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**OAK RIDGE
Y-12
PLANT**

MARTIN MARIETTA

**IRIDIUM ALLOY CLAD VENT SET
MANUFACTURING QUALIFICATION STUDIES**

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INTRODUCTION

In 1987 the Department of Energy-Office of Special Applications (DOE-OSA) decided to transfer the iridium alloy Clad Vent Set (CVS) manufacturing for the General Purpose Heat Source (GPHS) program from EG&G Mound Applied Technologies, Inc. (EG&G-MAT) to the Oak Ridge Y-12 Plant operated by Martin Marietta Energy Systems, Inc. (Energy Systems). The reason for this transfer was to consolidate the GPHS program iridium hardware manufacturing. The CVS starting stock of iridium powder, foil, and blanks were already being manufactured at another Energy Systems facility - the Oak Ridge National Laboratory (ORNL).

Since 1987 CVS manufacturing technology transfer efforts have taken place between EG&G-MAT and Energy Systems. EG&G-MAT retained all of their tooling, but they supplied all the necessary product drawings, specifications, and procedures, as well as their tooling drawings. Most of the tooling designs and processing steps were duplicated at the Y-12 Plant. Minor changes were required in both tooling design and processing steps, to accommodate particular health, safety, environmental, and manufacturing requirements at the Y-12 Plant.

In order to evaluate the effects of the key Y-12 Plant processing modifications, four joint Y-12 Plant/EG&G-MAT iridium CVS manufacturing qualification studies were organized. The successful completion of these studies allowed the Y-12 Plant to commence pilot production of CVS components for the CRAF and CASSINI missions. The CVS cup metallurgical qualification work will be presented here.

METHODS

The cup metallurgical comparisons consisted of evaluating porosity and inclusion contents, grain size and microstructural attributes, and microhardness of both the Y-12 Plant and EG&G-MAT products. Initially, the Y-12 Plant product was four new process (vacuum arc remelted, extruded, and rolled) and one old process (arc cast and rolled) iridium alloy, DOP-26, nonflight quality cups. These cups were recrystallized for one hour at 1573° K in vacuum (1.3×10^{-2} Pa or better). This was performed in a Y-12 Plant Development Division furnace because the new Y-12 Plant production high temperature vacuum furnaces were not available at the time. The EG&G-MAT product was five old process, flight quality cups recrystallized using the same parameters described above. The EG&G-MAT cups were also outgassed for one hour at 1773°K in vacuum. The Y-12 Plant cups were not outgassed because both Energy Systems and EG&G-MAT personnel concurred that the 1773°K outgassing step was probably unnecessary or

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at least excessive (with regard to temperature). It should be noted that an optional 1498°K outgassing step has been retained in the Y-12 Plant assembly operations.

The cups were electrical discharge machined (EDM) into 12 equal pole-to-equator sections at each facility. Each section was sequentially numbered from 1 to 12 as it was machined. The EDM edges were mechanically removed by sanding. Cup sections 2 and 8 were metallographically prepared and analyzed at the Y-12 Plant, while sections 1 and 7 were prepared and analyzed at EG&G-MAT. The remaining sections were used for other qualification studies.

Microhardness readings were taken at EG&G-MAT using their standard 200 g test load. All samples tested at the Y-12 Plant were tested at both 200 g and 1000 g test loads. This was done with the intent to qualify the 1000 g test load for production use.

All the results from the analyses at each site were compared. These comparisons indicated that follow-up work was required to achieve full recrystallization in non-outgassed new process material. It was also evident that additional microhardness testing was required because of the use of an improperly calibrated microhardness tester at the Y-12 Plant. This follow-up work was conducted at the Y-12 Plant on new process material recrystallized for one hour at 1648°K in one of the new production high temperature vacuum furnaces.

Before any of these follow-up heat treat runs were made one of the new production vacuum furnaces was zone certified. The zone certification was conducted at 1648°K using eight thermocouples. Three of these thermocouples were the regular furnace thermocouples arranged at different heights. One of each of the five auxiliary thermocouples were placed in the corners and center of the hearth. Five tungsten weights were used to hold the thermocouples in position and to serve as heat treat mass simulators. During the zone certification the furnace thermocouples ranged from 1646 to 1649°K while the auxiliary hearth thermocouples ranged from 1656 to 1660°K. The five hearth thermocouples were approximately 10°K hotter because of their proximity to the bottom furnace elements. The cup heat treat procedure calls for 1648 ± 25°K; therefore, these minor gradients were deemed acceptable.

Once the zone certification was successfully completed, three additional heat treat runs were made with new process cups (for destructive testing) as well as scrap cups used to simulate the effects of a full heat treat group. The first additional heat treat run was made with three new process cups (from blanks E1-5-1, 2, and 3) and twenty-five scrap cups. All of these cups were placed on and covered with graphite felt. The second heat treat run was made with two new process cups (from blanks E1-10-6 and -7) and 22 scrap cups. This run was made using a tantalum box and cover lined on the inside bottom with graphite felt. The reason for using the tantalum box was to minimize the use of graphite felt in the high temperature vacuum furnace in order to better protect the iridium and the furnace itself from potential contamination.

All five of the new process cups from the first two additional heat treat runs were EDMed into sections. Six sections from each cup were evaluated for extent of recrystallization and grain size only. Since all five cups were found to be fully recrystallized, the third and final follow-up heat treat run was made with three new process cups (from blanks D1-10-4, CR3-13-1 and -6) and 33 scrap cups. This run was also made using the tantalum box and cover lined on the inside bottom with graphite felt. The two blanks from the CR3 ingot were intentionally chosen as material produced using the most recent blank processing improvements (primarily improved

vacuum arc remelting/extrusion/rolling practices). Again, after heat treating the three cups were EDMed into sections. Six sections from each cup were given a full metallurgical evaluation to determine how well they conformed to the metallography requirements of Energy Systems specification, GPHS-M-188, Revision B (equivalent to EG&G-MAT's SPA790433, Revision E). These requirements are listed below.

Metallography Requirements - GPHS-M-188, Revision B

Hardness. Microhardness of the fully heat-treated cup shall be between 250 and 350 diamond pyramid hardness (DPH). This test shall be performed with a diamond pyramid indenter at the load of 1000 g according to ASTM E 92-82. The averages of three hardness values normal to the centerline shall be taken at the pole, radius, and equator of at least two sample sections. The hardness shall be taken on unetched surfaces of mounted sections.

Microstructure. Grain size of the fully heat-treated cup shall not exceed ASTM grain size 3, and any internal defects shall be kept within acceptable limits as specified in the following table.

POROSITY

No single void greater than 10 microns. Maximum concentration of porosity shall not exceed the level grouping shown in ASTM B276-79, AO2.

INCLUSIONS

There shall be no continuous stringer longer than 0.25 mm. The combined length of all continuous stringers and individual inclusion in any 6.4 mm length of a sample shall not exceed a length of 0.4 mm. Use ASTM E45-81 for method.

There shall be no cracks or laminations.

The microstructure of entire cross-sectional area of the four sections shall be examined. Any anomalous metallurgical conditions shall be noted and photographed.

RESULTS AND DISCUSSION

The comparison evaluations done on the sections from the five cups produced by each facility indicated excellent agreement with regard to porosity and inclusion contents. There were no out-of-specification conditions noted for any of these ten cups nor for the three follow-up cups that were also evaluated for internal defects.

The evaluations for grain size and microstructural attributes such as elongated or duplex grain structure and cracks or delaminations were also in excellent agreement. As stated previously the new process material did not fully recrystallize at 1573°K (see Figure 1). This is consistent with earlier work at EG&G-MAT (Forrest et al. 1986). Figure 2 shows that new process material heat treated at 1648°K, but not outgassed, does fully recrystallize. Table 1 shows that material in this condition has the same or slightly finer grain size than any of the other material conditions.

There were no consistent grain size patterns evident with regard to cup location/orientation within the Y-12 Plant production furnace nor in relation to position (pole, radius, or equator) within sections. Cracks or delaminations were found only in some of the non-flight quality original study material.

The Y-12 microhardness data from the original four cups made at the Y-12 Plant from new process material are not included in Table 1 because of (1) the previously mentioned microhardness tester calibration problems and (2) the inhomogeneous microstructure from incomplete recrystallization. The valid microhardness ranges shown in Table 1 indicate that the new process material tends to be slightly harder than the old process material. The microhardness differences between loads at specific positions (same cup and same position) ranged from 24 to 106 DPH, but they were typically around 60 DPH. The 1000 g test load always produced lower values. This is consistent with earlier unpublished findings by T. K. Roche at ORNL and is best explained by the high elastic modulus, approximately 517 GPa, for iridium.

A statistical analysis was performed on both sets of microhardness data (200 g and 1000 g test load) from the last 18 cup sections. There were no statistically significant differences found among cups or locations using the 200 g test load. The overall average was 341 ± 78 DPH (3 sigma limits). This implies limits of 260 to 420 DPH versus the present specification of 250 to 350 DPH. Based on the present specification limits, 36% of the reported positional (pole, radius, or equator) averages would be expected to be outside the specification limits.

Using the 1000 g test load there were no statistical differences in microhardness among the cups; however, there were some minor differences between locations. The poles averaged 289 ± 49 (3 sigma limits) versus 272 ± 40 DPH at the radius and 272 ± 47 DPH at the equator. The overall average was 278 ± 50 DPH. This implies limits of 230 to 330 DPH. Based on the present specification limits, 5% of the positional averages would be expected to be outside the specification limits. The 1000 g load tended to reduce the variability in the data compared to the 200 g load because of the reduced elastic modulus effects, as well as the increased operator ease in reading the indentations.

CONCLUSIONS

The initial Y-12 Plant and EG&G-MAT cup metallurgical comparisons in conjunction with the Y-12 Plant follow-up work has confirmed/established agreement between the Y-12 Plant and EG&G-MAT on the specified cup metallurgical criteria. Energy Systems and EG&G-MAT personnel concurred that the use of a 1000 g test load for microhardness testing will be acceptable for the iridium CVS cups. Additional 1000 g test load data will be collected and analyzed to determine whether or not the microhardness requirements should be modified. The Y-12 Plant iridium CVS manufacturing processes are now considered fully qualified.

Reference

Mary A. Forest, James R. McDougal, and Ranny W. Saylor, General Purpose Heat Source (GPHS), Clad Vent Set (CVS) Formability Study, MLM-3395, Monsanto Research Corporation, Mound Facility, Miamisburg, Ohio (November 1986).

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Figure 1. PN 3792-14-3460-2 from Work Request 5628 (blank BR6-8--5)
Incomplete recrystallization in radius region of cup
formed from new process material. Recrystallized at
1573°K for 1 hour - not outgassed. Magnification 100X.

Figure 2. PN 3886-40-1494-7 from Work Request 5628 (blank CR3-13-1)
Typical microstructure in polar region of cup formed from improved
new process material. Recrystallized at 1648°K for 1 hour -
not outgassed. ASTM 7 Magnification 100X.

Table 1. Microstructural Data for Various Material/Test Conditions

Blank Process	Cup Fabrication Facility	Heat Treat Condition (°K)	Testing Facility	ASTM Grain Size Number		Vickers Microhardness (DPH)	
				Range	Average	200 g Load	1000 g Load
Old	EG&G - MAT	1573/1773	EG&G - MAT	---	---	294-349	---
Old*	EG&G - MAT	1573/1773	EG&G - MAT	---	7	251-337	---
Old	EG&G - MAT	1573/1773	Y-12 Plant	5-1/2 - 6-1/2	6	---	---
Old	Y-12 Plant	1573	EG&G - MAT	6-1/2 - 7	6-1/2	307-333	---
New*	EG&G - MAT	1573/1773	EG&G - MAT	---	7	286-366	---
New*	EG&G - MAT	1573	EG&G - MAT	NFR**	7	306-391	---
New	Y-12 Plant	1573	EG&G - MAT	NFR**	6-1/2-9	304-357	---
New	Y-12 Plant	1648	Y-12 Plant	6-1/2 - 7-1/2	7+	291-403	252-318

* Data from Forrest et al. 1986.

** NFR - Not fully recrystallized.

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