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2021 Report - SRNL Aging and Lifetimes program tritium aging studies on structural alloys

Timothy M. Krentz

Anastasia Mullins

January 2022

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PREFACE OR ACKNOWLEDGEMENTS

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EXECUTIVE SUMMARY

This report documents work performed in fiscal year 2021 at SRNL in support of the Aging and Lifetimes program. This work is part of an on-going collaboration between SRNL and SNL to understand tritium embrittlement of structural metals used in Gas Transfer System reservoirs, thereby informing lifetime assessments and predictions. In this effort, test coupons are pre-charged with tritium and allowed to age in a freezer to minimize tritium diffusion and off-gassing while allowing for born-in helium levels to systematically increase. Coupons are then removed periodically and tested to develop an understanding of how mechanical properties degrade as helium levels increase. This report summarizes test results from coupons which are in various stages of aging as they were tritium pre-charged in previous years.

Tests were performed on 304L that was previously tritium pre-charged and allowed to age to nominally 300 appm helium. Specimens from this study were previously tested at 100 appm nominal helium content, and this new test point expands that study. Tensile testing of these heat treated 304L tubes (and welded tubes) aged to 100 appm helium were completed which provided a unique study of a diverse set of microstructures and yield strengths ranging from 200-800 MPa. Additionally, 304L notched and smooth tensile specimens aged to 300 appm helium were tensile tested to predetermined strains to provide for a systematic microscopy investigation of aging effects on microstructural damage in partnership with SNL. Testing was performed on 304L electron beam (EB) weld coupons as well as 21-6-9 annealed coupons that were thermally pre-charged in 2020. Tests on the EB welds were completed at 100 appm helium contents, and tests on the annealed 21-6-9 material were completed at 100, 200, and 300 appm helium. Table 0-1 provides the status of the ongoing tritium coupon aging studies. The table includes the year tritium pre-charging took place, the material, and the targeted helium concentrations (appm) for testing.

Table 0-1. Status of Ongoing Tritium Coupon Aging Programs

Material	Tritium Pre-charged (year)	Mechanical Testing of Aged Specimens with Born-in Helium (appm)				
		100	200	300	600	1000
304L weld/HAZ	2017	DNM	NP	Complete	Complete	Planned
21-6-9 weld/HAZ*	2017	DNM	NP	Complete	Complete	Planned
304L tube, tensile	2019	Complete	NP	Complete	Planned	Planned
304L E-beam welds	2020	Complete	NP	Planned	Planned	Planned
21-6-9 Annealed	2020	Complete	Complete	Complete	Planned	Planned

DNM = did not measure

NP = not planned

Complete = new testing completed in 2021

*21-6-9 HAZ has higher tritium solubility than 304L and thus reaches relevant Helium concentrations in shorter aging times

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LIST OF ABBREVIATIONS

SRNL	Savannah River National Laboratory
SNL (/CA)	Sandia National Laboratories (California campus)
FZ	Fusion Zone (as of a weld)
HAZ	Heat Affected Zone (as of a weld)
appm	Atomic parts per million

1.0 Introduction

Tritium reservoirs, constructed from forged austenitic stainless steels, are filled at the Savannah River Site. These materials are selected for their good compatibility with hydrogen isotopes. Nonetheless, embrittlement occurs when hydrogen and its isotopes, including tritium, dissolve into the metal's atomic lattice, thereby changing deformation processes. Additionally, tritium decays to relatively insoluble helium, becoming trapped in the microstructure and creating nanoscale bubbles that progressively reduce fracture resistance as helium builds up over time. Cracks have been observed in storage vessels after extended service [1-5]. The consequences of a leak in service are high, and thus information on changing mechanical properties over time is critical for predicting safe service life. The focus of Savannah River National Laboratory's (SRNL) ongoing program on tritium impacts on structural materials is to gather data on the properties of stockpile relevant materials as tritium-decay-helium builds in order to inform lifetime assessment and prediction of tritium storage vessels.

Tritium embrittlement has been studied at SRNL through tensile and fracture testing of austenitic stainless steel base metal forgings, providing necessary inputs for design guidance [6, 7]. These historical studies progressed from examinations of representative materials, to actual stockpile forgings, and variations on forging techniques as well as joining technologies. These data, combined with testing at SNL/CA on hydrogen-pre-charged materials, have provided the basis of our current understanding of hydrogen-isotope embrittlement. SRNL's ongoing efforts with SNL/CA aim to expand our understanding of tritium embrittlement and thus support SNL in providing the experimental data necessary to develop quantitative models for describing tritium embrittlement and predicting component lifetimes.

Areas of current study include a systematic experiment on the impact of varying microstructures in 304L stainless steel tubing and its orbital welds on their sensitivity to tritium embrittlement. Testing on smooth and notched 304L tensile test specimens after tritium exposure and aging expands the available data on flow curves, plastic deformation, and triaxial stress states in this material. Starting in FY22 are studies on the mechanical response in tension and fracture testing of electron beam (EB) welded 304L stainless steel after tritium exposure and the tensile behavior of annealed 21-6-9 stainless steel at relatively early ages after tritium exposure. These new programs will provide data on the performance of EB welds, a prevalent welding technique used in all future GTS reservoir designs and help to explain large losses in fracture toughness observed in the fusion zones (FZ) and heat affected zones (HAZ) of 21-6-9 stainless steel welds. These ongoing studies build on the above-mentioned historical studies, and those studies progressed from representative material studies to realistic engineering studies on forgings and joining technology, these studies take a strategy of meeting the current data requirements necessitated by changing manufacturing technologies and progressing on to fundamental science which deepens understanding of the mechanics of materials after tritium exposure. Prospective future studies were laid out in [7].

2.0 Experimental Procedure

2.1 Type 304L Stainless Steel Tube tensile testing

Testing was conducted on commercially available 304L stainless steel tubing received in a strain-hardened condition. Samples were heat treated to vary the microstructure and strength of the tubing without changing the composition. Further details on the heat treatment, thermal pre-charging with tritium, and initial testing can be found in [8-11]. For reference, the microstructures of the as-received and heat treated materials are shown in Figure 2-1. Additional details of the preparation and testing of samples in the non-charged and hydrogen-pre-charged condition are described in [8, 9]. The test setup can be seen in the left panel of Figure 2-2.

**Figure 2-1 – Micrographs of 304L tube microstructures based on different heat treatments.
Reproduced with permission from SNL/CA through SAND2020-8528 PE [12].**

2.2 Type 304L Stainless Steel Tensile Specimens

Samples of the type 304L stainless steel in the smooth tensile and notched tensile specimen geometries were supplied by SNL/CA. Notched tensile specimens experience complex triaxial stress states in testing enabling the study of the process of void nucleation, while smooth tensile specimens inform the development of flow curves of tritium embrittled material for computational modeling. Interrupted tests were also completed to enable microstructural characterization to advance understanding of deformation processes in tritium-exposed structural materials.

Forged 304L tensile specimens (both smooth and notched) were tritium-pre-charged and aged to 300 appm helium followed by testing at SRNL. Details on the conditions of the tritium pre-charging, refer to SRNL-STI-2018-00036 [6] and details on the first round of testing at nominally 100 appm helium were reported in SRNL-STI-2021-00046. Typical test setups are shown in Figure 2-2. Tests were conducted to 5%, 10%, and 20% true strain as well as to failure. Notched tensile specimens were tested with a 12.7 mm gauge length, 0.0254 mm/minute displacement rate, to a drop from peak load of 9% and 12%, as well as to failure.

Figure 2-2. SRNL's radiological hood with tube specimen (left) and tensile test setup (right) used in testing samples pre-charged with tritium

2.3 Testing of 304L EB welds

The preparation and thermal pre-charging with tritium of EB weld 304L stainless steel specimens is described in SRNL-STI-2021-00046 [11].

2.3.1 304L smooth tensile EB weld specimens

Tensile tests on the 304L stainless steel EB weld specimens were conducted at room temperature in air using a screw-driven testing machine in displacement control while measuring load, crosshead displacement, and extension. Smooth tensile specimens were tested with a 12.7 mm gauge length and 1.27 mm/minute displacement rate. A drawing of the specimen geometry is shown on the left side of Figure 2-3. These tests were conducted 5 months after the initial tritium thermal pre-charge to allow the specimens to develop nominally 100 appm internal helium.

2.3.2 304L EB welded arc shaped fracture mechanics specimens

Fracture tests on the 304L stainless steel EB weld specimens were conducted at room temperature in air using a screw-driven testing machine in displacement control while measuring load, crosshead displacement, crack opening displacement via clip on gage, and electrical potential drop for determination of crack extension (see Figure 2-4 for an image of such a specimen under test). A drawing of the specimen geometry is shown on the right side of Figure 2-3. These tests were also conducted 5 months after the initial tritium thermal pre-charge to allow the specimens to develop nominally 100 appm internal helium.

Figure 2-3 – Drawings of threaded tensile specimens (left) and arc shaped fracture mechanics specimens (right)

Figure 2-4 – Arc shaped fracture mechanics specimen under test

2.4 Tensile testing of Annealed 21-6-9 material

The preparation and thermal pre-charging with tritium of these annealed 21-6-9 stainless steel specimens is described in SRNL-STI-2021-00046 [11]. Tensile tests on the annealed 21-6-9 specimens were conducted at room temperature in air using a screw-driven testing machine in displacement control while measuring load, crosshead displacement, and extension. Smooth tensile specimens were tested with a 12.7 mm gauge length, 1.27 mm/minute displacement rate. As above, tests were conducted to 5% and 20% true strain as well as to failure. A drawing of the specimen geometry is shown on the left side of Figure 2-3. Specimens were tested at time points corresponding to calculated values of 100, 200, and 300 appm helium.

3.0 Results and Discussion

3.1 304L tube tensile samples

The results in this section expand upon work presented and published in the proceedings of the 3rd Asia Pacific Symposium on Tritium Science as “Tritium embrittlement of austenitic stainless-steel tubing at low helium contents” [10]. Prior studies at Sandia National Laboratories on the effects of hydrogen [8, 9] demonstrated that internal hydrogen-induced losses in ductility could be correlated with differences in yield strength despite significant microstructural differences in compositionally identical material. Heat treatments were used to generate the microstructures shown in Figure 2-1 from as-manufactured tubing.

Additional tests have now been completed on longer aged specimens containing nominally 300 appm helium. The tensile response of each condition and age in the As-Received and 1116K heat treatment samples are shown in Figure 3-1.

Figure 3-1 Engineering Stress-Strain Curves for the As-Received and 1116 K Heat treatment conditions at all Hydrogen-Isotope exposure conditions and ages

Yield, ultimate tensile strength, and elongation values for all samples, as well as reduction of area measurements on non-charged and hydrogen-pre-charged samples, are tabulated in Appendix A. Reduction in outer diameter measurements are also given for the thermally tritium pre-charged and aged specimens. It should be noted that reduction of area measurements have not been completed on tritium exposed samples as taking optical images are challenging due to limitations handling the material. Therefore reduction in outer diameter were measured as a surrogate as this could be completed using a caliper immediately following completion of the test.

Figure 3-2 shows the change in yield strength compared to the noncharged condition for both hydrogen-pre-charged and tritium-aged samples at two different ages corresponding to nominally 100 and 300 appm helium. The yield and tensile strengths of the tritium-aged condition with only 100 appm helium are lower than the hydrogen pre-charged condition. However, the hydrogen pre-charging conditions results in a nominal hydrogen content of 7700 appm, while the tritium-aged condition retains 3600 appm of tritium and develops 100 appm helium. Data from the literature indicate an approximately linear relationship between strength (as well as ductility) and hydrogen isotope concentration [13]. Therefore, the higher yield strength in the hydrogen-precharged samples is expected due to the higher hydrogen-isotope content. As well, low levels of just a few hundred appm helium or less can have significant impacts on the fracture resistance of austenitic stainless steels similar to that seen in steels with significantly higher hydrogen isotope contents [14, 15]. The effects of this helium sourced hardening can be seen in the second test of tritium pre-charged and aged specimens with nominally 300 appm helium.

Figure 3-2 – Absolute changes in yield strength over the noncharged condition for thermally hydrogen pre-charged and tritium pre-charged tube samples. Reproduced in part from [10]

Figure 3-3 – Percent Elongation results from uncharged, hydrogen pre-charged, and tritium pre-charged specimens at two ages corresponding to 100 and 300 nominal appm helium

Shown in Figure 3-3 is the percent elongation for all 304L tube specimens tested to date, highlighting the increase in elongation seen with hydrogen charging and with tritium charging with 100 appm helium. In the 300 appm helium condition, the as-received specimens displayed increasing elongation to failure and

the 866K, 1000K, and 1033K conditions show minimal change. However, the fully recrystallized and fully annealed samples show a significant loss in elongation in the 300 appm helium condition.

These data reveal microstructural dependence on mechanical response after exposure to hydrogen isotopes and helium produced by tritium decay. Generally, heavily annealed specimens with less cold work (e.g. 1116K and 1311K conditions) display a greater sensitivity to the effects of hydrogen isotopes and helium compared to material with a greater degree of work hardening (e.g. as-received, 866K, 1000K, 1033K). Microstructural characterization on tensile specimens tested to failure and in interrupted tests in subsequent years will help in explaining this behavior.

3.1.1 304L Weld Tube Tensile Test Results

Figure 3-4 – Plots of Engineering Stress Strain

Several 304L tubes were welded and tested in the 100 and 300 appm helium conditions. The results are shown in Figure 3-4. Welds with 300 appm helium displayed lower strains to failure compared to 100 appm helium which is consistent with the trends observe on the fully annealed and fully recrystallized samples shown in Figure 3-3. Welds often have lower strength than their base metals due to the high amount of heat input during the welding process. Therefore, the mechanical properties are more likely to resemble annealed microstructures. In the as-received (AR) welded tube, the weld fusion zone has significantly lower strength compared to the base material. This localizes plastic deformation to the fusion zone, because the stresses never exceed the yield strength of the base material. In the case of the fully annealed weld sample, the weld and base material have similar properties, thus the strength and ductility properties reflect the behavior of the annealed tubing.

3.2 304L notched and smooth tensile samples

Results from the smooth tensile tests are shown in Table 3-1. Tests on the smooth tensile coupons were either to nominally failure or interrupted at 5, 10, 20% strain. The tests to failure are being used to help develop and calibrate plastic flow models at SNL/CA for various amounts of helium content. A microscopy

study will be performed on the interrupted smooth tensile coupons in order to understand how damage evolves with increasing amounts of helium in the microstructure. The notched tensile coupons were tested to either failure or interrupted immediately before failure. The notch provides elevated triaxial stress and will therefore affect the damage evolution compared to a smooth specimen. Increasing helium content increases both yield and ultimate tensile strength, as would be expected. Microscopy investigations are planned on both the failed and interrupted notched samples to understand how damage evolves in this modified stress state. Further testing of these sample groups is scheduled for August 2022 to investigate helium levels of 600 appm, with higher concentrations of helium in subsequent experiments.

Table 3-1 – Mechanical parameters measured in tensile testing of notched and smooth 304L stainless steel

Helium [appm]	UTS [MPa]	Yield [MPa]	Modulus [GPa]	Reduction in Area
100	635	466	196	66%
300	645	497	201	69%

3.3 Fracture testing of EB Welds

The J-integral versus crack length increase (J vs. da) curves are created from the rising displacement data described above as prescribed in ASTM E1820-20. These data represent a measure of the resistance to crack growth, or the energy associated with a certain amount of crack extension. J_Q values, the material fracture toughness, are taken as the point where the J vs. da curve crosses the 0.2 mm offset of the crack tip blunting line, constructed from the tensile properties taken from tritium charged and aged tensile coupons described below. Three replicates of each sample were tested, and the resulting crack growth resistance curves are shown together in Figure 3-5. The average calculated fracture resistance from this round of testing was 489 kJ/m².

Figure 3-5 – Fracture Resistance curves calculated for 3 tests with EB weld material thermally pre-charged with nominally 3700 appm tritium and aged to develop nominally 100 appm of helium

3.4 Tensile Testing of EB Welds

Engineering stress strain curves from the tensile testing of EB weld specimens thermally pre-charged with tritium and aged to 100 appm of helium are shown in Figure 3-6.

Figure 3-6 – Engineering stress strain curves from 3 tests with EB weld material thermally pre-charged with nominally 3700 appm tritium and aged to develop nominally 100 appm of helium

3.5 Tensile testing of Annealed 21-6-9 material

Previous reports (Annual aging studies report SRNL-STI-2018-00036 [7], and SRNL-STI-2020-00002 [16]) discussed the preparation and initial fracture testing results from tritium-pre-charged 304L/308L FZ and HAZ and 21-6-9/308L FZ and HAZ samples. Longer aging conditions were tested in 2020 in the same fashion as reported in those previous documents, at approximately 3 years of aging, leading to nominally 600 and 980 appm helium in the 304L and 21-6-9 samples respectively. Data show that the fracture resistance of 21-6-9 HAZ material decreased abruptly (Figure 3-7) with increasing helium concentration.

This series of tensile tests of annealed 21-6-9 material was pursued at several lower helium concentrations to attempt to investigate the very large drop in fracture resistance in the 21-6-9 weld HAZ material. Above testing in 304L tube orbital welds has indicated that fully annealed material can be used as a physical model for weld HAZ material. Figure 3-8 summarizes select parameters from these tensile tests. Yield strength significantly increases with helium content, while ultimate tensile strength increases modestly. Further microstructural investigations are ongoing to further examine these samples, and later tests are planned at ages sufficient to develop greater helium contents.

Figure 3-7 – Fracture resistance values for 304L and 21-6-9 weld FZ and HAZ. The 21-6-9 HAZ displays lower toughness with helium accumulation compared to the other tested samples

Figure 3-8 – Yield Strength, Ultimate Tensile Strength (left) and Percent Reduction in Area (right) after aging to 100, 200, and 300 appm helium in 21-6-9 stainless steel after pre-charging with tritium

4.0 Conclusions

- Tensile testing on 304L in tubes, notched tensile specimens, and smooth tensile specimens were completed following tritium pre-charging and aging to nominally 300 appm helium. Increasing helium content caused further hardening and in fully annealed microstructures, lower ductility. Additional samples are still aging and will be tested in out years to evaluate the effects as still higher helium content is built in.
- Three rounds of tensile tests (to failure and interrupted) were conducted this year on annealed 21-6-9 tensile specimens at 100, 200, and 300 appm helium. These specimens were charged in 2021 to examine 21-6-9 stainless steel to try and elucidate the mechanism of the pronounced degradation due to helium seen in 21-6-9/308L HAZ fracture testing. Results showed significantly increasing yield strength, and ultimate tensile strength that increased on average with helium content. The percent reduction in area in specimens pulled to failure was not monotonically varying with helium content. Further testing is planned with specimens of this material at higher helium contents in the future.
- Testing was conducted on 304L electron beam welded tensile and fracture specimens at 100 appm helium content. The average fracture resistance measured was 489 kJ/m². Further tests of these materials are planned in out years after more helium is built in.
- Future studies will include testing at higher helium content in ongoing sample sets, testing across the whole stockpile to target sequence (STS) temperature range, and investigations into fundamental plastic deformation mechanisms via single and oligocrystal specimens.

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Appendix A. Tabulated Results from Tensile Testing

Table 5-1 – Mechanical Properties of 304L Tubing

Includes hydrogen test data from SNL/CA for completeness, reproduced from [10]

Microstructure	Environmental condition (appm)	Yield Strength (MPa)	Tensile Strength (MPa)	Total Elongation (%)	Reduction of Area*
Strain-hardened (as-received)	Non-charged	697	857	23	0.71
	H-pre-charged	788	945	27	0.50
	T + 100 He	802	907	27	0.21
	T + 300 He	742	898	29	0.20
Partially recovered	Non-charged	678	854	27	0.68
	H-pre-charged	711	915	32	0.50
	T + 100 He	706	874	34	0.23
	T + 300 He	726	880	33.5	0.20
Fully recovered	Non-charged	576	780	36	0.64
	H-pre-charged	625	850	41	0.54
	T + 100 He	609	798	45	0.29
	T + 300 He	645	808	45	0.26
Partially recrystallized	Non-charged	377	692	47	0.70
	H-pre-charged	448	770	53	0.58
	T + 100 He	437	726	54	0.36
	T + 300 He	474	732	51.5	0.34
Fully recrystallized	Non-charged	228	614	63	0.76
	H-pre-charged	293	688	67	0.65
	T + 100 He	285	641	69	0.41
	T + 300 He	326	650	46.5	0.39
Annealed	Non-charged	179	565	64	0.80
	H-pre-charged	237	636	71	0.66
	T + 100 He	226	593	74	0.45

T + 300 He	255	587	44	0.40
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*Note: RA values for Tritium pre-charged specimens correspond to reduction in outer diameter measurements, which might not perfectly reflect reduction in area in a tube specimen geometry.

Table 5-2 – Mechanical Properties of Welded 304L Tubing
Includes hydrogen test data from SNL/CA for completeness, reproduced from [10]

Microstructure	Environmental condition (appm)	Yield Strength (MPa)	Tensile Strength (MPa)	Total Elongation (%)	Reduction of Area*
As-received tubing welded	Non-charged	248	566	15	0.82
	H-pre-charged	287	631	15	0.62
	T + 100 He	287	600	15	0.28
	T + 300 He	314	606	13.5	0.26
fully recovered tubing welded	Non-charged	256	577	16	0.82
	H-pre-charged	296	651	15	0.67
	T + 100 He	281	594	15	0.29
	T + 300 He	311	608	14.5	0.31
Fully annealed tubing welded	Non-charged	178	561	59	0.78
	H-pre-charged	239	630	67	0.65
	T + 100 He	239	601	68	0.43
	T + 300 He	586	260	44.5	0.40

Table 5-3 – Mechanical Properties of Electron Beam Welded 304L Stainless Steel

Environmental condition (appm)	Yield Strength (MPa)	Tensile Strength (MPa)	Total Elongation (%)	Reduction of Area
T + 100 He	440	651	27	0.57

Table 5-4 – Mechanical Properties of Annealed 21-6-9 Stainless Steel

Environmental condition (appm)	Test Termination	Yield Strength (MPa)	Tensile Strength (MPa)	Total Elongation (%)	Reduction of Area
T + 100 He	5% Strain	498	645	5	0.05
	20% Strain	492	750	19	0.16

	To Failure	491	791	55	0.50
	5% Strain	511	651	5	n/m
T + 200 He	20% Strain	513	771	22	n/m
	To Failure	516	800	54	0.52
	5% Strain	533	666	5	0.04
T + 300 He	20% Strain	538	783	22	0.17
	To Failure	534	807	55	0.52

Appendix B. Compiled Electron Beam Welded 304L Stainless Steel Fracture Test Curves