Contract No:

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

Disclaimer:

SRNL-STI-2017-00416

Keywords: *Mechanical Properties, Type 304L Stainless Steel, Type 316L Stainless Steel, Type 21-6-9 Stainless Steel, Hydrogen Embrittlement, J-Integral, Helium Embrittlement, High-Energy-Rate Forging*

Retention: *Permanent*

2017 Status Report - Tritium Aging Studies on Stainless Steel: Effect of Hydrogen, Tritium and Decay Helium on the Fracture-Toughness Properties of Stem, Cup, and Block Forgings

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Publication Date: June 2017

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U. S. Department of Energy

Savannah River National Laboratory
Savannah River Nuclear Solutions, LLC
Aiken, SC 29808

Prepared for the U.S. Department of Energy under contract number DE-AC09-08SR22470.

Printed in the United States of America

Prepared for
U.S. Department of Energy

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I. SUMMARY

The materials of construction of tritium reservoirs are forged stainless steels. During service, the structural properties of the stainless steel change over time because of the diffusion of tritium into the reservoir wall and its radioactive decay to helium-3. This aging effect can cause cracks to initiate and grow which could result in a tritium leak or delayed failure of a tritium reservoir. Numerous factors affect the tendency for crack formation and propagation and are being investigated in this program. The goal of the research is to provide relevant fracture mechanics data that can be used by the design agencies in their assessments of tritium reservoir structural integrity. In this status report, new experimental results are presented on the effects of tritium and decay helium on the cracking properties of specimens taken from actual tritium reservoir forgings instead of the experimental forgings of past programs. The properties measured are more representative of actual reservoir properties because the microstructure of the specimens tested are more like that of the actual tritium reservoirs. The program was designed to measure the effects of material variables on tritium compatibility and includes two stainless steels (Type 304L and 316L stainless steel), multiple yield strengths (360-500 MPa), and multiple forging shapes (Stem, Cup, and Block).

Although the study is still underway, a number of conclusions can be drawn from the results to date. First, all forgings exhibited very high fracture toughness behavior. The Type 304L stainless steel block forging had the highest fracture-toughness values. For the low yield strength heat, fracture-toughness values averaged more than 2150 kJ/m^2. The high strength heat had fracture-toughness values averaging about 68% of the low-strength forging, i.e., 1500 kJ/m^2. Type 316L stainless steel stem forgings also have very high fracture toughness with values exceeding 1500 kJ/m^2 on average. The Type 316L stainless steel cup forging had the lowest fracture-toughness value, 54% of the block forging. However, the values still exceeded 1200 kJ/m^2. The reason for the reduced toughness of the cup forging is because of large strain variation required to form the cup shape of the forging and its higher overall yield strength.

Hydrogen precharging reduced the fracture toughness of all of the forgings but there were some key differences. In general, Type 316L stainless steel is more resistant to hydrogen effects than Type 304L stainless steel. Also, the lower yield strength forgings were more resistant than the higher strength ones. The stem forging was the most resistant to the hydrogen precharging effect with toughness values averaging 1116 kJ/m^2. The cup and block forgings had fracture-toughness values between 737-821 kJ/m^2 after hydrogen precharging. Overall, hydrogen reduced the fracture-toughness to values that were between 34-51% of the fracture-toughness value of the as-forged low yield strength block forging. Tritium-precharging caused an even larger drop in toughness because of the additional effect of decay helium.

Tritium-precharged steels were aged to build-in 600 appm (atomic parts per million) of decay helium which reduced the average fracture-toughness values of the three forgings to values that ranged between 12% and 23% of the as-forged low yield-strength block forging. The stem forging had the highest fracture toughness (~500 kJ/m^2) after tritium precharging and aging and the cup had the lowest value (265 kJ/m^2). Examinations of the fracture modes indicate that the hydrogen isotopes and helium cause a fracture mode change from microvoid nucleation and growth process to one that is characterized by quasi-cleavage and twin boundary fracture.

II. INTRODUCTION

Tritium reservoirs are constructed from forged stainless steels and filled and stored at the Savannah River Site. The vessels are constructed from forged stainless steels because of their good compatibility with tritium. These steels are highly resistant to, but not immune from, the embrittling effects of hydrogen isotopes and helium from tritium decay. Cracking in storage vessels has been observed after extended service times and material properties like ductility, elongation-to-failure, and fracture toughness are reduced with time as tritium and its radioactive decay product, He^3, slowly accumulate within the vessel walls during service (1-8). Because of these tritium aging effects, one of the primary interests of the Savannah River Site's Enhanced Surveillance Program task is to measure tritium effects on steel behavior and fracture-toughness values for use by the Design Agencies for fracture modeling, reservoir life prediction, and safety margin calculations (9 - 25).

New experimental research and development programs are underway and are described in recent reports (22-25). These programs are first-of-a-kind because they set out to measure tritium and decay helium effects on the cracking properties of stainless steels using actual tritium reservoir forgings instead of the experimental forgings of past programs. In this way, the properties measured will be more representative of actual reservoir properties because the microstructure of the specimens will be more like that of the forged reservoirs. The test matrices for the various programs are designed to measure the effects of specific forging variables on tritium compatibility and were described earlier (22). The programs include three heats of stainless steel, multiple yield strengths, four different forging processes, and four different reservoir forgings.

In this report, the results to date on the effect of tritium and decay helium on the fracture-toughness properties of stem, cup, and block forgings are reported. An earlier study reported on the fracture-toughness properties in the as-forged and hydrogen-precharged conditions (23). The results from that report were converted to SI units and included here for convenience and comparison between hydrogen and tritium effects. The stem and cup forgings were made from a single heat of Type 316L while the block forgings were made from two heats of Type 304L stainless steel. For type 316L forgings, the properties were measured for specimens cut in two different orientations from the stem and cup portions of the forging. Fracture-toughness properties were also measured for Type 304L block forgings having two different yield strengths. For both forgings, tritium and decay helium effects on toughness were measured by thermally precharging specimens with tritium and then aging to build-in helium from tritium decay.

III. EXPERIMENTAL PROCEDURE

Table I lists the compositions of the stainless steels used in this study. The Type 316L stainless steel was in the form of a cylindrical-cup forging. The Type 304L stainless steels were in the form of two cylindrical block forgings. Arc-shaped fracture-toughness specimens shown in Figure 1 were cut from the forgings. For the Type 316L forgings, specimens were cut from the cup portion in the CR-orientation and from the stem in the CL-orientation (Figure 2). Figure 1 depicts the location and orientation of the specimens cut from the forging. Also shown in Figure 2 is the specimen-identification scheme that was used to track the original location and orientation of each specimen from the forging. Figure 3 is a photograph of the as-cut forging and specimens. Similarly shaped specimens were cut from two Type 304L stainless steel cylindrical block forgings shown in by Figure 4. The Type 304L forgings were produced to have two different nominal yield strengths: 410 MPa (LY) and 480 MPa (HY). Figure 5 shows a photograph of the as-cut forgings and specimens. Finally, round-tensile specimens were machined from the stem, cup, and block forgings for verifying the as-forged mechanical properties (Table II) and are shown in Figures 6 and 7. Additional details of the test matrices for the stem, cup, and block forging studies including tritium-precharging schedules and experimental plans are given in the technology development plan (22). The results on the fracture-toughness measurements for the as-machined- and hydrogen-precharged specimens are included here for convenience but more details are given in Reference 23.

Table I - Compositions of Types 316L and 304L Stainless Steel Forgings (Weight %)

Material	MCN	Forging	Cr	Ni	Mn	P	Si	Co	Mo	C	S	N	O	Al
316L Stem & Cup	200948	7K0010	16.6	12.9	.71	.011	.51	.029	2.3	.009	.004	.036	.001	.003
304L Block LY	200952	11459	18.6	9.5	1.7	-	.57	.061	.098	.022	.001	-	-	-
304L Block HY	200952	11460	18.6	9.5	1.7	-	.57	.061	.098	.022	.001	-	-	-

Table II Room Temperature Mechanical Properties of Stem, Cup, and Block Forgings

Material	MCN	Forging / Direction	Yield Strength MPa	Ultimate Strength MPa	Elongation %
316L Stem	200948	7K0010 – Longitudinal	364	574	52.0
316L Stem	200948	7K0010 - Cylindrical	398	610	60.7
316L Cup	200948	7K0010 - Longitudinal	496	678	50.8
304L Block LY	200952	11459 - Longitudinal	413	616	67.6
304L Block LY	200952	11459 - Cylindrical	416	657	58.1
304L Block HY	200952	11460 - Longitudinal	465	647	56.8
304L Block HY	200952	11460 - Cylindrical	494	703	53.5

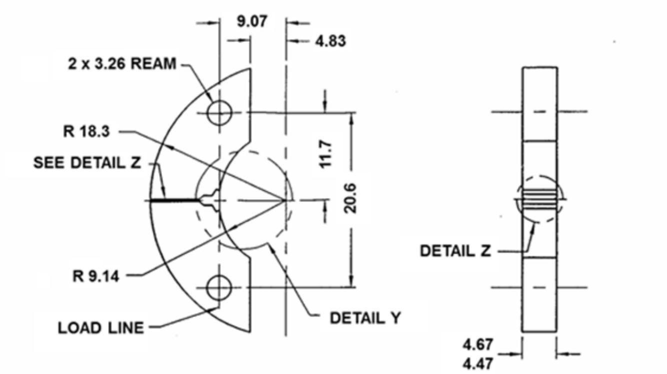

Figure 1. Shape and Dimensions of Fracture-Toughness Specimen in mm.

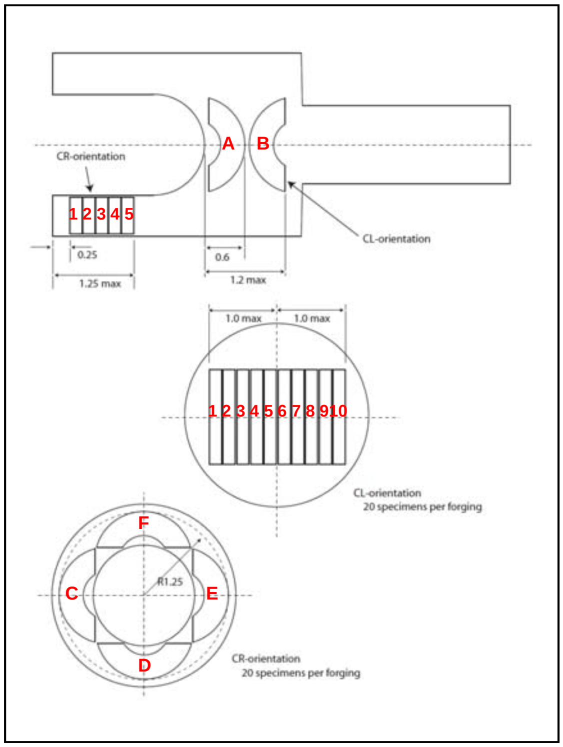

Figure 2. Fracture-Toughness Specimen Location and Orientation - Type 316L Forging: Specimens Labeled "A" or "B" were Cut from the Stem Portion of the Forging and Specimens Labeled "C", "D", "E", or "F" from the Cup Portion of the Forging.

Figure 3. Photograph of Type 316L Stainless Steel Cup Forging Showing As-Cut Specimens.

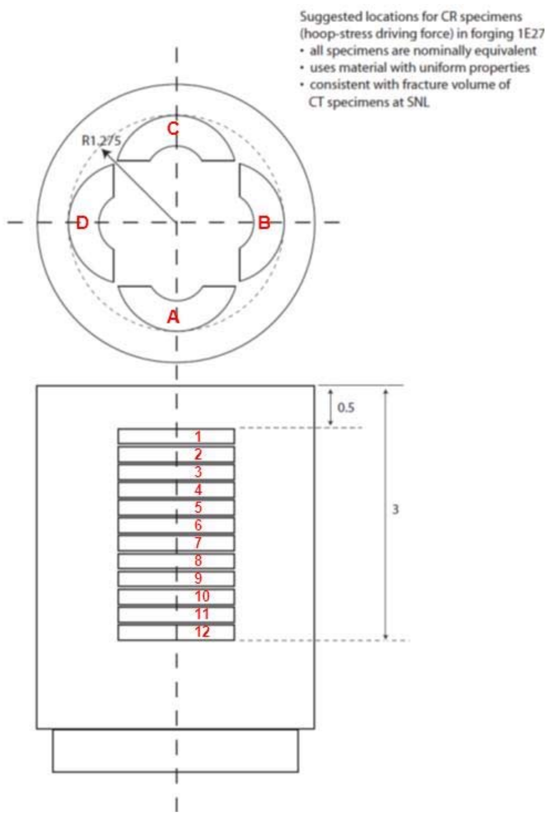

Figure 4. Fracture-Toughness Specimen Location and Orientation For Type 304L Cylindrical Block Forging.

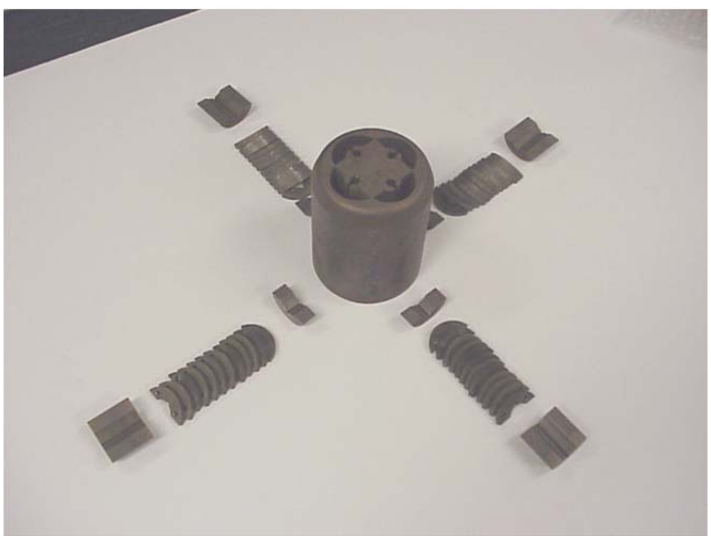

Figure 5. Type 304L Cylindrical Block Forging and As-Cut Fracture-Toughness Specimens.

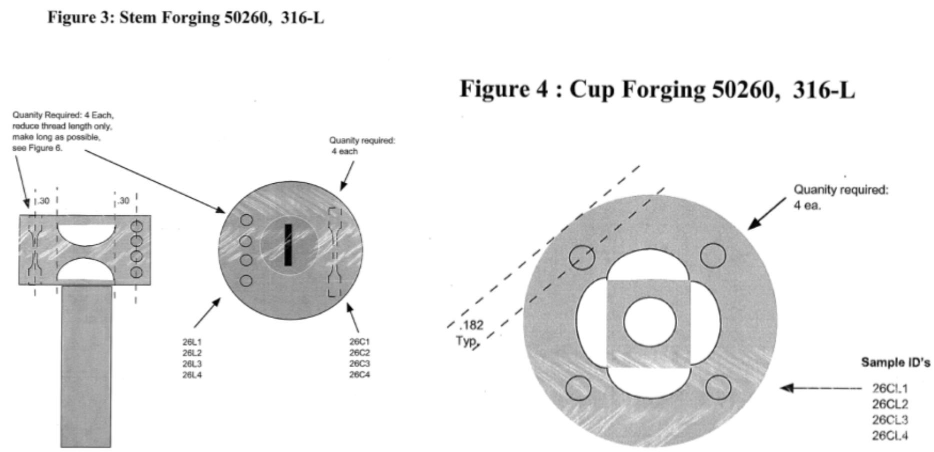

Figure 6. Orientation of Tensile Specimens Cut From Type 316L Stem and Cup Forgings.

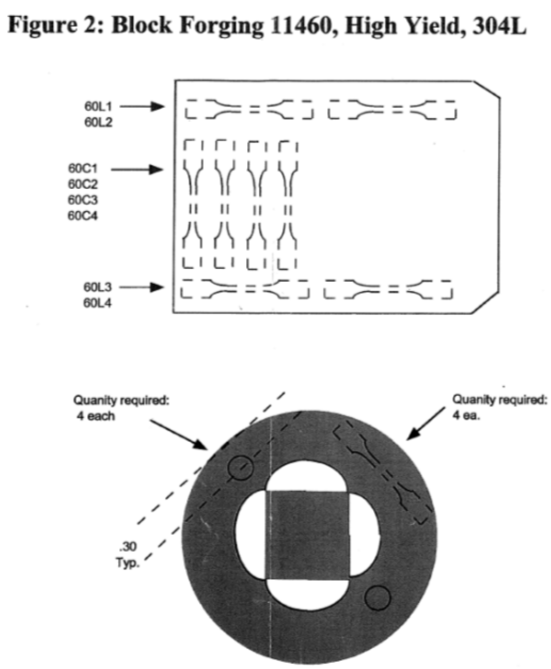

Figure 7. Orientation of Tensile Specimens Cut From Type 304L Block Forgings.

Some of the specimens cut from the Type 316L stainless steel forgings and the Type 304L block forgings were precharged with hydrogen or tritium gas at 623 K and an over-pressure of 34.5 MPa and then stored in air at 193 K. The storage temperature was chosen so as to minimize tritium off-gassing loss and to allow for the build-in of helium from tritium decay until testing is performed (this process sometimes takes years to accomplish). The hydrogen-isotope content of the precharged specimens is estimated by using established hydrogen solubility values to be 3700 atomic parts per million (appm) for Types 304L and 316L stainless steels (26). Decay helium content was developed during storage at -80°C for 45 months prior to testing. Tritium-charged specimens could not be rapidly quenched from the precharging temperature and so the tritium contents tend to be much lower than calculated. Instead, the specimen tritium and decay helium concentrations were estimated from the measured decay helium content of a high-energy-rate forged Type 304L specimen given a similar exposure. The tritium exposure conditions are estimated to be sufficient to uniformly saturate the test specimens throughout with a tritium content of approximately 1600 atomic parts per million (appm). Storage for 45 months before testing resulted in an estimated decay helium content of 600 appm uniformly distributed in the test specimen.

The elastic-plastic J-integral was evaluated for all specimens at ambient temperature by loading to failure at 0.002 mm/s while monitoring load, load-line displacement and crack extension (using a DC potential-drop technique). Two-to-three tests are planned for each condition and the data analyzed according to ASTM E1820 (27). Not all tests have been completed yet. Tritium-exposed specimens tested to date are reported here. For all test conditions, the requirements for the uncracked ligament and thickness were not satisfied; therefore, all fracture-toughness values are reported as unqualified J_Q values. While all fracture surfaces showed uniform crack fronts, only tritium-exposed specimens showed no evidence of shear lips along the sides of the specimens (implying plane-strain conditions prevailed for these specimens).

The J-Integral versus crack length increase (J-R) curves were constructed from the data using ASTM E1820 (27). Fracture-toughness values are determined by using the intercept of an offset line with the J-R curve as shown in Figure 8 which shows data on the effect of tritium from an earlier study (24, 25). The offset line has a slope that is proportional to the flow strength of the material. As the material yields before cracking the crack tip blunts and changes shape. In effect, the ASTM procedure is determining the point at which the crack begins to grow after blunting has occurred. This study included materials having a range of flow strengths with an overall average of 80 ksi. For the stem, cup, and block forgings, the best-fit slope for the blunting line was 2.5 x Flow Strength. These best-fit values were used for all specimens to determine fracture-toughness values to avoid later complications in the analysis because hydrogen, tritium, and decay helium all affect flow strength, and tensile specimens would not be available for each condition. The blunting lines are shown for the J-R Curve results to show the goodness of fit to the data. No attempt was made at this time to quantify the fracture-toughness differences as a function of blunting-line slope. In general, fracture-toughness values determined with steeper sloped blunting lines are lower and therefore, more conservative. In these high work-hardenable stainless steels, the J-R curve clearly deviates away from the lower sloped blunting lines as the material in front of the crack work hardens prior to crack extension. Because of this, the fracture-toughness properties reported here should be conservative.

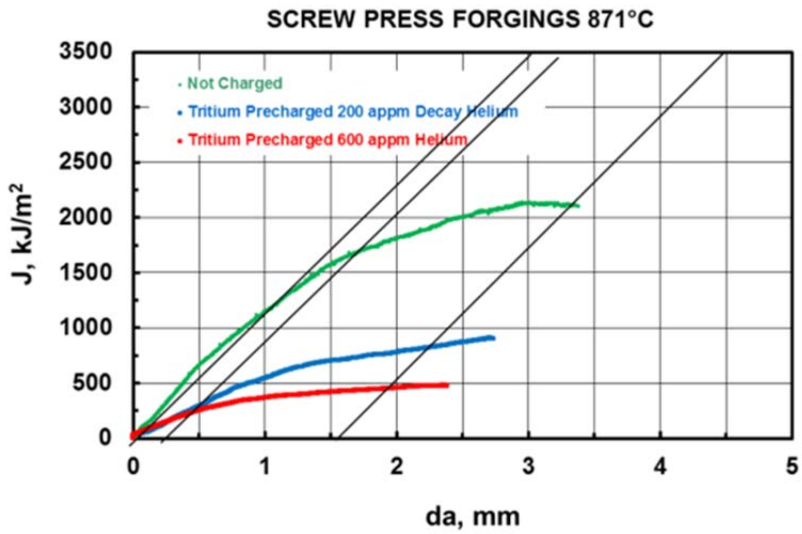

Figure 8. Typical J-R curves for As-received and Tritium-Precharged Specimens. J_Q Values Shown Were Determined from the Intercept of the J-R Curve with the 0.2 mm Offset Line (24).

IV. EXPERIMENTAL RESULTS

Table III lists the fracture-toughness values for the stem, cup, and block forgings. The table includes the SI values for not-charged and hydrogen-precharged values from an earlier report (23) and the values for the tritium-precharged specimens measured to date. The tritium-precharged matrix has not yet been completed and a more complete data table will be reported at a later date.

Table III – Fracture-Toughness Values of Stem, Cup, and Block Forgings

Specimen	Source	J_Q kJ/m^2	AVG kJ/m^2	StDev kJ/m^2	Fraction of Low-Yield Strength Block As-Forged Toughness
Specimens Not Charged					
26AL11	Stem	1804	1519	403	.69
26BL13	Stem	1234			
26RC6	Cup	1353	1205	164	.54
26RD8	Cup	975			
26RE10	Cup	1286			
26RF4	Cup	1207			
59RC1	Block LY	2223	2191	109	1.0
59RA1	Block LY	2069			
59RB6	Block LY	2280			
60RC11	Block HY	1846	1498	492	.68
60RB6	Block HY	1150			
Specimens Pre-Charged with Hydrogen Gas					
Specimen	**Source**	**J_Q kJ/m^2**	**AVG kJ/m^2**	**StDev kJ/m^2**	**Fraction of Low-Yield Strength Block As-Forged Toughness**
26AL2	Stem	1013	1116	169	.51
26AL8	Stem	1312			
26BL4	Stem	1024			
26RC4	Cup	796	821	24	.37
26RD1	Cup	823			
26RF2	Cup	843			
59RA9	Block LY	706	737	42	.34
59RB2	Block LY	720			
59RD4	Block LY	785			
60RA9	Block HY	735	760	36	.35
60RB2	Block HY	743			
60RD4	Block HY	802			
Specimens Precharged with Tritium					
Specimen	**Source**	**J_Q kJ/m^2**	**AVG kJ/m^2**	**StDev kJ/m^2**	**Fraction of Low-Yield Strength Block As-Forged Toughness**
26AL13	Stem	496			.23
26RD9	Cup	277	265	88	.12
26RE6*	Cup	347			
26RF8*	Cup	172			
59RB8	Block LY	337	374	80	.17
59RA5	Block LY	465			
59RC1	Block LY	320			
60RA5	Block HY	380	389	9	.18
60RC7	Block HY	398			
60RB8	Block HY	387			

*3.4 x 10^{-5} mm/s crosshead speed; all other specimens tested at 2.0 x 10^{-3} mm/s.

Type 304L Block Forgings

The Type 304L low-strength forging had the highest fracture-toughness properties with values averaging 2191±109 kJ/m^2. This value represents a convenient baseline to compare all of the materials and precharging conditions and Table III lists the fraction toughness values as a fraction of this value. Figure 9 shows J-R behavior for selected specimens from this forging for not-charged, hydrogen-precharged, and tritium-precharged conditions. The hydrogen-precharged specimens had lower fracture-toughness values and the J-R curves tended to flatten out earlier than the non-charged specimens. For tritium-precharged specimens the effect was similar but even more pronounced. Hydrogen precharging reduced the fracture-toughness value of the low-strength block forging to an average value of 737±42 kJ/m^2. Tritium precharging reduced the fracture-toughness value to 374±80 kJ/m^2. Thus, the hydrogen-precharged specimens had fracture-toughness values that were 0.34 of the baseline value; while, the tritium-precharged specimens had fracture-toughness values 0.17 of the baseline value.

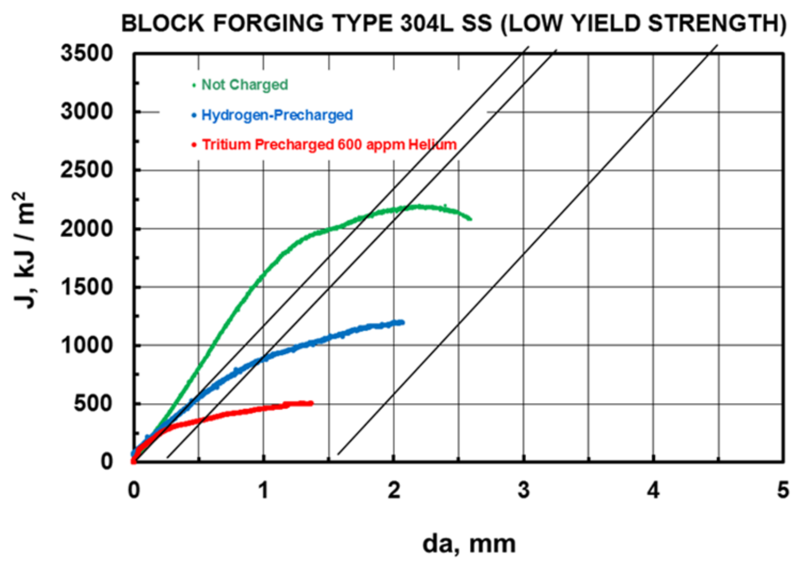

Figure 9. J-R Curves for the Low Strength Type 304L Block Forging Before and After Hydrogen and Tritium Precharging and Aging.

The large effect of hydrogen on fracture toughness is accompanied by a change in fracture mode. Figure 10 shows a comparison between the not-charged specimens and the hydrogen-charged specimen for the low yield strength block forging. Notice that the fracture mode of the as-forged specimens is characterized by microvoids that have nucleated and coalesced to cause failure, the typical fracture mode of stainless steels at ambient temperature. For hydrogen-precharged specimens, Figure 10 shows the fracture appearance tends to be flatter, with less evidence of microvoid nucleation and growth, and more evidence for quasi-cleavage and twin-boundary parting. There also tends to be more secondary cracking into the plane of fracture. The examination of fracture surfaces of tritium-exposed specimens have not yet been conducted.

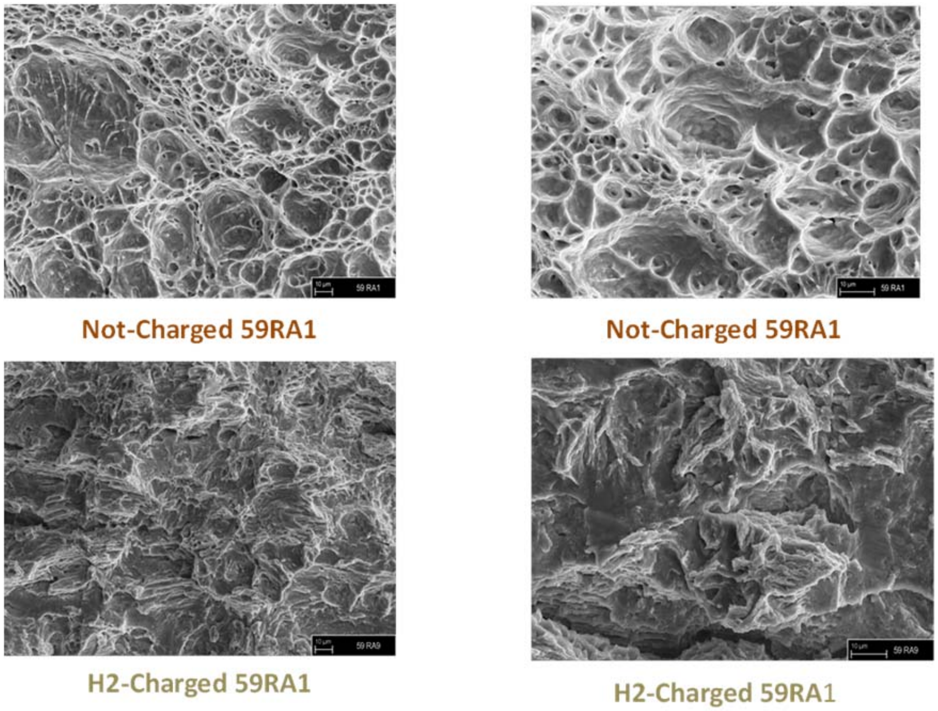

Figure 10. Fracture Appearance of Not-Charged and Hydrogen-Precharged Specimens Taken From the Low Yield Strength Type 304L Stainless Steel Block Forging.

Figure 11 shows the J-R behavior for selected specimens taken from the high strength forging. The high strength forging had fracture-toughness values on average of 1498±492 kJ/m^2 (Table III) which is about 0.68 of the low strength forging value. Hydrogen-precharged values averaged 760±36 kJ/m^2; while, tritium-precharged values averaged 380±9 kJ/m^2. These values are very similar to the hydrogen and tritium-precharged low strength forging values. Again, the J-R behavior exhibited a more flattened curve, particularly for the tritium-precharged specimens. Figure 12 shows the fracture appearance of the not-charge and hydrogen-precharged high strength forging and the overall effect of hydrogen is much like it was in the low strength specimens.

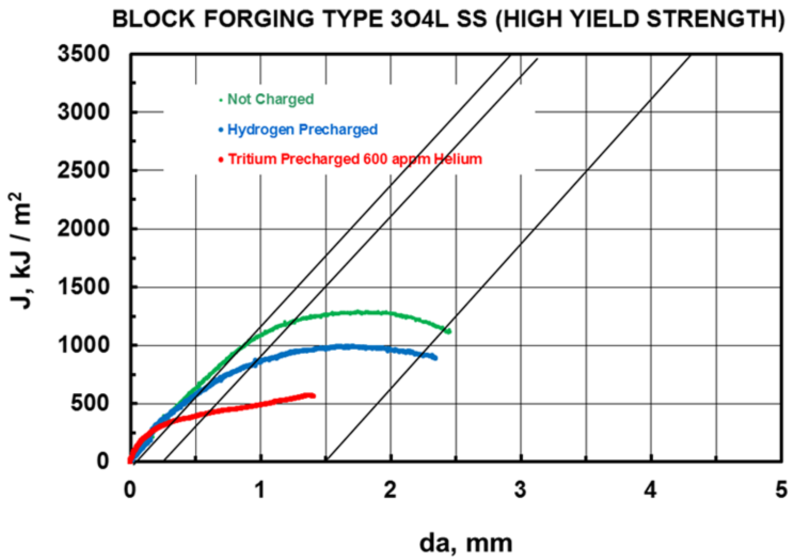

Figure 11. J-R Curves for the High Strength Type 304L Block Forging Before and After Hydrogen and Tritium Precharging and Aging.

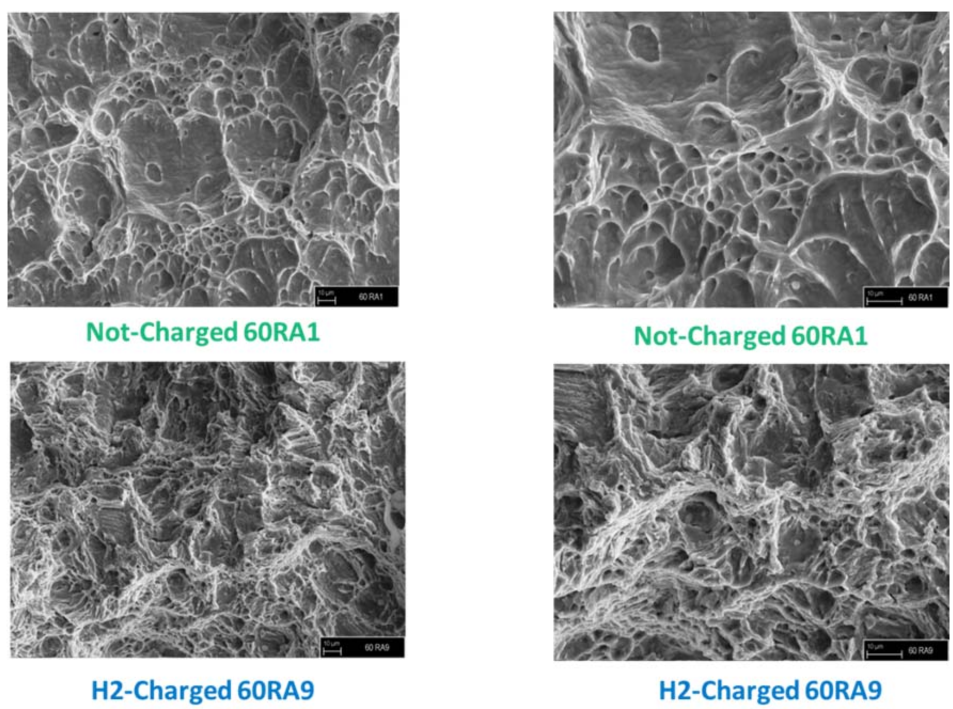

Figure 12. Fracture Appearance of Not-Charged and Hydrogen-Precharged Specimens Taken From the High Yield Strength Type 304L Stainless Steel Block Forging.

Type 316L Stem and Cup Forgings

Figure 13 shows the typical J-R curves calculated from the load-displacement-crack length records for selected non-charged, hydrogen-precharged, and tritium-precharged specimens taken from the Type 316L stem forging and Table III shows the average values. Note that only one tritium-precharged specimen has been tested so far out of the group of stem forgings. Not-charged specimen had high fracture-toughness values, 1519±403 kJ/m^2 which equates to 0.69 of the baseline value. Hydrogen precharging caused a reduction in fracture toughness of the stem forgings to 1116±169 kJ/m^2 or 0.51 of the baseline. The only tritium specimen tested had a fracture-toughness value of 496 kJ/m^2 or 0.23 of the baseline. The results to date indicate that the Type 316L stainless steel stem forging was the most resistant to hydrogen and tritium effects on toughness. The fracture modes of the not-charged and hydrogen charged stem forging is shown in Figure 14 and show a similar appearance as the block forgings.

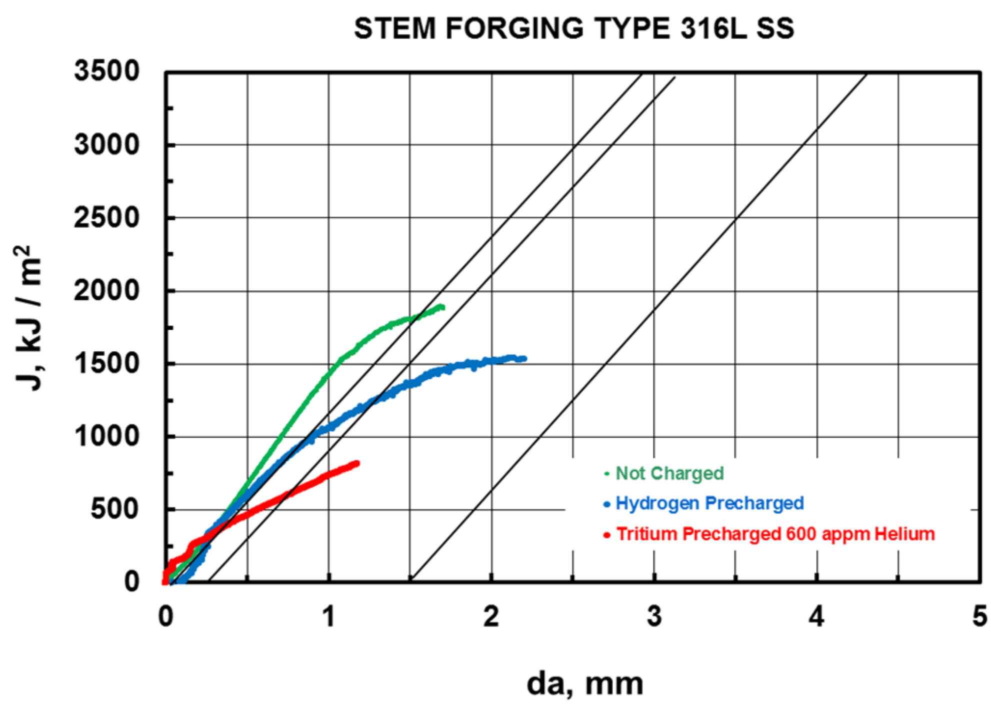

Figure 13. J-R Curves for Type 316L Stem Forging Before and After Hydrogen and Tritium Precharging and Aging.

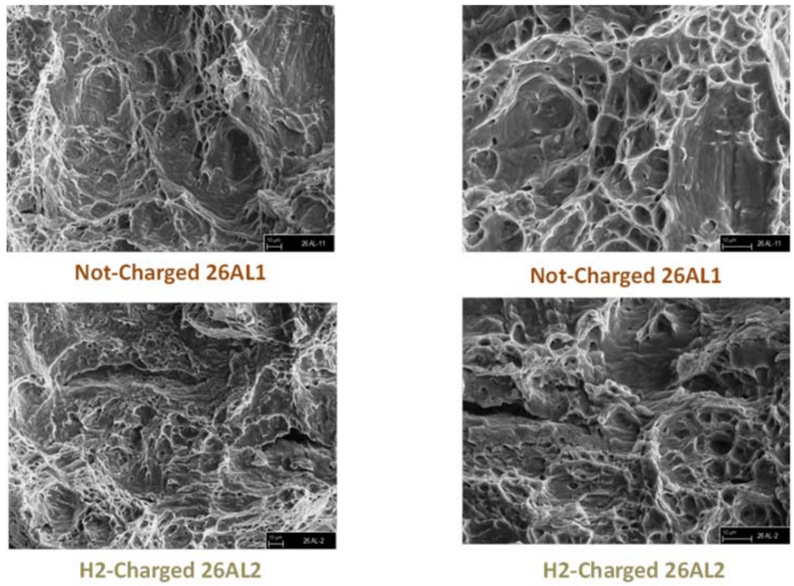

Figure 14. Fracture Appearance of Not-Charged and Hydrogen-Precharged Specimens Taken From the Type 316L Stainless Steel Stem Forging.

Figure 15 shows the J-R curves for specimens cut from the Type 316L stainless steel cup forging. Table III shows the average values and indicates that not-charged specimen had fracture-toughness values, 1205±164 kJ/m^2 which equates to 0.54 of the baseline value. Hydrogen precharging caused a reduction in fracture toughness of the stem forgings to 821±24 kJ/m^2 or 0.37 of the baseline. The tritium specimens tested had an average fracture-toughness value of 277 kJ/m^2 or 0.23 of the baseline. The as-forged cup forgings had fracture-toughness values that were lower than the fracture-toughness values for specimens taken from the stem section of the same forging (Table III). The most likely reason for the difference is that the cup portion is strained more than the stem or block during the forging operation. The fracture modes before and after hydrogen precharging are shown in Figure 16 and were similar in appearance to the stem forging.

The results to date are summarized in Figure 17. The bar chart shows the fracture-toughness values for the stem, cup and block forgings and includes the not-charged, hydrogen-precharged and tritium-precharged conditions. The figure shows the reduction in toughness values after hydrogen precharging and the further reduction that occurs from the tritium-precharging-and-aging.

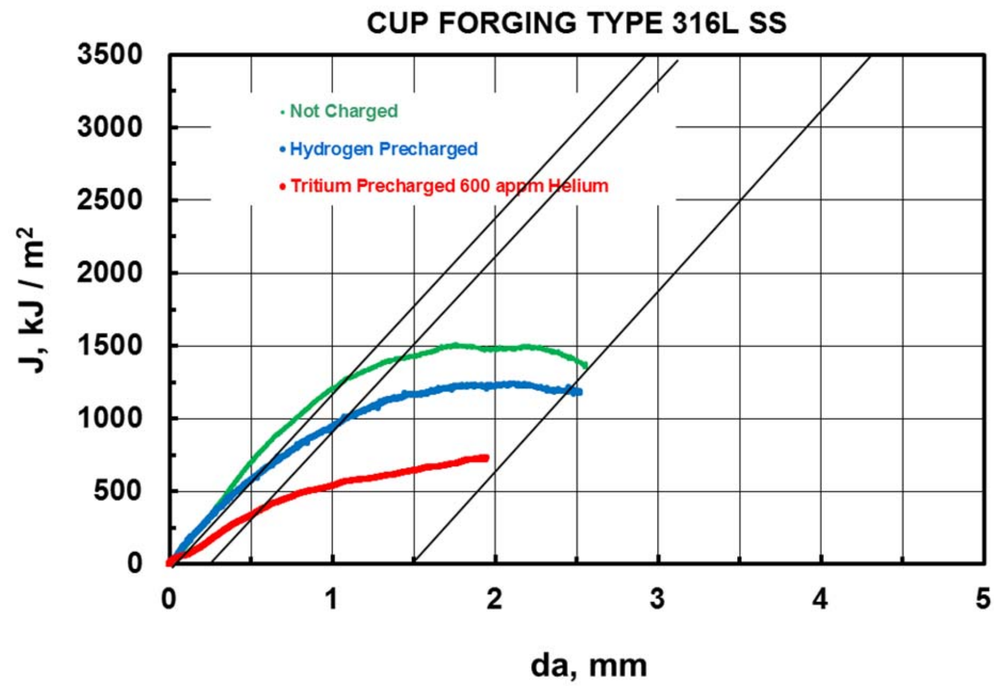

Figure 15. J-R Curves for Type 316L Cup Forging Before and After Hydrogen and Tritium Precharging and Aging.

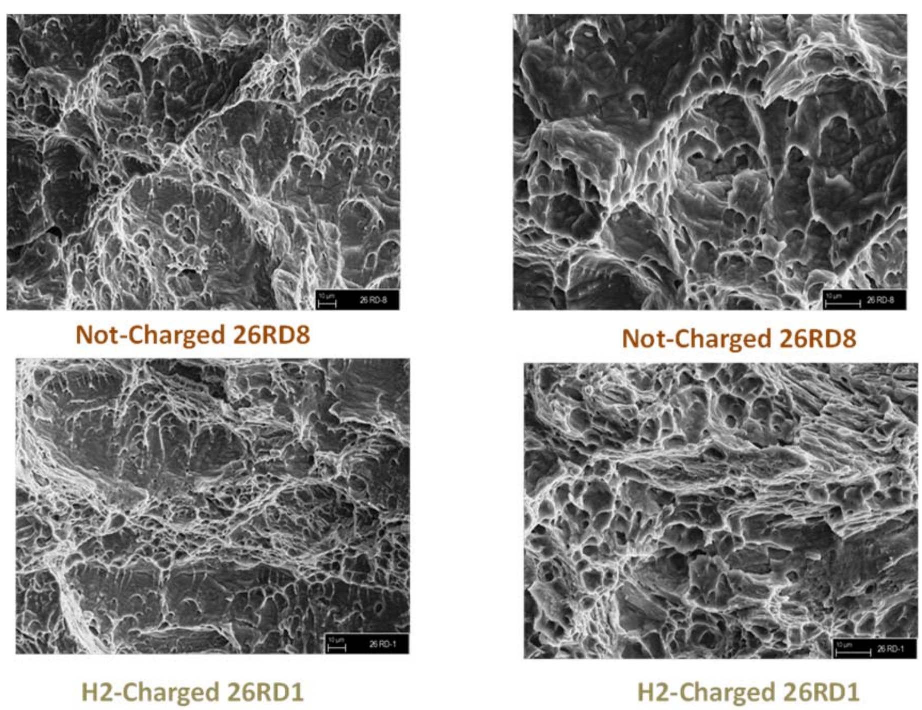

Figure 16. Fracture Appearance of Not-Charged and Hydrogen-Precharged Specimens Taken From the Type 316L Stainless Steel Cup Forging.

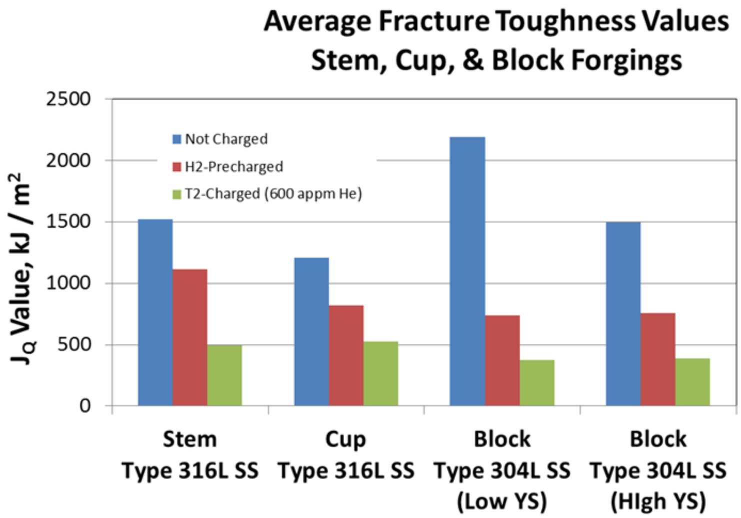

Figure 17. Average Fracture-Toughness Values for Stem, Cup, and Block Forgings

V. Summary and Conclusions

The effects of hydrogen and tritium on the fracture-toughness properties of Type 316L stem and cup forgings and Type 304L stainless steel block forgings were measured. The following are the main conclusions that were drawn from the work to date:

1. The fracture toughness of the stem cup and block forgings were very high and exceeded 1200 kJ/m^2 on average. The fracture toughness of specimens cut from the low yield strength Type 304L stainless steel block forging had the highest fracture toughness and the Type 316L stainless steel cup forging had the lowest fracture toughness.

2. Hydrogen precharging reduced the fracture toughness of the stem, cup, and block forgings to values between 34-51% of the baseline value measured in specimens cut form the low yield strength Type 304L stainless steel block forging. Type 316L stainless steel was more resistant to toughness reductions by hydrogen than Type 304L stainless steel. Hydrogen caused fracture mode changes from microvoid coalescence to quasi-cleavage and twin boundary fracture.

3. Tritium precharging reduced the fracture-toughness values more than hydrogen precharging because of the effects of helium from radioactive decay of tritium. The fracture-toughness properties of tritium-precharged forgings ranged from 12% to 23% of the baseline values.

VI. FUTURE WORK

The fracture-toughness measurements on tritium-precharged stem, cup, and block forgings will continue. The current matrix will be completed and reported in the future. Fractography of tritium-precharged specimens will also be conducted soon. Measurements will be conducted on specimens with higher helium during the out-years as helium builds in from tritium decay. Plans include fracture-toughness measurements on tritium-precharged specimens at slower and faster crosshead speeds. Also underway are tritium precharging runs on Types 304L and 21-6-9 stainless steel weldments and heat-affected-zone specimens. Initial tests are scheduled for October, 2017.

VII. ACKNOWLEDGEMENTS

The forgings used in this study were provided by Chris San Marchi of Sandia National Laboratory in Livermore California. Jim Wilderman assisted in conducting the mechanical tests of the tritium-exposed specimens. Henry Ajo conducted the scanning electron microscopy. Chad Sweeney, Calvin Clamp, Dante Pilgrim, and Ken Imrich made invaluable contributions in preparing tritium-precharging vessels and tritium-precharging procedures; conducting specimen recovery; and transporting specimens to SRNL after the tritium-precharging runs.

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