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ORNL/TM-2010/14

Metallurgical Evaluation of Grit Blasted Versus Non-Grit Blasted Iridium Alloy Clad Vent Set Cup Surfaces

February 2010

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UT-BATTELLE

ORNL-27 (4-00)

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ORNL/TM-2010/14

Materials Science and Technology Division

METALLURGICAL EVALUATION OF GRIT BLASTED VERSUS NON-GRIT BLASTED IRIDIUM ALLOY CLAD VENT SET CUP SURFACES

George B. Ulrich and Hu F. Longmire

Date Published: February 2010

Prepared by
OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831-6283
managed by
UT-BATTELLE, LLC
for the
U.S. DEPARTMENT OF ENERGY
under contract DE-AC05-00OR22725

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ACRONYMS

ASTM	American Society for Testing and Materials
CVS	Clad Vent Set
EDM	electrical discharge machined
h	hour
HV	Vickers hardness
min	minute
ORNL	Oak Ridge National Laboratory

METALLURGICAL EVALUATION OF GRIT BLASTED VERSUS NON-GRIT BLASTED IRIDIUM ALLOY CLAD VENT SET CUP SURFACES*

George B. Ulrich and Hu F. Longmire

ABSTRACT

Metallurgical evaluations were conducted to determine what, if any, grain size differences exist between grit blasted and non-grit blasted DOP-26 iridium alloy cup surfaces and if grit blasting imparts sufficient compressive cold work to induce abnormal grain growth during subsequent temperature exposures. Metallographic measurements indicated that grit blasting cold worked the outside cup surface to a depth of approximately 19 µm. Subsequent processing through the air burn-off (635°C/4h) and vacuum outgassing (1250°C/1h) operations was found to uniformly recrystallize the cold worked surface to produce grains with an average diameter of approximately 8.5 µm (American Society for Testing and Materials (ASTM) grain size number 11). Follow-on heat treatments at 1375°C, 1500°C, and 1900°C for durations ranging from 1 min to 70 h yielded uniform grain sizes and no abnormal grain growth from grit blasting. Abnormal grain growth was noted at the 1500°C and 1900°C heat treatments in areas of cold work from excessive clamping during sample preparation.

1. INTRODUCTION

DOP-26 iridium alloy (nominally 0.3% tungsten, 0.006% thorium, and 0.005% aluminum by weight) cups (Fig. 1) are used for primary containment of plutonium-238 dioxide pellets. The encapsulated pellets (fueled Clad Vent Sets) are used in radioisotope power systems usually to power instrumentation for National Aeronautic and Space Administration deep-space missions. The Clad Vent Set (CVS) manufacturing process/procedure flow for cups is shown in Fig. 2. Blanks (52 mm diameter x 0.65 mm thick) are received in the wrought (80% reduction after an intermediate 1300°C/1 h recrystallization heat treatment) and vacuum stress relieved (900°C/1 h) condition. The blanks are encapsulated in tantalum and stainless steel then warm-formed (925°C/10 min preheat) in two deep draw operations. After cup forming the encapsulation materials are chemically removed and the iridium cups are acid cleaned prior to recrystallization at 1375°C/1 h in vacuum [$\leq 1 \times 10^{-4}$ Torr]).

One cup from a heat treat lot (group of cups recrystallized together) is destructively tested (D-tested) for metallurgical and chemical analyses per the Oak Ridge National Laboratory (ORNL) Deep Drawn Iridium Alloy Cups Specification GPHS-M-188, Rev. 8. Each D-test cup is electrical discharge machined (EDM) into 12 samples (cup segments). The samples are deburred by hand to remove the EDM layer prior to additional sample preparation. The D-test cup may be selected any time after the post-deep-draw recrystallization heat treat operation. Many D-test cups are selected for testing after the dye penetrant inspection/polishing operations as shown in Fig. 2, but some are selected after the grit blasting operation depending on production needs/availability.

The grit blasting operation is conducted with tungsten carbide powder procured per ORNL Specification GPHS-M-189, Rev. 6. The powder is sieved to Tyler series -65 mesh (212 µm)/+ 150 mesh (106 µm). A dedicated Empire Abrasive Equipment Company (Langhorne, PA) Pro-Finish Model PF-2636 grit blaster is used at a line pressure of 50 ± 5 psi for approximately 25 s with the nozzle approximately 4” from a cup. One cup at a time is placed in a polyvinyl chloride fixture (T2E139776, Rev. F) for consistent holding and masking.

*Research sponsored by the U.S. Department of Energy, Office of Space and Defense Power Systems, under contract with UT-Battelle, LLC.

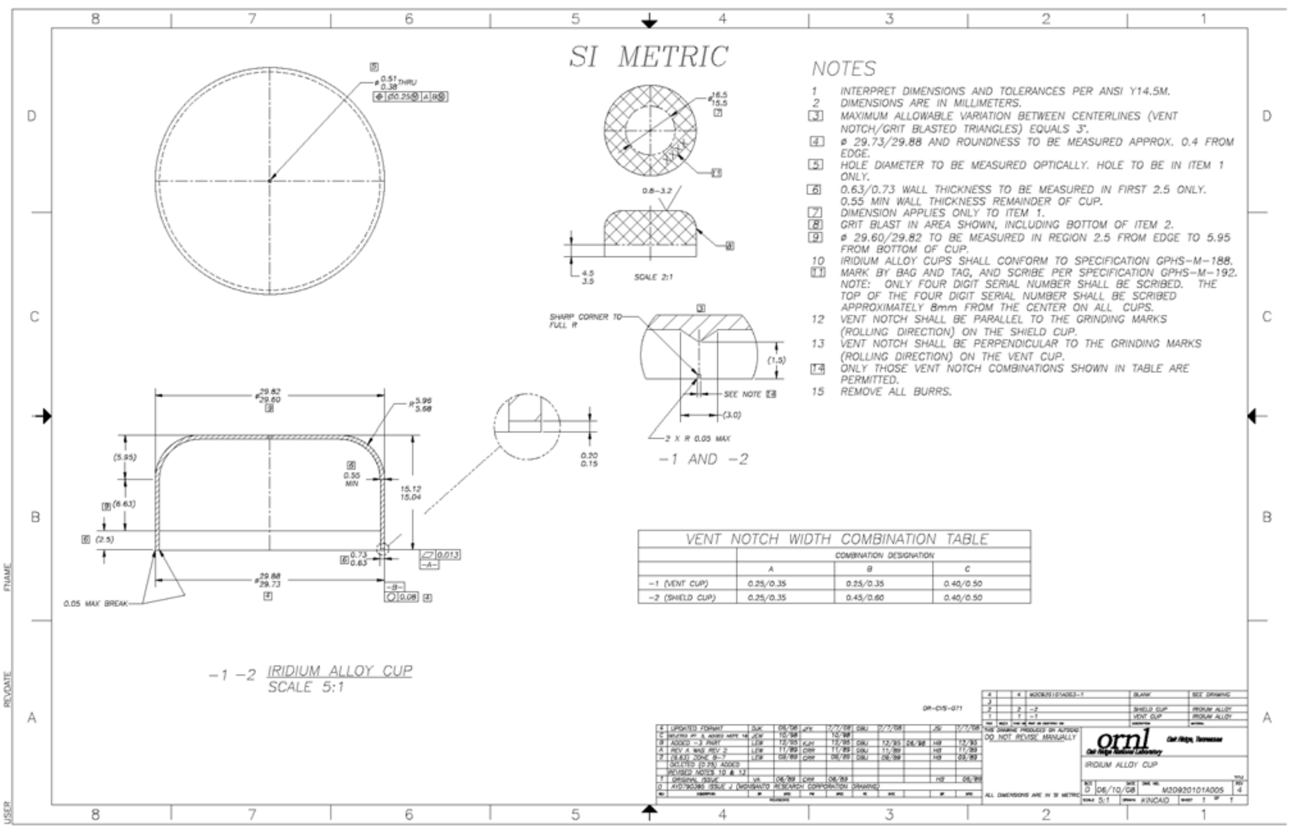

Fig. 1. Iridium alloy cup drawing.

- Receive Blank in wrought & stress relieved condition
- Engrave Blank GPHS-XF-3624/25
- Clean-Ir, Ta, SS GPHS-Y-005/-007
- Weld Blank Assembly GPHS-K-001
- First-form GPHS-XF-3624/25
- Fabricate Mild Steel Cup GPHS-FABR-7735/7735A
- Second-form GPHS-XF-3624/25
- Trim GPHS-XF-3624/25
- Acid Strip GPHS-Y-006
- Clean GPHS-Y-005
- Store Archive Rings GPHS-XF-3624/25
- Recrystallize GPHS-Y-002
- Size GPHS-XF-3624/25
- Grind GPHS-XF-3624/25
- Lap GPHS-XF-3624/25

- Vent Notch GPHS-XF-3624/25
- Polish GPHS-XF-3624/25
- Penetrant Inspection GPHS-Y-010
- Polish GPHS-XF-3624/25
- Section D-test GPHS-XF-3624/25A
- Certify Carbon GPHS-Y-021 or MST-MatP-SOG-110
- Certify Oxygen GPHS-Y-022 or MST-MatP-SOG-111
- Certify GDMS MET-MatP-SOP-79
- Certify Metallography MET-MetL-QA-2
- EDM Vent Hole (3624) GPHS-XF-3624/25
- Clean GPHS-Y-005
- WC Grit Impurity Cert. ACO-SOW-001
- Grit Blast GPHS-XF-3624/25
- Flatten GPHS-XF-3624/25
- Engrave Cup (final) GPHS-XF-3624/25

- Cup Wt./Fab. Audit GPHS-XF-3624/25
- Dimensional Inspection GPHS-C-3624/25
- Rework-Fab Operations GPHS-XF-3624/25
- Dimensional Inspection GPHS-C-3624/25
- Clean GPHS-Y-005
- Cert. Review Assign - 9753/9754
- Air Burn-Off (witness specimen or assembly) GPHS-Y-002
- Vacuum Outgas (witness specimen or assembly) GPHS-Y-002

Fig. 2. Iridium alloy cup CVS manufacturing process/procedure flow.

The fixture is placed on a rotary table and manually rotated while the grit blast nozzle is manually held and moved to ensure full coverage of the exposed surfaces of the cup. Note: Grit blasting is done on the outer surfaces of the cups for emissivity purposes in use.

2. PROCEDURE

A total of eight D-test samples from cup 3625-05-5104 (blank G3-14-4) and nine samples from cup 3625-05-5164 (blank G2-3-7) were metallurgically evaluated in various processing conditions. Cup 3625-05-5104 was processed through the Rework-Fab Operations (see Fig. 2) for re-sizing prior to D-test sectioning while cup 3625-05-5164 was processed through the Engrave Cup (final) operation (Fig. 2) without any rework before sectioning. Each sample was manually deburred after it was EDM from the cup to remove the EDM-affected edges. The samples were then prepared metallographically (mount, grind, polish, and etch) and evaluated for depth of cold work, grain size, and microhardness (Vickers using 1000 g load – average of three indentations at each location) at the pole, radius, and equator locations per ORNL procedure MET-MetL-QA-2, Rev. 9 as appropriate. Cold worked areas were characterized by visible slip lines near the outer cup surface. These slip lines appeared as wavy slip which was easily distinguished from straight scratch marks that sometimes remained as artifacts of polishing. The microhardness measurements were from the interior or bulk region - not the cold worked surface region.

Four original D-test samples from each of the two grit blasted cups were evaluated at the grit blasted surface for depth of cold work and overall for grain size and microhardness. The cold work depth values consisted of an average of three measurements from a 500X magnification image. Two other D-test samples (from each cup) that had been used as witness specimens for the air burn-off (635°C/4 h) and vacuum outgas (1250°C/1 h) operations were evaluated for grain sizes on the grit blasted (outside) and the non-grit blasted (inside) surfaces and overall for grain size and microhardness. (Note: witness specimens are no longer utilized in CVS manufacturing. Witness specimens were extra D-test samples that accompanied finished assemblies through the air burn-off and vacuum outgassing operations.) Additional used witness specimens were given additional vacuum heat treatments at 1375°C/1 h, 1500°C/1 h, 1500°C/70 h, 1900°C/1 min, or 1900°C/2 h and evaluated for grain sizes on the grit blasted and the non-grit blasted surfaces and overall for grain size and microhardness. Note: The selection of the additional heat treatment temperatures and times was arbitrary.

3. RESULTS AND DISCUSSION

3.1 RECRYSTALLIZED AND GRIT BLASTED CONDITION

A summary of the combined results for the four original D-test samples from each of the two grit blasted cups (total of 8 samples) are shown in Table 1. The table includes the combined averages, standard deviations, relative standard deviations (standard deviation ÷ average x 100), maximums, minimums, and ranges by position for all eight samples. The results indicate that the depth of cold work averages about 19 μm regardless of position. The depth of cold work minimum and maximum values show the variability inherent in the manual grit blast operation with overall minimum and maximum depths of 11 and 36 μm, respectively. Figs. 3 and 4 are photomicrographs showing the typical grit blasted (outside) and non-grit blasted (inside) surfaces, respectively. Table 1 shows that the overall (excluding the cold worked surface) grain sizes are uniform – typically near an ASTM grain size number 6. Also the overall microhardness values (Vickers indenter with 1000 gram-force) are fairly consistent with overall minimums and maximums of 231 and 271 HV, respectively. The radius position with a

Table 1. Metallurgical Summary for Samples (8 total) in the Recrystallized and Grit Blasted Condition

	Depth of cold work @ grit blast edge (µm)			Overall ASTM Grain Size Number			Overall Microhardness (HV 1000 gf)		
	Pole	Radius	Equator	Pole	Radius	Equator	Pole	Radius	Equator
Av	17	21	19	6.1	6.1	5.9	241	258	244
SD	6	5	8	0.3	0.4	0.4	2	7	10
RSD (%)	35	24	42	5	7	7	1	3	4
Max	29	31	36	5.5	5.5	5.5	243	271	259
Min	11	14	12	6.5	6.5	6.5	236	249	231
Range	18	17	24	1.0	1.0	1.0	7	22	28

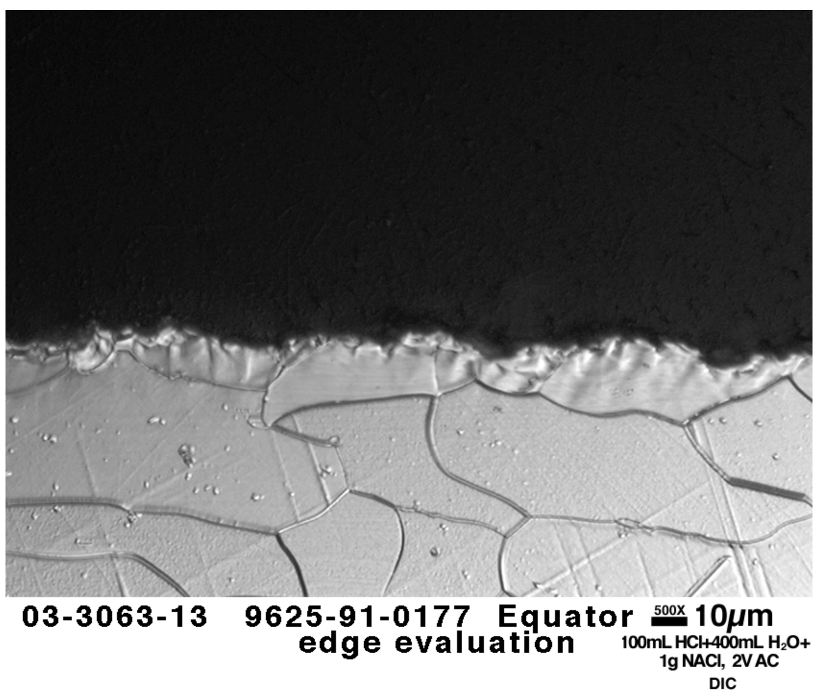

Fig. 3. Photomicrograph at 500X magnification showing typical cold worked surface from grit blasting on outside of iridium alloy cup.

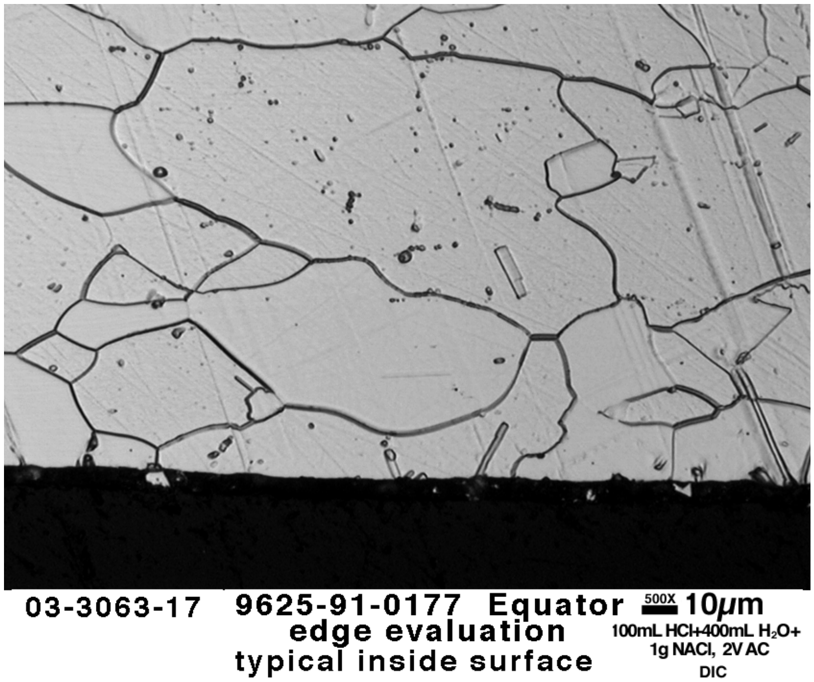

Fig. 4. Photomicrograph at 500X magnification showing typical non-grit blasted (inside) surface of iridium alloy cup.

grand average of 258 HV is slightly elevated from the pole grand average of 241 HV and the equator grand average of 244. This may be explained by the cup cold sizing operation (see Fig. 2 – immediately after the recrystallization operation or part of the final rework) imparting more cold work in the radius than anywhere else.

3.2 RECRYSTALLIZED GRIT BLASTED, AIR BURN-OFF, AND VACUUM OUTGASSED CONDITION
(SAME AS FINISHED CVS ASSEMBLY AND/OR USED WITNESS SPECIMEN)

A summary of the combined results at the outside and inside surfaces for two used witness specimens (recrystallized, grit blasted, air burn-off, and vacuum outgassed condition which is the same condition of finished CVS assemblies) from both of the grit blasted cups are shown in Table 2. The table includes the combined averages, standard deviations, relative standard deviations, maximums, minimums, and ranges by position and surface (outside grit blasted or inside non-grit blasted) for all four samples. Also Figs. 5 and 6 are photomicrographs showing the typical overall grain structure and the fine-grained outside surface, respectively, for one of these samples. The results show that the outside grit blasted (cold worked) surface is recrystallized by the 1250°C/1 h vacuum outgas operation. The outside surface grain size diameter averages about 9 µm regardless of position versus about 33 µm at the inside surface. These average grain size diameters convert to ASTM grain size numbers of 11 and 7 for the outside and inside surfaces, respectively. Recrystallization and probably some grain growth occurred during the original 1375°C/1 h “recrystallization” operation at the inside surfaces (as well as the rest of the cup/sample) while little or no grain growth, only recrystallization, occurred at the grit blasted (cold worked) outside surfaces during the subsequent 1250°C/1 h vacuum outgas operation.

Table 2. Metallurgical Summary for Outside and Inside Surfaces of Samples in the Recrystallized, Grit Blasted, Air Burn-off, and Vacuum Outgassed Condition (Used Witness Specimens – Similar to CVS Assembly)

Surface	Average Grain Size Diameter (μm)			Approximate ASTM Grain Size Number		
Outside	**Pole**	**Radius**	**Equator**	**Pole**	**Radius**	**Equator**
Av	8	10	8	11.1	10.8	10.9
SD	3	4	1	1.6	1.0	0.5
RSD (%)	38	40	13	14	9	5
Max	12	15	10	9.3	9.3	10.4
Min	4	7	7	13.0	11.5	11.6
Range	8	8	3	3.7	2.2	1.2
Inside	**Pole**	**Radius**	**Equator**	**Pole**	**Radius**	**Equator**
Av	30	32	36	7.4	7.1	6.6
SD	11	10	4	1.2	0.9	0.2
RSD (%)	37	31	11	16	13	3
Max	38	46	40	6.5	6.1	6.4
Min	16	24	32	9.1	8.0	6.9
Range	22	22	8	2.6	1.9	0.5

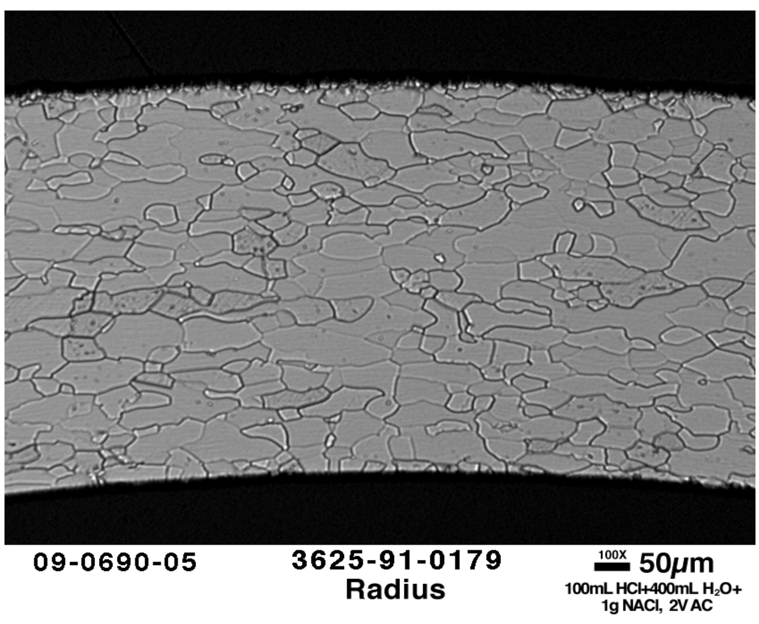

Fig. 5. Photomicrograph at 100X magnification showing fine-grained outside (upper) surface vs. the overall grain size, plus the apparent tendency to have fine grains at the inside (lower) surface for an iridium alloy cup sample in the recrystallized, grit blasted, air burn-off, and vacuum outgassed condition.

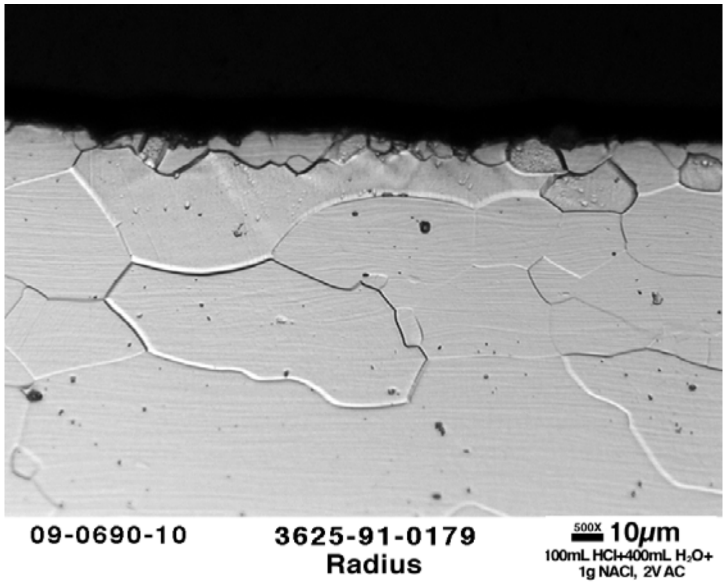

Fig. 6. Photomicrograph at 500X magnification showing fine-grained outside (upper) surface vs. the overall grain size for an iridium alloy cup sample in the recrystallized, grit blasted, air burn-off, and vacuum outgassed condition.

Table 3 summarizes the overall or bulk results for the same samples/condition in Table 2. Comparing the inside surface results in Table 2 to the overall results in Table 3 and/or the overall results in Table 1 indicates that the grain sizes are slightly finer at the inside surfaces, i.e. ASTM grain size numbers of approximately 7 versus 6. This may be a free surface effect, i.e. grains restrained or limited by the free surface, resulting in slightly finer grains. Or there is the possibility that the apparent slight grain size difference between the inside surface and the overall structure is more related to the difficulty in judging grain size near a surface, i.e. there is little or no difference. The overall microhardnesses after the additional vacuum outgassing in Table 3 are approximately 20 HV lower than those after the recrystallization/grit blast operations (shown in Table 1). Most likely cup processing operations (see Fig. 2) after the recrystallization operation through the grit blasting/rework operations, particularly cold sizing, impart a small amount of cold work throughout the overall microstructure. This lightly cold worked condition is typically below any critical deformation level (< 2.5%) for recrystallization or abnormal grain growth in the bulk structure.[1-7] Thus, the 1250°C vacuum outgassing operation serves as a stress relief anneal (recovery only) yielding a lower microhardness for the bulk structure. Note: The ORNL metallography procedure, MET-MetL-QA-2, requires that the microhardness indentations be properly spaced to avoid edge and indentation interactions. Additional tabulations were made to verify that no biases (from improper spacing or any other cause) existed for the three indentations made at each pole, radius, and equator position. Also the overall grain sizes and microhardnesses in Table 3 at the equator in the grit blasted and non-grit blasted regions indicate that grit blasting has little or no effect on the overall/bulk features.

Table 3. Metallurgical Summary for Overall (Bulk Portion of) Samples in the Recrystallized, Grit Blasted, Air Burn-off, and Vacuum Outgassed Condition (Used Witness Specimens –Similar to CVS Assembly)

	Overall ASTM Grain Size Number				Overall Microhardness (HV 1000 gf)			
	Pole	Radius	Equator	Equator non-grit blast region	Pole	Radius	Equator	Equator non-grit blast region
Av	6.6	6.4	6.3	6.1	225	233	223	215
SD	0.3	0.3	0.3	0.5	10	2	2	2
RSD (%)	5	5	5	8	4	1	1	1
Max	6.5	6.0	6.0	5.5	241	235	225	218
Min	7.0	6.5	6.5	6.5	218	230	221	213
Range	0.5	0.5	0.5	1.0	23	5	4	5

3.3 RECRYSTALLIZED, GRIT BLASTED, AIR BURN-OFF, VACUUM OUTGASSED AND SUBSEQUENT HEAT TREATMENT CONDITIONS

Five used witness specimens (recrystallized, grit blasted, air burn-off, and vacuum outgassed condition) were subsequently given a final heat treatment at 1375°C/1 h, 1500°C/1 h, 1500°C/70 h, 1900°C/1 min, or 1900°C/2 h to see if abnormal grain growth would occur via secondary recrystallization.[1,2] The final heat treatment at 1375°C/1 h clearly retained the finer outside surface grains as shown in Table 4 and Figs. 7 and 8. The sample with the final heat treatment at 1500°C/1 h retained some fine grains at the surface (polar location in Table 4 and Fig. 9), but many of the grains had sizes similar to the inside surface grains and the overall structure (Fig. 10 and Table 4). Table 5 shows that even though the grain sizes coarsened (ASTM grain size numbers and grains per thickness decreased) as the final heat treatment temperatures and/or times increased, the microhardnesses remained uniform. This indicates that the 1375°C/1 h recrystallization operation is a full anneal. Note: The microhardnesses in Table 1 (as-recrystallized plus light cold work from subsequent processing) are slightly higher than those in Table 3 (recrystallized, lightly cold worked, stress relieved with 1250°C/1 h vacuum outgas) and Table 5 as expected. The final heat treatments at 1500°C/70 h, 1900°C/1 min, and 1900°C/2 h generally showed uniform grain sizes between the outside and inside surfaces within each sample (Figs. 11, 12 and 13, respectively), except for some anomalous areas that will be addressed further.

Table 4. Metallurgical Summary for the Outside and Inside Surfaces of Samples Given Subsequent Final Heat Treatments after the Recrystallization, Grit Blast, Air Burn-off, and Vacuum Outgas Operations

Final Heat Treatment	Surface	Average Grain Size Diameter (µm)			Approximate ASTM Grain Size Number		
		Pole	Radius	Equator	Pole	Radius	Equator
1375°C/1h	Outside	5	12	11	12.3	9.9	10.1
	Inside	19	29	25	8.5	7.3	7.7
1500°C/1h	Outside	43	40	43	5.9	6.3	5.9
	Inside	38	45	48	6.5	6.0	5.8

Fig. 7. Photomicrograph at 100X magnification showing fine-grained outside (upper) surface vs. the overall grain size for a used witness specimen (recrystallized, grit blasted, air burn-off, and vacuum outgassed) and subsequently final heat treated at 1375°C/1 h.

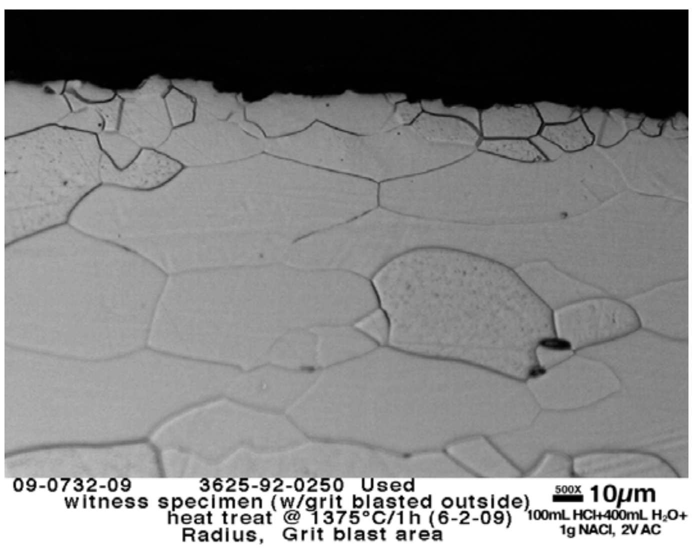

Fig. 8. Photomicrograph at 500X magnification showing fine-grained outside (upper) surface vs. the overall grain size for a used witness specimen (recrystallized, grit blasted, air burn-off, and vacuum outgassed) and subsequently final heat treated at 1375°C/1 h.

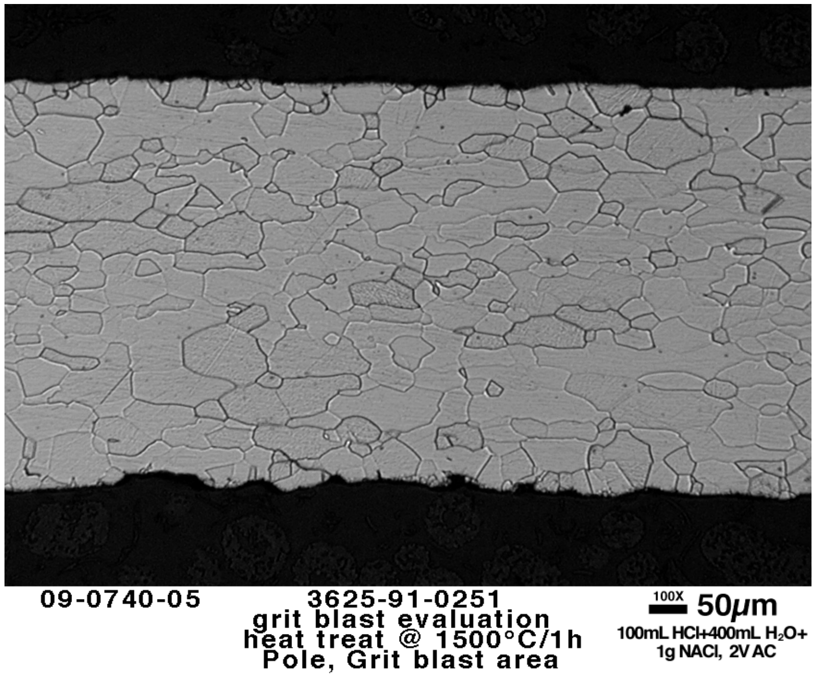

Fig. 9. Photomicrograph at 100X magnification showing fine-grained outside (upper) surface vs. the overall grain size for a used witness specimen (recrystallized, grit blasted, air burn-off, and vacuum outgassed) and subsequently final heat treated at 1500°C/1 h.

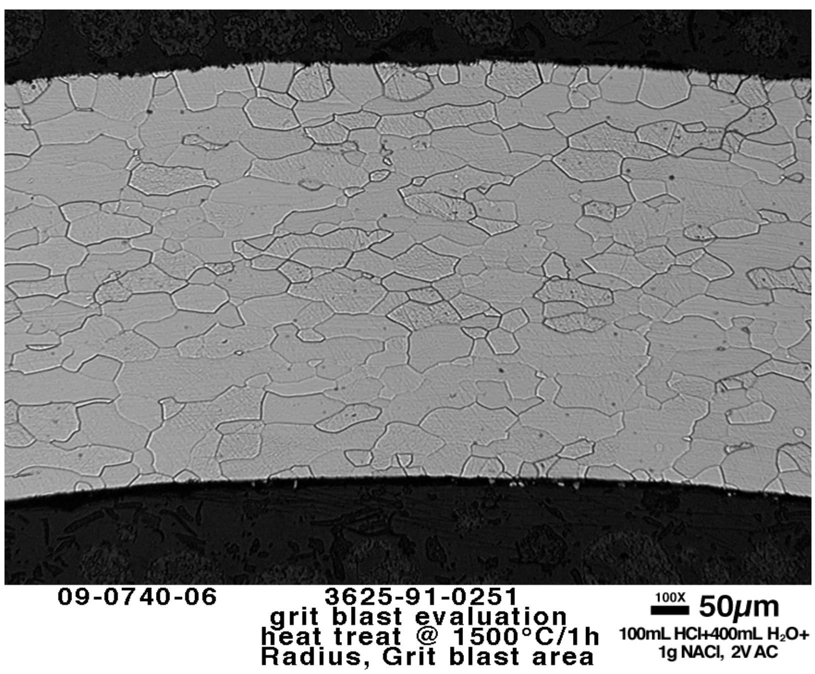

Fig. 10. Photomicrograph at 100X magnification showing a fairly uniform grain size on the outside (upper), inside (lower), and overall for a used witness specimen (recrystallized, grit blasted, air burn-off, and vacuum outgassed) and subsequently final heat treated at 1500°C/1 h.

Table 5. Metallurgical Summary for Overall (Bulk Portion of) Samples Given Subsequent Final Heat Treatments after the Recrystallization, Grit Blast, Air Burn-off, and Vacuum Outgas Operations

Final Heat Treatment	Overall ASTM Grain Size Number				Overall Microhardness HV 1000 gf		
	Pole	Radius	Equator	Grains Per Thickness	Pole	Radius	Equator
1375°C/1 h	7.0	6.5	6.0	25	223	241	231
1500°C/1 h	6.5	6.0	5.5	19	259	236	228
1500°C/70 h	4.0	4.0	4.0	10	219	216	214
1900°C/1 min	2.5	3.0	2.5	6	226	225	221
1900°C/2 h	1.0	0.0	1.0	2	219	239	230

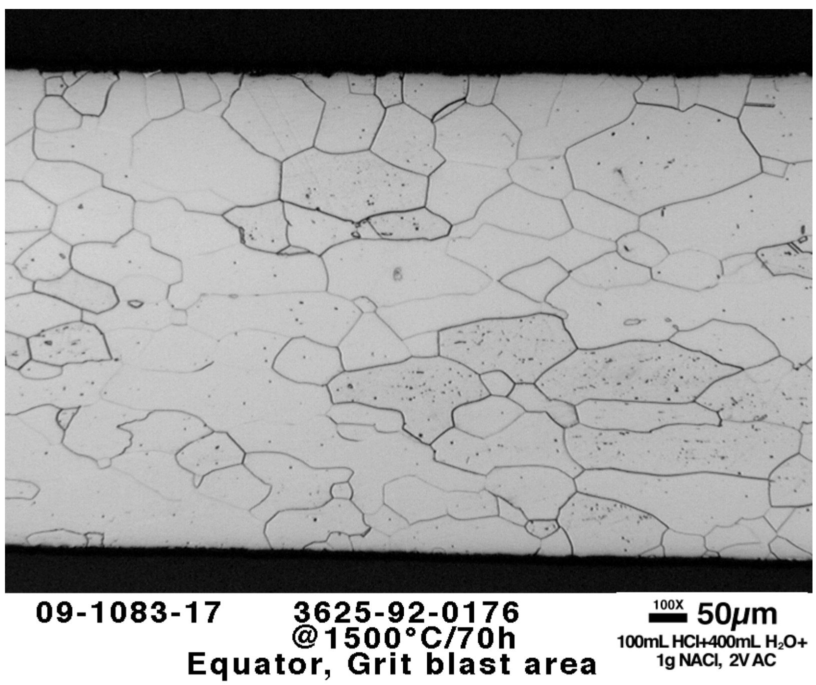

Fig. 11. Photomicrograph at 100X magnification showing a fairly uniform grain size on the outside (upper), inside (lower), and overall for a used witness specimen (recrystallized, grit blasted, air burn-off, and vacuum outgassed) and subsequently final heat treated at 1500°C/70 h.

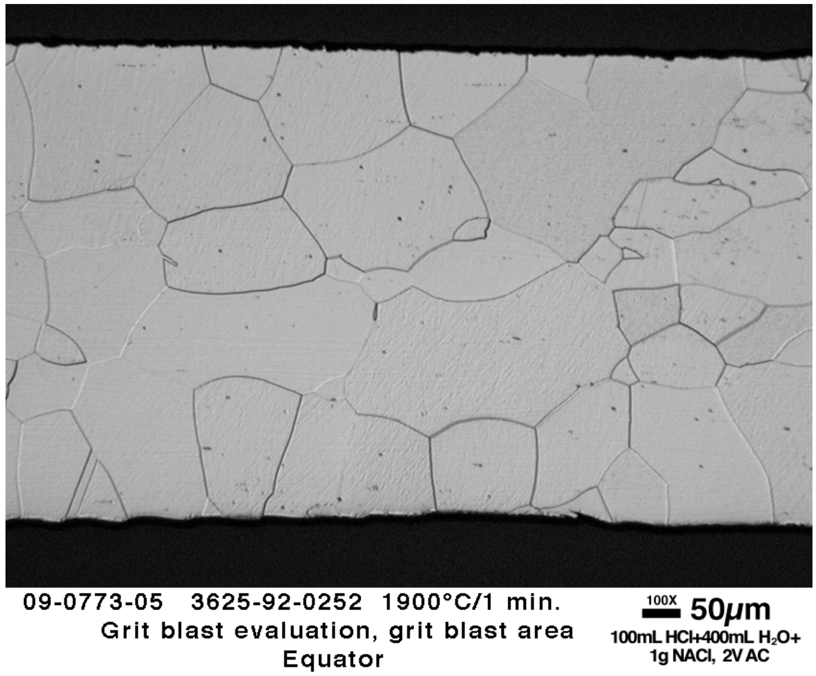

Fig. 12. Photomicrograph at 100X magnification showing a fairly uniform grain size on the outside (upper), inside (lower), and overall for a used witness specimen (recrystallized, grit blasted, air burn-off, and vacuum outgassed) and subsequently final heat treated at 1900°C/1 min.

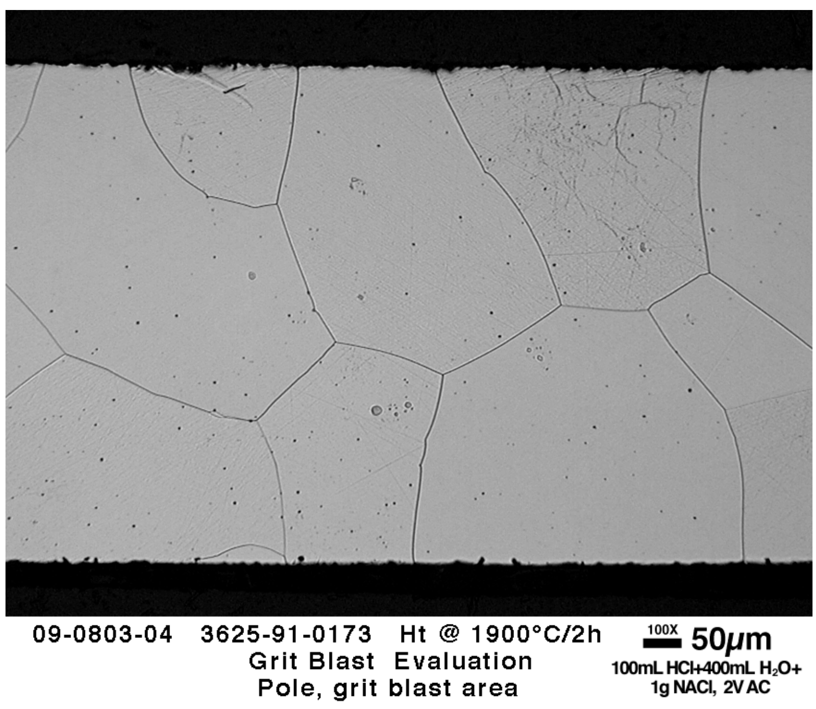

Fig. 13. Photomicrograph at 100X magnification showing a fairly uniform grain size on the outside (upper), inside (lower), and overall for a used witness specimen (recrystallized, grit blasted, air burn-off, and vacuum outgassed) and subsequently final heat treated at 1900°C/2 h.

3.4 ANOMALOUS CLAMPED (DURING DEBURRING) AREAS OF USED WITNESS SPECIMENS SUBSEQUENTLY GIVEN VARIOUS HEAT TREATMENTS

Anomalous areas consisting of locally coarsened grains were found in the polar regions of some used witness specimens subsequently heat treated as described in the previous section. D-test samples (from recrystallized and grit blasted cups) were clamped for deburring during sample preparation. These samples were deburred on their edges after they were removed via EDM from the cup. The deburring operation consisted of clamping each sample in the polar region with hemostat pliers so that a hand-held motorized rotary tool could be used to remove the EDM surface from the sample edges. The clamping force must be sufficient to keep the sample from moving during deburring, but if the force is excessive it will compressively deform and/or damage the sample. If this occurs in a CVS production certification sample, the deformed/damaged area can be excluded from metallurgical analysis. A typical reduced sample thickness region from clamping with hemostat pliers and the resultant surface deformation and damage is shown in Figs. 14 and 15 for a used witness specimen (recrystallized, grit blasted, clamped, air burn-off, and vacuum outgassed).

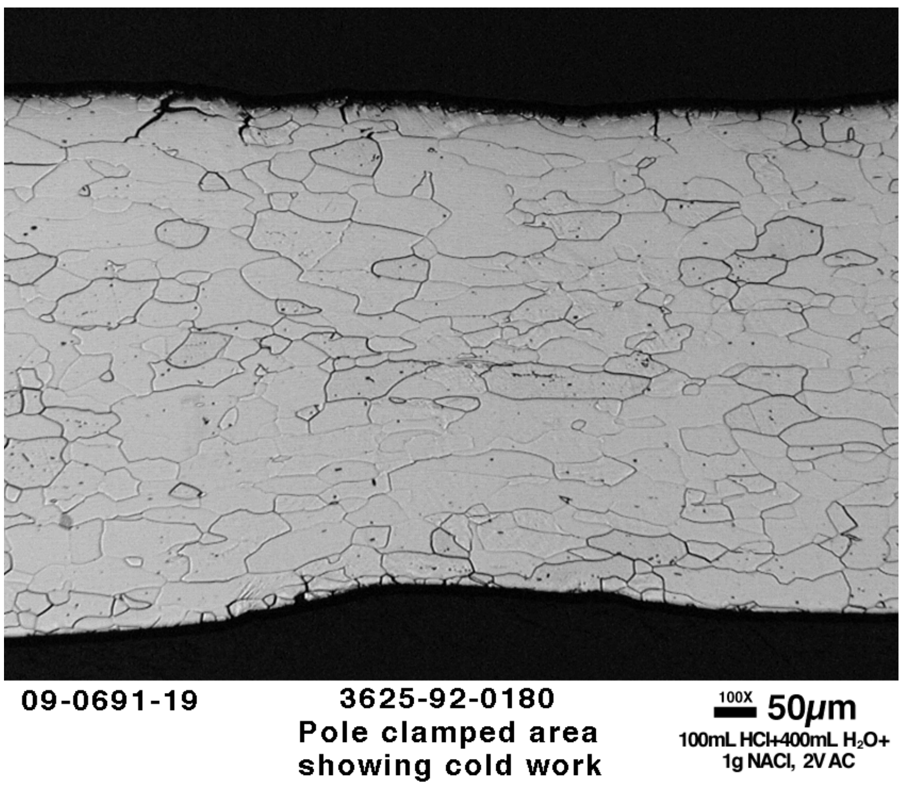

Fig. 14. Photomicrograph at 100X magnification showing a typical clamped area with deformation and some grain boundary separation on the outside (upper) and inside (lower) surfaces of a used witness specimen (recrystallized, grit blasted, clamped, air burn-off, and vacuum outgassed) – no grain growth.

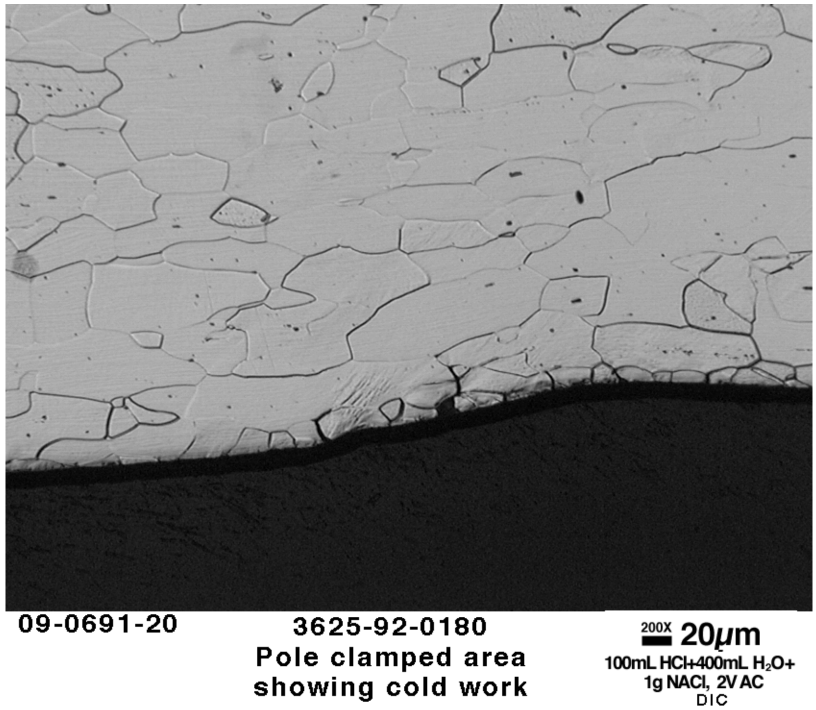

Fig. 15. Photomicrograph at 200X magnification of the clamped area on the inside (lower) surface of figure 14 clearly showing grain deformation and some grain boundary separation.

The compressive clamping forces for samples subsequently given a final heat treatment typically resulted in thickness reductions or strains of about 12% as shown in Table 6. Also the number of grains per thickness for the clamped and non-clamped areas is shown in Table 6. No grain growth, normal or abnormal, occurred anywhere in the sample given a subsequent final heat treatment at 1375°C/1 h as shown in Fig. 16. Abnormal grain growth was noted in the clamped areas of samples given subsequent final heat treatments at 1500°C/1 h, 1500°C/70 h, and 1900°C/2 h as shown in Figs. 17 – 19, respectively. This is reinforced in Table 6 this by showing the reduced number of grains per thickness in the clamped versus the non-clamped areas of these samples. The abnormal grain growth is readily discernible in Fig. 17. Very large grains are evident in Figs. 18 and 19 which should be compared to Figs. 11 and 13, respectively, to see the true extent of the abnormal grain growth. The sample heat treated at 1900°C/1 min was not clamped excessively during sample deburring; therefore, no abnormal grain growth was noted.

Table 6. Clamping Strain and Number of Grains Through the Thickness in Deformed Regions of Final Heat Treatment (after the Recrystallization, Grit Blast, Air Burn-off, and Vacuum Outgas Operations) Samples

Final Heat Treatment	Clamping Strain (% Reduction in Thickness)	Grains Per Thickness in Clamped Area	Grains Per Thickness in Non-Clamped Area
1375°C/1h	10	25	25
1500°C/1h	13	10	19
1500°C/70h	11	4	10
1900°C/1 min	None	--	6
1900°C/2 h	14	1	2

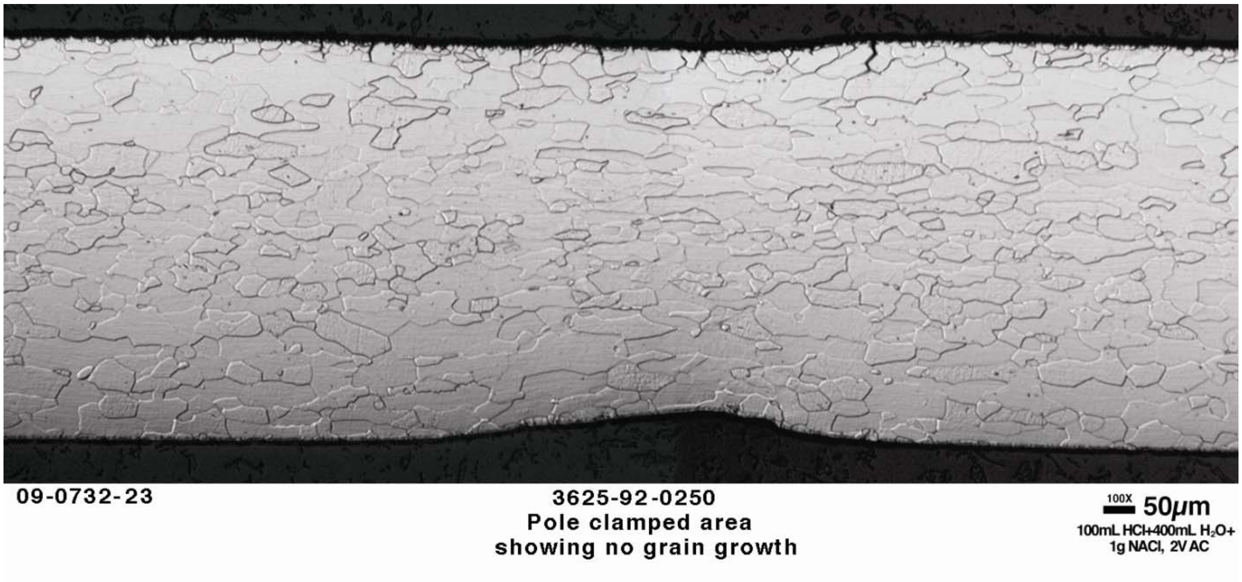

Fig. 16. Photomicrograph at 100X magnification showing the clamped area of the sample heat treated at 1375°C/1 h after recrystallization, grit blast, clamping, air burn-off, and vacuum outgas. Some grain boundary separation is evident on the outside (upper) surface, but no grain growth has occurred.

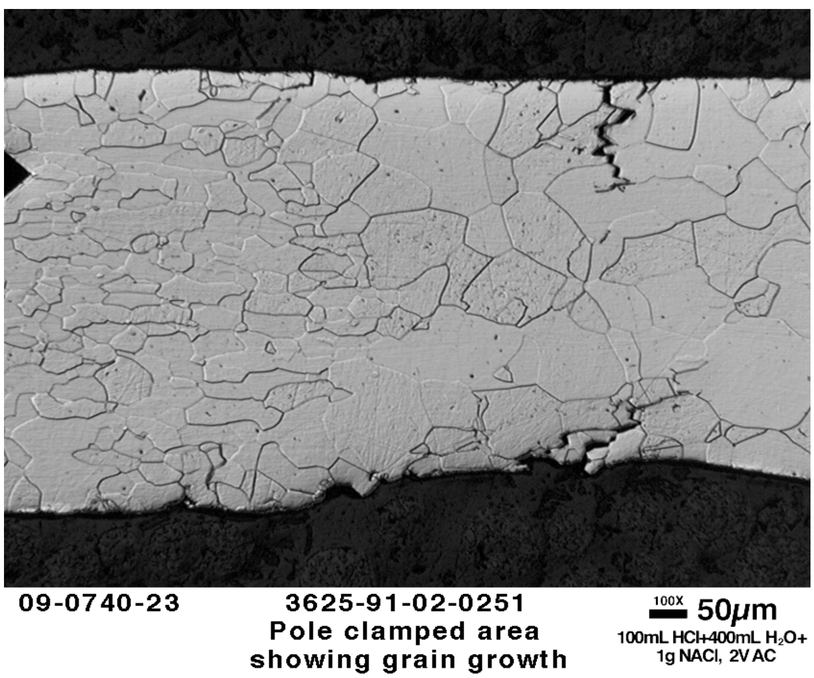

Fig. 17. Photomicrograph at 100X magnification showing the clamped area of the sample heat treated at 1500°C/1 h after recrystallization, grit blast, clamping, air burn-off, and vacuum outgas. Reduced thickness and grain boundary separation are evident as well as grain growth.

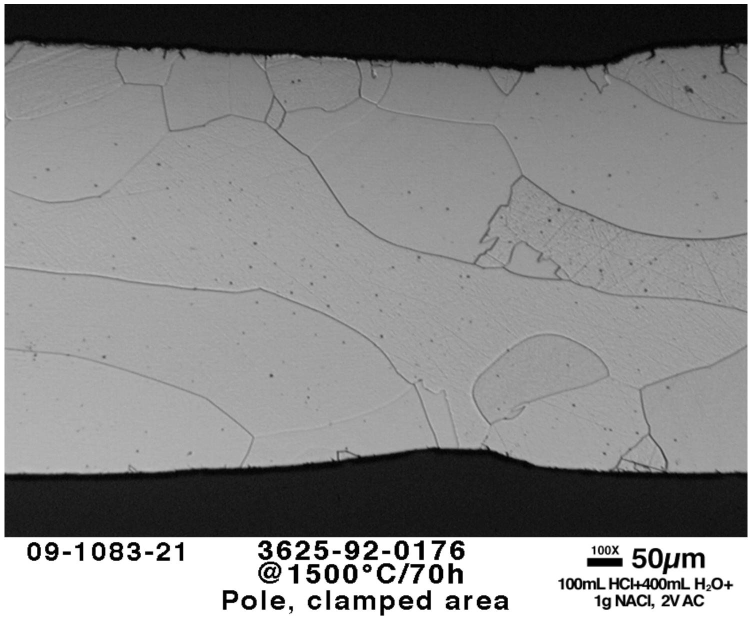

Fig. 18. Photomicrograph at 100X magnification showing the clamped area of the sample heat treated at 1500°C/70 h after recrystallization, grit blast, clamping, air burn-off, and vacuum outgas. Reduced thickness and some grain boundary separation are evident as well as enlarged grains. Compare to figure 11 for abnormal grain growth.

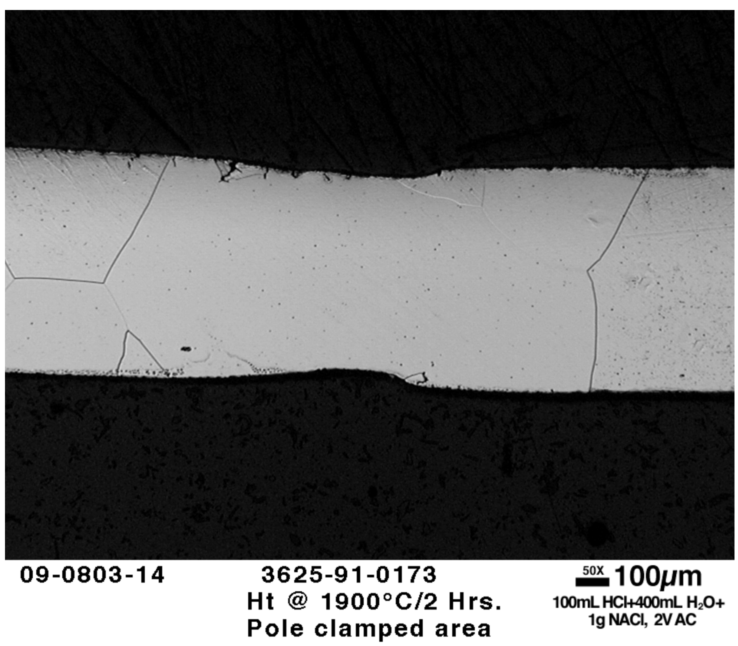

Fig. 19. Photomicrograph at 100X magnification showing the clamped area of the sample heat treated at 1900°C/2 h after recrystallization, grit blast, clamping, air burn-off, and vacuum outgas. Reduced thickness and some grain boundary (former) separation are evident as well as the single grain in this area. Compare to figure 13 for abnormal grain growth.

3.5 COMPARISON OF CVS CUP PRODUCTION METALLURGICAL RESULTS BEFORE AND AFTER GRIT BLASTING

Table 7 shows production destructive test results for iridium alloy cups tested for the years 1999 through 2009. This summary separates cups sampled and tested after (9 cups) and before (28 cups) the grit blasting operation. The results indicate that the overall (bulk – not surface) grain sizes and microhardnesses are similar whether the cups are sampled after or before grit blasting. The slightly larger ranges for grain sizes and microhardnesses of cups sampled before grit blasting are most likely related to the larger (3X) sample size.

Table 7. Metallurgical Summary for Iridium Alloy CVS Cup Production Destructive Test Results for the Years 1999 – 2009

	Overall ASTM Grain Size Number			Overall Microhardness (HV 1000 gf)		
Statistic	Pole	Radius	Equator	Pole	Radius	Equator
	Sampled Before Grit Blast Operation (9 cups)					
Av	6.3	6.3	6.2	237	258	243
D	0.5	0.4	0.4	9	12	11
RSD (%)	8	6	6	4	5	5
Max	5.5	5.5	5.5	265	275	264
Min	7.5	7.5	7.0	222	230	225
Range	2.0	2.0	1.5	43	45	39
	Sampled Before Grit Blast Operation (28 cups)					
v	6.4	6.5	6.3	240	250	246
SD	0.5	0.5	0.5	13	11	11
RSD (%)	8	8	8	5	4	4
Max	5.5	5.0	5.0	290	287	275
Min	7.5	8.0	8.0	220	229	224
Range	2.0	3.0	3.0	70	58	51

4. CONCLUSIONS

1. The CVS cup grit blasting operation imparts cold work on the outside cup surface that ranges from 11 to 36 μm deep with an average depth of 19 μm.

2. The 1250°C/1 h vacuum outgassing operation recrystallizes the grit blasted surface to a fine grain size of approximately 9 μm diameter (ASTM grain size number 11) and it serves as a stress relief anneal for the bulk structure.

3. The 1375°C/1 h recrystallization operation is a full anneal.

4. Abnormal grain growth does not occur in "standard-processed" CVS cup material with heat treatments up to 1900°C/2 h.

5. Abnormal grain growth does occur in areas with compressive strains of 10% after heat treatment as low as 1500°C/1 h.

6. CVS cup production grain size and microhardness results are similar whether sampling is after or before grit blasting.

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