microhardness measurements were also made on MPT-11-33 and PB-07-13. MPT-11-33 exhibited similar increased hardness in the banded areas, ~39 points on the Vickers scale. However, PB-07-13, which had been tested at 1100°C, exhibited virtually no differences in microhardness between the bands and regions of rapid solidification.

Electron probe microanalyses were not made on MPT-11-33 or PB-07-13 because of time constraints.

3.3 Hardness Characterization

Microhardness traces were made across the thicknesses of the caps, domes, side walls, and weld fusion zones of each capsule. All measurements were made using the Vickers scale and a 50-gram load. The microhardness traces are shown in Figures 19 to 30. All of the traces plot hardness as a function of position in the wall thickness, starting at the outer surface and moving toward the inner surface.

The average hardnesses for each part are given in Table 1. All of these hardnesses are consistent with the Vickers 230 to 280 range reported for annealed T-111.

Table 1. Means and Standard Deviations of Vickers Microhardnesses Measurements from Side Walls, Domes, Caps, and Welds*

	PB-08-03	MPT-11-33	PB-07-13
Side wall	250 13.5	246 11.3	237 8.7
Dome	256 13.1	251 12.1	245 5.0
Cap	245 8.4	242 9.0	241 6.7
Weld	260 14.2	265 24.2	242 9.1

* 50-gram load.

The average hardnesses of materials from capsules PB-08-03 and MPT-11-33, which were pressure-burst tested at 1010°C, are also very consistent with the hardnesses reported for materials from previous pressure-burst tests. A consistent hardness rank order was observed in these parts: the weld was hardest, followed by the dome, side wall, and cap. Some evidence was also observed of through-thickness hardness variations. In the weld, higher hardnesses were observed in the bands, as described in the previous section. In the base metals, higher hardnesses were generally observed in areas where the grain boundaries were free of second phases and, hence, difficult to etch. This is consistent with what has been reported previously for other capsules.

Capsule PB-07-13, which was pressure-burst tested at 1100°C, exhibited hardnesses slightly lower than those of the other two capsules, particularly in the weld and dome regions. There was also less evidence of hardness variations in the PB-07-13 materials, as evidenced by the lower standard deviations in Table 1.

Figure 19. Vickers microhardness trace across side wall of capsule PB-08-03.

Figure 20. Vickers microhardness trace across dome of capsule PB-08-03.

Figure 21. Vickers microhardness trace across cap of capsule PB-08-03.

Figure 22. Vickers microhardness trace across weld of capsule PB-08-03.

Figure 23. Vickers microhardness trace across side wall of capsule MPT-11-33.

Figure 24. Vickers microhardness trace across dome of capsule MPT-11-33.

Figure 25. Vickers microhardness trace across cap of capsule MPT-11-33.

Figure 26. Vickers microhardness trace across weld of capsule MPT-11-33.

Figure 27. Vickers microhardness trace across side wall of capsule PB-07-13.

Figure 28. Vickers microhardness trace across dome of capsule PB-07-13.

Figure 29. Vickers microhardness trace across cap of capsule PB-07-13.

Figure 30. Vickers microhardness trace across weld of capsule PB-07-13.

3.4 Chemical Characterization

Oxygen and nitrogen analyses were made of samples from the cap, dome, and side-wall regions of each capsule. The results are shown in Tables 2 and 3.

Table 2. Oxygen Analyses from Side Walls, Domes, Caps, and Welds (ppm)

	PB-08-03	MPT-11-33	PB-07-13
Side wall	89	66	89
Dome	112	71	76
Cap	74	96	59
Mean = 81 ppm, Std. Dev. = 16.6			

No significant capsule-to-capsule variations are apparent in these gas analysis results. The overall averages were 81 ppm oxygen and 15 ppm nitrogen.

Table 3. Nitrogen Analyses from Side Walls, Domes, Caps, and Welds

	PB-08-03	MPT-11-33	PB-07-13
Side wall	9	16	15
Dome	12	15	16
Cap	10	26	15
Mean = 15 ppm, Std. Dev. = 4.9			

These oxygen contents are approximately 40% lower than those reported by Mound for T-111 from previous burst tests. Statistical analysis indicated this difference to be significant with greater than 99% confidence.

4.0 Summary And Conclusions

The observations of these three capsules enable several important conclusions to be drawn, and point out a number of areas where increased understanding is needed. A summary of these observations follows.

4.1 Failure Mode

The predominant failure mode in these capsules is classic elevated temperature stress rupture in the weld regions. The large grains in the weld fusion zone and heat affected region make the weld area preferentially prone to grain boundary sliding and intergranular stress rupture failure when pressurized capsules are exposed to simulated fire testing. Grain boundary sliding that eventually results in failure along these boundaries is commonly observed in most metals subjected to high stresses at elevated temperatures. Two of the primary metallurgical approaches to increasing resistance to this type

of failure are to avoid large grains and to align grain boundaries parallel to the applied stress axis. The closure welds between the cap and side wall provide exactly the opposite—very large grains with boundaries aligned perpendicular to the stress axis. As a result, it is not surprising that all three capsules (as well as most of those tested previously) failed by classic intergranular stress rupture in the weld region.

While analyses indicate that higher stresses are present at the center of the dome, and some evidence was seen of grain boundary sliding in this area, the fine grains present in the base metal provide much greater resistance to stress rupture failure. Hence, efforts to model capsule end-of-life should concentrate on the weak-link weld region, and incorporate creep and stress rupture properties characteristic of the coarse-grained weld material.

4.2 Metallurgical Variability and Changes During Testing

A considerable amount of variability is present in the microstructures of the materials, particularly the "second phases" sometimes seen decorating the grain boundaries, and the dendritic substructure present in the weld fusion zone. These appear to be related to variations in hardness and presumably other properties. However, the nature of these features is not yet well understood, nor is it completely clear what gives rise to these variations or how they influence properties. For example, it seems likely that the formation and resolution of the "grain boundary phases" can be influenced by temperature excursions associated with both welding and pressure-burst testing (particularly at 1100°C). However, it is not yet clear why "virgin base metal" areas with "clean" grain boundaries exhibit higher hardnesses than similar appearing regions following pressure-burst testing at 1100°C.

In addition, the weld fusion zone substructure and its changes during pressure-burst testing are not well understood. It *does* appear likely, however, that metallurgical changes occur differently at the 1010 and 1100°C pressure-burst temperatures. This could have significant implications regarding capsule life prediction based on extrapolation of pressure-burst failure times obtained at different temperatures. It seems clear that additional work is needed to

better understand the metallurgy of T-111 and how it changes during processing, stockpile life, and elevated temperature excursions.

4.3 Differences in Welding Procedures

Different welding procedures were used in manufacturing the capsules—capsule MPT-11-33 was welded with a different ratio of pulse rate-to-travel speed than the other two capsules. It is not clear whether other changes were made over the years in which the capsules were made, nor how such changes may influence pressure-burst test results. A search of the production records to document such changes seems an appropriate first step.

4.4 Differences in Oxygen Analysis Results from SNL/NM and Mound Laboratories

There are consistent and statistically significant differences in oxygen analytical results obtained by SNL/NM and Mound. SNL/NM's oxygen analyses on the three capsules examined here, as well as one previously analyzed capsule, are lower by approximately 40% than those reported by Mound for 13 capsules. This could result from an analytical procedure difference between the SNL/NM and Mound analytical laboratories.

APPENDIX A. PRESSURE-BURST TEST POST-MORTEM
EVALUATION DATA SHEET CAPSULE PB-08-03

FABRICATION DATA

- Strength Member Body, Serial No. 765 T-111 Lot 8
- Strength Member Cap, Serial No. 682 T-111 Lot 8
- Tantalum Pellet Identification TP-057
- Pressure-burst Capsule Tube Identification Lot 7
- Pressure-burst Capsule Fabrication Date Code F78
- Pressure: 1195.7 psia @ 23.4°C = 5192 psia @ 1010°C

TIME AT TEMPERATURE

- Capsule Placed on Test 08/16/79 (date) • Containment Temperature 410°C
- Capsule Removed from Test 01/19/88 (date)
- Total Time on Test 8.42 (years)

TEST DATA

- Rupture Time 523 hours
- Rupture Location Weld Tail-Out Region

POST RUPTURE DATA

- **Metallographic Evaluations (include appropriate photographs):**

Leaking occurred in the weld tail-out region, consistent with the bubble-leak test indication. The leak path developed as a result of intergranular stress rupture crack formation along grain boundaries in the coarse-grained weld fusion zone and adjacent heat affected zone. Variations in metallographic etching response occurred at some grain boundaries and within the weld fusion zone.

- **Microhardness Values:**

Side wall 250 ±13.5 DPH
Dome 256 ±13.1 DPH
Cap 245 ±8.4 DPH

- **T-111 Raw Material Values:**

Vendor 232 DPH
Mound 247-248 DPH

Comments concerning microhardness gradients:

Base metal hardnesses tended to be somewhat higher in slow etching areas where the grain boundaries were not decorated by second phases. Weld fusion zone hardness exhibited considerable scatter: bands marking the onset of solidification following a weld pulse were ~47 points harder than steady state solidification regions.

- **Grain Size Determination:**

Side wall 7-7.5 ASTM
Dome 8-8.5 ASTM
Cap 7.5-8 ASTM

- **T-111 Raw Material Values:**

Vendor 8 ASTM
Mound 8 ASTM

- **O₂/N₂ Values:**

	Side wall	Dome	Cap	Vendor	Mound	
Oxygen	<u>89</u>	<u>112</u>	<u>75</u>	<u>32</u>	<u>10</u>	ppm
Nitrogen	<u>9</u>	<u>12</u>	<u>10</u>	<u>28</u>	<u>28</u>	ppm

- **T-111 Raw Material Values:**

- **Scanning Electron Microscope and Ancillary Equipment Evaluations (optional):**

Intergranular cracking was observed along the weld fusion zone/side wall boundary around the entire perimeter of the capsule interior. No evidence of cracking was seen elsewhere. Evidence of elevated temperature grain boundary sliding was observed in the coarse grained weld region, as well as in the fine grained dome region.

- **Remarks:**

Intergranular cracking was observed along the weld fusion zone/side wall boundary around the entire perimeter of the capsule interior. No evidence of cracking was seen elsewhere. Evidence of elevated temperature grain boundary sliding was observed in the coarse-grained weld region, as well as in the fine-grained dome region.

**APPENDIX B. PRESSURE-BURST TEST POST-MORTEM
EVALUATION DATA SHEET CAPSULE MPT-11-33**

FABRICATION DATA

• Strength Member Body, Serial No.	<u>3528</u>	T-111 Lot	<u>Lot 11</u>
• Strength Member Cap, Serial No.	<u>3121</u>	T-111 Lot	<u>Lot 11</u>
• Tantalum Pellet Identification	<u>LTP 15</u>		
• Pressure-burst Capsule Tube Identification	<u>Lot 7</u>		
• Pressure-burst Capsule Fabrication Date	<u>Code K 84</u>		
• Pressure: <u>1206</u> psia @ <u>25.6°C</u>	= <u>5192</u> psia @ <u>1010°C</u>		

TIME AT TEMPERATURE

• Capsule Placed on Test	<u>07/16/85</u> (date)	• Containment Temperature	<u>210°C</u>
• Capsule Removed from Test	<u>01/19/88</u> (date)		
• Total Time on Test	<u>2.5</u> (years)		

TEST DATA

• Rupture Time	<u>On test for 739 hours, capsule was not tested to failure</u>
• Rupture Location	<u>N/A</u>

POST RUPTURE DATA

• **Metallographic Evaluations (include appropriate photographs):**

No leak path was found, consistent with expectations from the burst test results. Intergranular stress rupture crack formation had begun in the coarse-grained weld fusion zone and adjacent heat affected zone at the inside surface, but cracking had proceeded less than 25% through the weld. Variations in metallographic etching response occurred at some grain boundaries and within the weld fusion zone.

• Microhardness Values:	T-111 Raw Material Values:
Side wall <u>246 ±11.3</u> DPH	Vendor <u>228-252</u> DPH
Dome <u>251 ±9.1</u> DPH	Mound <u>228-254</u> DPH
Cap <u>242±9.0</u> DPH	

Comments concerning microhardness gradients:

Base metal hardnesses tended to be somewhat higher in slow etching areas where the grain boundaries were not decorated by second phases. Weld fusion zone hardness exhibited considerable scatter: bands marking the onset of solidification following a weld pulse were ~39 points harder than steady state solidification regions.

• Grain Size Determination:	T-111 Raw Material Values:
Side wall <u>7.5-8.0</u> ASTM	Vendor <u>7.0</u> ASTM
Dome <u>8.0</u> ASTM	Mound <u>8.0</u> ASTM
Cap <u>6.5-7.0</u> ASTM	

• O₂/N₂ Values:	T-111 Raw Material Values:
Side wall Dome Cap Vendor Mound	
Oxygen <u>66</u> <u>71</u> <u>96</u> <u>38-40</u> <u>16-23</u> ppm	
Nitrogen <u>16</u> <u>15</u> <u>26</u> <u>6-7</u> <u>8-10</u> ppm	

• **Scanning Electron Microscope and Ancillary Equipment Evaluations (optional):**

Intergranular cracking was observed along the weld fusion zone/side wall boundary on the inside surface. This was most pronounced beneath the weld tail-out region, where ~50% of the interface was cracked. No evidence of cracking was seen elsewhere on the capsule side wall, dome, or cap. Evidence of elevated temperature grain boundary sliding was observed in the coarse-grained weld region, as well as in the fine-grained dome region.

• **Remarks:**

Elevated temperature grain boundary sliding and intergranular stress rupture had begun in this capsule, but had not yet resulted in leaking. Stress rupture cracking was concentrated in the weld tail-out region, but this cracking was confined to near the inside surface, and had not grown into the portion of the fusion zone corresponding to the tail-out passes.

**APPENDIX C. PRESSURE-BURST TEST POST-MORTEM
EVALUATION DATA SHEET CAPSULE PB-07-13**

FABRICATION DATA

• Strength Member Body, Serial No.	<u>629</u>	T-111 Lot	<u>Lot 7</u>
• Strength Member Cap, Serial No.	<u>632</u>	T-111 Lot	<u>Lot 7</u>
• Tantalum Pellet Identification	<u>TP-040</u>		
• Pressure-burst Capsule Tube Identification	<u>Lot 7</u>		
• Pressure-burst Capsule Fabrication Date	<u>Code B78</u>		
• Pressure: <u>1204</u> psia @ <u>24.5°C</u>	= <u>5192</u> psia @ <u>1010°C</u>		

TIME AT TEMPERATURE

• Capsule Placed on Test	<u>05/16/79</u>	(date)	• Containment Temperature	<u>210°C</u>
• Capsule Removed from Test	<u>08/26/87</u>	(date)		
• Total Time on Test	<u>8.25</u>	(years)		

TEST DATA

• Rupture Time	<u>Total time on test at 1100°C was 640 hours. Failure likely at 16 hours</u>
• Rupture Location	<u>Weld tail-out region</u>

POST RUPTURE DATA

- **Metallographic Evaluations (include appropriate photographs):**
Leaking occurred in the weld tail-out region, consistent with the bubble-leak test indication, as a result of intergranular stress rupture crack formation along grain boundaries in the coarse-grained weld fusion zone and adjacent heat affected zone. Metallographic etching response was very different from capsules PB-08-03 and MPT-11-33 because a) the bands in the weld fusion zone etched much darker and more rapidly, and b) grain boundaries in the base metals etched more slowly and were relatively free of second phases.

• Microhardness Values:	T-111 Raw Material Values:
Side wall <u>237 ±8.7</u> DPH	Vendor <u>244-247</u> DPH
Dome <u>245 ±5.0</u> DPH	Mound <u>246-248</u> DPH
Cap <u>241 ±9.1</u> DPH	

Comments concerning microhardness gradients:

Base metal hardnesses were relatively uniform, consistent with the uniform etching response, but lower hardnesses were observed in regions of PB-08-03 and MPT-11-33 where grain boundaries were free of second phases. Similarly, the weld fusion zone hardness exhibited very little scatter. Bands marking the onset of solidification following a weld pulse, despite their darker etching, exhibited the same hardness as the steady-state solidification regions.

• Grain Size Determination	T-111 Raw Material Values:
Side wall <u>7.0-7.5</u> ASTM	(Vendor) <u>8.5</u> ASTM
Dome <u>8.5</u> ASTM	(Mound) <u>8.0</u> ASTM
Cap <u>7.5-8.0</u> ASTM	

• O₂/N₂ Values:	T-111 Raw Material Values:
Side wall Dome Cap	Vendor Mound
Oxygen <u>89</u> <u>76</u> <u>59</u>	<u>28</u> <u>42</u> ppm
Nitrogen <u>15</u> <u>16</u> <u>15</u>	<u>17</u> <u>28</u> ppm

- **Scanning Electron Microscope and Ancillary Equipment Evaluations (optional):**
Intergranular cracking was observed along the weld fusion zone/side wall boundary around the entire perimeter of the capsule interior. No evidence of cracking was seen elsewhere. Evidence of elevated temperature grain boundary sliding was observed in the coarse-grained weld region, as well as in the fine-grained dome region.

- **Remarks:**
The capsule failed because of elevated temperature grain boundary sliding resulting in eventual intergranular stress rupture. This occurred preferentially in the weld region, rather than in the more highly stressed dome region, because the very large grains in the weld region make this the metallurgical weak link for elevated temperature stress rupture failure. Exposure to the higher 1100°C testing temperature appears to have produced significant metallurgical changes. This could complicate prediction of failure times based on extrapolation of results from tests at other temperatures.

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