Comprehensive Characterization of Helium-induced Degradation of the Friction Stir Weld on Neutron Irradiated 304L Stainless Steel

W. Tang
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September 2023

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# CONTENTS

LIST OF FIGURES .................................................................................................................. v
LIST OF TABLES ....................................................................................................................... vii
ABBREVIATED TERMS .......................................................................................................... viii
EXECUTIVE SUMMARY ......................................................................................................... x

1. INTRODUCTION .............................................................................................................. 1
   1.1 Accumulation of helium in the in-core materials and its impact on welding ...................... 1
   1.2 Friction stir welding as a promising solution ..................................................................... 2
   1.3 Recent achievements on irradiated material FSW ............................................................. 3

2. MATERIALS, EXPERIMENTAL METHODS, AND MAJOR RESULTS OF PREVIOUS
   CHARACTERIZATION .......................................................................................................... 3
   2.1 Custom 304L stainless steel composition, fabricating, and processing .............................. 3
   2.2 Major results from previous SEM microstructure characterization of the 304D-5-14
       specimen ............................................................................................................................ 5
   2.3 Microhardness testing ....................................................................................................... 10
   2.4 DIC tensile testing ............................................................................................................ 11
   2.5 Fractography analysis ...................................................................................................... 13

3. RESULTS AND DISCUSSION .......................................................................................... 13
   3.1 Microhardness distributions ............................................................................................. 13
   3.2 Tensile properties and local plastic deformation .............................................................. 15
   3.3 Fractography analysis ...................................................................................................... 22
       3.3.1 Analysis of the specimen fracture area ....................................................................... 22
       3.3.2 Helium-related features ............................................................................................ 24
       3.3.3 Cracking and fracture events at the gauges portion of the tested specimens ............... 25

4. CONCLUSIONS AND FUTURE WORK ........................................................................... 28

5. ACKNOWLEDGMENTS ..................................................................................................... 29

REFERENCES ......................................................................................................................... 29
LIST OF FIGURES

Figure 1. Helium-induced cracks in the weld HAZ of stainless steel containing 8.3 appm He [2]. Note many cracks exceed 1 mm in length and the total length of macroscopic cracks is well above ~5-6 mm. ......................................................... 2

Figure 2. Reference microstructure for 304D custom heat (24-appm B-enriched, measured ~15.6 appm He in another coupon with the same heat): EBSD Inverse Pole Figure (IPF), Image Quality (IQ), Phase, and Kernel Average Misorientation (KAM) maps. One may see annealed austenite structure with a minor amount of ferrite. Many ferrite grains have a specific elongated shape with a long axis oriented in the horizontal direction. The IQ map shows the reduced pattern quality around many ferritic grains (no such effect was observed in 304C heat); reduced Kikuchi pattern quality may suggest some element segregation effects. The KAM map reveals no plastic strain (fully annealed conditions). Amount of ferrite for this scan <0.2-0.3%. Scan size: 300× 250 µm; EBSD step (pitch) size: 0.5 µm. ................................................................................. 4

Figure 3. Cross section of the friction stir weld performed on the 304L SS containing ~10 appm helium. No macroscopic cracks or crack-like defects (e.g., ~0.5 mm or so) were observed in the SZ or TMAZ. Note the SEM image has some minor distortion due to the low magnification level. Left side: FSW advancing side; right side: FSW retreating side [17]. .............. 6

Figure 4. Schematic of the SZ, TMAZ, HAZ, and GM in an irradiated 304L SS friction stir weld. .............. 6

Figure 5. Grain structures in different metallurgical zones of the 304D-5 friction stir weld. The scale bar for grain structure transitions SEM/EBSD image (Top left) is 1 mm and that for the rest SEM/EBSD images is 100 µm [17]. ............................................................................. 6

Figure 6. SEM images along the 304D-5-14 SZ centerline [17]. .......................................................... 7

Figure 7. Individual helium bubbles, helium bubble chains, and micro-cracks in the 304D-5-14 TMAZ [17] .................................................................................................................. 8

Figure 8. Clustering of the helium-induced damage in TMAZ. Dashed ovals at right show the clusters. (Left) BSE image; (right) SE [17]. ................................................................. 9

Figure 9. SEM pictures of the 304L SS BMs contains different amount of helium [17].......................... 10

Figure 10. Vickers microhardness measurement lines ........................................................................... 11

Figure 11. Miniature tensile specimen drawing (unit: mm) .................................................................. 11

Figure 12. Miniature tensile specimen mapping on the 304D-5-14 friction stir weld specimen .......... 12

Figure 13. The wire EDM system installed in LAMDA to manufacture specimens from radioactive material .................................................................................................................. 12

Figure 14. Miniature tensile specimen extracted from the 304D-5-14 friction stir weld metallographic specimen. ............................................................................................................. 13

Figure 15. 304D-5-14 friction stir weld specimen horizontal Vickers microhardness distributions. ....... 14

Figure 16. 304D-5-14 friction stir weld specimen vertical Vickers microhardness distributions. ......... 15

Figure 17. 304D-5-14 MS images before testing (top), at UTS (middle), and before failure (bottom). ............................................................................................................................ 16

Figure 18. 304D-5-14 MS images with digital extensometer before testing (top), at UTS (middle), and before specimen failure (bottom). ........................................................................ 17

Figure 19. 304D-5-14 tensile curves of all friction stir weld miniature tensile specimens. ..................... 18

Figure 20. Local strain εx distribution of 304D-5-14 MS specimen at different tensile stages. .......... 19

Figure 21. Local strain εx distribution of 304D-5-14 BR specimen at different tensile stages .......... 20

Figure 22. Local strain εx distribution of 304D-5-14 TA specimen at different tensile stages .......... 21

Figure 23. Local strain εx distribution of 304D-5-14 MR specimen at different tensile stages .......... 22

Figure 24. Low-magnification (800×) SEM images of the fracture regions of the tested tensile specimens. Material condition IDs and image order (e.g., “TA”) correspond to Figure 12
and Table 2. One may see dominating ductile fracture with minor features (black arrows) likely related to boron-rich regions.

Figure 25. High-magnification (nominal magnification 10,000×) SEM images of typical fracture surface appearance. For material condition IDs see Figure 12 and Table 2.

Figure 26. Fracture surface features showing internal cracks, large pores, and cleavage-like spots. “MS”-condition image (i) represents typical the ductile fracture for comparison. Note magnification varies for some images; “TR”-condition is shown twice.

Figure 27. Gauge portions of the tensile specimens after mechanical testing and fracture. Black arrows mark local fracture events (mostly along the gauge edges); the “EDM”-label marks the separation and peeling-off of the EDM-induced layer. Note pronounced delayering at the surface of the TR specimen (c). For material condition IDs see Figure 12 and Table 2, pre-specimen geometry is shown in Figure 11.

Figure 28. Examples of localized fracture at the specimen gauges: a) The MA specimen, fracture at the specimen edge; b) The MR specimen, fracture at the specimen surface, also note a thin wire-like crack. Tensile direction is vertical in this Figure.

Figure 29. Delayering and multiple cracks at the surface of the TR specimen. This phenomenon is difficult to fully explain now.
LIST OF TABLES

Table 1. Chemical composition of the 304L SS, wt.% [17] ................................................................. 4
Table 2. DIC tensile specimen IDs, locations, and gauge area metallurgical zones .......................... 12
Table 3. 304D-5-14 friction stir weld specimen tensile properties .................................................. 18
<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Full Form</th>
</tr>
</thead>
<tbody>
<tr>
<td>304L SS</td>
<td>304L stainless steel</td>
</tr>
<tr>
<td>appm</td>
<td>atom parts per million</td>
</tr>
<tr>
<td>BM</td>
<td>base metal</td>
</tr>
<tr>
<td>BSE</td>
<td>backscatter electron</td>
</tr>
<tr>
<td>dpa</td>
<td>displacement per atom</td>
</tr>
<tr>
<td>EBSD</td>
<td>electron backscatter diffraction</td>
</tr>
<tr>
<td>EDM</td>
<td>electrical discharge machining</td>
</tr>
<tr>
<td>FSW</td>
<td>friction stir welding</td>
</tr>
<tr>
<td>GB</td>
<td>grain boundary</td>
</tr>
<tr>
<td>GTAW</td>
<td>gas-tungsten arc welding</td>
</tr>
<tr>
<td>HAZ</td>
<td>heat affected zone</td>
</tr>
<tr>
<td>He</td>
<td>helium</td>
</tr>
<tr>
<td>HFIR</td>
<td>High Flux Isotope Reactor</td>
</tr>
<tr>
<td>IMET</td>
<td>Irradiated Material Examination and Testing facility</td>
</tr>
<tr>
<td>IPF</td>
<td>inverse pole figure</td>
</tr>
<tr>
<td>IQ</td>
<td>image quality</td>
</tr>
<tr>
<td>KAM</td>
<td>kernel average misorientation</td>
</tr>
<tr>
<td>LAMDA</td>
<td>Low Activation Materials Development and Analysis Laboratory</td>
</tr>
<tr>
<td>LWR</td>
<td>light water reactor</td>
</tr>
<tr>
<td>LWRS</td>
<td>Light Water Reactor Sustainability program</td>
</tr>
<tr>
<td>MA</td>
<td>middle advancing side</td>
</tr>
<tr>
<td>MR</td>
<td>middle retreating side</td>
</tr>
<tr>
<td>MS</td>
<td>middle stir zone</td>
</tr>
<tr>
<td>Ni</td>
<td>Nickel</td>
</tr>
<tr>
<td>NPP</td>
<td>nuclear power plant</td>
</tr>
<tr>
<td>ORNL</td>
<td>Oak Ridge National Laboratory</td>
</tr>
<tr>
<td>PCBN</td>
<td>Polycrystalline Cubic Boron Nitride</td>
</tr>
<tr>
<td>REDC</td>
<td>Radiochemical Engineering Development Center</td>
</tr>
<tr>
<td>rpm</td>
<td>rotation per minute</td>
</tr>
<tr>
<td>SE</td>
<td>secondary electron</td>
</tr>
<tr>
<td>SEM</td>
<td>scanning electron microscope/microscopy</td>
</tr>
<tr>
<td>SS</td>
<td>stainless steel</td>
</tr>
<tr>
<td>SZ</td>
<td>stir zone</td>
</tr>
<tr>
<td>TA</td>
<td>top advancing side</td>
</tr>
<tr>
<td>TEM</td>
<td>transmission electron microscope/microscopy</td>
</tr>
<tr>
<td>TMAZ</td>
<td>thermo-mechanically affected zone</td>
</tr>
<tr>
<td>TR</td>
<td>top retreating side</td>
</tr>
<tr>
<td>TS</td>
<td>top stir zone</td>
</tr>
<tr>
<td>UTS</td>
<td>ultimate tensile stress</td>
</tr>
<tr>
<td>wppm</td>
<td>weight parts per million</td>
</tr>
<tr>
<td>YS</td>
<td>yield stress</td>
</tr>
</tbody>
</table>
EXECUTIVE SUMMARY

The report describes new experimental results on the mechanical performance of the friction stir welds made on neutron-irradiated 304L stainless steel (SS) with helium. The report focuses on helium-related issues, e.g., the helium-induced degradation in the welded joint, aiming at the need to repair irradiated components of nuclear power plants (NPP). The friction stir welds analyzed here were produced at ORNL previously, and initial characterization work was performed, mostly addressing the presence or absence of macroscopic cracks and helium bubbles, and microstructure. The present work attempts to perform a more comprehensive study to assess mechanical performance, i.e., microhardness distribution, tensile properties, tensile deformation behaviors, and fractography analysis.

Section 1 briefly describes issues associated with transmutation-induced helium and its impact on weldability; the promising solution for repairing irradiated parts and components using friction stir welding (FSW); the recent FSW on irradiated 304L SS containing different amounts of helium.

Section 2 describes the studied materials (custom steel heats with ~10 atom parts per million (appm) He); introduces FSW tool and parameters; summarizes previous microstructure characterization results including helium-induced damage (Helium bubbles, bubble chains, and micro-cracks); presents experiments performed in this report, i.e., horizontal and vertical Vickers microhardness test, DIC tensile tests of miniature specimens extracted from the friction stir weld at different metallurgical zones, and fractography analysis of the broken tensile specimens.

Section 3 presents experimental results from microhardness test, DIC tensile test, and fractographic analysis; analyzes FSW thermal-mechanical histories effects on hardness, tensile properties, and tensile strain distribution; discusses helium-induced damage to friction stir weld mechanical behavior and fracture mechanisms.

Section 4 summarizes the work performed and suggests future work steps, which include further characterization on helium-induced damages, such as in-situ test, Thermal mechanical affected zone (TMAZ)/Heat affected zone (HAZ) specimen strain bands and fracture features mechanism, and re-irradiation experiments as a way to ensure the friction stir weld performance in a real NPP.
1. INTRODUCTION

1.1 ACCUMULATION OF HELIUM IN THE IN-CORE MATERIALS AND ITS IMPACT ON WELDING

Fusion welding is widely used in nuclear power plant (NPP) construction, and common welding techniques, such as gas tungsten arc welding (GTAW) and gas metal arc welding (GMAW), are routinely employed to weld pristine, nonirradiated materials, providing high-quality joints. However, neutron irradiation and overall harsh in-reactor environment (i.e., temperature, radiation, and mechanical stress fields, elevated pressure, and potentially corrosive environment) compromise material performance. Over time, some parts and components may require repair or replacement that ideally relies on welding techniques. Thus, replacement and repair welding are essential to ensure the long-term viability, competitiveness, and safe lifetime extensions of the existing US reactor fleet.

Fusion repair welding of NPP irradiated components meets one specific issue: helium-induced degradation and/or cracking. During nuclear reactor operation, neutron fluxes stimulate transmutation reactions in the NPP structural materials and lead to helium accumulation, mainly because of the transmutation of boron and nickel.

\[ ^{10}\text{B} + n \rightarrow ^{7}\text{Li} + ^{4}\text{He} \]  
\[ ^{58}\text{Ni} + n \rightarrow ^{59}\text{Ni} + \gamma \]  
\[ ^{59}\text{Ni} + n \rightarrow ^{56}\text{Fe} + ^{4}\text{He} \]

Helium accumulation and helium-related issues are especially severe in water-moderated reactors, due to the “soft” neutron spectra, with a high fraction of thermal neutrons.

Stainless steels are widely used in nuclear reactor as structural materials. The presence of Ni in stainless steel with B impurities makes helium accumulation unavoidable. Unfortunately, helium is practically insoluble in steel. With the high heat input in fusion repair welding of irradiated stainless steels, helium migrates to grain boundaries (GBs) and tends to form bubbles, drastically embrittle the GB and reduce the GB strength. Moreover, the local transient elevated temperatures during fusion welding result in tensile stresses in the weld and nearby areas, high enough to initiate crack formation on the helium-compromised GBs and lead to the crack propagation in the heat-affected zone (HAZ) and/or weld zone, i.e., helium-induced cracking.

Even 1–3 appm of helium is usually enough to cause helium-induced cracking in welding, and austenitic steels with 5–15+ appm (atom parts per million) of helium are often considered nonweldable with today’s fusion welding techniques. In short, helium accumulates at rates of roughly 0.2–1 appm per dpa, and 1 dpa usually means ~1 year of service for in-core components. The rates may be an order of magnitude lower for the peripheral components or higher in the core, but, generalizing, critical helium concentrations (above 1–3 appm) may be reached even for peripheral parts within ~10–20 years. This time is much lower than the planned life span of typical NPPs (40 years), even without the extension (usually extended to 60–80 years). Thus, helium-related issues prohibit fusion welding repair, and, without any doubt, this is inevitable during NPP operation.

As an example, in 1986, the GTAW technique was used to repair the water leakage of a Savannah River National Laboratory reactor, and many cracks were presented at the HAZ after repair welding attempts. Those cracks resulted in the reactor’s permanent shutdown. Investigation showed that these were helium-induced cracks, and helium concentration in the repaired water tank wall was ~3 appm [1]. An example of
a fusion weld cross-section with helium-induced cracks in the HAZ is shown in Figure 1 [2]; the helium amount was 8.3 appm. Studies have shown that elevated temperatures and the appearance of tensile stress during fusion welding cooling down are two key factors in helium-induced crack formation and propagation [3].

![Figure 1. Helium-induced cracks in the weld HAZ of stainless steel containing 8.3 appm He [2]. Note many cracks exceed 1 mm in length and the total length of macroscopic cracks is well above ~5-6 mm.](image)

Thus, as discussed above, nickel is a common element in many stainless steels, and boron is an impurity that is difficult to avoid in commercial production; hence, helium accumulation is unavoidable and at some point, it will reach a critical level to compromise the material’s weldability during a reactor’s service life.

### 1.2 FRICTION STIR WELDING AS A PROMISING SOLUTION

Friction Stir Welding (FSW) is an advanced manufacturing technology [4,5] with peak temperatures much lower than the material bulk melting point (generally between 0.6–0.8 Tₘ). During FSW, the material is heated up by friction between the welding tool and welded material, as well as by plastic deformation of the material being welded. Heated material flows/deforms around the welding tool and forms a joint, as the result of mechanical mixing and recrystallization [6]; tool wear during high temperature materials FSW is often mentioned as a minor side effect[7].

In general, a friction stir weld contains four metallurgical zones, stir zone (SZ), thermo-mechanical affected zone (TMAZ), HAZ, and base metal (BM). The SZ is the material directly affected by the FSW tool, usually, underneath the FSW tool shoulder, experiences elevated temperature and large plastic deformation, and is generally recrystallized during the cooling process. The TMAZ is located on both sides of the SZ, experiencing both high temperature and some plastic deformation but the grain elongation is still seen after the FSW. The HAZ is further out from the TMAZ, receiving sufficient thermal input that affected the material microstructure/properties but there is no plastic deformation. For the part in which material microstructure/mechanical properties are not affected by the FSW heat input and plastic deformation is called BM. In a friction stir weld cross section, the side where the FSW tool rotating and traveling are along the same welding direction is called the advancing side, and the side where the two directions are opposite to each other is called the retreating side.

FSW is arguably the most recent significant invention in welding technologies [6], and it has been widely studied and developed with different materials, from nonferrous metals to ferrous metals [8-10] and from steels to high alloy materials [11-13]. In many applications, FSW demonstrated outstanding weldability to
form high-quality joints. Lower peak temperatures and shorter time at elevated temperature, compared to traditional fusion welding approaches, significantly reduces the intensity of diffusion-based processes, including helium migration. Lower temperatures may also reduce tensile thermal stresses and, in turn, cracking. These considerations make the FSW a potential candidate to weld irradiated material with high helium content.

1.3 RECENT ACHIEVEMENTS ON IRRADIATED MATERIAL FSW

Although being promising, the FSW had not been used for joining irradiated materials until Oak Ridge National Laboratory’s (ORNL’s) team, including the present report authors, who were the major contributors to FSW and characterization efforts, successfully demonstrated FSW on irradiated 304L stainless steel (304L SS) for the first time at the end of 2017 [6,14,15].

This development of irradiated 304L SS FSW and subsequent initial characterization [6,14] was a joint effort of several organizations, programs, and projects, including ORNL, the US Department of Energy Light Water Reactor Sustainability Program (LWRS), and Electric Power Research Institute (EPRI). Within this effort, the custom 304L SS coupons with different controlled amounts of boron were irradiated at the High Flux Isotope Reactor (HFIR) at ORNL, to receive controlled amounts of transmuted helium from boron. The irradiated 304L SS coupons were then friction stir welded at Radiochemical Engineering Development Center (REDC) of ORNL. Up to now, FSW has been applied on three irradiated 304L SS coupons that contained ~5, ~10, and ~20 weight parts per million (wppm) of boron before irradiation, respectively. Metallographic specimens were removed from all three frictions stir welds at Irradiated Material Examination and Testing (IMET) Facility of ORNL [16]. Preliminary characterization on one specimen from each of the ~10 and ~20 wppm friction stir welds showed that no macro helium-induced cracking was observed at the weld cross section [6,14-17]. Currently, the rest of the welded specimens, as well as leftover coupon blocks, are stored at ORNL to support future investigations (including current work).

The most recent report of the major characterizations including helium effects, microstructure characterization, and mechanical properties of irradiated 304L SS friction stir welds can be found at [17]. The report focused on microstructure conditions, grain size, plastic strain gradients, the morphology of the helium-induced damage, and deformation behavior of the friction stir weld containing ~5.2 appm helium, as well as, the microstructure and helium induced damage of the friction stir weld containing ~10 appm helium.

The present report attempts to continue the characterization of helium-containing friction stir welds, with a particular focus on microhardness distribution, tensile properties, tensile plastic strain distributions, and helium-induced degradation on the friction stir weld containing ~10 appm helium (20 wppm of boron prior to the irradiation).

2. MATERIALS, EXPERIMENTAL METHODS, AND MAJOR RESULTS OF PREVIOUS CHARACTERIZATION

2.1 CUSTOM 304L STAINLESS STEEL COMPOSITION, FABRICATING, AND PROCESSING

All 304L SS coupons with different levels of boron, including the one characterized in this report 304D-5, were irradiated at ORNL High Flux Isotope Reactor (HFIR) for three operation cycles (22 – 26 days/cycle) in 2014 [18]. Chemical compositions of the 304D heat material prior to the irradiation are shown in Table 1. The 304L SS coupon dimensions were 76 mm × 56 mm × 8.9 mm. Through fitting
tests, 45 stainless steel coupons were placed in the VXF-16, VXF-17, VXF-19, and VXF-21 large bores at HFIR for irradiation. In addition, neutronics calculations with different codes were performed to determine the amount of irradiation required to generate the targeted helium level in each specimen. The irradiation strategy was developed which was based on completely transmuting boron to helium while keep the burning of nickel to a minimal amount.

Table 1. Chemical composition of the 304L SS, wt.% [17]

<table>
<thead>
<tr>
<th>Type</th>
<th>Heat name</th>
<th>B (wppm)</th>
<th>Fe</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>Cu</th>
<th>N</th>
<th>P</th>
<th>S</th>
</tr>
</thead>
<tbody>
<tr>
<td>Custom B- enriched 304L</td>
<td>304D</td>
<td>24</td>
<td>Bal.</td>
<td>0.01</td>
<td>1.53</td>
<td>0.49</td>
<td>19.33</td>
<td>10.41</td>
<td>0.04</td>
<td>0.05</td>
<td>0.035</td>
<td>&lt;0.001</td>
<td>0.002</td>
</tr>
</tbody>
</table>

Figure 2. Reference microstructure for 304D custom heat (24-appm B-enriched, measured ~15.6 appm He in another coupon with the same heat): EBSD Inverse Pole Figure (IPF), Image Quality (IQ), Phase, and Kernel Average Misorientation (KAM) maps. One may see annealed austenite structure with a minor amount of ferrite. Many ferrite grains have a specific elongated shape with a long axis oriented in the horizontal direction. The IQ map shows the reduced pattern quality around many ferritic grains (no such effect was observed in 304C heat); reduced Kikuchi pattern quality may suggest some element segregation effects. The KAM map reveals no plastic strain (fully annealed conditions). Amount of ferrite for this scan <0.2-0.3%. Scan size: 300×250 µm; EBSD step (pitch) size: 0.5 µm.
Figure 2 shows a typical microstructure of the studied materials (304D heat) after the neutron-irradiation. One may see a well-annealed austenite structure with multiple annealing twins. The analysis showed no pronounced texture in the austenitic matrix. A limited amount of retained ferrite (much below 1%) presents in the structure with some insignificant variations between locations and heats; retained ferrite presence is typical for many 300-series steels and, as believed, should be out of concern for the present work purposes. Ferrite grains sometimes appeared as bands or chains elongated in one direction — most likely, the former hot rolling direction.

The FSW was performed on the irradiated 304D-5 coupon with an unirradiated 304L SS tab at each end of the irradiated coupon. As mentioned above, the irradiated 304L SS coupon dimensions were 76.2 mm × 55.9 mm × 8.9 mm, and the tab dimensions were 38.1 mm × 55.9 mm × 8.9 mm. A Polycrystalline Cubic Boron Nitride (PCBN) tool was used in FSW, and the FSW started in the beginning tab, went through the irradiated coupon along the length (55.9 mm) direction, and stopped in the end tab. FSW parameters were 400 rpm rotation rate, 50.8 mm/min welding speed with position control mode and argon cover gas [14].

After FSW, metallographic specimens (thin slices) were cut from the FSW in cell 6 at the IMET facility of ORNL using a bandsaw. The center of the metallographic specimen was aligned with the center of the friction stir weld, and each metallographic specimen contains SZ, TMAZ, HAZ, and BM. The dimensions of the slices were 30.5 mm × 8.9 mm × 2.5 mm. The friction stir weld was located in the middle of the 30.5 mm long slice. The slices were transferred to ORNL’s Low Activation Materials Development and Analysis (LAMDA) facility for further preparation and analysis.

The selected thin slice (ID: 304D-5-14), which was located in the middle section along the friction stir weld length, were epoxy-mounted, mechanically ground, and polished using standard metallography procedures.

2.2 MAJOR RESULTS FROM PREVIOUS SEM MICROSTRUCTURE CHARACTERIZATION OF THE 304D-5-14 SPECIMEN

The cross section of the friction stir weld is shown in Figure 3 [17]. The SEM image brightness and contrast were adjusted to highlight any potential defect such as cracks and discontinuities in the structure. As expected, macroscopic defects (e.g., a millimeter in length or so) should be easy to detect at the given conditions. Analyzing the image, it is a solid metallic surface without any visible cracks or voids.
Figure 3. Cross section of the friction stir weld performed on the 304L SS containing ~10 appm helium. No macroscopic cracks or crack-like defects (e.g., ~0.5 mm or so) were observed in the SZ or TMAZ. Note the SEM image has some minor distortion due to the low magnification level. Left side: FSW advancing side; right side: FSW retreating side [17].

Without etching, it is hard to see the friction stir weld shape and boundary visually. However, with some visible features such as the edges of the undercut at the specimen surface and the root tip, the typical shape of a 304L SS friction stir weld made by the same FSW tool, and the SEM observation of the 304L SS friction stir weld specimen, the schematic SZ and TMAZ shape were plotted on a specimen cross section shown in Figure 4.

Figure 4. Schematic of the SZ, TMAZ, HAZ, and GM in an irradiated 304L SS friction stir weld.

Figure 5. Grain structures in different metallurgical zones of the 304D-5 friction stir weld. The scale bar for grain structure transitions SEM/EBSD image (Top left) is 1 mm and that for the rest SEM/EBSD images is 100 μm [17].
Typical friction stir weld microstructures were observed in different metallurgical zones of the 304D-5-14 specimen, as shown in Figure 5 [17]. The SZ microstructure (The two images on the right) represented roughly shaped, sometimes equiaxed grains, approximately 20-40 µm in size. The SZ area was practically ferrite-free because of the experience at elevated temperature during FSW. At the SZ root, grains in the SZ were very fine due to the low heat input in FSW, and grains turned much larger in the TMAZ right outside of the SZ root (Middle bottom). Grain structure transitions (Left top image) were clearly shown from the SZ to TMAZ, and HAZ. The TMAZ was featured with elongated grain structures without recrystallization or full recrystallization, while the HAZ grain shapes and sizes were close to the reference BM. The reference BM (Bottom left) exhibited larger grain structures than those in the SZ.

Most SEM images inside the 304D-5-14 SZ show specific void-like or bubble-like features (~1–2 µm in size or smaller) of dark color, Figure 6 [17]. The observed features had a round shape (likely, spherical in 3D) and significantly varied size. Their density also varies across the SZ, suggesting their appearance was
sensitive to the local conditions. BM location revealed much smaller counts, and the features presented differently. A significant fraction of the observed round features may be helium bubbles; however, EELS spectroscopy [19] should be used to confirm.

In the TMAZ, individual helium bubbles, helium bubble chains, and several micro-cracks were observed, Figure 7 [17]. As they are shown, individual helium bubbles appeared inside grains and on grain boundaries, and the helium bubble chains and the micro-cracks are along grain boundaries. It is also noticed that the compromised grain boundaries are in a small fraction in those pictures.

(a) Individual helium bubbles and helium bubble chains in the TMAZ on the 304D-5-14 weld advancing side. Left, BSE image; Right, SE image.

(b) Individual helium bubbles and micro-cracks in the TMAZ on the 304D-5-14 weld retreating side. Left, BSE image; Right, SE image.

Figure 7. Individual helium bubbles, helium bubble chains, and micro-cracks in the 304D-5-14 TMAZ [17]
In this 304D-5-14 friction stir weld specimen, helium-induced features often formed specific clusters or “clouds” of large pores and bubbles, as seen in Figure 8 [17]. These clusters are often associated with the retained ferrite grains and preexisting metallurgical inclusions. Such feature was not observed in the lower helium content (~5.2 appm) friction stir weld reported previously.

Figure 8. Clustering of the helium-induced damage in TMAZ. Dashed ovals at right show the clusters. (Left) BSE image; (right) SE [17].

BM SEM observation also showed larger voids like feature in the 304D-5-14 specimen (~10 appm helium) than that with lower helium content 304C-5-14 specimen (~5.2 appm). The comparison of the current ~10 appm helium and the ~5.2 appm helium 304L SS BMs under SEM are shown in Figure 9 [17]. The hypothesis of the helium bubble clusters or clouds in the high helium content 304D-5-14 specimen is the preexisting inhomogeneity of the boron distribution in the base material. Boron segregations (likely, elevated boron levels in the retained ferrite and metallurgical inclusions) generated, in turn, inhomogeneous helium distribution during irradiation in the HFIR. Such boron/helium distribution segregations reflected in the friction stir weld microstructures.
(a) 304D-5-14 specimen containing ~10 appm helium

(b) 304C-5-14 specimen containing ~5.2 appm helium

Figure 9. SEM pictures of the 304L SS BMs contains different amount of helium [17]

2.3 MICROHARDNESS TESTING

Previous SEM characterization identified the friction stir weld shape, location, and boundaries [17]. Follow the results, Vickers microhardness tests were performed on the 304D-5-14 friction stir weld specimen along five horizontal lines and three vertical lines, Figure 10. The three horizontal lines crossed the whole specimen, and they were ¼, ½, and ¾ of the SZ depth from the weld top surface, respectively. From the left to the right, the three horizontal lines started at HAZ on the friction stir weld advancing side, went through TMAZ on the advancing side, SZ, TMAZ on the retreating side, and ended at HAZ on the retreating side. The two short horizontal lines located at the bottom of the specimen left and right corners, and measurements from the two short horizontal lines represent the BM Vickers hardness. The three vertical measurement lines crossed the specimen from the top to the bottom; the middle line went through the weld root tip, and the other two located on the advancing and retreating sides of the weld, respectively. From the top to bottom, the three vertical lines started at SZ, went through TMAZ and HAZ, and the bottom, either HAZ or BM. The Vickers microhardness tests were performed with 200 g force and 10 s dwell time, with indentation spacing of 250 μm.
2.4 DIC TENSILE TESTING

The drawing of the miniature tensile specimens used in this study is shown in Figure 11. Such a small size of specimen can be extracted out at different metallurgical zones of the metallographic specimen 304D-5-14. The miniature tensile specimen mapping on the metallographic specimen cross section is shown in Figure 12. As they are shown in Figure 12, the long-axis of the top row specimens is at ¼ SZ depth, and the long-axis of the 2nd row specimens is at 5/8 SZ depth, and that of the bottom specimen is at the SZ root tip. Moreover, specimen gauge areas of the left, middle, and right specimens at the top two rows are located in the TMAZ/HAZ on the advancing side, SZ, and TMAZ/HAZ on the retreating side, respectively. The center of the bottom tensile specimen is at the SZ root tip. A summary of the specimen IDs and locations are listed in Table 2.

![Figure 10. Vickers microhardness measurement lines](image)

![Figure 11. Miniature tensile specimen drawing (unit: mm)](image)
Figure 12. Miniature tensile specimen mapping on the 304D-5-14 friction stir weld specimen.

Table 2. DIC tensile specimen IDs, locations, and gauge area metallurgical zones

<table>
<thead>
<tr>
<th>Specimen ID</th>
<th>Specimen height location</th>
<th>Gauge area metallurgical zones</th>
</tr>
</thead>
<tbody>
<tr>
<td>304D-5-14 TA</td>
<td>¼ SZ depth (Top row)</td>
<td>TMAZ/HAZ on the advancing Side</td>
</tr>
<tr>
<td>304D-5-14 TS</td>
<td>¼ SZ depth (Top row)</td>
<td>SZ</td>
</tr>
<tr>
<td>304D-5-14 TR</td>
<td>¼ SZ depth (Top row)</td>
<td>TMAZ/HAZ on the retreating Side</td>
</tr>
<tr>
<td>304D-5-14 MA</td>
<td>5/8 SZ depth (Middle row)</td>
<td>TMAZ/HAZ on the advancing Side</td>
</tr>
<tr>
<td>304D-5-14 MS</td>
<td>5/8 SZ depth (Middle row)</td>
<td>SZ</td>
</tr>
<tr>
<td>304D-5-14 MR</td>
<td>SZ depth (Bottom row)</td>
<td>TMAZ/HAZ on the retreating Side</td>
</tr>
<tr>
<td>304D-5-14 BR</td>
<td>SZ depth (Bottom row)</td>
<td>SZ and TMAZ/HAZ on both sides</td>
</tr>
</tbody>
</table>

The mount material was removed from the 304D-5-14 metallographic specimen after microstructure characterization and microhardness tests. Wire electric discharge machining (EDM) was used to extract miniature tensile specimens at different locations, and the wire EDM system in LAMDA of ORNL is shown in Figure 13. An extracted miniature tensile specimen is shown in Figure 14.

Figure 13. The wire EDM system installed in LAMDA to manufacture specimens from radioactive material.
After the miniature tensile specimen extraction, they were cleaned using acetone and painted with black and white spackle patterns. The MTS Insight 10 tensile frame with a 2.5kN loadcell was used for tensile testing at room temperature at LAMDA. During tensile testing, the specimen surface with painted spackle pattern faced to the camera for taking images. The tensile strain rate was set to $0.001 \text{s}^{-1}$, and the camera took one image per second.

### 2.5 Fractography Analysis

After the mechanical tests, the tensile specimens were cleaned with high-purity acetone and then subjected to an ultrasonic bath in distilled water. This process was repeated at least three times to ensure that most, if not all, of the paint was removed. Fractographic analysis was carried out using a TESCAN MIRA3 SEM. The specimens were mounted on holders inclined at 45 degrees, which enabled the analysis of both the fracture areas and tensile portions (at $-45$-degree and $+45$-degree tilts, respectively). SEM images were captured using a 5 kV voltage and a beam current of approximately 40 pA.

### 3. Results and Discussion

#### 3.1 Microhardness Distributions

Microhardness distributions of the three horizontal measurement lines across the friction stir weld SZ, TMAZ, and HAZ are shown in Figure 15. The BM hardness (223.5 VHN) is represented by a straight dash line, with the average value of the two bottom line measurements.
Hardness values in the SZ, TMAZ and HAZ measured along the ¼ and ½ SZ depth were lower than that of the BM, and the lowest hardness values were measured in the TMAZ/HAZ area. It was also noticed that the hardness values in the SZ and TMAZ/HAZ along the ½ SZ depth line are slightly higher than those along the ¼ SZ depth line, respectively. Such difference could be caused by higher peak temperatures hence larger grain sizes at locations closer to the tool shoulder (Specimen top surface), which generated the most FSW heat input. One hardness value along the ½ SZ depth is obviously higher than the other measurements along the same line, and higher than the BM hardness, but the reason stays unknown.

The SZ hardness values along the ¾ SZ depth measurement line were higher than SZ hardness at higher locations (¼ and ½ lines), and some of them were even higher than the BM hardness. The high SZ hardness close to weld root was due to the fine recrystallized grain sizes resulted from low heat input during FSW (Figure 5). The SZ hardness large variation along the ¾ SZ depth line indicated grain sizes along that line were not uniform, because of different thermal and mechanical histories in FSW. On the other hand, the TMAZ/HAZ hardness values along the ¾ SZ depth line were close or just slightly higher than those along the ¼ and ½ SZ depth lines, and such small differences were caused by the different peak temperature reached in the FSW.

The hardness values and distributions can be modified by adopting different FSW tool designs and select different processing parameters, which mainly control the heat input and plastic deformation in FSW.

Microhardness distributions along the three vertical measurement lines are shown in Figure 16, and the length and width ratio of the picture shown in Figure 16 is changed to fit in the hardness plots. For all three lines hardness results, measurements close to specimen top (the left edge of the picture) showed SZ hardness, and those close to specimen bottom (the right edge in the picture) indicated HAZ and TMAZ hardness. The SZ middle line stayed in the SZ the longest because it went through the SZ root tip, while the SZ left line was on the advancing side and the SZ right line was on the retreating side of the friction stir weld.
From Figure 16, hardness increased at locations within 1 – 1.25 mm from the specimen top, where we observed fine grain structures due to excessive plastic deformation by the FSW tool shoulder. Except one measurement at this area, SZ left and SZ right line hardness are all lower than that of the BM, no matter the measurement was in the SZ, TMAZ, or HAZ. In addition, hardness values close to the bottom started to increase towards the BM value, which meant the measuring was moving close to the FSW unaffected BM zone. For SZ middle line hardness distribution, beside the hardness increase close to the top, there were a significant hardness increases at locations 5 – 7 mm away from the specimen top. Such hardness increases (peak value 288 VHN) was caused by the extremely fine grain structures at the weld root area presented in Figure 5.

![Graph showing microhardness distribution](image)

**Figure 16.** 304D-5-14 friction stir weld specimen vertical Vickers microhardness distributions.

Overall, the microhardness distributions of the 304D-5-14 specimen corresponded with the microstructure observation [17]. Higher hardness values were measured at the top and at the weld root of the SZ, where fine grain structures were observed. In addition, the ¾ SZ depth horizontal line was ~5.25 mm from the specimen top surface with the hardness increase in the SZ, which agreed with the vertical SZ middle line hardness results, i.e., significant hardness increasing at locations 5 – 7 mm from the top. Therefore, with the current FSW tool and parameters, the bottom ¼ SZ area exhibited high hardness as a result of the thermal-mechanical effects in FSW.

### 3.2 TENSILE PROPERTIES AND LOCAL PLASTIC DEFORMATION

Hundreds of images were taken at different load/stress levels during each DIC tensile test. Images with spackle pattern of miniature tensile specimen 304D-5-14 MS (5/8 SZ depth in the SZ) at the moment before testing, at the tensile load of ultimate tensile strength (UTS) stress, and right before specimen
failure are shown in Figure 17. As it is shown in the images, the specimen had significant amount of deformation/elongation during the tensile test, and exhibited obvious necking before it broke, indicating good ductility in the SZ.

![Image showing specimens at different stages](image_url)

**Figure 17.** 304D-5-14 MS images before testing (top), at UTS (middle), and before failure (bottom).

A 2.5 mm long line was applied in the gauge area on the 1st image of each specimen as the digital extensometer. With the software processing, the length of the digital extensometer could be calculated for every image taken in the tensile test. Therefore, elongations, obtained from the initial digital extensometer length and its changes during the test, can be obtained at corresponding stress levels. The initial extensometer (Red line in the specimen gauge area), and appearances at UTS and right before the failure of the 304D-5-14 MS specimen are shown in Figure 18. Clearly the digital extensometer had large extension before the specimen fail.
Correlating elongations obtained from the digital extensometer and recorded tensile stresses, tensile curves and tensile properties were obtained. All tensile curves and tensile properties of the seven miniature tensile specimens are shown in Figure 19 and listed in Table 3, respectively. Despite helium bubbles in the SZ (Figure 6) and helium bubbles, bubble chains and micro-cracks in the TMAZ (Figure 7), all tested specimens exhibited higher yield strength and comparable or higher ultimate strength with annealed conventional 304L SS (210 MPa yield strength and 564 MPa ultimate strength). Except the specimens at the top retreating side (304D-5-14 TR) and at the weld root (304D-5-14 BR), all specimen elongations were also higher than the conventional 304L SS elongation, 58% (Note there is also an extensometer length difference between the current experiment (2.5 mm long) and conventional 304L SS (50 mm long)). The BM tensile elongation of the irradiated 304L SS with ~5.2 appm helium was 71.3% [20]. Moreover, the uniform elongation of each tensile curve possessed a big ratio of the total elongation, indicating excellent ability staying in uniform deformation to resist necking and rapid fracture propagation. The lowest elongation was measured from the specimen extracted at the weld root, which also presented the highest yield strength and ultimate strength. Such relatively high strength and low elongation properties were results of the super fine grain sizes in the SZ root, which caused dislocation movement difficulty in the SZ and strain localization in the TMAZ during the tensile process.

Therefore, although helium bubbles, helium bubbles chains and a few micro-cracks were presented in the SZ, TMAZ, and HAZ, they didn’t cause catastrophic effects on the friction stir weld strength and ductility.
Figure 19. 304D-5-14 tensile curves of all friction stir weld miniature tensile specimens.

Table 3. 304D-5-14 friction stir weld specimen tensile properties.

<table>
<thead>
<tr>
<th>Specimen ID</th>
<th>Yield strength, MPa</th>
<th>Ultimate strength, MPa</th>
<th>Uniform elongation, %</th>
<th>Total elongation, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>304D-5-14 TA</td>
<td>329.3</td>
<td>571.7</td>
<td>51.5</td>
<td>64.8</td>
</tr>
<tr>
<td>304D-5-14 TS</td>
<td>327.9</td>
<td>536.3</td>
<td>46.9</td>
<td>62.4</td>
</tr>
<tr>
<td>304D-5-14 TR</td>
<td>320.3</td>
<td>518.4</td>
<td>30.3</td>
<td>43.3</td>
</tr>
<tr>
<td>304D-5-14 MA</td>
<td>312.2</td>
<td>565.7</td>
<td>56.6</td>
<td>71.8</td>
</tr>
<tr>
<td>304D-5-14 MS</td>
<td>380.6</td>
<td>614.4</td>
<td>47.5</td>
<td>63.3</td>
</tr>
<tr>
<td>304D-5-14 MR</td>
<td>323.7</td>
<td>536.9</td>
<td>55.4</td>
<td>73.8</td>
</tr>
<tr>
<td>304D-5-14 BR</td>
<td>462.6</td>
<td>677.5</td>
<td>18.2</td>
<td>32.1</td>
</tr>
</tbody>
</table>

By applying 2D analysis on the DIC results, local strain distributions and changes in the specimen gauge area were obtained.

The 304D-5-14 MS specimen (Middle stir zone) strain $\varepsilon_x$ (strain along the pulling direction) distributions at different tensile stages, including global yield, uniform deformation, UTS, and necking, are shown in Figure 20. The green dash lines in the tensile curve plot illustrate corresponding tensile stages of the strain distribution images. From Figure 20, plastic strains were mainly within the specimen gauge area after yielding, and the peak strains (Red color) were distributed at the right half of the gauge area. After the tensile stress reached to UTS, plastic strain localized in a small area of the previous wide peak strain zone, and the specimen broke at the same location by the end of tensile test.
The bottom root specimen 304D-5-14 BR strain $\varepsilon_x$ 2D distributions at different tensile stages, including at yield, between yield and UTS, at UTS, and during necking, are shown in Figure 21. For the 304D-5-14 BR specimen, strain localization occurred shortly after the tensile stress reached to yield and before it reached to UTS. This is due to the inhomogeneous microstructures and hardness distributions at the SZ root area. Inside the SZ root, grains are super fine with high hardness, but grains turned much larger in the TMAZ right outside of the SZ root with low hardness (Figure 4, Figure 15, and Figure 16). Therefore, in the tensile test, plastic deformation was localized at the softer TMAZ soon after the tensile stress reached to yield stress (YS), and specimen failed at the same location by the end of the test.
Figure 21. Local strain $\varepsilon_x$ distribution of 304D-5-14 BR specimen at different tensile stages.

The strain $\varepsilon_x$ distributions of the TMAZ/HAZ specimens at the top advancing side and the middle retreating side are shown in Figure 22 and Figure 23, respectively. Unlike the relatively uniform strain distribution of the SZ specimen (Figure 20) and the early stain localization of the bottom root specimen (Figure 21), multiple plastic strain bands, with about 45 degrees to the specimen gauge length directions, were presented soon after the yielding of the TMAZ/HAZ specimens. Tensile strains inside the strain bands are much larger than those outside of the strain band, though they might be within the same metallurgical zone. Those strain bands continue existed when tensile stresses were between YS and UTS, and some strain bands even presented when the stresses reached to UTS with strains $> 0.5$ in Figure 22 and Figure 23. At the UTS, the strain band widths were about 60 – 120 $\mu$m for both specimens. Further studies are needed to identify the mechanism of the strain bands present, and that could be related with the FSW thermal and mechanical histories in the TMAZ and HAZ, the FSW tool determined TMAZ and HAZ geometry, and/or irradiation effects such as helium aggregation.
Figure 22. Local strain $\varepsilon_x$ distribution of 304D-5-14 TA specimen at different tensile stages.

YS = 329.3 MPa
$\varepsilon_x = 0.2$

UTS = 571.7 MPa
$\varepsilon_x = 0.4$

$\varepsilon_x = 0.55$
3.3 FRACTOGRAPHY ANALYSIS

3.3.1 Analysis of the specimen fracture area

Figure 24 provides an overview of the fracture surface for all tested specimens. In most of the specimens, the fracture area assumes a square-like shape with dimensions roughly measuring 300 by 300 µm. This significantly smaller cross-sectional area (300 by 300 µm, as compared to ~600 by 600 µm in Figure 11) and the consequent high area reduction imply the high ductility of the friction stir welded material. The fracture surface predominantly exhibits signs of ductile fracture, evident from the ductile dimples (covering the majority of the surface) or ductile shear (occupying a smaller region).

Certain features, not associated with ductile fracture (e.g., deep holes, pores, and crack-like structures, highlighted by black arrows in Figure 24), were also observed. However, their frequency and overall surface area coverage were minimal, typically one or two per specimen, with the notable exception of the TR specimen, as shown in Figure 24a. The TR specimen exhibited several crack-like features.
Figure 24. Low-magnification (800×) SEM images of the fracture regions of the tested tensile specimens. Material condition IDs and image order (e.g., “TA”) correspond to Figure 12 and Table 2. One may see dominating ductile fracture with minor features (black arrows) likely related to boron-rich regions.
Figure 25. High-magnification (nominal magnification 10,000×) SEM images of typical fracture surface appearance. For material condition IDs see Figure 12 and Table 2.

Figure 25 provides a close-up of the typical fracture surface. The dominant features are ductile dimples, which slightly vary in size based on different metallurgical locations. While ductile dimples or ductile shear predominantly characterize the TR specimen (as seen in Figure 25c), there’s also evidence of ductile tearing intertwined with features that are believed to be related to the initiation and growth of internal cracks (indicated by the black arrow).

3.3.2 Helium-related features

Figure 26 displays features that are believed to be related to the presence of helium (boron) rich regions and compromised grain boundaries. The most commonly observed features are deep, trench-like structures formed at compromised boundaries containing chains of helium bubbles, as depicted in Figure 26 (a,b,c). The deep holes shown in Figure 26 (d,e) are potentially indicative of coarse inclusions or large helium bubbles. The unique “ductile bridges” or “pillars” shown in Figure 26f underscore the intricate geometry of the internal damage. Minor cleavage areas, as seen in Figure 26g, likely correspond to helium-rich zones. Deep localized fractures, presented in Figure 26h, are relatively infrequent and are presumably due to metallurgical inclusions.
In all cases, except for the TR specimen, such features constitute only a minor portion of the observed area.

Figure 26. Fracture surface features showing internal cracks, large pores, and cleavage-like spots. “MS”-condition image (i) represents typical the ductile fracture for comparison. Note magnification varies for some images; “TR”-condition is shown twice.

3.3.3 Cracking and fracture events at the gauges portion of the tested specimens

Figure 27 illustrates the condition of the specimen gauge sections following the tensile tests and subsequent fracture. Typically, it’s easy to discern both the necking and the fracture regions. Intriguingly, the majority of the specimens indicates localized fracture along their edges, highlighted at specific spots by black arrows. The distribution of these spots seems random; they do not align into a regular zigzag or any ordered pattern, which would be characteristic of conventional strain-induced shear bands. It is probable that these localized fracture spots are associated with helium-rich zones (Figure 9(a)) possibly generated in the BM production and compromised grain boundaries.

The presence of these localized fracture spots aligns with the generally erratic pattern of strain localization, as unveiled by DIC analysis (refer to Section 3.2). Notably, both the MS and BR specimens visually exhibited no such fracture spots, likely indicating the lack of helium-rich regions.
The TR specimen, on the other hand, exhibited an unexpectedly poor surface quality, manifesting multiple crack formations and delamination effects, as seen in Figure 27c. To prevent the formation of debris and dislodged particles, cleaning was executed with extreme care, which resulted in some paint residue within several cracks. While multiple cracking is typically indicative of premature fracture and diminished engineering ductility, the ductility for this specimen remains high, as detailed in Table 3, with a total elongation value surpassing 40%. Further investigation is required to shed light on this unexpected behavior.

Figure 27. Gauge portions of the tensile specimens after mechanical testing and fracture. Black arrows mark local fracture events (mostly along the gauge edges); the “EDM”-label marks the separation and peeling-off of the EDM-induced layer. Note pronounced delayering at the surface of the TR specimen (c). For material condition IDs see Figure 12 and Table 2, pre-specimen geometry is shown in Figure 11.

Figure 28 shows several examples of the fracture events along the gauge. The specific appearance of these areas points out strong variations in the material conditions. Generally ductile material (slightly deformed austenite in TMAZ) contains specific brittle “clusters” or “kernels,” easily fracturing in a brittle way.
Figure 28. Examples of localized fracture at the specimen gauges: a) The MA specimen, fracture at the specimen edge; b) The MR specimen, fracture at the specimen surface, also note a thin wire-like crack. Tensile direction is vertical in this Figure.

Figure 29. Delayering and multiple cracks at the surface of the TR specimen. This phenomenon is difficult to fully explain now.

Figure 29 shows additional images of the TR specimen, taken from a different specimen half. One may see a striking overlapping of pronounced ductility and ductile fracture (see also Figure 24 and Figure 25) and multiple delamination and, probably, cleavage processes. Such phenomena could be expected for composite materials, combining ductile/soft and brittle/hard phases or alloy components. It is an attractive idea to perform in-situ test with an identical specimen to investigate the phenomenon in more detail.
4. CONCLUSIONS AND FUTURE WORK

The present work describes experimental results on the microhardness distributions and tensile performance of the friction stir weld made on neutron-irradiated austenitic 304L SS with ~ 10 appm helium. The report evaluates microhardness, tensile curves, tensile properties, and strain distributions in the SZ, TMAZ and HAZ of the friction stir welds with helium, as well as the effects of the FSW and helium-induced damage on friction stir weld specimen mechanical behaviors.

It was shown that the grain size dominated the hardness in the SZ zone, higher than BM hardness values were obtained in an area close to the weld top surface and at the SZ root, where small grains and super fine grains were observed. With FSW thermal and mechanical effects, hardness in the TMAZ and HAZ were lower than that of the BM.

Tensile tests on miniature tensile specimens extracted from the SZ, SZ root, and TMAZ/HAZ exhibited very good strengths and elongations, comparable or higher than those of conventional 304L SS, respectively. Therefore, helium-induced damages (helium bubbles, helium bubbles chains and a few micro-cracks presented in the SZ, TMAZ, and HAZ) after FSW didn’t result in catastrophic degradation on the friction stir weld strength and ductility.

With the DIC assistance, tensile strain distributions of irradiated 304L SS friction stir weld specimens at different metallurgical zones were presented. When tensile stresses were between the YS and UTS, SZ specimens showed uniform strain distribution, SZ root specimen demonstrated early strain localization, and TMAZ/HAZ specimens exhibited multiple strain bands. Those local strain behaviors were related with FSW tool design, FSW thermal and mechanical histories, irradiation, and/or helium evolution during FSW. Further studies are needed to identify mechanisms of the strain band development in the TMAZ/HAZ specimens.

Fractography analysis showed mostly ductile fracture with some brittle areas, related, as believed, to the helium-rich spots. From the BM observation, such helium enriched spots might be produced artificially in the BM production but needs further characterization to determine. Helium-rich spots led to localized fracture effects along the specimen edges and probably in the bulk. Helium-related issues were the most pronounced in the TR specimen, leading to delamination effects.

It is important to underline the following: whereas the results mentioned and the data provided are believed to be accurate, more work is necessary to understand, quantify, and explain the observed features and peculiarities and compare them with the literature. The work conducted herein, the selected experimental tools and approaches, and the amount of data obtained all were a result of a compromise between scientific and practical importance and available funds.

Future works of this study include helium-induced damages further characterization, such as in-situ test and transmission electron microscope/microscopy (TEM) characterizations, TMAZ/HAZ specimen strain bands and fracture mechanism investigation, and friction stir weld re-irradiation and characterization.

Although more work is necessary, the obtained results show limited helium-induced degradation in the friction stir weldments and their good mechanical performance, compared to the conventional welding techniques. It makes the FSW a very promising technological approach for repair welding materials with high helium content.
5. ACKNOWLEDGMENTS

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