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**SUMMARY OF DECONTAMINATION COVER
MANUFACTURING EXPERIENCE**

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February 1995

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MANUFACTURING EXPERIENCE**

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SUMMARY

Decontamination cover forming cracks and vent cup assembly leaks through the decontamination covers were early manufacturing problems. The decontamination cover total manufacturing process yield was as low as 55%. Applicable tooling and procedures were examined. All manufacturing steps from foil fabrication to final assembly leak testing were considered as possible causes or contributing factors to these problems. The following principal changes were made to correct these problems:

1. the foil annealing temperature was reduced from 1375° to 1250°C,
2. the decontamination cover fabrication procedure (~~including visual inspection for surface imperfections and elimination of superfluous operations~~) was improved,
3. the postforming dye penetrant inspection procedure was revised for increased sensitivity,
4. a postforming (prewelding) 1250°C/1 h vacuum stress-relief operation was added,
5. a poststress relief (prewelding) decontamination cover piece-part leak test was implemented,
6. the hold-down fixture used during the decontamination cover-to-cup weld was modified, and concomitantly
7. the foil fabrication process was changed from the extruding and rolling of 63-mm-diam vacuum arc-remelted ingots (extrusion process) to the rolling of 19-mm-square arc-melted drop castings (drop cast process),

Since these changes were incorporated, the decontamination cover total manufacturing process yield has been 91%. Most importantly, more than 99% of the decontamination covers welded onto vent cup assemblies were acceptable. The drastic yield improvement is attributed primarily to the change in the foil annealing temperature from 1375° to 1250°C and secondarily to the improvements in the decontamination cover fabrication procedure.

INTRODUCTION

Matched pairs of iridium alloy Clad Vent Set (CVS) cups are used to encapsulate individual $^{238}\text{PuO}_2$ fuel pellets for the U.S. Department of Energy (Radioisotope Power Systems Division) General Purpose Heat Source - Radioisotope Thermoelectric Generator (GPHS-RTG) program. The heat from the radioactive decay of the ^{238}Pu is used to produce electrical power thermoelectrically for deep space missions such as the upcoming National Aeronautics and Space Administration Cassini mission to Saturn and the ongoing Galileo and Ulysses missions. The fueled capsules are prepared in a cooperative effort by several organizations. CVSs are produced by Martin Marietta Energy Systems, Inc. (Energy Systems). The $^{238}\text{PuO}_2$ pellet fabrication, fueled CVS closure weld, and decontamination of the external CVS surfaces are performed at the Los Alamos National Laboratory (LANL). The fueled CVSs are assembled into GPHS-RTG units and tested at EG&G-Mound Applied Technologies, Inc. (EG&G-MAT).

A decontamination cover (Fig. 1) is electron-beam welded (by Energy Systems) over the vent hole in each vent cup assembly portion of a CVS, preventing decontamination solutions used at LANL from entering the vent hole. The decontamination cover is removed at EG&G-MAT before each CVS is assembled into a GPHS-RTG.

The decontamination cover total manufacturing process yield during early production for the Cassini mission was as low as 55%, as shown in Table 1. This yield was in contrast to the approximately 85% yield for the pre-production (Batch 2) parts. Table 1 also shows the individual process yields for early production. Most of these losses resulted from through-cracks in the dome of the decontamination cover. Of course, the most costly losses were those found during leak testing after the decontamination cover had been electron beam welded onto the vent cup assembly.

All finished vent cup assemblies must have leak rates less than or equal to 1×10^{-6} standard cm^3/s as determined by a helium mass spectrometer leak detector at 2-atm helium differential pressure. Calculations indicate that a $1\text{-}\mu\text{m}$ -diam hole would produce this leak rate under these conditions, assuming a tube defect geometry and viscous flow.¹ However, subsequent dye penetrant and/or visual (with up to 30X magnification and filtered light) inspection of leaking decontamination covers revealed very narrow through-cracks up to ~ 3.2 mm long. Metallographic examination of these cracks showed them to be intergranular (see Fig. 2) and oriented parallel to the foil rolling direction.

Fig. 1. Decontamination cover and vent cup assembly for GPHS-RTG.

Table 1. Process yields* for decontamination cover manufacturing

Process	Early production yields (%)	Later production yields (%)
Shop visual inspection	70	99
Dye penetrant inspection	86	95
Dimensional inspection	100	100
Preassembly leak test	96	100
Preassembly visual examination	100	98
Assembly leak test	95	99
Total	55	91

*Rounded to the nearest whole number

Fig. 2. Cracks in dome of decontamination cover (100 X).

The following five items were identified as potential contributors to the poor production yield:

1. material [extrusion process foil (63-mm-diam vacuum arc-remelted ingots that were extruded and rolled) versus drop cast process foil (19-mm-square arc-melted drop castings that were rolled)],
2. foil annealing temperature (had been changed to 1375°C to match the temperature required to achieve recrystallization in the cup versus the previous annealing temperature of 1300°C used for the pre-production parts),
3. decontamination cover fabrication procedure (did not require adequate visual examination for surface imperfections before blanking, before forming, and after forming, also forming pressure was not correctly stated and blank flattening, outer diameter (OD) grinding and sizing steps were superfluous operations that had the potential for creating additional surface imperfections),
4. elimination of the prewelding 1500°C "outgas" operation performed during previous CVS manufacturing at EG&G-MAT for the Galileo/Ulysses missions, and
5. weld tooling.

These items were investigated before the production procedures were modified. Also, piece-part nondestructive inspection techniques such as visual inspection, dye penetrant inspection, and leak testing were evaluated with the idea of removing questionable decontamination covers before they were welded onto vent cup assemblies.

PROCEDURE

The initial "fixes" to the decontamination cover manufacturing problem(s) were to implement a piece-part leak-testing operation and a 1500°C "outgassing"--or, more appropriately, stress relieving--operation before welding a decontamination cover to a vent cup assembly. The piece-part leak-testing operation was the first processing change made in response to the problem of leaking assemblies. Although this operation was not in place at the very beginning of early production, it is listed as an early production process with its own yield in Table 1. The 1500°C operation was added because EG&G-MAT had used it during previous CVS production and had not reported cracking or leak-test problems with decontamination covers. The EG&G-MAT decontamination cover piece-part yield was reported to be 92%² with drop cast process foil annealed at 1300°C.

The piece-part leak test and 1500°C stress-relief changes were followed quickly by an evaluation of the effects of different annealing temperatures on formability as well as different annealing/stress-relieving temperature combinations on postweld leak-test results. Concurrently, an evaluation of the decontamination cover dye penetrant inspection was conducted. Also, the tantalum external tip (or hold-down pin) used to hold the decontamination cover during the decontamination cover-to-cup electron-beam weld was redesigned.

The annealing and stress-relieving evaluations led to changing the foil annealing temperature from 1375° to 1250°C for 1 h in vacuum. The postforming (prewelding) stress-relief temperature was changed from the interim 1500° to 1250°C for 1 h in vacuum. The sensitivity of the dye penetrant inspection was increased so that potential "leakers" would be removed from the production stream early. The piece-part leak-testing operation was maintained as a final screen for leakers before assembly welding. The hold-down pin used to keep the decontamination cover in place during the decontamination cover-to-cup electron-beam weld was modified so that the pin contacted only the outer flat and not the dome as the previous tooling did.

The decontamination cover fabrication procedure (GPHS-XF-3619) underwent several modifications (revisions C, D, E, and F) over a period of one year. Very few decontamination covers were fabricated during this one year transition period, therefore the differences between fabrication procedure revisions B and F were the most critical and useful. The key modifications were as follows: (1) visual examination for surface imperfections was done using 5X to 30X magnification at *all* critical steps - before blanking, before forming, and after forming, (2) blanks found to have minor surface imperfections could be converted to frit vent components instead of scrapping them, and (3) the blank flattening, OD grinding, and sizing operations were eliminated as superfluous and potential sources of additional surface imperfections. Note that the forming pressure was corrected in the procedure to be 1000 psi which had been used all along instead of the erroneously stated 250 psi.

Coincidentally, almost half of the parts subsequently produced using all of these modifications were from foil made using the drop cast process rather than the extrusion process. The production data were analyzed statistically to determine and separate the effect(s) of all of the aforementioned modifications on the process yields.

RESULTS AND DISCUSSION

Since the aforementioned changes were made, the decontamination cover total manufacturing process yield has been 91%, as shown in Table 1. Most importantly, only 3 vent cup assemblies (of 444 total) have failed the critical assembly leak test during this later production versus 11 (of 204 total) during early production. Note that more than twice as many assemblies were produced during later production than during early production. Examination of the data in Table 1 indicates that drastic yield improvements were achieved in both the shop visual and the dye penetrant inspections. These drastic improvements, combined with notable improvements in the preassembly and assembly leak-test yields and a slight reduction in yield for the preassembly visual exam, brought the total process yield to a respectable 91%.

The yield improvements for the shop visual and dye penetrant inspections and both leak tests indicate that the foil fabrication process, the foil annealing temperature, the decontamination cover fabrication procedure, or a combination of these three items changed to make these improvements. Without improvements in at least one of these items, the later production yields for the shop visual and dye penetrant inspections should have been the same as or lower than the early production yields. Review of the processing records for these three items showed that changes were made for all three in later production. However, separating the effects of these three items from each other and from the effects of all of the other changes was quite laborious but fruitful.

The data for both the dye penetrant inspection and the leak-test operations (combined piece part and assembly leak tests) were the keys to separating the effects of all of the changes. These data, shown in Table 2, indicate that the greatest yield improvements occurred with the reduction in the annealing temperature. Additional significant improvements in the yields occurred when the fabrication procedure was revised. Changing from the extrusion process foil to the drop cast process foil apparently offered no improvement. Unfortunately, none of the drop cast process foil was annealed at 1375°C. However, drop cast process foil annealed at 1300°C fabricated into decontamination covers by Energy Systems (for Cassini pre-production) per a procedure similar to revision B had yields of 92% for dye penetrant inspection and 93% for the assembly leak test. It is believed that the EG&G-MAT production yields for the Galileo and Ulysses missions (also using drop cast process foil annealed at 1300°C and a fabrication procedure somewhat similar to revision B) were comparable or higher.

Table 2. Dye penetrant and leak-test yields by foil fabrication process, annealing temperature and decontamination cover fabrication procedure

Manufacturing condition	Dye penetrant $\frac{\text{Accept}}{\text{total}}$ (%)	Leak test $\frac{\text{Accept}}{\text{total}}$ (%)
Extrusion process 1375°C anneal Revision B fabrication	$\frac{113}{145}$ (78)	$\frac{75}{88}$ (85)
Extrusion process 1250°C Revision B fabrication	$\frac{239}{269}$ (89)	$\frac{215}{223}$ (96)
Extrusion process 1250°C Revision C thru F fabrication	$\frac{83}{85}$ (98)	$\frac{67}{68}$ (99)
Drop cast process 1250°C Revision C thru F fabrication	$\frac{290}{306}$ (95)	$\frac{278}{279}$ (99+)

It is expected that the lower annealing temperature (1250° or maybe even 1300° vs 1375°C) produces a finer grain size, which accounts for the significant reduction in the incidence of cracking. Note that the metallurgical requirement for the foil after annealing is that it be 100% recrystallized with an American Society for Testing and Materials (ASTM) grain size of 5.0 or finer. The production certification records generally indicate that foil annealed at 1250°C had a finer grain size (ASTM 6.5 to 8.5) than foil annealed at 1375°C (ASTM grain size of 6.5 to 7.5). Note that the drop cast process foil annealed at 1300°C which was used with good yields for Cassini Batch 2 prove-in parts had an ASTM grain size of 7.0. Earlier work³ with extrusion process foil had shown as well that the lower annealing temperature yielded both a finer grain size and a reduction in the incidence of cracking during forming. Also, the frit vent cover disc parts are susceptible to intergranular cracking problems during the edge pressing operation, which is done after the 1900°C/2 h diffusion-bonding step. The 1900°C/2 h exposure produces a "bamboo structure" in which the grains are very large with single, straight-line grain boundaries through the thickness. Thus, grain size plays an important role in the cold formability of iridium alloy foil.

Changing the decontamination cover fabrication procedure apparently produced notable yield improvements for both dye penetrant inspection and leak testing (Table 2). The specific changes most responsible for these improvements are probably the visual examinations at 5X to 30X magnification before and after blanking. These two visual examinations coupled with the fact that blanks found to contain minor surface defects

could be converted to frit vent components would account for the 99% yield for the shop visual inspection during later production (Table 1). The visual examination at 5X to 30X magnification after forming would have reduced this yield if flaws had been manifest after forming. Elimination of the superfluous operations of flattening, OD grinding, and sizing, would have reduced the potential for introducing additional surface flaws and thus may have been partly responsible for the high yields for the dye penetrant and leak test operations shown in Table 2.

Efforts were made to improve the surface condition of the wrought foil. Although it is possible that these efforts were partially successful, the results were not quantified. Nevertheless, if the foil surface condition did improve with time, this would account at least partially for the yield improvements in Tables 1 and 2. Formerly the foil surface condition was inspected visually with 5X magnification and with dye penetrant. All imperfections were marked. Since this meant that virtually every area of a piece of foil had some kind of imperfection on one side or the other the results were rather useless. Because of this the general foil surface inspection was stopped. Thus it is conceivable that the efforts to improve the foil surface condition were successful making the material less susceptible to cracking during decontamination cover manufacturing. However, improvements in the foil surface condition could not be separated from improvements in the foil selection methods for fabricating decontamination covers.

Table 2 indicates that the foil fabrication process, extrusion or drop cast, had little or no effect on the decontamination cover yields for the dye penetrant and leak test operations. This is contrary to GPHS program production lore at Energy Systems. It was believed that the drop cast process foil was more forgiving than the extrusion process foil. Analyses of the decontamination cover production data indicate that this belief stemmed from the fact that the poor early production yields were all with extrusion process foil while the good later production yields were mostly with drop cast process foil. Without analyzing the data further it would be easy to conclude that the early yield problems were material-related. However, Table 2 indicates that the changes in the annealing temperature and the fabrication procedure were responsible for the improved yields.

CONCLUSIONS

Numerous changes were made in the decontamination cover manufacturing process. These changes improved the decontamination cover total manufacturing process yield from 55% during early production to 91% during later production. Most importantly, more than 99% of the decontamination covers welded onto vent cup assemblies were acceptable. The drastic yield improvement is attributed primarily to the change in the foil annealing temperature from 1375° to 1250°C and secondarily to the modifications in the decontamination cover fabrication procedure.

FUTURE WORK

The authors recommend that a grain size and microindentation hardness (200-g load) study be conducted with sections of drop cast process foil annealed at various temperatures (i.e., 1150°, 1225°, 1375°, and 1500°C). These results would be compared with existing results for extrusion process foil. Material annealed at the two most promising temperatures would be formed into decontamination covers and then visually and dye penetrant inspected for cracks. This information would be used to determine whether the foil-grain-size requirement should be modified. Fortunately, only a procedure change, not a specification change, would be necessary.

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