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MLM-MU-76-70-0003

MILLIWATT GENERATOR HEAT SOURCE
PROGRESS REPORT

July 16, 1976 - October 15, 1976

Wayne R. Amos
Wayne R. Amos
Program Manager

JUL 01 1976

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MOUND LABORATORY

Miamisburg, Ohio

operated by

MONSANTO RESEARCH CORPORATION

a subsidiary of Monsanto Company

for the

UNITED STATES ENERGY RESEARCH

AND DEVELOPMENT ADMINISTRATION

U. S. Government Contract No. EY-76-C-04-0053

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MILLIWATT GENERATOR HEAT SOURCE PROGRESS REPORT

I. Introduction

Activities at MRC associated with the Milliwatt Generator Heat Source efforts over the period July 16, 1976, to October 15, 1976, are presented below.

II. Post Mortem Analyses of SN-5 and SN-7 (P. E. Teaney and R. L. Wise)

Post mortem analyses of two "prototype" MWG heat sources (SN-5 and -7) were conducted during disassembly of the heat sources for fuel recovery. The heat sources were fabricated during August, 1971, and were used as prototype heat sources in RTG tests at both Mound and Sandia Laboratories. The capsule design is the same as for the "S" type compatibility tests which consists of two-layer capsules containing hafnium as a getter for oxygen. The inner capsule is 0.040" thick Ta-10W alloy with a 0.060" thick cap and a 0.010" thick shim. The outer layer capsule is 0.020" thick Hastelloy-C with a 0.055" thick trepan-type cap. Each heat source contained approximately five watts of fuel.

Both heat source assemblies were integral prior to disassembly. Metallographic examination of the cross-sectioned specimens revealed no evidence of fuel/Ta-10W reaction except in specimen SN-7 where fuel was melted to the sidewall of the liner during welding. This area of the specimen is shown in the photomicrograph in Figure 1. A band of fuel is located just below the weld and extends around the inside circumference of the liner. The reaction shown in the photomicrograph occurred when fuel was melted during closure of the liner and approximately 10 μ m of the container dissolved in the molten PuO_2 . A similar but less evident band of fuel existed in SN-5, but no fuel/Ta-10W reaction was observed in the cross-sectioned specimen. No detrimental effects of long-term aging were observed in either specimen.

Microhardness traverses were conducted across the sidewall and dome of the inner capsule from each heat source. The

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250X (As Polished)

Photomicrograph SN-7 Showing Ta-10W/PuO₂ Reaction
Which Occurred During Welding

FIGURE 1

results are shown in Figures 2 and 3 for SN-5 and SN-7, respectively. Results for each specimen are nearly identical except that values for the sidewall of SN-7 are slightly higher than those for SN-5. Although the liners are not embrittled, values for the sidewalls of the specimens are somewhat higher than normal. (Normal hardness for annealed Ta-10W liners is 193-223 VHN.)

Samples from the sidewall and dome of each specimen were submitted for oxygen and nitrogen analyses. The values obtained are shown in Table I along with average hardness values for the specimens. Although the values are low, the correlation between oxygen content and microhardness suggests that differences in values for the different areas of the specimens are due in part to oxygen pick-up which occurred either during fabrication or aging. However, metallographic examination of the specimens in the etched condition revealed a difference in the microstructure of the specimens which would also contribute to the differences in microhardness.

Photomicrographs of the cap, dome, and sidewall of specimen SN-7 are shown in Figure 4. The grains in the caps of the specimens are nearly equiaxial, indicating annealed material which would be expected to have a low microhardness. The microstructure of the dome is indicative of material which has only partially recovered after being worked and the material would be expected to show an increased hardness over the fully annealed material. Very little recovery of the worked material is indicated in the sidewalls of the specimens. In the sidewall photomicrographs, (at the site of the microhardness traverse), higher microhardness values would be expected in this material even if the oxygen content were equal in all locations. Hardware fabrication reports for the specimens do not indicate that the material was annealed after fabrication.

The post mortem analyses revealed no evidence of reactions or adverse effects which could be attributed to the various conditions and the approximately five years of aging to which the heat sources were subjected.

FIGURE 2. MICROHARDNESS VALUES ON SN-5

FIGURE 3. MICROHARDNESS VALUES ON SN-7

↑
Outside Edge Cap 125X (Etched)

Dome 125X (Etched)

Sidewall 125X (Etched)

Photomicrographs of SN-7
Showing Differences in
Microstructure

FIGURE 4

TABLE I

OXYGEN, NITROGEN AND AVERAGE VICKERS HARDNESS
VALUES FOR MWG "PROTOTYPE" HEAT SOURCES

<u>Sample</u>	<u>Location</u>	<u>O₂ (PPM)</u>	<u>N₂ (PPM)</u>	<u>Avg VHN</u>
SN-5	Sidewall	110	2	276
	Dome	57	0	212
	Cap	-	-	189
SN-7	Sidewall	182	1	293
	Dome	62	0	226
	Cap	-	-	191

III. Evaluation of "S" Type Compatibility Tests (P. E. Teaney and R. L. Wise)

Metallographic evaluation of the "S" type compatibility tests was completed. The last of three specimens was evaluated after three years at 600°C. The "S" type test matrix is shown in Table II. The first two "S" type specimens were evaluated after 60 days at 800°C and 900°C, respectively. (Section III, MWG Heat Source Progress Report, June 16 - July 15, 1973). These capsules differ from present heat sources in that they are two layer capsules employing a Ta-10W liner, hafnium was used as a getter instead of yttrium, and the specimens were not pretreated. The capsule design is the same as for SN-5 and SN-7 described in Section II of this report and is shown in Figure 5.

The specimen that was tested for three years at 600°C was cross-sectioned and examined on the metallograph in the as-polished condition. The dome and cap of the specimen were smooth and free of any signs of reaction. However, the side-walls of the specimen were somewhat more irregular along the inside edge than the dome and lid, suggesting the possibility of slight intergranular attack at the inside surface

TABLE II

FUEL/T-111 COMPATIBILITY TEST MATRIX
"S" TYPE CAPSULES*

<u>Temperature</u> (°C)	<u>Date Due Out of Furnace</u>	
	<u>60 Days</u>	<u>3 Years</u>
600		C
800	C	
900	C	

*Special Tests - Full size capsules with Hf getter
to produce $\text{PuO}_{1.75}$ (Ta-10W Liner)

C = Metallography Completed

M = In Metallography

FIGURE 5. CAPSULE DESIGN FOR "S" TYPE COMPATIBILITY TESTS

of the sidewall. The sidewall of the specimen is shown in the two photomicrographs in Figure 6. The intergranular attack, if any, appeared to be limited to 10 μm or less. Measurement of the sidewall thickness after testing indicated a thickness of 39.05 mils. Measurements were not made prior to testing. However, the capsule was fabricated from approximately 40 mil material.

A spherical void approximately 173 μm in diameter was observed in the cross-section of the weld of the liner (Figure 7). The fabrication report indicates some difficulty was encountered with some weld blow-outs during fabrication, but that the welds resealed and no repair was needed. It was felt that the blow-out problem was caused by outgassing of the fuel due to insufficient clearance between the weld and fuel. The void in the weld obviously resulted from entrapment of the gas in the weld during solidification.

Microhardness traverses were conducted across the cap, sidewall and dome of the specimen and the results are shown in Figure 8. Results were similar to those reported for SN-5 and SN-7 in this report (Section II) and for the 60 day "S" type samples reported previously, with hardness values for the sidewall somewhat higher than those for the dome and cap.

Samples of the sidewall and bottom of the capsule were analyzed for oxygen and nitrogen. The results are given in Table III along with results for the 60 day "S" type samples. The values are low indicating very little oxygen pick-up during the three years at 600°C. The indicated oxygen pick-up for the specimen falls about midway between those for the specimens tested for 60 days at 800°C and 900°C, respectively. There is not enough difference between the oxygen content of the sidewall and dome of the specimen to account for the 55 VHN difference in microhardness.

The specimens were examined in the etched condition to determine if the difference in microhardness could be related to the microstructure. The results were very similar to those reported for SN-5 and -7 with the photomicrographs of SN-7 in Figure 4 being typical for the "S" type samples as well as SN-5. Again, the microhardness values appeared to be related more to the microstructure of the material than to the small quantity of oxygen absorption during the test.

← Ta-10W/Fuel Interface

125X (As Polished)

↑
Sidewalls
↓

Photomicrographs Showing Irregular
Appearance of Fuel/Ta-10W Interface
of "S" Type Specimen (3 years at
600°C)

FIGURE 6

250X (As Polished)

↑
Ta-10W/Fuel Interface

Photomicrograph of "S" Type Specimen
after 3 years at 600°C

FIGURE 7

FIGURE 8. MICROHARDNESS VALUES ON MF-1

TABLE III

OXYGEN, NITROGEN AND AVERAGE VICKERS HARDNESS
VALUES FOR "S" TYPE COMPATIBILITY TESTS

<u>Specimen</u>	<u>Sample Location</u>	<u>O₂ (PPM)</u>	<u>N₂ (PPM)</u>	<u>Avg. Hardness (VHN)</u>
3 Yrs. @ 600°C	Liner Bottom	81	1	214
	Liner Sidewall	86	0	269
60 Days @ 800°C	Liner Bottom	39	8	232
	Liner Sidewall	12	9	264
60 Days @ 900°C	Liner Bottom	206	6	276
	Liner Sidewall	160	3	318

Overall evaluation of the "S" type compatibility specimens indicated that fuel/Ta-10W reactions were limited to 10 μm or less in all three specimens. Although some very slight oxygen pick-up did occur in the specimens tested for 60 days at 900°C and for three years at 600°C, the quantity was insufficient to threaten the ductility of the capsules.

IV. Fuel Aging Studies (P. E. Teaney and C. B. Chadwell)

Plutonia fines generation data were obtained for specimens aged up to 15 months at 450°C after pretreatment. Fines analyses are shown in Table IV, along with data for previously reported specimens (Section IV, MWG Heat Source Progress Report Jan. 16 - April 15, 1976). It is apparent by these data that after the slight initial generation of fines during the first six months of aging at 450°C, no additional fines are generated through 15 months of aging at 450°C.

Photomicrographs, showing the appearance of the fuel microstructure prior to testing, after pretreatment, and at each

TABLE IV
FUEL AGING STUDIES
(Plutonia Fines Data)

<u>Sample Number</u>	<u>Test Conditions</u>	<u>Weight Percent ^{238}Pu Fines</u>		
		<u>< 5 μm</u>	<u>5-10 μm</u>	<u>10-20 μm</u>
P-438	Baseline Fuel for MF-29C through -38C	3.83×10^{-3}	4.3×10^{-4}	
P-445	Baseline Fuel for MF-39C through -47C	1.73×10^{-3}	5.9×10^{-4}	
MF-37C	Pretreated Fuel	6.5×10^{-4}	4.9×10^{-4}	
MF-38C	Pretreated Fuel	1.36×10^{-3}	7.0×10^{-4}	
MF-29C	Impacted Fuel (149 meters/sec at 450°C)	4.08×10^{-2}	1.0×10^{-4}	
P-445 (aged)	Aged 10 months in a 20-30 gram batch	4.96×10^{-3}	6.6×10^{-4}	
MF-39C	Aged 6 months at 450°C (after pretreatment)	1.53×10^{-2}	2.98×10^{-2}	1.32×10^{-2}
MF-40C	Aged 6 months at 450°C (after pretreatment)	2.25×10^{-2}	1.74×10^{-2}	1.24×10^{-2}
MF-41C	Aged 9 months at 450°C (after pretreatment)	1.60×10^{-2}	3.34×10^{-2}	4.34×10^{-2}
MF-42C	Aged 9 months at 450°C (after pretreatment)	2.52×10^{-2}	2.08×10^{-2}	3.14×10^{-2}
MF-43C	Aged 15 months at 450°C (after pretreatment)	2.27×10^{-2}	1.01×10^{-2}	1.22×10^{-2}
MF-44C	Aged 15 months at 450°C (after pretreatment)	9.33×10^{-3}	1.13×10^{-2}	3.11×10^{-2}

additional stage of aging at 450°C are shown in Figures 9 through 13. Comparison of the fuel microstructure prior to testing (Figure 9) with the microstructure of the fuel after pretreatment (Figure 10) shows that during pretreatment, helium in the fuel migrated to the grain boundaries, forming small round voids which delineate the boundaries. The two phase appearance of several of the fuel shards in both the aged and pretreated specimens is indicative of substoichiometric fuel. Analyses of previous fuel has indicated that the lighter (acicular-structured) phase is PuO_2 and that the darker phase is Pu_2O_3 . Examples of the substoichiometric fuel is particularly evident in the etched photomicrographs in Figure 10. Except for possible clues to the mode of break-up, these figures should not be used to estimate the generation of fines. The pressure mounting technique used for metallographic processing of the samples makes this determination from the photomicrographs impossible.

The status of the fuel aging studies is summarized in Table V. The final six samples in the test matrix were scheduled to be tested for > 15 months depending on results of previous tests. Because the plateau in fines generation is continuing, testing of these specimens will be extended to 24 months in order to obtain longer term data.

V. Process Engineering

For FY-77 the programmatic formal process engineering includes a) a thermodynamic study of the $\text{PuO}_2/\text{Y/T-111}$ system, b) an extension of compatibility studies and c) studies concerning the moisture content of plutonia fuel. Details of the work to date follow:

a) Thermodynamic Studies (D. R. Schaeffer)

An effort to establish the thermodynamics of the $^{238}\text{PuO}_2/\text{Y/T-111}$ system has been initiated. The program objectives include the determination of the free energy of formation of HfO_2 in T-111. This will necessitate finding the activity of hafnium in T-111 and the partial pressure of oxygen in the system. Additional studies might include investigating the variation of the partial pressure of oxygen in the

125X (As Polished)

Photomicrographs of Baseline
Fuel (P-445) Used for Fuel Aging
Study Showing Appearance of
Microstructure Prior to Test

FIGURE 9

250X (As Polished)

110X (Etched)

130X (As Polished)

210X (Etched)

Photomicrographs Showing
Fuel after 1 hour at
1350°C Pretreatment

FIGURE 10

125X

150X

220X

Photomicrographs of Fuel
from MF-39C and -40C
Showing Fuel Microstructure
after Aging 6 months at
450°C

FIGURE 11

150X

150X

150X

Photomicrographs of Fuel
from MF-41C and -42C
Showing Fuel Microstructure
after Aging 9 months at
450°C

FIGURE 12

130X (As Polished)

150X (As Polished)

230X (As Polished)

Photomicrographs of Fuel
from MF-43C and -44C
Showing Fuel Microstructure
after 15 months Aging at
450°C

FIGURE 13

TABLE V
FUEL AGING STUDIES

<u>Sample Description</u>	<u>Type of Sample</u>	<u>No. Samples</u>	<u>Age</u>	<u>Analyses to be Performed</u>	<u>Status of Metallography and Fines Analysis</u>
Baseline Fuel	A	2	-	<5 μ fines, fuel microstructure	Completed
Pretreatment Effects	B	2	-	<5 μ fines, fuel microstructure	Completed
Impact Effects on Non-aged Fuel	C	2*	-	<5 μ fines, >5 μ fines distribution	Completed
Aging Effects	C	2	6 mo at 450°C	<5 μ fines, fuel microstructure	Completed
	C	2	$\geq$ 9 mo at 450°C	<5 μ fines, fuel microstructure	Completed
	C	2	$\geq$ 12 mo at 450°C**	<5 μ fines, fuel microstructure	Completed
	C	2	$\geq$ 15 mo at 450°C***	<5 μ fines, fuel microstructure	Off Test on 2/07/77
Impact Effects on Aged Fuel	C	2	$\geq$ 15 mo at 450°C***	<5 μ fines, >5 μ fines distribution	Off Test on 2/07/77
Thermal Ramp (to 1000°C) Effects on Aged Fuel	C	2	$\geq$ 15 mo at 450°C***	<5 μ fines, fuel microstructure	Off Test on 2/07/77

A - PuO₂ shards, no Y

B - PuO₂ shards with Y, encapsulated in liner and strength member, pretreated

C - PuO₂ shards with Y, encapsulated through clad, pretreated in strength member

*Impact data agreed with previous impact data, therefore, second capsule will be reserved for other tests.

**Testing extended to 15 months

***Testing extended to 24 months

system with the amount of yttrium in the system and determining the effect of changing the liner material from T-111 to Ta-10W.

A literature search has begun to determine experimental methods.

b) Compatibility Studies (R. L. Wise and E. Stacy)

This study is designed to correlate the compatibility results of the preliminary sample matrix shown in Table VI with those already available on the T-111 compatibility samples specifically designed for the MWG Heat Source Program. The samples listed in Table VI have been exposed to temperatures in the 800° to 1200°C range and the examination of these samples will allow accurate long-term compatibility projections for the heat source and will provide useful information regarding other potential heat source materials.

Metallographic examination has begun on the Group H samples, while the samples from Groups A, B, C and E are being processed for metallographic examination.

c) Moisture Content of Plutonia (W. A. Zantotelli, Jr.)

The determination of the moisture content of plutonia fuel is being studied in an attempt to more clearly define the production fuel pre-loading procedures. After completion of a literature search it was concluded that a Consolidated Electronics Corporation Model 36-321A-MA moisture analyzer would be utilized, which provides both a direct measurement and high sensitivity.

The instrument consists of a furnace for heating the sample, an electrolytic cell for absorbing the vaporized water and electrolyzing it to hydrogen and oxygen, and an integrating system for measuring the current used in electrolysis. The sample is heated in a flow of dry nitrogen with the evolved water vapor passing into a phosphorous pentoxide cell. Phosphoric acid is formed

TABLE VI
COMPATIBILITY SAMPLE MATRIX

<u>Group</u>	<u>Metal</u>	<u>Fuel</u>	<u>Test Temperature (°C)</u>
A	Ta	SRP	1200
B	T-111	Production	1200
C	Ta-10W/T-111	SRP	1200
D	Ta	PuO _{1.81}	1200
E	Ta-10W	SRP	800
F	Ta-10W	SRP	1200
G	Ta-10W	PuO _{1.81}	1200
H	Ta-10W	SRP + Additive	1200
I	Ta-10W	PuO _{1.81}	1000
J	Ta-10W	Production	1700
K	Ta-10W	Production	1900
L	Mo-50Re	SRP	800
M	Mo-50Re	SRP	1000
N	Mo-50Re	SRP	1200
1	Pt	PuO _{2-x}	1000
2	Pt-20Rh	SRP	1000
3	Pt-40Rh	SRP	800
4	Pt-40Rh	SRP	1000
5	Pt-40Rh	SRP	1200
6	Pt/Pt-40Rh	Production	1200
7	Ti	SRP	800
8	Ti	SRP	1000
9	Hastelloy X	Production	800
10	Haynes 25	Production	800

and electrolyzed. The integrator measures the amount of current necessary for this electrolysis and records the moisture in micrograms of water. The maximum sensitivity of commercial analyzers is 1 ppm water. From literature descriptions of glovebox operations, a sensitivity of 6 ppm should be possible.

Experimental plans include subjecting a series of low fired (700°C) and high fired (900°C) plutonia samples to environments of 100% humidity, no humidity, and typical glovebox humidity (40-60%). Sample weights will range up to a maximum of a heat source fuel loading charge. After installation of the moisture analyzer in the glovebox, the unit will be standardized. Analysis of the samples will follow, with 1000°C heating to assure the chemisorbed and physically adsorbed moisture is collected.

The proposed installation is shown in Figure 14, with the initial sample analysis scheduled for February-March, 1977.

VI. Pressure Burst Testing (D. R. Schaeffer and R. L. Wise)

As part of the evaluation of the pulse arc welding technique (Section V, MWG Progress Report, April 16-July 15, 1976), four simulated 25-year, pulse arc welded capsules [MPT-105C(E) through -108C(E)] were pressure burst tested. The results are given in Table VII. Failure of the pulse arc welded capsules occurred after a small leak developed in the sidewall or bottom (see Figures 15 through 18), whereas the previously tested capsules welded with a continuous arc normally failed in the weld. Comparison of the rupture times of the pulse and continuous arc welded capsules is made in Figure 19. This figure shows that the time to rupture for the pulse arc welded capsules is significantly less than that for the continuous arc welded capsules except for MPT-108C(E) which failed prematurely due to observed inclusions in the sidewall.

Further comparison of the welding techniques was made by dimensionally measuring five pulse arc welded and four continuous arc welded capsules to determine the amount of permanent deformation experienced by each capsule as a comparison of its relative creep behavior. The results of the measurements are included in Table VII. The measurements reveal that the relative creep behavior of the

FIGURE 14. SCHEMATIC REPRESENTATION OF MODIFIED MOISTURE ANALYZER

TABLE VII

RUPTURE AND DIMENSIONAL DATA ON PRESSURE BURST CAPSULES

(SIMULATED 25 YEAR DESIGN)

Capsule No.	Weld	Internal Pressure (PSIA)	Time To Rupture (Hours)	Length (In.)	Diameter (In.) (See Sketch at Bottom)			Outward Cap Deflection (In.)
					#1 Position	#2 Position	#3 Position	
MPT-99C(E)	Continuous	0	0	0.707	0.691	0.692	0.693	.0002
MPT-100C(E)	Continuous	5995	162.86	0.751	0.694 0.691*	0.703	0.694 0.695*	.0012
MPT-101C(E) (Stem Failure)	Continuous	7994	3.12	0.747	0.694 0.690*	0.705	0.694 0.695*	.0010
MPT-102C(E)	Continuous	7003	55.28	0.754	0.695 0.691*	0.705	0.693 0.695*	.0015
MPT-105C(E)	Pulse	6996	28.10	0.744	0.693 0.691*	0.703	0.696 0.698*	.0006
MPT-106C(E)	Pulse	7501	11.27	0.741	0.695 0.693*	0.707	0.697 0.699*	.0012
MPT-107C(E)	Pulse	6510	32.44	0.743	0.695 0.692*	0.703	0.698 0.698*	-
MPT-108C(E)	Pulse	7281	14.00	0.744	0.697 0.693*	0.705	0.699 0.699*	.0006
MPT-109C(E)	Pulse	0	0	0.707	0.692 0.693*	0.694	0.698 0.700*	.0004

*Original Measurements

MPT-105C(E)
6996 psia at 1000°C
28.1-hour Rupture (at
arrow)

FIGURE 15

MPT-106C(E)
7501 psia at 1000°C
11.3-hour Rupture (at
arrow)

FIGURE 16

MPT-107C(E)
6510 psia at 1000°C
32.4-hour Rupture (at arrow)

FIGURE 17

MPT-108C(E)
7281 psia at 1000°C
14.0-hour Rupture (at arrow)

FIGURE 18

FIGURE 19. LOG INTERNAL PRESSURE VS LOG RUPTURE TIME

continuous and pulse arc welded capsules is similar with the possible exception being the outward deflection of the cap. However, it should be noted that these measurements were made with the capsules at atmospheric pressure and that differences might exist if the capsules were at the test conditions of 6000 to 7000 psia and 1000°C.

Insight into possible differences in deformation behavior is provided by the absence of the circumferential crack in the weld fillet in the two pulse welded capsules which were sectioned [MPT-107C(E) and MPT-108C(E)], which would make a more rigid configuration.

In an initial effort to determine why the pulse arc welded capsules failed sooner than the capsules welded with a continuous arc, MPT-108C(E) was sectioned and a visual examination of the inside revealed three 0.125-inch long cracks. One of the cracks was in the sidewall and was responsible for the capsule failure during the pressure burst test. The two other cracks were in the radial portion of the bottom and were located circumferentially around the capsule.

Oxygen and nitrogen determinations were made on MPT-108C(E). Found were 100 ppm oxygen and 24 ppm nitrogen, which is similar to the continuous arc weld capsules.

VII. Scanning Electron Microscopic Examination of Failed Pulse Arc Welded Capsules (R. E. Zielinski)

The exterior surfaces of capsules MPT-105C(E) and MPT-106C(E) were examined after failure using the SEM. The exterior surfaces of the weld beads appeared undistorted and exhibited very uniform down slopes (Figure 20). There was no indication of failure in the weld zones although some anomalies were observed in the caps of both capsules. Figure 21 (top) shows an area where two inclusions were located in the cap of MPT-105C(E). One inclusion was visible at the cap/weld bead boundary, while the other was located in the cap proper. The second inclusion shown in Figure 21 (bottom) was rather extensive and had reacted with the T-111. The inclusion as others located in the past consisted primarily of iron and silicon. The discontinuity was optically visible and should have been visible after welding if it was present at that time. Since it was not reported prior to testing it has to be concluded that the discontinuity appeared during testing. Differences in thermal expansion or gases formed during the thermal excursion could have caused the removal of the T-111 surface covering the inclusion.

MPT-105C(E)

MPT-106C(E)

Weld Bead Downslopes

FIGURE 20

FIGURE 21
MPT-105C(E) Cap Inclusions

A similar phenomenon was observed on the cap of MPT-106C(E) where a line was observed in the cap heat affected zone (Figure 22, top-left). This line was a linear depression which was exposed and contained iron/silicon particles (Figure 22, top-right). Several nearly circular surface stains were also observed on the cap of MPT-106C(E) (Figure 22, bottom). Iron was detected in small amounts at the dark boundaries of these stains.

The next region examined on both capsules was the bottom extending from the radius to the center. Both capsules appeared very similar in this region with extensive grain boundary separation (Figures 23 and 24). This the first time that such a phenomenon has been observed during the examination of pressure burst capsules. It is random and it apparently results from the bottoms of the capsules stretching during testing since no interior weld bead cracks were observed in these capsules.

The point of failure was identified in MPT-105C(E) as being situated in the center of the bottom (Figure 25, top-left). A higher magnification photograph (Figure 25, top-right) of the failed region showed one extensive and several minor linear crack paths. Most of these cracks are again filled with iron/silicon inclusions (Figure 25, bottom).

The examination of the interior surface of MPT-107C(E) identified the crack (Figure 26, top-left) that was directly opposite the point of failure located on the exterior portion of the capsule. The crack contained pieces of included material (iron/silicon) and grains not typical of the parent material (Figure 26, top-right and bottom). These grains appeared soft or layered indicating that they had reacted with some foreign material.

Several other cracks were observed in the interior bottom. These cracks ran in patterns with a major crack surrounded by several minor cracks (Figure 27, top). Most of the major cracks had portions of inclusions visible in them (Figure 27, bottom) as well as soft grains (Figure 28, top) and surface reaction zones (Figure 28, bottom) present. The reaction product was predominantly tantalum.

It appears that the weld regions of the capsules are becoming more structurally sound and the areas where inclusions are present are now the weakest region of the capsules.

MPT-106C(E) Cap

FIGURE 22

Grain Boundary Separations in
Bottoms of MPT-105C(E) and -106C(E)

FIGURE 23

Higher Magnifications of
Figure 23 (MPT-105C(E) and
-106C(E)

FIGURE 24

MPT-105C(E) Failure Region

FIGURE 25

MPT-107C(E) Interior Surface

FIGURE 26

MPT-107C(E) Interior Cracks
at Bottom

FIGURE 27

Soft Grains and Surface Reaction
Zones at Cracks

FIGURE 28

VIII. Hardware Anomalies (R. E. Zielinski)

Two strength member die blanks from Lot 7 T-111 were examined on the SEM because numerous indications were observed during dye penetrant examination. The majority of indications were identified as embedded material (Figure 29), the others being areas where iron/silicon inclusions (Figure 30, top) were present. The embedded material was T-111 that had been forced into the surface of the sheet material. No other elements were observed in the embedded material except at the boundary where titanium and calcium were observed (Figure 30, bottom). The appearance of titanium and calcium could not be explained since no impurity particles were observed.

Further examination of one of the die blanks produced an interesting result. Several other embedded particles were observed in areas where no dye indications were observed. These areas appeared similar to those where indications were observed (Figure 31) and the fact that they were not detected would indicate that dye penetrant inspection is not a 100% effective means of evaluating hardware material.

The die blanks from the original group that had no indications were processed into strength member bodies. The dye penetrant examination of these bodies after sizing indicated that numerous indications were present on most of the bodies. The indications were strange in that dye did not flow from them yet they glowed under the ultraviolet light.

One of the bodies was examined using the SEM. This examination showed the presence of small particles in most of the indications (Figure 32). The others had iron/silicon inclusions visible. The composition of the particles was determined by x-ray analysis to be titanium, calcium, and silicon. In a side study the particles were identified as the dye developer. This study also explained the presence of titanium and calcium at the indication boundaries found in the original die blanks.

To evaluate the defects which were hidden by the dye developer, the body was subjected to the normal hardware acid cleaning process and reexamined on the SEM.

Reexamination of the body indicated the presence of several different types of imperfections. Additional inclusions became visible in addition to differences in surface texture (Figure 33). Numerous surface stains also became visible

Imbedded Material

FIGURE 29

Silicon/Iron Inclusions

Tantalum and Calcium at
Boundaries

FIGURE 30

Imbedded Material Not
Detected by Dye Penetrant

FIGURE 31

Small Particles in
Dye Penetrant Indications

FIGURE 32

Surface Texture Variances
FIGURE 33

(Figure 34, top) and an x-ray examination of these stained regions indicated the presence of iron, nickel, and chromium. In Figure 34 (top) an apparent crack is also visible. Examination of this area in the backscatter mode on the SEM indicated, however, that the depression was rather shallow (Figure 34, bottom). This was not the case for all the indications, with one so deep that the bottom of the hole could not be observed.

In summary, it appears that the dye indications found on the strength member bodies exposed several different types of defects, and effects of these defects on the behavior of the capsule cannot be evaluated at the present time. This analysis of the blanks and bodies has indicated that dye penetrant examination is not completely effective in locating hardware anomalies. On the other hand, several types of anomalies have been identified as being present in the material and the effects of these anomalies on the behavior of the capsules should be addressed.

IX. Scanning Electron Microscopic Examination of MPT-87C(E)
(R. E. Zielinski)

Pressure burst capsule MPT-87C(E) was received from Sandia Laboratories (Albuquerque) and the failure region examined. The appearance of the failure zone on the interior surface of the capsule is shown in Figure 35 (top). This crack contained iron/silicon inclusions, portions of which are visible (Figure 35, bottom).

The failure zone on the exterior portion was not as clearly defined and the capsule was dipped in an etching solution in an attempt to clean the surface. After cleaning, the exterior failure zone appeared as a reaction zone (Figure 36, top-left). This zone was rather porous and was composed of the structure shown in Figure 36 (top-right and bottom). The reaction product was composed of iron and hafnium.

This is the first time such a reaction zone has been observed in the T-111 hardware. Another observation regarding this capsule was that no extensive weld body heat affected zone crack was observed.

Crack and Shallow Depression

FIGURE 34

FIGURE 35

MPT-87C(E) Failure Zone

MPT-87C(E) Failure Zone after Etching

FIGURE 36

X. Examination of Norton Ta-10W Sheet Material (E. Stacy)

Because silicon and iron inclusions were found in rupture areas of pressure burst capsules, a study was initiated to determine whether these may have been introduced into the T-111 during the sheet fabrication at Norton Metal. For comparisons, Ta-10W sheet was evaluated for presence of inclusions, as well as pure tantalum sheet, which Norton agreed to evaluate.

A random material sample was cropped from a sheet of Norton produced Ta-10W (approximately 24 mils thickness). The sample was cleaned by wiping the surfaces with ethanol. Using the SEM, suspect surface impurities were observed but could not be identified by Energy Dispersive X-Ray Analysis (EDXRA).

A light chemical etch (a maximum material removal of 1 mil total) was used to remove the surface film. SEM evaluation revealed numerous inclusions embedded in the sheet surface (Figure 37, top) which were randomly dispersed over the total sheet surface. X-ray analysis identified these inclusions as primarily iron with a minor constituent of silicon (Figure 37, bottom). Numerous surface cracks and voids were also observed (Figure 38), indicating removal of the included material during the cleaning or etching of the sample.

Five additional etchings were performed (total of 11 mils) with SEM/EDXRA evaluations after each etching process. In summary, there were a few silicon/calcium inclusions, randomly dispersed tantalum spheres, a slight staining which was concentrated in the grain boundaries, and sparsely scattered silicon/iron inclusions.

XI. Tooling (J. E. Newland)

All the necessary FY-77 reimbursable hardware has been fabricated. The in-process gages are presently undergoing inspection in the gage lab. Rework and refinement of these, however, will not occur until the reimbursable hardware dimensional data have been evaluated statistically. At that time the Sandia metric drawings will be upgraded to the production design. (Because the metric gages have not been available, the reimbursable hardware fabricated to date has been to the English system.)

Inclusions Imbedded in
Ta-10W Sheet

FIGURE 37

Cracks and Voids in
Ta-10W Sheet

FIGURE 38

The liner cap swage and trim dies have been demonstrated successfully and swaged liner caps are being utilized for reimbursable hardware. The strength member cap swage process has been changed. A separate die was designed and fabricated to swage the chamfer. This is then followed by swaging the tenon. The dies have been demonstrated successfully, and swaged caps will be used for the reimbursable pressure burst capsules.

Work is continuing on the clad cap swaging operations. The swaging die for the boss has been demonstrated successfully and development is underway on the swaging operation for the tenon. Work has not yet been done on the trim die. If the clad cap swaging operations prove successful, there will be a programmatic savings of 11,000 manhours by swaging rather than machining.

XII. Encapsulation (C. E. Burgan)

The following units were fabricated and shipped during the report period as part of Sandia's reimbursable requirements.

<u>Quantity</u>	<u>Serial No.</u>	<u>Description</u>	<u>Desti- nation</u>	<u>Ship Date</u>
14	MF-79C, -81C through -86C, and -88C through -94C	16-year single channel, fueled, 3.5 watts	GEND	08/12/76
2	MF-2F and -3F	25-year single channel, fueled, 4.0 watts	SLA	09/30/76
7	MF-4F through -10F	25-year single channel, fueled, 4.0 watts	GEND	10/01/76
6	MT-1F, -2F, -7F, -9F, -10F, and -15F	25-year single channel, tungsten- molybdenum fuel simulant	SLA	10/04/76

Additionally, there were 10 tungsten-molybdenum filled simulant units fabricated (MT-3F, -4F, -5F, -6F, -8F, -11F, -12F, -13F, -14F, and -16F). These, also part of SLA's reimbursable requirements, will be impacted at various angles and velocities with Mound's impact gun.

Presently being fabricated are another 22 fueled units of the 25-year design (MF-11F through -32F). Ten of these will be shipped to GEND for their formal feasibility run, while the others will fulfill the remainder of SLA's FY-77 fueled unit reimbursable requirements. Two of the latter units will be impacted (at Mound) followed by determination of plutonia fines content.

XIII. Impact Gun (C. G. Anderson, F. E. Klapheke, and J. E. Newland)

Installation and check-out of the impact gun in Building 50 is nearing completion. After reaction mass hydraulic problems and velocity measurement problems were resolved, several shots at various velocities were successfully fired. The alpha box-passbox system has been put into location and is now undergoing helium leak checking. Gun operation with simulant units is still planned for November, 1976, with the fueled units to follow.

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