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Milliwatt Generator Heat Source Progress
Report: January-December 1989

Ranny W. Saylor

September 10, 1990

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MOUND

operated by

EG&G MOUND APPLIED TECHNOLOGIES

P.O. Box 3000, Miamisburg, Ohio 45343-3000

for the

U. S. DEPARTMENT OF ENERGY

Contract No. DE-AC04-88DP43495

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Summary

- All hardware shipments to LANL were made on or ahead of schedule, thus satisfying all War Reserve and other hardware/yttrium requirements for the reporting period January–December 1989.
- A special investigation was conducted to determine the source of a surface “staining” problem observed on some T-111 components. Although the cause was not positively identified, the actions taken as a result of the investigation dramatically reduced both the frequency of occurrence and the magnitude of the problem.
- Hardware fabrication activities continued in an efficient and timely manner, with production losses at a minimal level. During the reporting period, a 99.4% utilization yield was realized, with a 0.8 dollar percent defectiveness.

MWG Hardware Shipments and Production

J. A. Sohn and P. E. Teaney

All shipments to LANL were made on or before the required shipping dates during the reporting period of January–December 1989. War Reserve hardware and yttrium shipments are summarized in Table 1. In addition, 145 example welds and 208 evaluation–use–only components were shipped to LANL.

Production of hardware for 1989 was completed on schedule, and 1990 hardware production was started as scheduled.

Special Investigation

R. W. Saylor, P. E. Teaney, and S. Napier

A sporadically occurring “staining” problem with T-111 components was first observed on 84 Linear Bodies following final heat treatment on September 27, 1988. All of these components appeared darker than normal WR product, and most

TABLE 1 – MWG HARDWARE SHIPMENTS: JANUARY–DECEMBER 1989

Components	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Total
Liner Shim	40	40			35		110	70		40	50	50	435
Liner Cap	50	50			35		135	70		60	55	50	505
Liner Body	40	40			25		80	70		60	60	50	425
SM Cap	55	55			20		55	70			50	50	355
SM Body	50	50			15		55	70			40	50	330
Clad Cap	75	75			25		65	70				50	360
Clad Body	13	51		53		48	34	48	58				305
Yttrium (g)	25	25			25		25	50		25	25	25	225

of them displayed heavily darkened areas in a pattern similar in appearance to fingerprints. Because the time of final heat treatment closely coincided with a process change in the NDT laboratory (dye penetrant inspection materials), it was initially assumed that the new NDT materials were not completely removed by the normal solvent/acid cleaning subsequently performed in the cleaning laboratory. The NDT and cleaning laboratory personnel were instructed to give the components a thorough visual inspection before transferring them to the next operation and to contact the program engineers regarding any "unusual" product. Several procedural changes were also implemented to ensure maximum effectiveness of the ultrasonic solvent cleaning and acid pickling operations. New tantalum trays were also fabricated for the cleaning operations.

Following the above occurrence, an occasional group of T-111 components was observed to have a white haze residue remaining after the solvent/acid cleaning. This residue required hand-rubbing with acetone-soaked pads and, in some cases, scrubbing with scouring powder (an MWG-approved cleaning agent) for removal. As some of these components were Liner and Strength Member Caps, and were not exposed to the new NDT materials, the overall manufacturing process was reviewed for the source of "contamination". In this review, it was observed that Cimflo-10 (forming paste) was not being completely removed by the ultrasonic Freon cleaning in the forming/machining area. Operating personnel were instructed

to thoroughly inspect for cleanliness before transferring the product to the next operation.

The next T-111 product to experience this problem was Liner Shims. As the Shim manufacturing processes are quite simple (blank, inspect, and clean/ heat-treat), the machining and NDT areas, as well as Cimflo-10 forming paste, were eliminated as potential problem sources. During this same time, it was learned that other WR programs at Mound had experienced a Freon contamination problem caused by long-term storage of Freon in plastic containers. Although plastic containers were used in the MWG processing area for dispensing Freon, long-term storage in such containers was not the practice. To eliminate this possible source of contamination, Freon was replaced with alcohol for cleaning operations. When the Freon supply was analyzed, only traces of polyethylene were found.

To determine the presence or absence of any hydrocarbon contamination from the Freon supply, a liner shim was examined by Auger Electron Spectroscopy (AES) and Energy Dispersive Spectroscopy (EDS). AES was used to examine the surface of the "contaminated" shim (up to a few monolayers in depth) while EDS was used to examine the subsurface (up to a micron deep).

The composition of the surface, both off and on the stain, is shown in Figure 1. The spectra from which these compositions were determined are shown in Figures 2 and 3.

Figure 1 – Surface composition of Liner Shim, both off and on stained area.

Figure 2 – Liner Shim area not stained.

Figure 3 – Liner Shim stained area.

These surfaces are quite comparable, being composed of carbon, tantalum, and oxygen. Such a composition is indicative of a tantalum oxide surface with some residual carbon. The level of carbon is that typical of a sample exposed to laboratory environments. A little more than a monolayer of contamination would have masked the tantalum and only carbon would have been seen in the spectra.

The corresponding compositions of the subsurface, off and on the stain, are shown in Figure 4. The subsurface is composed of oxygen and tantalum; no carbon was detectable. Such a ratio of oxygen is typical of layers of tantalum oxide on tantalum metal. Any polyethylene film would have been represented by an increased carbon peak intensity.

The investigation was then focused toward events (that is, processes, materials, and work areas) common to all T-111 products:

- Lot 12 T-111 starting materials
- Material Control storage area
- Masking tape (used to protect sheet materials during shearing)
- Curtis oil (used in the blanking operation)
- Freon (currently alcohol) cleaning in forming/machining area
- Production Inspection operations
- QC Inspection operations
- Cleaning/heat-treating operations

A test matrix was used to determine whether limited exposure to the above

Figure 4 – Subsurface composition of Liner Shim.

processing materials would account for the staining condition. Five Strength Member Caps were individually “soaked” in Curtis oil, ethyl alcohol, and Freon, as well as Cimflo-10, and 1,1,1-trichloroethane (machining coolant). One cap was not exposed to any of these materials and served as a control sample. All parts were then processed through the MWG cleaning and heat treating operations.

Visual examinations showed a slightly darker area on the top of the cap which was exposed only to the Curtis oil. However, all parts displayed a surface texture that is typical of the stained condition. No processing technique was identified that explains the sporadic appearance of the white haze and the subsequent darkening of T-111 components during heat treating.

Strength Member Caps (SMC) 5644 and 5670 were fully processed WR components ready for shipment and considered to be examples of the more questionable product regarding surface “cleanliness”. The caps were sampled for engineering evaluation and subjected to AES examination. Summaries of the surface composition of SMC 5644 (top and bottom) and SMC 5670 (top and bottom) are shown in Figures 5 and 6, respectively. Related spectra are shown in Figures 7 through 10. As shown, all the surfaces are comparable in that they are composed of tantalum, hafnium, and oxygen along with some carbon. No other contaminants were detected. The amount of carbon is typical of a surface that has been exposed to normal room atmospheres and is the same amount seen on most “clean” surfaces analyzed. A summary of

Figure 5 – Strength Member Cap 5644 top and bottom surfaces.

Figure 6 – Strength Member Cap 5670 top and bottom surfaces.

Figure 7 – Strength Member Cap 5644 top surface.

Figure 8 – Strength Member Cap 5644 bottom surface.

Figure 9 – Strength Member Cap 5670 top surface.

Figure 10 – Strength Member Cap 5670 bottom surface.

the relative thicknesses of the carbon and oxygen contaminants for each sample side is shown in Figure 11. Related profiles are shown in Figures 12 through 15. The estimated carbon/oxygen thicknesses were in the 50–100 Å range, only slightly thicker than expected for a metal surface exposed to air. This small increase in carbon/oxygen thickness may account for the darkened appearance of the materials.

A recent observation suggests that the oxidation can be attributed to the hafnium-enriched surfaces and the vendor processing of Lot 12 T-111. It was noticed that some sheets of T-111 material received from Bonded Storage displayed characteristics similar to the observed

component staining. These sheets showed a discoloration pattern where overhanging edges jutted from the stack, creating areas that were more exposed to the environment than those in intimate contact with the next sheet.

It should also be noted that high temperature and humidity levels were present for several weeks in the Material Control storage area during the replacement of the HVAC system in the summer of 1988. The absence of a controlled environment may have contributed to a slight surface oxidation of some starting materials having high surface concentrations of hafnium. Previous evaluations of Lot 12 T-111 40-mil sheet materials have shown varying levels of hafnium content on or near the

Figure 11 – Depth comparison of Strength Member Caps 5644 and 5670.

Figure 12 - Strength Member Cap 5644 top surface.

Figure 13 - Strength Member Cap 5644 bottom surface.

Figure 14 - Strength Member Cap 5670 top surface.

Figure 15 - Strength Member Cap 5670 bottom surface.

surface of some starting materials. In order to ensure that routine machining and polishing operations were not depleting the overall hafnium ratio, analyses were conducted before and after these operations. No significant loss of hafnium was observed (Table 2).

Presently, all sheets of T-111 materials are being reviewed by program engineers before they are released to production. The surfaces are prepared by polishing with silicon-carbide paper to remove residues after the blanking operation. Final product is closely scrutinized for "cleanliness" and reprocessed or downgraded, as appropriate. Also, the manufacturing processes are

reviewed whenever unusual cleaning problems occur. Taking these actions and paying special attention to product cleanliness after each operation have reduced the staining problem dramatically, both in frequency of occurrence and magnitude. However, the cause and the corrective action are not completely resolved at this time.

Hardware Fabrication and Quality

T. A. Knox

Hardware fabrication continued to be conducted in an efficient and timely manner. Production losses were maintained at a minimal level. Approximately

TABLE 2 - COMPARISON OF THE BULK HAFNIUM AND TUNGSTEN CONTENTS BEFORE AND AFTER REMOVAL OF SURFACE CONTAINING HAFNIUM PARTICLE CONCENTRATIONS ON LOT 12 T-111 SHEET.

Results for 100 mil T-111 sheet*			Results for 40 mil T-111 sheet*		
Sheet No.	Hafnium (Wt. %)	Tungsten (Wt. %)	Sheet No.	Hafnium (Wt. %)	Tungsten (Wt. %)
3	1.9	7.7	11	2.0	8.1
	1.9	8.0		1.9	8.1
4	2.0	7.9	53	2.0	8.1
	2.0	7.8		2.0	8.0
10	1.9	7.9	75	1.9	8.2
	1.9	7.7		2.0	8.2
11	1.9	7.7			
	1.9	7.7			

* Shaded results are after removal of 0.0015" of material from each surface of the 100 mil sheet and 0.0005" of material from each surface of the 40 mil sheet. This approximates the amount of material removed during component fabrication.

3,650 pieces of hardware were accepted by DOE for WR shipment, and approximately 200 hardware components were dispositioned as example weld units, thus satisfying all programmatic needs for CY 1989.

The annual report for MC2893A hardware is summarized in Table 3. The overall utilization yield for the reporting period (January–December 1989) was 99.4%. The dollar value of the hardware accepted was \$649,120; the cost incurred for defective hardware during the period was \$5,134. A hardware rejection summary is shown in

Table 4, along with reasons for rejection. This provides information on the areas of the fabrication process that can be revised to improve process yields. The hardware quality index report given in Table 5 lists the lot submittals by Mound to DOE/DAO and the quality rating for the reporting period.

Reference

1. E. A. Mershad, *Milliwatt Generator Heat Source Progress Report: July–December 1986*, MLM–ML–87–46–002, Monsanto Research Corporation, Miamisburg, Ohio (June 15, 1987), 17 pp.

TABLE 3 – MC2893A QUALITY REPORT FOR CY 1989

Component	Process Yields	CY 1989	To Date
Liner Shim 291669-00 A01102	Fabricated Prime Reject Utilization Yield%	538 538 0 100	5278 5272 6 99.9
Liner Cap 291670-00 A01103	Fabricated Prime Example Weld Reject Utilization Yield%	540 532 7 1 99.8	5390 5264 108 18 99.7
Liner Body 291671-00 A01104	Fabricated Prime Example Weld Reject Utilization Yield%	532 523 8 1 99.8	5781 5346 382 53 99.1
SM Cap 291672-00 A01105	Fabricated Prime Example Weld Reject Utilization Yield%	628 606 18 4 99.4	6154 5763 293 98 98.4
SM Body 291673-00 A01106	Fabricated Prime Example Weld Reject Utilization Yield%	605 470 125 10 98.3	6435 5524 725 186 97.1
Clad Cap 291674-00 A01107	Fabricated Prime Example Weld Reject Utilization Yield%	718 685 30 3 99.6	5608 5350 220 38 99.3
Clad Body 291675-00 A01108	Fabricated Prime Example Weld Reject Utilization Yield%	314 300 9 5 98.4	5630 5385 164 81 98.6
Percent Overall Utilization Yield		99.4	98.7
Dollar Value of Fabricated Hardware		649,120	6,027,742
Dollar Cost of Defectiveness		5,134	69,619
Dollar Percent Defectiveness		0.8	1.1

TABLE 4 – MC2893A HARDWARE REJECTION SUMMARY

Component	Rejected Quantity	Reason for Rejection
Liner Shim (291669-00)	0	none
Liner Cap (291670-00)	1	dimensional ^b
Liner Body (291671-00)	1	visual ^b
SM Cap (291672-00)	4	visual ^b
SM Body (291673-00)	10	dye penetrant ^a
Clad Cap (291674-00)	3	dimensional ^b
Clad Body (291675-00)	5	dye penetrant ^a
^a Material-related ^b Operator-related		

TABLE 5 – CERTIFICATE OF INSPECTION REPORT

C of I Number	Date Accepted	Component	Lot Size	Defectiveness	% Lot Acceptance
88-7110	01/14/89	Liner Shim	88	None	100
88-7111	01/12/89	Clad Body	13	None	100
88-7113	01/12/89	Liner Body	89	Rejected*	0
88-7114	01/12/89	SM Cap	78	None	100
88-7117	01/18/89	SM Body	120	None	100
88-7128	02/09/89	Liner Body	89	None	100
88-7129	02/15/89	Clad Body	51	None	100
88-7130	02/15/89	Clad Cap	13	None	100
88-7150	03/14/89	Liner Shim	90	None	100
88-7161	04/17/89	Clad Cap	74	None	100
88-7162	04/17/89	Clad Body	53	None	100
88-7166	04/18/89	Liner Cap	44	None	100
88-7167	04/18/89	Liner Cap	44	None	100
88-7168	04/21/89	Liner Shim	180	None	100
88-7169	04/21/89	Liner Shim	180	None	100
88-7173	05/03/89	Clad Cap	124	None	100
88-7185	06/07/89	Clad Body	48	None	100
88-7191	06/23/89	Clad Body	34	None	100
88-7198	07/07/89	Liner Body	87	None	100
88-7199	07/07/89	SM Body	73	None	100
88-7217	09/10/89	Clad Body	48	None	100
88-7222	08/18/89	SM Cap	89	None	100
88-7223	08/17/89	Clad Cap	89	None	100
88-7231	09/20/89	Liner Body	88	None	100
88-7232	09/25/89	Clad Body	58	None	100
88-7245	10/30/89	Liner Body	261	None	100
88-7247	11/01/89	Liner Cap	179	None	100
88-7252	11/16/89	SM Cap	88	None	100
88-7261	12/05/89	SM Body	121	None	100
88-7263	12/11/89	SM Body	81	None	100
88-7264	12/15/89	SM Body	34	None	100
88-7265	12/15/89	Clad Cap	176	None	100
88-7267	12/20/89	Clad Cap	90	None	100
88-7268	12/20/89	Liner Cap	175	None	100

*Gaging discrepancy; required reworking/recertification of gages

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