EFFECT OF PWHT AND FILLER METAL ON STRESS RELAXATION CRACKING SUSCEPTIBILITY IN 347H STAINLESS STEEL WELDS FOR ELEVATED TEMPERATURE SERVICE

by

Timothy J. Pickle
A thesis submitted to the Faculty and the Board of Trustees of the Colorado School of Mines in partial fulfillment of the requirements for the degree of Master of Science (Metallurgical and Materials Engineering).

Golden, Colorado

Date ______________________________

Signed: ______________________________

Timothy Pickle

Signed: ______________________________

Dr. Zhenzhen Yu
Director of Center for Welding, Joining, and Coatings Research
Thesis Advisor

Golden, Colorado

Date ______________________________

Signed: ______________________________

Dr. Ivar Reimanis
Interim Department Head
George S. Ansell Department of Metallurgical and Materials Engineering
ABSTRACT

347H stainless steel (SS) welds, used in commercial concentrating solar power (CSP) thermal energy storage at temperatures of 565 °C, are reported to be susceptible to stress-relaxation cracking (SRC) within months or years of service. Without prior stress relief via post weld heat treatment (PWHT), stress relaxation cracking may occur. The welded heat-affected zone (HAZ) and fusion zone (FZ) of 347H SS, particularly in thick sections greater than a half-inch, may be susceptible to reheat cracking during PWHT. The goal of this project is to determine the effect of PWHT and alternative alloys/filler consumables on reheat cracking in 347H SS welds to avoid SRC during service.

Welding experiments on 347H SS substrates, using GTAW and SMAW, were completed to duplicate the susceptible microstructure for crack susceptibility testing using E347 and E16.8.2 weld consumables. Finite element modeling (FEM) was performed to reveal residual stress profiles. Reheat crack tests, using a Gleeble© 3500, were performed as a function of temperature, stress/strain, and microstructure. Four different sets of microstructures and materials were investigated: 1) simulated HAZ on 347H SS substrate material, 2) simulated HAZ on a modified 316L substrate material, 3) transverse cross-welded samples of E347-347H SS, and 4) transverse cross-welded samples of E16.8.2-347H SS. Finally, elevated temperature tensile tests at 600°C using the Gleeble© 3500 were used to determine the effect of PWHT on mechanical properties for all four sets of materials.

The reheat crack tests indicate more rapid failure in higher pre-strains/stresses and higher test temperatures. The E347 FZ was demonstrated to be the most susceptible to cracking. The 347H HAZ samples exhibited a higher critical stress to failure threshold than E347 FZ and is deemed to be less susceptible to reheat cracking. Plastic strain (1%) in the E16.8.2 FZ was needed to cause cracking. Furthermore, failure occurred preferentially in the 347H HAZ instead of the E16.8.2 FZ. No cracking was detected in the modified 316L SS samples up to 16% strain. Careful PWHT design is needed to prevent cracking in 347H welds, and E16.8.2 consumable demonstrates to be the best SRC mitigation technique in FZ among the filler tested.
# TABLE OF CONTENTS

ABSTRACT .................................................................................................................................................. iii

LIST OF FIGURES .................................................................................................................................... viii

LIST OF TABLES ...................................................................................................................................... xvii

LIST OF ABBREVIATIONS ...................................................................................................................... xix

ACKNOWLEDGMENTS ............................................................................................................................. xx

CHAPTER 1: INTRODUCTION ................................................................................................................ 1

CHAPTER 2: LITERATURE REVIEW .......................................................................................................... 3

2.1 Composition and Microstructure of 347H SS .................................................................................. 3

2.1.1 CALPHAD Simulations of Weld Microstructure ......................................................................... 6

2.1.2 Solubility limits of Nb (C, N) in γ-austenite .............................................................................. 9

2.2 Elevated Temperature Mechanical Properties ............................................................................... 10

2.3 Recovery, Recrystallization, and Grain Growth in 347H welds ...................................................... 12

2.4 Weldability Issues with E347-347H SS Welds ............................................................................... 15

2.4.1 Liquation Cracking Susceptibility of 347H SS .......................................................................... 18

2.4.2 Embrittlement in 347 SS Welds .................................................................................................. 21

2.4.3 Ductility Dip Cracking (“Micro fissuring”) .................................................................................. 24

2.4.4 SRC and Reheat Cracking in 347H Welds .................................................................................. 26

2.4.5 Gleeble Testing Methodologies for SRC Simulation and Contributing Factors ............... 32

2.5 Mitigation Techniques for SRC of 347H SS Welds ............................................................... 36

2.5.1 Effects of PWHT ....................................................................................................................... 36

2.5.2 Alternative Materials: Weld Consumables ............................................................................... 40

2.5.3 Alternative Materials: Substrate ............................................................................................... 43

CHAPTER 3: EXPERIMENTAL AND MODEL METHODS ................................................................. 45

3.1 Welding methodology .................................................................................................................... 47

3.1.1 Weld Setup ............................................................................................................................... 48

3.1.2 Weld parameters for all welds ................................................................................................. 48

3.2 Finite element modeling (FEM) of weld simulations .................................................................... 50

3.2.1 Model setup, including mesh and boundary conditions ......................................................... 50

3.2.2 Goldak moving heat source weld model .................................................................................. 52

3.2.3 Material properties .................................................................................................................. 54

3.2.4 Setup of PWHT model ............................................................................................................ 55
3.3 Gleeble SRC test methodology ................................................................. 56
  3.3.1 Sample Geometries .................................................................................. 57
  3.3.2 Reheat cracking Gleeble test procedure .................................................. 60
  3.3.3 Steps 1-2: HAZ thermal cycles ............................................................. 62
  3.3.4 Steps 3-4: Apply stress at room temperature .......................................... 64
  3.3.5 Step 5: Stroke calibration for heating up step at 58°C/minute .................... 65
  3.3.6 Experimental Variables ......................................................................... 66
  3.4 Gleeble FEM ............................................................................................... 67
  3.5 Gleeble Thermomechanical Testing at 600°C with and without PWHT ........ 70

CHAPTER 4: WELD EXPERIMENTAL AND FINITE ELEMENT MODEL RESULTS ...... 72
  4.1 Weld Experiment Characterization .............................................................. 72
    4.1.1 2” thick-31 pass weld (W1.1) ................................................................. 72
    4.1.2 2” thick-40 pass weld (W1.2-3) .............................................................. 75
    4.1.3 1” thick-16 pass welds (W.3 and W.4) .................................................... 77
  4.2 Weld Finite Element Modeling Results ....................................................... 82
    4.2.1 As-Welded 2” FEM Stress Results ....................................................... 83
    4.2.2 Transverse direction neutron diffraction measurements on 2” thick welds 85
    4.2.3 PWHT effect on stress in weld FEM ..................................................... 86
    4.2.4 Effect of joint geometry and plate thickness on residual stress ............... 87

CHAPTER 5: STRESS RELAXATION/REHEAT CRACK GLEEBLE RESULTS AND
  DISCUSSION ..................................................................................................... 89
  5.1 347H HAZ SRC results ................................................................................ 89
    5.1.1 HAZ microstructures prior to SRC testing .......................................... 89
    5.1.2 Reheat crack susceptibility of 347H HAZ .............................................. 91
  5.2 Gleeble FEM Results and Comparison with Experimental Results ............... 104
  5.3 E347-347H cross welded SRC results ....................................................... 115
    5.3.1 Gleeble reheat cracking results ............................................................ 115
    5.3.2 Comparison between 347H HAZ and E347 WM ................................. 117
    5.3.3 Metallurgical characterization of E347 WM samples ............................. 121
  5.4 FE Analysis of Failure Susceptibility ......................................................... 127
  5.5 NUCL 167 SPH (316L w/ B) SRC results .................................................. 130
  5.5 E16.8.2-347H cross welded SRC results .................................................... 133
LIST OF FIGURES

Figure 1.1: NREL Gen 3 Roadmap TES Schematic [1] ................................................................. 1
Figure 2.1: 347H as-received from a 1” thick plate solution annealed at 1100°C for 30 min [12] 5
Figure 2.2: STEM/HAADF images and corresponding Nb elemental maps showing Nb-C precipitation in grain boundaries (a-b) and dislocation within grains (c-d) in as-received material (solution annealed condition for a 0.04 wt% C heat) [12] ....... 5
Figure 2.3: Pseudo-binary phase diagram of 347H SS of typical composition (0.05C-0.58Nb) ... 7
Figure 2.4: Thermodynamic equilibrium solubility diagram of Nb in elevated temperature phases in 347H SS of typical composition (0.05C-0.58Nb) ......................................................... 8
Figure 2.5: CALPHAD calculated TTT of max 50% volume fraction Nb (C, N) precipitation in $\gamma$-matrix in 347H SS with starting high ($2 \times 10^{16}$ m$^{-2}$) and low ($2 \times 10^{12}$ m$^{-2}$) dislocation densities and 50% diluted WM using E309L filler and 347H composition (which may occur in arc welding along fusion line) ........................................... 8
Figure 2.6: NbC solubility diagram in $\gamma$-austenite ($\log_{10}[\text{Nb}][\text{C}]=2.26-(6770/T_{\text{K}})$) ............. 9
Figure 2.7: Calculated equilibrium weight fraction of Nb (C,N) v. temperature for 0.04 and 0.11 wt% N and 0.02-0.06 wt% C contents [13] ................................................................. 10
Figure 2.8: Maximum allowable stress values (S) from ASME BP&V code Section II-D. All of the values were taken from Table 1A for plate SA-240 designations with higher stress values. [11] ........................................................................................................ 11
Figure 2.9: Creep rupture database for 347H SS in time to rupture for constant engineering stress for service temperature (565°C) up to 816°C [14-16] .......................................................... 12
Figure 2.10: Load relaxation curves after 0.8 true compressive strain for determining recovery(rev) and recrystallization (rex) times in 347H SS for temperatures of 1150-1230°C [17]. Time from beginning to the first inflection point determines the time to recovery, while the time from the first inflection point to the second inflection point is complete recrystallization. ........................................................................ 13
Figure 2.11: Fraction of recrystallization vs. relaxation time for different temperatures in 347H SS at 1150, 1170, 1190, 1210, and 1230°C [17] ................................................................. 14
Figure 2.12: Microstructures after grain coarsening with temperatures of 1000-1270°C for 60 minutes after being hot forged [18] .................................................................................. 14
Figure 2.13: Grain growth kinetics in the specimens during different soakings [18] .................. 15
Figure 2.14: 347H SS GTAW autogenous weld (190 A, 4 IPM, ~50 kJ/in.) ............................ 17
Figure 2.15: Example of liquation crack and eutectic in spot transverse restraint and hot ductility tests and EDX intensity map indicating Nb enrichment in liquation secondary phases compared with the base metal at the crack tips [19] ......................................................... 20
Figure 2.16: Liquation detection as a function of the Cullen and Freeman parameter $(1/2\text{Nb}/(30\text{C}+50\text{N}))$ [22] ................................................................................................. 20
Figure 2.17: Total crack length measured for Varestraint tests of various 347 SS heats with different C and F parameters [21].

Figure 2.18: Total crack length (TCL) from spot transvarestraint results of 3.75% augmented strain plotted with the zero ductility temperature (ZDT) determined from Gleeble hot ductility tests [19].

Figure 2.19: a) Ductility dip in room temperature tensile tests, and b) toughness dip in room temperature Charpy V-notch tests as a function of the LMP (varying PWHT aging temperatures and times) of E347 2” thick welds with four different compositions and time [24].

Figure 2.20: Ductility contours from elevated temperature tensile tests after solution heat treatments at 1050-1325°C and subsequent aging (700-900 °C) for 18%Cr-12%Ni-1%Nb SS [28].

Figure 2.21: Fissure bend test results on shielded metal arc welding (SMAW) welds of various fillers and ferrite content [21]. The plot resembles the number of fissure counts measured optically versus ferrite number of various E3XX weld consumables.

Figure 2.22: Reheat crack (or stress relief cracking) in 347H SS weld metal [9].

Figure 2.23: Stress relaxation cracking failure in superheat tubes by Xcel Energy [34].

Figure 2.24: SRC crack in 347H SS after 6 months of service at 600-650°C with a) presence of metallic filament and Cr-rich oxides and b) creep cavity formation ahead of SRC crack in [8].

Figure 2.25: Schematic of PFZ along grain boundaries due to solute depletion [4].

Figure 2.26: SEM image showing PFZ in Inconel 740H weld metal near the grain boundary. The mismatch of strengthening mechanisms enhance the susceptibility to SRC in PFZ region [33].

Figure 2.27: STEM-EDS maps of Cr and Nb near a creep cavity ahead of a toe crack in the fillet of a 347H weld showing Cr depletion and Cr rich carbide (likely Cr23C6) along a grain boundary with the presence of a Nb rich carbonitride (likely Nb (C, N)) and a PFZ less than 500 nm wide [35]. Image used with permission of collaborative research study funded by the Electric Power Research Institute and executed by Dr. Geoff West of Warwick University.

Figure 2.28: Macro crack in a) HAZ, b) closeup of crack, c) region of dark bands representing α’ martensite [36].

Figure 2.29: 347H SS Secondary intergranular cracks ahead of fracture surface a) under 800°C and 10% plastic strain and b) under 700°C and 10% plastic strain, and fractography analysis using SEM for 700°C and 10% plastic strain case: c) mixed-mode fracture surface showing micro voids on surface with grain facets, and d) void along grain boundaries [6].

Figure 2.30: Literature survey of SRC susceptibility curves reported as temperature vs. time from various SRC tests, and service failures [6, 9, 38].

Figure 2.31: SRC Gleeble methodology developed by Lehigh University [6].
Figure 2.32: Stress relief cracking results for 347H with 10% CW condition [6]........................ 35
Figure 2.33: SRC susceptibility for various materials, stress, and temperature conditions [6].... 35
Figure 2.34: a) Effect of temperature (at one hour approximation) on stress relief of Type 347 steel [42, 43], and b) stress relaxation measurements for the weld centerline of 1” pipe 0.84 inch thick [38, 41]................................................................. 38
Figure 2.35: Multi-step PWHT overlayed on crack and precipitation c-curves [2, 3, 38]........... 39
Figure 2.36: Example of 3-step PWHT treatment used for thick 347H SS welds [44]............ 39
Figure 2.37: Schaefer Delong diagram with Etsy Ni\textsubscript{eq} equation and experimental composition points and boxes representative of “acceptable” composition window for E16.8.2 [46] ........................................................................................................................................ 42
Figure 2.38: Charpy impact toughness in AW condition, HT-4 hrs., and HT-168 hours for four different cross welded sets. The 347H welds were aged at 1650°F (900°C) [40].............. 43
Figure 2.39: Relationship between Charpy impact energy and lateral expansion of E16-8-2T, E308LT, E316LT, and E316LMn T FCAW weld metals at -196°C [46] ............................ 43
Figure 2.40: Pseudo-binary phase diagram of modified 316L predicted from CALPHAD simulations using Thermocalc© software (B~ 0.0008 wt%) ..................................................... 44
Figure 3.1 Critical path of experimental and modeling tasks for answering research questions. Welding experiments were first completed to help complete the other tasks in the project, including the Gleeble experiments, needed to gather the critical stress/strain maps for PWHT and weld design.................................................................................................................. 46
Figure 3.2 SolidWorks Assembly of the experimental setup of a 2” thick weld....................... 48
Figure 3.3 Closeup SolidWorks cross section of weld and backing bar showing machined grooves for water lines and gas lines for back purging with Argon gas......................... 48
Figure 3.4 (a) FE model mesh of whole 12” x 12” model, (b) meshes of weld zone separated by pass number, and (c) cross section of weld zone of 40-pass experimental sample... 50
Figure 3.5 Boundary conditions for (a) clamped and (b) unclamped models ....................... 52
Figure 3.6 (a) Goldak heat source model [52] and (b) example temperature contour during welding simulation.................................................................................................................. 53
Figure 3.7 Temperature profiles experienced by (a) the whole plate, and (b) a cross sectional view and (c) three nodes as marked in (b) during pass 31 of the 40-pass weld........ 53
Figure 3.8 True stress strain curves used in the materials database using Gleeble data (black and red) and literature data (grey) [14]................................................................. 55
Figure 3.9 Flow chart of stress and strain calculation during the PWHT simulation using creep law in ABAQUS.................................................................................................................. 56
Figure 3.10 (a) Gleeble experiment setup using stainless steel (SS) grips and c-gauge, and (b) geometry of Gleeble cylindrical samples adapted from ASTM E8 sub-size requirements [61]........................................................................................................ 58
Figure 3.11 Gleeble sample extracted from 1” thick weld in transverse direction................. 59
Figure 3.12 Dimensions of the dog-bone flat tensile sample for NUCL 167 SPH samples (inches)
........................................................................................................................................... 60

Figure 3.13 Six step Gleeble reheat crack test methodology displayed with stress, stroke, and
temperature as a function of time for HAZ simulations (1335°C HAZ peak
temperature, 0.174 pre-strain, and 800°C reheat temperature condition shown).
(NOTE: WM tests use the same methodology, except steps 1-2 (HAZ thermal cycle))
........................................................................................................................................... 61

Figure 3.14 Thermal cycles used for generating HAZ microstructures in 347H with 1150,1275,
and 1335 °C peak temperature (details listed on steps 1-2 for reheat crack test)...... 62

Figure 3.15 Continuous cooling transformation (CCT) curve calculations, using Thermocalc®,
showing three different cooling rates, including the 347H HAZ cooling rate of
23°C/s with respect to a 0.001 volume fraction detection of grain boundary (GB) Nb
(C,N) and dislocation Nb (C,N) precipitation in γ-austenite using 347H experimental
composition......................................................................................................................... 63

Figure 3.16 Room temperature true stress vs. true strain results for all four sets of materials..... 65

Figure 3.17 Stroke vs. temperature under a zero-force condition for all experimental Gleeble
samples to account for thermal expansion................................................................. 66

Figure 3.18 FEA simulation model of Gleeble test sample........................................... 68

Figure 3.19 Example of creep strain and strain rate collected from c-gauge during steady state
creep at isothermal holding step for 750°C/ 0.174 strain condition (sample 5)....... 69

Figure 3.20 Creep data from Gleeble experiments used for FEM................................. 70

Figure 3.21 Thermomechanical test heat treatments prior to tensile test at 600°C ............ 71

Figure 4.1 Microstructure representation of 2”- 31-pass weld: (a) macro cross section of weld,
(b) FZ microstructure of pass 30 indicating columnar morphology , (c)
microstructure of intersection between pass 26 and 30 showing reduction of ferrite in
pass 30 due to reheating effects, (c) transition microstructure of FZ and HAZ of pass
26, and (d) FZ microstructure of root pass showing equiaxed microstructure. ....... 73

Figure 4.2 Ferrite scan across the top layer of 31pass-2” thick weld ............................... 74

Figure 4.3 HAZ-BM microstructure adjacent to pass 29 going from the FZ boundary, CGHAZ
(1) to the BM (10)................................................................................................................. 75

Figure 4.4 Example of weld (W.1-2) a) after completion of root pass from top view, b) top view
after completion of pass 40, and c) side view after completion of 40 passes showing
angular deformation (measured to be 14°) ................................................................. 76

Figure 4.5 W.1-3 microstructure after PWHT (875°C/3 hours) of (a) bulk 40 pass weld, and (b)
intersection between top layer and second sub-surface layer............................... 77

Figure 4.6 Cross section of E347-347H SS weld (W.4-4) with Gleeble sample extraction
location outlined (top) and microstructure reconstruction in gauge section of Gleeble
samples (bottom) (TD=transverse direction; ND= normal direction;
LD=Longitudinal direction)............................................................................................... 78
Figure 4.7 Cross section of E16.8.2-347H SS weld (W.3-1) with Gleeble sample extraction location outlined (top) and microstructure reconstruction in gauge section of Gleeble samples (bottom) (TD=transverse direction; ND= normal direction; LD=Longitudinal direction). ................................................................. 78

Figure 4.8 Weld morphology of center of weld metal in pass 15 (representative of the higher ferrite regions) in both (a) W.4-4 (E347) weld and (b) W.3-1 (E16.8.2). The δ-ferrite etches dark and γ-austenite is the light phase. .......................................................................................... 79

Figure 4.9 WRC 1992 diagram [64] with predicted points for undiluted weld metal and 347H base metal (batch #2) .......................................................................................................................... 80

Figure 4.10 Average hardness profile (five data points) from BM to FZ (pass 15) for both E347 (W.4-4) and E16.8.2 (W.3-1) welds ........................................................................................................... 81

Figure 4.11 Effect of E347 multi-pass microstructure on microhardness in passes 9 and 11 (gauge section of Gleeble samples): (a) Multi-pass microstructure of W4.4 passes 9 and 11 showing three diamond Vickers’s indent rows spaced 150µm from each other, and (b) microhardness contour map corresponding to measurements using a 300 g load. Microhardness reached peak hardness values of ~240 HV in upper regions of pass 9 and lower δ-ferrite regions of reheated pass 9 and pass 10 (outlined by red box). ....................................................................................... 82

Figure 4.12 As-welded von Mises residual stress (MPa) and three principal stress directions in clamped (a-d) and unclamped (e-h) condition for the rear left quarter of the model. x (i.e., 11), y (i.e., 22) and z (i.e., 33) axis represents transverse, longitudinal and normal direction, respectively. .............................................................................. 84

Figure 4.13 Effective plastic strain and three principal strains in the as-welded condition in unclamped (a-d) condition for the rear left quarter of the model. x (i.e., 11), y (i.e., 22) and z (i.e., 33) axis represents TD, LD, and ND, respectively ...................................................................................... 85

Figure 4.14 TD elastic strain comparison for the TD (ε11) in the (a) FEM and (b) neutron diffraction experiments. Orders of strain are comparable, with peak tensile strains near root and compressive strain in the mid-thickness ................................................. 86

Figure 4.15 (a) Temperature distribution upon reaching 950°C on surface, (b) von Mises stress contour upon reaching 950°C (MPa), (c) overall effective plastic strain contour upon reaching 950°C, (d) von Mises stress history of high stress area (circled location in contours) for the duration of 4 hour PWHT at 950°C and cooling and (d) strain history of the high stress area for the duration of the whole PWHT ...................... 87

Figure 4.16 2D stress contours of welds with different conditions: (a) 2 inch thick plate with a single-V groove; (b) 0.5 inch plate with a single-V groove; and 1 inch plate with a (c) single-V groove, (d) double-V groove, and (e) J groove ........................................ 88

Figure 5.1 (a) 347H SS base metal of location with Nb-rich oriented precipitates (unetched-SEI) (b) Liquation evidence after 1335°C peak HAZ thermal cycle and 600 °C for 9 hours (electro etched-SEI) .................................................................................. 90

Figure 5.2 Liquation evidence during the HAZ thermal cycle (a) After a 1335°C peak temperature CGHAZ simulation only (un-etched polarized LOM), and (b) Specimen
3 (see Table 5.1) after completion of SRC test for 9 h at 600°C (center of specimen (electro etched)-FESEM SEI. The microstructures are representative of an eutectic Nb (C,N)/γ microstructure due to grain boundary liquation, with light phase being Nb (C,N) and surrounding darker grains being γ-austenite. Figure 5.3 EDS map of Nb(C,N)/γ-austenite liquation microstructure due to 1335°C HAZ, (a) SEI image, (b) Fe map, (c) Nb map, (d) Cr map, (e) Ni map (need to include in results as the first results to show).

Figure 5.4 Comparison of different pre-strains for 950°C Gleeble tests, showing failure at 0.08 (S35) and 0.1 strains (S12), with no failure during cooling from 0.05 strain test (S34). Figure 5.5 Comparison of different pre-strains for 1050°C Gleeble tests, showing failure at 0.05 (S28) and 0.1 strains (S13), with no failure during cooling from 0.04 (S33), 0.025 (S32), and 0.01 (S31) strain tests.

Figure 5.6 Stress relaxation curves of the 347H HAZ samples to show the effect of temperature and pre-strain on stress evolution during step 6 for 550-1050°C temperature conditions.

Figure 5.7 Summary of single-step SRC susceptibility curves for 347H HAZ with three variables including stress/strain, temperature, and peak HAZ temperature.

Figure 5.8 Susceptibility map as a function of initial stress at room temperature in step 3 and testing temperature for 347H HAZ samples.

Figure 5.9 Susceptibility map as a function of starting stress at temperature in step 6 and testing temperature for 347H HAZ samples with corresponding critical samples at 800, 950 and 1050°C showing presence of microcracks and void formation along grain boundaries.

Figure 5.10 Presence of micro voids on GB (a) in the center of the gauge section of specimen 10 (800°C/0.1 strain). FESM SEI image (20 kV and 8.5mm WD) and (b) voids along with polarized image of microcracks on GB with TRIP α’ martensite surrounding the microcracks and presence of liquation (Nb (C, N)/γ eutectic microstructures) within vicinity of the microcracks. Tensile axis is shown to be perpendicular to the crack growth direction.

Figure 5.11 Comparison of microhardness in a variety of microstructures of 347H SS.

Figure 5.12 Fracture surfaces in center of cross section of 0.174 strain conditions for (a-b) 800°C, (c) 750°C (fracture parallel to Nb-rich oriented particles), and (d) 850°C samples.

Figure 5.13 Representation of unetched secondary cracks of S5 (800°C-0.174 strain) showing coarse intergranular GB Nb (C, N) along crack path ahead of primary fracture surfaces for sub-crack 1 (a-b) and sub-crack 2 (c-d).

Figure 5.14 900°C with 0.174 strain. (a) edge of sample, (b) center of sample. Failed at 5.8 min after reaching 900 °C. FESEM, electroetched with 50% nitric acid solution: 900°C with 0.1 strain. (c) edge of sample, (d) center of sample failed at 31 min after reaching 900 °C.SEM-SEI, electroetched with 50% nitric acid solution.
Figure 5.15 EDS Line scan across Nb (C, N) of on grain boundary close to primary fracture surface of Sample 8 (900°C/0.174 pre-strain) ................................................................. 104

Figure 5.16 Stress and temperature contours for an example of a 0.1 pre-strained sample heated up to 1050°C ............................................................... 105

Figure 5.17 Stress and temperature evolution in center of gauge section during Gleeble test with a pre-strain of 0.08 (~415 MPa) and peak temperature of 900 °C, including FEM stress contours and stress validation with experiment ........................................... 106

Figure 5.18 Original mesh and example of stress and temperature evolution during Gleeble test with a pre-strain of 0.08 (~415 MPa) and peak temperature of 900 °C (sample 36) with stress contours at each step ............................................................... 108

Figure 5.19 Plastic, thermal, and creep strain development from steps 3-6 for 900°C and 0.08 pre-strain (sample 36): (a) plastic strain contours at each step and (b) calculation of strain components of sample in center of gauge section as a function of time ...... 109

Figure 5.20 (a) Comparison of stress evolution between FE prediction and Gleeble experimental results and (b) calculation of strain components in center of gauge section as a function of time for 950°C 0.08 pre-strain condition (sample 35) .......................... 110

Figure 5.21 Susceptibility map as a function of corrected pre-strain from Gleeble FEM predictions and temperature ........................................................................... 114

Figure 5.22 SRC susceptibility curves of E347-347H cross welded samples (all failures in WM) with various starting stresses (legend) and stress upon reaching temperature (labeled) for first batch of welds ........................................................................................................ 117

Figure 5.23 Starting stress at room temperature (step 3) vs. temperature for all samples, including failure at temp., failure upon cooling, and no failure ......................................................... 118

Figure 5.24 Stress upon reaching temperature (step 6) vs. temperature for all samples, including failure at temp., failure upon cooling, and no failure ......................................................... 118

Figure 5.25 Initial stress (step 3) vs. LMP for E347 WM and 347H HAZ samples ................. 119

Figure 5.26 Stress at elevated temperature (step 6) vs. LMP for E347 WM and 347H HAZ samples .................................................................................................................... 120

Figure 5.27 Stress relaxation percentage during heating for all Gleeble samples as a function of temperature .............................................................................. 121

Figure 5.28 Example of ductility dip crack (DDC) in reheated weld metal in 1000°C test with lowest stress condition (G.W.30) ................................................................................. 122

Figure 5.29 Fracture surfaces of 950°C sample with 518 MPa initial stress and 71 MPa stress at temperature (G.W.3) relative to the columnar orientation of the weld morphology and applied stress .................................................................................................................... 123

Figure 5.30 Microcracks in interdendritic regions of columnar solidification grains for 950°C sample with 437/75 MPa stress values (G.W.9) that failed at temperature .............. 124

Figure 5.31 Microcracks in interdendritic regions of columnar solidification grains for 800°C sample with 523/144 MPa stress values (G.W.6) that failed on cooling with some
indication of darker phase near the microcracks. The arrows are pointing to a darker etched phase (darker than δ-ferrite), which is likely sigma.  

Figure 5.32 EBSD representation of macro cracks in WM of GW24 (1050°C): (a) micrograph of microcrack, (b) inverse pole figure (IPF) map, (c) phase identification map, and (d) grain color map.

Figure 5.33 Histogram of misorientation angles between grain crack and no crack grain boundaries from EBSD IPF map of GW24 sample showing cracking between “high angle” solidification grain boundaries.

Figure 5.34 (a) 2-D von Mises and (b) plastic strain contours for high stress region area from FE simulation of 2” weld in unclamped condition combined with (c) initial stress vs. temperature susceptibility map of E347 WM and 347H HAZ.

Figure 5.35 Comparison of stress in (a) FEM simulation with (b) literature FEM [69] and (c) example of service failure in dissimilar weld (DSW) of stainless steel and P91 in similar location [70].

Figure 5.36 (a) 2D Von Mises stress and (b) plastic strain contours after reaching 950°C during PWHT simulation of unclamped condition combined with (c) stress at temperature vs. temperature susceptibility map of E347 WM and 347H HAZ.

Figure 5.37 Stress evolution as a function of temperature and time for three pre-strains of 0.165, 0.085, and 0.07 at 950°C for NUCL 167 SPH samples (no cracking).

Figure 5.38 Comparison of stress evolution as a function of temperature and time among HAZ of 347H and NUCL 167 SPH simulated with 1335°C peak temp and at a few similar starting stresses at room temperature (step 3).

Figure 5.39 Polarized LOM of electroetch (2V) with 40% nitric acid solution of 0.085 strain sample at 950°C (half width, center of gauge section) with signs of varying grain sizes and liquation along the rolling bands (due to HAZ thermal cycle).

Figure 5.40 Representation of cracks in E16.8.347H cross welded samples for Mo7, Mo11 and Mo10, including HAZ microcracks.

Figure 5.41 SRC testing results of E16.8.2-347H cross welded SRC samples with 6% strain showing failure in HAZ under 900 and 950°C, but surface cracks only in WM under 950 and 1050°C (NOTE: temperature of 347H HAZ is approximately 50°C lower than peak temperature in center of gauge sample).

Figure 5.42 Fracture surface analysis of Mo8 sample (950°C/6% strain) that failed in 347H HAZ where Top represents area closer to top surface of weld and Bottom represents more subsurface region.

Figure 5.43 Comparison of fracture surfaces of representative E347-347H cross welded SRC samples with fracture in the WM only, and those of E16.8.2-347H cross welded SRC samples with failure in the HAZ. All images have same scale.

Figure 5.44 LOM polarized image with 0.05µm colloidal silica polish of E16.8.2 WM in 1050°C, 6% strain sample (SSGB=solidification sub grain boundary;
SGB= solidification grain boundary). Cracks seem to propagate along higher energy SGB.

Figure 5.45 Comparison of stress relaxation between E16.8.2 (Mo6) and E347 (GW7) cross welded samples during 850°C, 1% strain test. Failure occurs in E347, but not in E16.8.2.

Figure 5.46 Comparison of stress relaxation between E16.8.2 (Mo5) and E347 (GW3) cross welded samples during 950°C, 1% strain test. Failure occurs in E347, but not in E16.8.2 after 2 hours and during cooling.

Figure 6.1 Comparison of 347H HAZ and E347-16 WM tensile test behavior at 600°C with and without PWHT (two conditions including 950°C/2 hr. and 1050°C/30 min). Two-step PWHT (750°C-2hr/1050°C-30min) and one test with an overheating PWHT to 1200°C is included for 347H HAZ.

Figure 6.2 Comparison of E347-16 and E16.8.2-15 WM tensile test behavior at 600°C with and without PWHT (two conditions including 950°C/2 hr. and 1050°C/30 min)

Figure 6.3 Comparison of NUCL 167 SPH (noted as 316L in the plot) HAZ and E16.8.2-15 WM tensile test behavior at 600°C with and without PWHT (both 950°C/2 hr. and 1050°C/30 min).

Figure 6.4 Comparison of 347H HAZ and NUCL 167 SPH (316L) HAZ tensile test behavior at 600°C with and without PWHT (both 950°C/2 hr. and 1050°C/30 min). Two-step PWHT (750°C-2hr/1050°C-30min) and one test with an overheating PWHT to 1200°C is included for 347H HAZ.

Figure 8.1 (a) Von Mises stress contour under as-welded condition after unclamping; (b) Temperature field after 4-hour holding at 950°C, (c) Stress relief along the heating with different heating rate. Solid and open symbols show failure and no failure at certain temperatures and stresses in experiment, respectively.

Figure A.1 Welding Procedure Specification (WPS) developed by Brahma Inc. used for hot tank seam/circumferential welds.

Figure A.2 PQR_81 used for developing WPS (3/8” weld).

Figure A.3 PQR_82 used for developing WPS (1” weld).

Figure B.1 Setup of 2” thick welds for HB-2B beamline at ORNL.

Figure B.2 A) 4 mm thick slice cut from bulk weld plate, b) 4mm thin sliver showing the relative 3 lines of interest, and C) comb shape assembly (x 2 combs) from each line.

Figure B.3 A) weld cross section showing the three sets of stress-free comb shapes from the 31-pass weld with the center of the cubes being the center of the beam locations.

Figure B.4 do-spacing of reference samples for all 3 lines as calculated from Bragg’s law based on the measured peak θ.

Figure B.5 Best case diffraction peak for TD showing low signal-to-noise ratios and fit to determine peak d-spacing value.

Figure B.6 d-spacing measurements of TD orientation (311 planes) for Line 1- top of weld.
Figure B.7 d-spacing measurements of TD orientation (311 planes) for Line 2-mid thickness. 162
Figure B.8 d-spacing measurements of TD orientation (311 planes) for Line 3-root .................. 163
Figure B.9 Calculated ε311, TD for AW and PWHT conditions for all three lines of data. The reference $d_0$ values are averaged for three regions each in WM, HAZ, and BM, with each line representing unique values dependent on values from Figure B.4......... 163
LIST OF TABLES

Table 2.1: Nominal composition of 347H alloy according to ASTM A240 [10] and composition of Gleeble samples (hot rolled plate) and all E347 WM .......................................................... 4
Table 2.2: Number of references corresponding to SRC failure in service [3] ......................... 16
Table 2.3: Summary of thermomechanical weldability Issues in 347H SS ......................... 17
Table 2.4: Summary of a variety of PWHT schedules using multiple steps from literature ...... 40
Table 2.5: Composition comparison in all weld metal of three electrodes that are candidates for welding of 347H SS substrates ................................................................. 41
Table 3.1 Chemical composition of main alloying elements of all experimental materials (including substrates and consumables) ............................................................. 45
Table 3.2 Welding experimental matrix (MA=metallurgical analysis; GE=Gleeble extraction; ND=neutron diffraction; ref ND-reference sample extraction for neutron diffraction) ............................................................. 47
Table 3.3 Weld parameters for 31-pass W.1-1 .......................................................................... 49
Table 3.4 Welding parameters for 2”-40 pass welds W1.2-3 and parameters used for 1”-16 pass welds W.3-W.4 ........................................................................................................... 49
Table 3.5 Material temperature dependent mechanical and thermal properties [53-55] ........ 54
Table 3.6 Stroke calibration values used for heating step for both 347H HAZ and E347/E16.8.2 WM SRC tests ................................................................................................. 66
Table 3.7 Creep parameters obtained from Gleeble experiments for temperatures ranging from 625-1075°C (grey-interpolated; white-experimentally fitted). Q-activation energy (assumed constant), R-gas constant ................................................................. 70
Table 5.1 Summary of Gleeble 347H SS HAZ SRC test variables and results (samples not listed were used for thermomechanical tensile tests) ...................................................... 95
Table 5.2 Strain components for three pre-strains of 0.1, 0.08, and 0.05 and testing temperatures at 1050, 950, and 900°C after step 3. LE=total strain; PE=plastic strain; EE=elastic strain; THE=thermal strain; CE=creep strain ................................................................. 111
Table 5.3 Strain components for three pre-strains of 0.1, 0.08, and 0.05 and testing temperatures at 1050, 950, and 900°C after 2 hours at step 6 (LE=total strain; PE=plastic strain; EE=elastic strain; THE=thermal strain; CE=creep strain) ................................................................. 111
Table 5.4 True plastic strain increase after heating step for all nine simulations ................ 112
Table 5.5 Summary of 347H SS SRC HAZ Gleeble results with updated true plastic strain from Gleeble model (blue highlights represent critical strain/stress) .......................... 113
Table 5.6 Summary of E347-16 WM Gleeble SRC test results (blue highlights represent critical stress values for those temperatures) ................................................................. 115
Table 5.7 Summarized results of E16.8.2-15-347H cross welded SRC Gleeble samples ....... 133
Table 6.1 Thermomechanical data of sample with and without PWHT pulled at 600°C after 4 hours service age (grey boxes represent two-step PWHT tests).............................. 139

Table C.1 Publication permissions given for figures in text from external sources (CCC-Copyright Clearance Center) and associated supplemental file names submitted in ProQuest .............................................................. 164
## LIST OF ABBREVIATIONS

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>CSM</td>
<td>Colorado School of Mines</td>
</tr>
<tr>
<td>CSP</td>
<td>concentrating solar power</td>
</tr>
<tr>
<td>CW</td>
<td>cold working</td>
</tr>
<tr>
<td>DDC</td>
<td>ductility dip cracking</td>
</tr>
<tr>
<td>DOE</td>
<td>Department of Energy</td>
</tr>
<tr>
<td>EDS</td>
<td>electron dispersive spectroscopy</td>
</tr>
<tr>
<td>ESEM</td>
<td>environmental scanning electron microscope</td>
</tr>
<tr>
<td>FEM</td>
<td>finite element model</td>
</tr>
<tr>
<td>FESEM</td>
<td>field emission scanning electron microscope</td>
</tr>
<tr>
<td>FZ</td>
<td>fusion zone</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>LMP</td>
<td>Larson Miller Parameter</td>
</tr>
<tr>
<td>LOM</td>
<td>light optical microscope</td>
</tr>
<tr>
<td>MGB</td>
<td>migrated grain boundary</td>
</tr>
<tr>
<td>NDE</td>
<td>non-destructive evaluation</td>
</tr>
<tr>
<td>NREL</td>
<td>National Renewable Energy Laboratory</td>
</tr>
<tr>
<td>ORNL</td>
<td>Oak Ridge National Energy Laboratory</td>
</tr>
<tr>
<td>PWHT</td>
<td>post weld heat treatment</td>
</tr>
<tr>
<td>SGB</td>
<td>solidification grain boundary</td>
</tr>
<tr>
<td>SMAW</td>
<td>shielded metal arc welding (stick welding)</td>
</tr>
<tr>
<td>SSGB</td>
<td>solidification sub-grain boundary</td>
</tr>
<tr>
<td>SS</td>
<td>stainless steel</td>
</tr>
<tr>
<td>TES</td>
<td>thermal energy storage</td>
</tr>
<tr>
<td>TTT</td>
<td>time-temperature-transformation</td>
</tr>
<tr>
<td>UTS</td>
<td>ultimate tensile strength</td>
</tr>
<tr>
<td>WM</td>
<td>weld metal</td>
</tr>
<tr>
<td>WPS</td>
<td>welding procedure specification</td>
</tr>
<tr>
<td>WSRF</td>
<td>weld strength reduction factor</td>
</tr>
<tr>
<td>YS</td>
<td>yield strength</td>
</tr>
</tbody>
</table>
ACKNOWLEDGMENTS

I would like to first and foremost thank my advisor, Dr. Zhenzhen Yu, for giving me the opportunity to work on this project in the Center for Welding, Joining, and Coatings Research (CWJCR). Her guidance and willingness to be available for many technical discussions have helped me to learn and have a wonderful graduate school experience. I thank her for encouraging me to finish a master’s degree as well as continuing as a PhD student under her advice.

I would also like to thank Dr. Judith Vidal from the National Renewable Energy Laboratory (NREL) for being the principal investigator of this project and for being a huge support and for participating in my committee. Dr. Chad Augustine, from NREL, also has helped manage the latter portions of this project, and I thank him for his support. I would also like to thank Dr. Stephen Liu and Dr. Kip Findley for agreeing to be on my committee and for contributing knowledge to this thesis. I have learned a lot from their expertise and courses.

I would like to also thank Dr. Yu Hong for providing help with the FEA simulation results that greatly contributed to the understanding of residual stress of our experimental welds and Gleeble simulations. I’d like to thank Karsten Anderson and Sarah Harling for their contribution to helping with the welding experiments, as their steady hands have helped produce high quality welds for this research.

I would like to thank other students and professors in the CWJCR for providing input to my project, including Dr. Jonah Klemm-Toole, Ethan Sullivan, Alexander Hansen, Abdelrahman Abdelmotagaly and many more. I’d like to thank Benjamin Schneiderman for traveling with me to assist in initial neutron diffraction experiments at ORNL. I’d like to thank all my friends and peers in the MME department for helping me in times where I needed assistance in both technical discussion and characterization, including Travis Marsh and Diptak Bhattacharya.

I want to give a huge thanks to my friends and family back home in Texas and California, including my eight siblings, mom, dad, and extended family. I love them dearly and am glad to have had their support my whole life. Travis Marsh and Alex Gonzales have been wonderful friends to live with during COVID-19, a very challenging time for everyone.

This research is supported by the Department of Energy with award # DE-EE00033458 and the National Renewable Energy Laboratory. This research used resources of the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory (ORNL). Many thanks to Dr. Jeff Bunn, Dr. Andrew Payzant, and Paris Cornwell for their assistance at the HIDRA beamline.
CHAPTER 1: INTRODUCTION

The energy industry has adopted a thermal energy storage (TES) system as a functioning form of renewable and clean energy in concentrating solar plants (CSP). Specifically, the National Renewable Energy Laboratory (NREL) has incorporated a system of solar salt hot-and-cold tanks for industry (e.g., 110 MW Crescent Dunes Solar Energy Project in Tonopah, NV) as seen in Figure 1.1 [1]. These molten nitrate salt thermal energy storage tanks are of considerable interest, because they allow for electricity generation when the sun is not shining, increasing the plant capacity factor, utilization of capital equipment, and total annual generation and reducing energy costs [1]. Due to the nature of the molten salt, the current temperatures experienced in the hot tanks during service are approximately 565 °C.

![Figure 1.1: NREL Gen 3 Roadmap TES Schematic [1]](image)

High temperature molten salts pose a potential corrosion issue. Therefore, current material selection for both the cold and hot TES tanks is 347H stabilized stainless steel (SS), since it is more resistive to sensitization (intergranular corrosion) and maintains better elevated temperature strength than other 300 series stainless steel grades (e.g. 304H), while being cheaper than Ni-based alloys [2]. However, weld fabrication for 347H SS and other austenitic stainless-steel grades incur significant amounts of residual stress, especially for thick sections, due to mechanical constraint and high coefficients of thermal expansion and contraction during weld thermal cycles. In addition, a constant high operating temperature contributes to potential, slow
long-range precipitation kinetics of the secondary phase, Nb (C, N), that may further increase the susceptibility of service-related issues, such as stress relaxation cracking (SRC).

The concern for SRC in 347H SS has been validated by multiple sources from both welds in service (multiple applications) and simulation of SRC conditions in labs [2-6]. There are multiple root causes that can accelerate the SRC mechanisms, namely stress, whether it be internal (residual stress), external applied pressure or a combination of both. Elevated temperatures, where creep strain may develop, and a susceptible microstructure (both the heat affected zone (HAZ) and E347 weld metal (WM)) combined with sufficient stress leads to SRC. The time to failure depends on the specific component (accounting for temperature, stress, and location) which can take a few months to a few years for this catastrophic failure to unfold [4].

Understanding general weldability (cracking susceptibility) of 347H SS is of importance; however, this project has a narrow focus on SRC and reheat cracking mechanisms.

The overarching goal of this project is to recommend SRC mitigation protocols to avoid failure of 347H welded manufacturing components, particularly hot tanks, in generation 2 concentrating solar power (CSP) plants. The specific objectives include:

1. Determine the susceptibility of 347/347H stainless steel (SS) and welds to SRC.
2. Evaluate welding protocols and post-processing, including post weld heat treatment (PWHT), employed for tank constructions that could increase SRC susceptibility.
3. Test new alloys and weld consumables commercially available that are resistant to SRC under the required operating conditions for service.

The hypothesis of this project is that weld parameter optimization and design, PWHT and alternative fillers (i.e., E16.8.2) are the most practical mitigation solutions to prevent SRC during elevated temperature service. However, PWHT is not a simple one-step procedure. PWHT may introduce reheat cracking if the heating rate, stress relaxation rate, temperatures and residual stresses are not carefully designed and analyzed as reported in industry and literature [7-9]. The research questions to be answered through this project are:

- What are the critical/threshold residual strain/strain level as a function of PWHT temperatures in 347H SS HAZ, E347 filler metal, E16.8.2 alternative filler, and alternative substrate compositions?
- What is the correlation between welding design and residual stress evolution?
CHAPTER 2: LITERATURE REVIEW

The background section consists of a broad literature review, including the weldability of 347H SS, metallurgical phenomena associated with 347H SS, effect of PWHT, and alternative materials that are less susceptible to SRC. The base microstructure of the 347H SS alloy, including CALPHAD simulations, are introduced first to explain the effect of temperature and composition on microstructure. A section on elevated temperature mechanical properties is then discussed to justify the use of 347H SS as a structural alloy for elevated temperature conditions compared to other 3XX SS grades. Then, a main section on weldability issues (i.e., susceptibility to cracking) of 347H SS is discussed, including all the potential cracking mechanisms and existing laboratory-scale testing methodology utilized to evaluate crack susceptibility. Following on weldability issues is a section on the corresponding mitigation solutions, particularly for SRC. The main mitigation approaches addressed are the use of PWHT and alternative weld consumables (for both existing and future tanks) and alternative plate grades (for construction of future new tanks).

2.1 Composition and Microstructure of 347H SS

The nominal alloying composition of AISI types 347 and 347H stainless steel listed in Table 2.1 corresponds to the allowable content in its definition space [10]. The “H” designation stands for carbon wt% higher than standard 347 SS (0.1 vs 0.08). The Nb content must be approximately eight times the carbon and nitrogen content in wt% in 347H SS, while ten times the carbon and nitrogen in standard 347 SS, with a maximum of 1% Nb [10]. 347H is intended for elevated temperature conditions above 540°C (up to 816°C max for section VIII applications) because of its exceptional maximum allowable stress (i.e. 109 MPa at 575°C when only accounting for short time yield strength but 92 MPa when accounting for creep from ASME BP&V Section II-D [11]). According to the same code, the maximum allowable stress in welded pipes would need a weld efficiency factor, strength of welded joint relative to the substrate strength, of 0.8-0.85 for single-V butt joints (~78 MPa peak allowable stress at 575°C). When external stresses during service (fluid pressures) are combined with weld induced residual stresses, the internal effective stresses could exceed the maximum allowable stresses during service temperatures. Minimizing residual stresses is critical to prevent stresses during service from approaching the designed mechanical strength for the corresponding thickness.
Table 2.1: Nominal composition of 347H alloy according to ASTM A240 [10] and composition of Gleeble samples (hot rolled plate) and all E347 WM

<table>
<thead>
<tr>
<th>wt %</th>
<th>C</th>
<th>N</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Mn</th>
<th>Nb</th>
<th>Si</th>
<th>S</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>347H ASTM A240</td>
<td>0.04-0.1</td>
<td>-</td>
<td>9-13</td>
<td>17-19</td>
<td>-</td>
<td>2</td>
<td>8 x (C+N), 1 max</td>
<td>0.75</td>
<td>0.03</td>
<td>0.045</td>
</tr>
<tr>
<td>347 ASTM A240</td>
<td>0.08</td>
<td>-</td>
<td>9-13</td>
<td>17-19</td>
<td>-</td>
<td>2</td>
<td>10 x (C+N), 1 max</td>
<td>0.75</td>
<td>0.03</td>
<td>0.045</td>
</tr>
<tr>
<td>Gleeble samples</td>
<td>0.05</td>
<td>0.03</td>
<td>9.1</td>
<td>17.3</td>
<td>0.3</td>
<td>1</td>
<td>0.58</td>
<td>0.5</td>
<td></td>
<td></td>
</tr>
<tr>
<td>E347 WM</td>
<td>0.03</td>
<td>0.03</td>
<td>10.1</td>
<td>19.5</td>
<td>0.2</td>
<td>1.5</td>
<td>0.36</td>
<td>0.5</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

1. Material Test Report (MTR) from Sandmeyer Steel Company  
2. Excalibur E347-16 all weld metal composition from Lincoln Electric Holdings, Inc.

The base microstructure of 347H typically consists of primary equiaxed austenite grains with secondary Nb (C,N) phase present along grain boundaries as shown in Figure 2.1, which is obtained from a 1” thick plate solution annealed at 1100°C for 30 min [12]. After holding at that temperature for 30 minutes, the plate is water quenched to room temperature to prevent M_{23}C_6 carbides forming along grain boundaries, i.e., sensitization that leads to loss of corrosion resistance along GB. The hot rolled annealed condition for plates is often recommended prior to welding of 347H plates. However, some cold working develops when rolling the plates into a round shape to meet the design specifications of the tank shell. A cold worked (CW) microstructure up to 10% has been reported to increase SRC susceptibility at 700-900°C by leading to more rapid intragranular Nb (C,N) nucleation, due to more nucleation sites with a higher dislocation density, upon reheating without sufficient strain recovery due to precipitate pinning of dislocations [6]. Strain distributes then along grain boundaries and increases intergranular crack susceptibility.

The addition of Nb allows for high temperature Nb (C, N) formation prior to the formation of M_{23}C_6. Scanning transmission electron microscopy (STEM) imaging of the dislocation substructure and GB showed formation of continuous NbC along GB and smaller NbC precipitates on dislocation cores of a 0.04 C wt% content as-received condition (as seen in Figure 2.2) [12]. The formation of Nb (C, N) provides the elevated temperature strength that is desirable in 347H over 304H SS. Nb (C, N) nucleation on dislocations (as seen in Figure 2.2c-d) contributes to intragranular strength and prevents strain recovery within the grains during stress.
relaxation, leading to strain accumulation along grain boundaries. The degree of $M_23C_6$ formation depends on the Nb/(C+N) ratio. A lower ratio (e.g., higher C content) increases the risk of more $M_23C_6$ formation but can be prevented with fast cooling rates and decreasing C content.

Figure 2.1: 347H as-received from a 1” thick plate solution annealed at 1100°C for 30 min [12]

Figure 2.2: STEM/HAADF images and corresponding Nb elemental maps showing Nb-C precipitation in grain boundaries (a-b) and dislocation within grains (c-d) in as-received material (solution annealed condition for a 0.04 wt% C heat) [12]
2.1.1 CALPHAD Simulations of Weld Microstructure

The solidification mode of 347H SS is generally ferritic-austenitic (FA), where ferrite begins to form first in the liquid followed by austenite. Then, Nb (C, N) nucleation happens right after complete solidification for the composition used, assuming equilibrium conditions. The distribution, size, and volume fraction of niobium carbonitrides in the HAZ and FZ will determine how the 347H SS welds behave during high temperature service conditions. The exposure to temperature and time has a significant impact on the precipitation behavior in both regions. Niobium carbonitrides are reported to be precipitated out from γ-austenite most actively between 700-1000°C [2]. An example of an equilibrium pseudo-binary phase diagram (showing elevated temperature phases) and solubility diagram of Nb in the main elevated temperature phases were calculated using Thermocalc© (Figure 2.3 and Figure 2.4), where Nb (C,N) may precipitate out right after solidification with a very low nucleation rate at ~1375°C. An example of a time-temperature-transformation (TTT) diagram of 50% equilibrium volume fraction of potential Nb (C, N) precipitation in a γ-matrix with an assumed grain size of 100µm was calculated using the precipitation module in Thermocalc© (Figure 2.5). The effect of dislocation density (increasing dislocation density with the black curve relative to the red curve) and modified 50% “diluted” weld metal composition using commercial E309L filler and 347H substrate compositions is shown in Figure 2.5. Reducing dislocation density (via recovery) and reducing the amount of Nb, C, and N (by modifying the weld metal composition) delays the time to Nb (C, N) precipitation, which may be synonymous to time to failure during SRC or reheat cracking. The sharp kinks are likely representative of a database discontinuity in material properties used calculated nucleation and growth rates that predict the TTT curves. The composition used for the Thermocalc© calculations was based on the 347H substrate Gleeble experiment composition (see Table 2.1).

A microstructure with sufficient temperature and time within the active precipitation temperature range (700-1000°C) would allow for sufficient nucleation and growth of Nb (C, N) precipitates within the austenite parent matrix (900°C being the most active nucleation temperature with growth rates increasing proportionally with increasing temperature). Temperatures above the active precipitation range (i.e., above 1150°C) will allow for partial dissolution of these precipitates; however, there may not be complete dissolution up to the solidus temperature. Nb (C, N) is thermodynamically stable out of solution within the γ-matrix
up to the solidus temperature, because maximum Nb solubility in $\gamma$-austenite is below the Nb bulk composition and Nb has a strong affinity for tying up free C and N not in solution. Temperatures below the active temperature range will essentially “lock-in” these precipitates that are out of solution. It has been reported that fine precipitates that are “locked-in” to the matrix lower the creep ductility of the material even though they increase elevated temperature creep strength; on the contrary, coarser matrix precipitates would allow for better creep ductility at high service temperatures [11]. Understanding the interaction and contribution of precipitates on high temperature mechanical properties has a significant impact on the life of the hot tank welds in service.

Figure 2.3: Pseudo-binary phase diagram of 347H SS of typical composition (0.05C-0.58Nb)
Figure 2.4: Thermodynamic equilibrium solubility diagram of Nb in elevated temperature phases in 347H SS of typical composition (0.05C-0.58Nb)

Figure 2.5: CALPHAD calculated TTT of max 50% volume fraction Nb (C, N) precipitation in γ-matrix in 347H SS with starting high \((2 \times 10^{16} \text{ m}^{-2})\) and low \((2 \times 10^{12} \text{ m}^{-2})\) dislocation densities and 50% diluted WM using E309L filler and 347H composition (which may occur in arc welding along fusion line)
2.1.2 Solubility limits of Nb (C, N) in γ-austenite

As cooling occurs from a continuous cast plate and during “solutionizing” heat treatment (1050-1100°C), γ-austenite and Nb (C, N) will be the primary phases upon cooling to room temperature. Nb (C, N) will not completely be dissolved in a solid solution with this composition. Complete dissolution of NbC in γ-austenite will not occur up to temperatures of 1400°C with a Nb content of 0.58 wt% and C content of 0.05wt% based on a Nb-C solubility diagram (see Figure 2.6). As temperature decreases, the volume fraction of NbC will increase. Since there is an excess amount of Nb relative to the stoichiometric ratio (Nb:C → 7:1) in wt% with the received composition, Nb segregation may lead to liquation on grain boundaries during high temperature weld thermal cycles. Studies on the amount of Nb, C, and N content in 347H SS influence the amount of Nb (C,N) content (Figure 2.7 [13]). A higher amount of C and N requires an increase in Nb to meet the 347H specification; therefore, Nb (C, N) content increases. With smaller amounts of Nb, C, and N, the Nb (C, N) may form after complete solidification (which is preferred for weldability). While more Nb (C, N) content would increase elevated temperature strength generally, the excessive amounts of Nb relative to C and N may increase liquation crack susceptibility in the partially melted zone (PMZ) of 347H SS welds if sufficient constraint exists.

Figure 2.6: NbC solubility diagram in γ-austenite (log10[Nb][C]=2.26-(6770/TK))
2.2 Elevated Temperature Mechanical Properties

The code used to determine the maximum allowable stress for commercial CSP tanks is ASME BP&V Section VIII-Div. 1 [11]. Figure 2.8 represents the maximum allowable stress that is dependent on the service temperature for 347, 347H, 347H welded pipe, 321H, 316H, 304H, and 316L. The sudden drop in allowable stress is attributed to time-dependent properties (i.e., creep). 347H SS maintains the highest allowable stress and creep resistance compared to other austenitic stainless steels for a service temperature of 565°C, which would be the best candidate for thickness reduction. Except for one 347H welded pipe (WP) comparison, all the values compared correspond to plate form with higher stress values allowed. 316L SS, as an example, has a maximum allowable recommended service temperature of 454°C according to ASME BP&V code Section II-D, which can be shown with no data points above 500°C seen in Figure 2.8 based on Table 1A in the code [11].
Figure 2.8: Maximum allowable stress values (S) from ASME BP&V code Section II-D. All of the values were taken from Table 1A for plate SA-240 designations with higher stress values. [11]

Creep rupture tests under constant load, on the other hand, provide information on a stress vs. time plot with different temperature dependent lines for a variety of 347H SS compositions in the solution annealed condition (Figure 2.9) [14-16]. As temperature increases, the time to rupture generally decreases with the same applied engineering stress. Mitigation of creep or creep-fatigue induced failure at service temperatures requires a significant reduction of total stress (mainly weld-induced residual stress) in the tank welds. For example, stresses above 300 MPa may contribute to a very short time to failure at 565°C (as early as 56 h for one heat), according to Figure 2.9. PWHT provides the most practical approach in mitigating creep, creep-fatigue, or stress relaxation induced failures of 347H SS welds. Reducing the maximum tensile stress at 565°C to stress values below creep rupture would help maintain a designed life cycle up to 30 years (262,000 hr.) (e.g., <90 MPa overall effective stresses for 600°C data points in Figure 2.9).
2.3 Recovery, Recrystallization, and Grain Growth in 347H welds

In the context of recovery, recrystallization, and grain growth, Nb (C, N) prevent these processes from taking place at low temperatures. Nb (C, N) help pin dislocations from movement, and thus, recovery and recrystallization are very dependent on precipitation kinetics. High temperatures (above 1100°C) are needed to fully achieve these states, particularly with high Nb (C, N) contents in 347H SS, but is dependent on whether cold working or hot deformation has taken place [17]. High dislocation density allows for Nb (C, N) to limit the recovery and recrystallization process. A study, using load relaxation curve, help determine a thermomechanical method for determining recovery and recrystallization as a function of temperature with different applied strains. A compressive true strain of 0.8 for a 18Cr-11Ni-0.06C-0.72Nb (in wt%) 347H SS alloy was applied at temperatures of 1150-1230 °C and then load was relaxed with a constant displacement to allow for static recovery and recrystallization [17]. The first linear slope in the load vs. time plot after hot deformation (seen in Figure 2.10) determines the time during recovery, and then the first inflection point corresponds to when recrystallization first takes place due to load softening. The time was taken between the first and
second inflection to determine the time for complete recrystallization, which is plotted in Figure 2.11.

Figure 2.10: Load relaxation curves after 0.8 true compressive strain for determining recovery (rev) and recrystallization (rex) times in 347H SS for temperatures of 1150-1230°C [17]. Time from beginning to the first inflection point determines the time to recovery, while the time from the first inflection point to the second inflection point is complete recrystallization.

While recovery and recrystallization may occur more during processing after continuous casting, most plate is received in the solution annealed condition prior to bending or weld fabrication. Pipe bends of plate may include recovery and recrystallization because of cold work. Regarding weld microstructures of solution annealed plate though, grain growth is more common in the coarse-grained heat affected zone (CGHAZ) and is dependent on the initial grain size. A study on grain growth after hot forging shows that temperatures 1170°C and below have no significant grain growth due to Nb (C,N) pinning effect (i.e. Zener pinning), while abnormal grain growth could still be observed around 1200-1270°C due to incomplete dissolution of Nb (C,N) at those temperatures [18]. While grain growth is dependent on temperature and time, a temperature of 1270°C quickly allows for coarsening to take place (from about 10µm to 60 µm within 10 minutes) prior to longer times up to 100 minutes (which coarsened to about 90 µm), as seen in Figure 2.12 and Figure 2.13. Times at high temperature during the weld thermal cycle (in the CGHAZ) are very short, and the temperature must be high to allow for grain growth to occur very fast (likely less than 30 seconds). A higher temperature 1) allows for more dissolution and coarsening of pre-existing GB carbonitrides that are pinning the grain boundary mobility, and 2)
faster coarsening rates. Understanding grain growth is important for consideration of PWHT. With this in mind, a PWHT treatment up to 1050°C for a couple hours may not lead to significant grain growth, while static recovery and recrystallization may still occur, which help contribute to a stress relief state in the 347H weldments.

Figure 2.11: Fraction of recrystallization vs. relaxation time for different temperatures in 347H SS at 1150, 1170, 1190, 1210, and 1230°C [17]

Figure 2.12: Microstructures after grain coarsening with temperatures of 1000-1270°C for 60 minutes after being hot forged [18]
2.4 Weldability Issues with E347-347H SS Welds

R.D. Thomas, Jr. (a former American Welding Society President) documented results from multiple industrial partners and papers on the phenomena of HAZ cracking in thick austenitic stainless steel welds from the 1950-1980s era that were in service [3]. His review covers cracking for 347, 321, 316Nb, 310, 309, 316, and 304 SS grades, which involved thick plates with high temperature service conditions but related to mechanical and not corrosion failures. Table 2.2 below shows the susceptibility range of these different SS grades related to HAZ cracking and the number of references citing failures relating to mechanical-based failures. While the elevated temperature strength and sensitization resistance of 347H is most desirable within the 3XX grades, there is a risk for weld failures. This study [2, 3] shows that 347 SS is the most susceptible grade due to its underlying strengthening mechanism: intragranular precipitation and weakened grain boundaries, while 304 is the least susceptible (ranking is not based on number of reported failure cases). Note that only mechanically based cracking was evaluated, not corrosion related. In essence, most of the failures reported 1) occurred in the HAZ, and 2) failed along the grain boundaries. Overall, the theories associated with the types of HAZ failures were 1) liquation cracking and 2) ductility-dip cracking, while 3) reheat cracking (or SRC) is most common among precipitation strengthened stainless steels, particularly 321 and 347 SS. Many of these failures that are reported in this study failed intergranularly in the HAZ while in service or during PWHT temperatures. However, Thomas points out that a carefully
designed PWHT and composition control (alloy design) could solve the issue of intergranular cracking in the HAZ of austenitic stainless steels [3].

Table 2.2: Number of references corresponding to SRC failure in service [3]

<table>
<thead>
<tr>
<th>Type</th>
<th>Number of References</th>
</tr>
</thead>
<tbody>
<tr>
<td>Most Susceptible</td>
<td></td>
</tr>
<tr>
<td>347</td>
<td>22</td>
</tr>
<tr>
<td>321</td>
<td>8</td>
</tr>
<tr>
<td>316Nb</td>
<td>3</td>
</tr>
<tr>
<td>310</td>
<td>4</td>
</tr>
<tr>
<td>309</td>
<td>1</td>
</tr>
<tr>
<td>316</td>
<td>14</td>
</tr>
<tr>
<td>Least Susceptible</td>
<td></td>
</tr>
<tr>
<td>304</td>
<td>9</td>
</tr>
</tbody>
</table>

Although cracking has been often reported in HAZ, there are several potential weldability issues in both the HAZ and other zones including partially melted zone (PMZ) and fusion zone (FZ) of 3XX austenitic stainless steels, especially in 347H. A representative weld microstructure of 347H generated by an arc energy of 50 kJ/in. is shown in Figure 2.14. The fine grain heat affected zone (FGHAZ) is generally not an area for crack initiation for 347H welds. The peak temperature experienced by CGHAZ could lead to dissolution of Nb (C, N). Dissolution of grain boundary (GB) carbonitrides during a weld thermal cycle in the CGHAZ allows for grain coarsening with less GB pinning effect, which reduces the strength and ductility of the CGHAZ relative to the FGHAZ. The PMZ experiences liquation along GBs due to depressed melting temperature of segregations rich in Nb. For instance, Nb (C, N) has a much lower melting point than the γ-matrix as shown in Figure 2.3. Therefore, PMZ may experience liquation cracking with the presence of sufficient cooling stresses above the solidus temperature of secondary phases. The FZ contains up to 10% volume fraction of retained δ-ferrite as the cooling rates are not usually slow enough (i.e., a non-equilibrium conditions) to allow for complete δ+γ+Nb (C, N)→γ+Nb (C, N) transformation during cooling. The characteristics of the PMZ, CGHAZ and FZ contribute to a variety of weldability issues.

For each potential weldability issue, there is one or a combination of factors: 1) composition, 2) thermal history including weld heat input and PWHT, 3) residual stress induced by the welding process, and 4) service-related factors (e.g., service temperatures, thermal cycling
and applied stress). The thermomechanical weldability issues associated with metallurgical nature are listed in Table 2.3. Some of the service-related weldability issues may not apply to this specific application, particularly corrosion related, e.g. “knife-line” corrosion [9], and therefore are not listed. Stress relaxation cracking (SRC) is the most likely weldability issue for failure in CSP tanks based on the service condition of an elevated temperature combined with the inherent residual stresses and susceptible weld microstructures.

![Image](image.png)

Figure 2.14: 347H SS GTAW autogenous weld (190 A, 4 IPM, ~50 kJ/in.)

Table 2.3: Summary of thermomechanical weldability Issues in 347H SS

<table>
<thead>
<tr>
<th>Failure Risk/Weldability Issue</th>
<th>Locations</th>
<th>Type of Cracking</th>
<th>When?</th>
<th>Mitigation Techniques</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td>Liquation Cracking</td>
<td>PMZ/FZ</td>
<td>Hot</td>
<td>Welding</td>
<td>Lower Cullen and Freeman parameter (1/2Nb/(30C+50N)) (composition control)</td>
<td>[19-22]</td>
</tr>
<tr>
<td>Embrittlement (including sigma and G -phases)</td>
<td>FZ</td>
<td>Warm</td>
<td>Service or PWHT</td>
<td>Reduction of δ-ferrite (composition control)</td>
<td>[23-25]</td>
</tr>
<tr>
<td>Ductility Dip Cracking</td>
<td>FZ (possibly HAZ)</td>
<td>Warm</td>
<td>Multi-pass welding and/or elevated T service</td>
<td>Tortuous grain boundaries (process control)</td>
<td>[9, 26-28]</td>
</tr>
<tr>
<td>Reheat cracking/ SRC</td>
<td>FZ, PMZ/HAZ</td>
<td>Warm</td>
<td>Service or PWHT</td>
<td>1. Residual stress management (process control-welding and PWHT) 2. Alternative materials (including weld consumables)</td>
<td>[3, 4, 6, 8, 9, 27, 29, 30]</td>
</tr>
</tbody>
</table>
2.4.1 Liquation Cracking Susceptibility of 347H SS

The main difference between solidification cracking and liquation cracking are the location and mechanisms. Solidification cracking typically occurs in the fusion zone centerline of a weld during solidification when a high-volume fraction of segregant impurities (e.g., S, P) or high partitioning coefficient elements (e.g., Nb) develop along primary solidification boundaries in the form of a continuous film while under a cooling strain. [19]). A solution for mitigating solidification cracking in stainless steels is to increase the tendency for δ-ferrite to form primarily in the melt (higher Cr_{eq}/Ni_{eq} ratio), since δ-ferrite has a higher solubility of S and P than γ-austenite. Liquation cracking is a form of hot cracking that initiates in the PMZ during cooling or reheated weld metal during multi-pass welding. The main characteristics needed for liquation cracking to develop are: 1) stress induced by contraction of a highly restrained weld during cooling, 2) a wide freezing temperature range due to constitutional liquation of low melting temperature phases or elements along grain boundaries [19]. The cracks developed are intergranular with evidence of Nb enrichment phases, deemed a eutectic (example seen in Figure 2.15).

The equilibrium solubility of Nb in δ-ferrite is approximately three times lower than Nb (C, N) in austenitic stainless steels and some Ni-base superalloys (see the three corresponding y axes in Figure 2.4 as a comparison). Carbon and nitrogen are present to tie up the Nb at high temperatures, where Nb (C, N) may form up to the solidus temperature. An excessive amount of Nb relative to C and N in terms of its stoichiometric ratio may lead to liquation with a liquation range of 60°C from 1290 to 1350°C for some alloys of 347 SS[19]). The sample with a 1350°C liquation temperature had minimal liquation within one grain adjacent to the fusion boundary, and the 1290°C liquation temperature sample had a liquation width of a few grains away from the fusion boundary. The backfilling phenomenon (i.e., grain boundary healing”) is limited and cannot heal completely the cracks with a lower liquation temperature. Cullen and Freeman developed a ratio of Nb relative to Nb and C and N (1/2Nb/(30C+50N)), defined as C & F parameter, that reveals the intensity of excessive Nb on liquation susceptibility on a range of 1315-1355°C (Figure 2.16 [22]). A lower C & F parameter decreases the risk for liquation cracking by reducing the width of liquation. Ferrite stringers present in the as-received (hot-rolled solution-annealed) plate can be sites for liquation in the PMZ. A relatively high concentration of Nb within these retained δ-ferrite stringers decreases the localized solidus
temperature. Reducing the C and F parameter to 0.1 and below by controlling the Nb content to a ratio of Nb/C less than 7.7 is recommended for any 347H grade. According to ASTM A240, the Nb is typically eight times the C+N content in wt%, since N also participates in forming Nb (C, N). Nb ratios are generally recommended to be lower in weld metal for similar reasons of reducing solidification cracking.

Weldability tests, including spot transvarestraint and hot ductility tests using the Gleeble, revealed the effects of composition on liquation cracking susceptibility [19, 21]. A spot Varestraint test essentially is three-point downward bending on a stationary GTAW weld as solidification is taking place along a bending block that direct cracks to develop only in the PMZ. Liquation will typically take place intergranularly along ferrite bands or pre-existing grain boundary Nb (C, N), and cracks will develop with a strain exceeding a critical threshold at elevated temperatures. A C and F parameter below 0.1 reduced the total crack lengths to below 0.1 mm (Figure 2.17 [21]) in the PMZ.

The brittle temperature region (BTR) is a low ductility region that ranges from the liquidus temperature to the solidus temperature, where the zero-ductility temperature (ZDT) is associated with the solidus temperature. The temperature range of the BTR is correlated to the width of the PMZ due to a temperature gradient, where the liquidus temperature represents the peak temperature at the PMZ/FZ boundary and the solidus temperature represents the peak temperature at the CGHAZ/PMZ boundary [7]. Hot ductility tests are used to rapidly heat a sample using the Gleeble to determine the ZDT upon heating and cooling from a zero-strength temperature (ZST), which is a temperature within the BTR that shows immediate failure upon pulling a tensile strain. Hot ductility tests were combined with spot transvarestraint tests to show the relationship between the ZDT (determined from the hot ductility tests) and total crack lengths (determined from spot transvarestraint tests) as illustrated in Figure 2.18 [19]. 347-C grade (highest P + S content out of the other grades examined) with a lowest ZDT exhibited the highest susceptibility to cracking with longest total crack length measurements. Reducing the width of the BTR and PMZ, by increasing the ZDT, generally reduces liquation cracking. ZDT can be increased by lowering C and F parameter of 347H grade and reducing P and S content.
Figure 2.15: Example of liquation crack and eutectic in spot transvarestraint and hot ductility tests and EDX intensity map indicating Nb enrichment in liquation secondary phases compared with the base metal at the crack tips [19]

Figure 2.16: Liquation detection as a function of the Cullen and Freeman parameter \((1/2\text{Nb}/(30\text{C}+50\text{N}))\) [22]
Figure 2.17: Total crack length measured for Varestraint tests of various 347 SS heats with different C and F parameters [21]

Figure 2.18: Total crack length (TCL) from spot transvarestraint results of 3.75% augmented strain plotted with the zero ductility temperature (ZDT) determined from Gleeble hot ductility tests [19]

2.4.2 Embrittlement in 347 SS Welds

PWHT can be the primary feasible method for reducing susceptibility SRC of 347H welds made with matching filler metal in service by 1) reducing residual stress, and 2) generating a less susceptible microstructure. However, there are certain temperature ranges where there are ductility drops in the WM (due to sigma embrittlement) and HAZ (due to strain aging). There are
two examples from literature that show the benefit of a 1050°C PWHT for both WM and HAZ, which is a standard solutionizing heat treatment for 347H SS.

The main challenge for materials with required strength at temperatures of 565°C is to prevent grain boundary embrittlement and drops in ductility below 30% based on handbook requirements[31], especially in heat affected zones and multi-pass welding fusion zones (FZ). For 347 SS weldments on 2-inch-thick plates, various PWHT at temperatures of 593-1066°C and annealing times of 8-150 hours were studied with four electrode compositions in literature [24]. Room temperature tensile and Charpy V-notch tests in all weld metals shown in Figure 2.19 reveal the effect of post weld heat treatment, including both temperature and time, on ductility (%) and impact toughness (J) at room temperature. The Larson Miller Parameter (LMP) tempering equation (Eq. 2.1) was used to account for both temperature and time in comparison to the as-welded condition:

$$LMP = \frac{(T(K) \times (20 + \log(t(hr))))}{1000}$$  \hspace{1cm} (2.1)

The four different weld metal compositions were quantified in the form of Cr$_{eq}$ and Ni$_{eq}$ ratios as a means of comparison to other weld metal mechanisms, particularly the formation of δ-ferrite during weld metal solidification. The Cr$_{eq}$ (Eq. 2.2) and Ni$_{eq}$ (Eq. 2.3) are:

$$Cr_{eq} = Cr + (0.7 * Nb)$$ \hspace{1cm} (2.2)

$$Ni_{eq} = Ni + (35 * C) + (20 * N)$$ \hspace{1cm} (2.3)

A higher Cr$_{eq}$/Ni$_{eq}$ ratio increases the tendency for more δ-ferrite to form during the welding process, while a lower ratio reduces the tendency for δ-ferrite and stabilizes the parent γ-austenite phase during solidification. The initial drop in ductility at lower temperatures occurs faster within weld metals with higher Cr$_{eq}$/Ni$_{eq}$ values (higher Cr and Nb content relative to C, N, and Ni) (Figure 2.19a), because σ-phase forms faster at lower temperatures with the presence of a higher amount of δ-ferrite. Figure 2.19b demonstrates a similar trend in the as-welded impact toughness as a function of Cr$_{eq}$/Ni$_{eq}$. The effect of micro fissuring in all austenitic weld metals is a common phenomenon and can contribute to microcracks during solidification that lead to reduced impact toughness [32]. While this may be the case for the as-welded condition, a lower Cr$_{eq}$/Ni$_{eq}$ ratio leads to a reduction in ductility at higher temperatures. The tests included all WM
in the longitudinal direction and can be assumed that dilution was negligible in the test region (i.e., minimum mixing with substrate).

![Graph A](image1.png)

![Graph B](image2.png)

Figure 2.19: a) Ductility dip in room temperature tensile tests, and b) toughness dip in room temperature Charpy v-notch tests as a function of the LMP (varying PWHT aging temperatures and times) of E347 2” thick welds with four different compositions and time [24].

A study on elevated temperature tensile testing after various heat treatments show the effects of strain aging temperature on ductility drops in the range of 700-900°C for a 18%Cr-12%Ni-1%Nb SS (older version of 347 with higher Ni and Nb contents) (Figure 2.20 [28]). Tensile specimens were solution heat treated ranging from temperatures of 1050-1325°C followed by aging at a variety of temperatures within 700-900°C for two hours. Then, specimens were pulled to failure at the aging temperature and the ductility was measured. Ductility contours
were placed in a plot of solution temperature vs. testing temperature to reveal the degree of ductility dip as a function of both variables. The highest degree of ductility dip occurs around 800°C aging temperature for all solution heat treat temperatures, while a higher solution temperature leads to a lower ductility for all testing temperatures. The explanation for these results pertains to the strain age embrittlement mechanism, where a higher solutionizing heat treatment will lead to dissolution of coarse Nb (C, N). During subsequent strain aging, formation of fine Nb (C, N) at dislocation cores during tensile testing at lower testing temperatures may lead to ductility dip.

![Ductility contours from elevated temperature tensile tests after solution heat treatments at 1050-1325°C and subsequent aging (700-900 °C) for 18%Cr-12%Ni-1%Nb SS [28]](image)

**Figure 2.20: Ductility contours from elevated temperature tensile tests after solution heat treatments at 1050-1325°C and subsequent aging (700-900 °C) for 18%Cr-12%Ni-1%Nb SS [28]**

### 2.4.3 Ductility Dip Cracking (“Micro fissuring”)

Ductility dip cracking (DDC) occurs typically in multi-pass welds during reheating from the weld process or elevated temperature service, where restraint and grain boundary tortuosity are low. DDC typically occurs on migrated grain boundaries in reheated weld metal, less tortuous grain boundaries with high concentrations of segregant elements (S+P) and second phases (or lack thereof) [7]. Some older reviews combine liquation cracking with DDC mechanisms seen in multi-pass welds [2]. However, Lippold distinguished the difference between the two crack types by describing their temperature differences [7]. Liquation cracking is associated with a BTR at elevated temperatures, while DDC is manifested in the form of a
ductility dip at solid state temperatures [7]. DDC may occur in 347H HAZ as well but is primarily associated with the multi-pass WM [27].

Strain-to-fracture tests are used to measure DDC susceptibility, which is an elevated temperature test where they apply a range of tensile plastic strains to determine when microcracks begin to develop (particularly on a spot weld microstructure [7]). Another literature example determined the susceptibility of a weld microstructure to DDC through bend test on multiple different austenitic weld filler multi-pass welds with varying ferrite content (Figure 2.21 [21]). Micro fissuring is defined as the formation of intergranular cracks due to grain boundary migration both during cooling after solidification and during reheating [32]. Lippold [7] states that “micro fissuring” is a representation of DDC and should be reported as DDC instead. Ferrite typically possesses more tortuous grain boundaries and higher solubility for segregating elements (e.g., sulfur and phosphorous) than austenite, providing better resistance to grain boundary migration and micro fissuring due to stress/strain distribution along straighter, low tortuous boundaries with respect to axis of maximum tensile stress. Hence, a high ferrite content reduces the tendency for DDC. Among fillers for 3xx stainless steels, E347 WM performed the worst and needed more ferrite content to reduce the degree of micro fissuring in reheated weld metal during multi-pass weld procedure. Reheated WM, depending on the peak temperature experienced and cooling rates, may solutionize ferrite content during a subsequent weld pass and reduce ferrite content. High-segregation elements (e.g., S, P) not in solution with austenite matrix, combined with sufficient cooling strains, lead to cracking along low tortuous migrated grain boundaries during reheating. High ferrite content reduces hot cracking and ductility dip cracking susceptibility; however, embrittlement may occur with excessive ferrite content due to elevated temperature service where sigma phase may begin to form via diffusive transformation. The best consumables that only need a small amount of ferrite to reduce cracking are E316 and E308. E16.8.2, which is a hybrid of 316 and 308, is an alternative filler with low ductility dip cracking and hot cracking susceptibility.
Figure 2.21: Fissure bend test results on shielded metal arc welding (SMAW) welds of various fillers and ferrite content [21]. The plot resembles the number of fissure counts measured optically versus ferrite number of various E3XX weld consumables.

2.4.4 SRC and Reheat Cracking in 347H Welds

Many cracking mechanisms are closely associated with high temperature failures in weldments and base materials, particularly creep, creep-fatigue, and thermal fatigue [4]. The most likely cracking mechanism for multi-pass weldments of 347/347H austenitic stainless steel is a reheat cracking mechanism. There are other types of names associated with reheat cracking mechanisms from reports and literature:

- Reheat cracking, a general term associated with cracking during PWHT
- Strain-age cracking, associated with Ni base alloys such as 740H
- Stress relief/relaxation cracking (SRC) that occurs during service

SRC is a high-temperature service related failure mechanism that occurs in precipitate-strengthened materials, notably 3XX stainless steels and Ni-base alloys [33]. About 50 failures related to SRC were reported in 800H, 16Cr13NiN, 617, 625, 304H, 321H, 347H, 310, 25/35Nb, and 601 (combination of 3XX SS and Ni-base alloys) [8]. SRC occurs normally in the weld metal and heat-affected-zone regions of high-temperature creep resistant materials. Figure
2.22 [9] demonstrates cracking in E347 WM during PWHT at 900°C, which can be observed with a branching nature with cracks occurring along solidification grain boundaries. Figure 2.23 [34] demonstrates an example of SRC cracks in a superheat tube lug after being in elevated temperature service of 595°C for a few months.

A list of characteristics of SRC have been collected from an extensive report of SRC susceptibility in many austenitic stainless steels and Ni-base alloys reported by the Netherlands Organization for Applied Scientific Research (also known as TNO) in which the 50 failures mentioned above correspond to the following fingerprints [4, 8]:

1. Failure occurs at operating temperatures between 500 and 750°C.
2. Failure occurs in new equipment mostly within 1 to 2 years.
3. Failure occurs after repair or modification often within 1 year.
4. Cracks are always located on grain boundaries, in front of cracks there are small-isolated creep like cavities (example of creep cavities ahead of SRC failure in 347H SS in Figure 2.24b).
5. The fracture surface of the material shows a brittle appearance with visually no deformation.
6. Cracks are present in the HAZ, weld metal and cold deformed areas (high dislocation density)
7. Mostly a metallic filament is present on cracked grain boundaries. This filament is enclosed by a Cr-rich oxide layer, which is low in Ni and Fe (see Figure 2.24a). The chemical composition of the metallic filament itself is material dependent, but always very low in Cr and high in Ni and Fe. Some other materials, like Inconel Alloy 617, have no metallic filament present. Sometimes referred to as “white-phase fracture” [5]. It should be noted that a Cr-rich oxide layer mainly develops in stress relaxation cracks over time after crack initiation in service, while a Cr-rich oxide is not likely seen in stress relief cracks from PWHT.
8. Cracks are only present in areas where the Vickers’s hardness is higher than 200 HV (load > 1 kg).
As mentioned earlier, the three main contributing factors are 1) residual stress, 2) susceptible microstructure, and 3) elevated temperature in creep dominated regimes. The three
factors together lead to reheat cracking or SRC. The metallurgical mechanisms contributing to cracking is hypothesized to be a combination of reprecipitation of niobium carbonitrides (Nb (C,N)) [6,7] in the grain interiors (in dislocation//s) and on the grain boundaries (as seen in Figure 2.2)). The reprecipitation of intragranular niobium carbonitrides during elevated T service or PWHT strengthens the grain interiors, while grain boundary carbonitrides have a low coherency with the γ-austenite grain boundaries. Therefore, a zone in between the grain interiors and grain boundaries is depleted of niobium carbonitrides, which is described as a precipitate free zone (PFZ). A microstructural evolution schematic illustrates the PFZ being between coarse intergranular precipitates and a zone parallel with a high density of relatively finer precipitates (with limited atomic mobility in comparison to grain boundaries) due to dislocation pile-ups and higher dislocation densities near grain boundaries (Figure 2.25 [4]). This location allows for strain localization to minimize the energy contribution from the residual stress, which eventually leads to cracking in this region [8,9]. Figure 2.26 below shows an example of a PFZ for a nickel-based alloy Inconel 740H in the weld metal with the secondary precipitate being γ' [33]. A PFZ is also shown near the crack tip and creep cavities of a SRC failure in a 347H SS HAZ failure (Figure 2.27 [35]). It should be noted that the PFZ for 347H SS welds in contrast to Inconel 740H is observed on a finer scale (< 500 nm), where a high-resolution transmission electron microscope (TEM) is needed to detect the PFZ. Also shown is a M23C6 carbide that resides on the same grain boundary where the PFZ is observed. The PFZ has been established as the main mechanism that can explain “intergranular” failure seen in SRC and reheat cracking examples.

![Figure 2.25: Schematic of PFZ along grain boundaries due to solute depletion [4]](image_url)
Figure 2.26: SEM image showing PFZ in Inconel 740H weld metal near the grain boundary. The mismatch of strengthening mechanisms enhance the susceptibility to SRC in PFZ region [33]

Figure 2.27: STEM-EDS maps of Cr and Nb near a creep cavity ahead of a toe crack in the fillet of a 347H weld showing Cr depletion and Cr rich carbide (likely Cr$_{23}$C$_6$) along a grain boundary with the presence of a Nb rich carbonitride (likely Nb (C, N)) and a PFZ less than 500 nm wide [35]. Image used with permission of collaborative research study funded by the Electric Power Research Institute and executed by Dr. Geoff West of Warwick University.

Secondary phases other than Nb (C, N) formed in the weld and HAZ require important consideration regarding SRC. For instance, α’ martensite may be present near the root HAZ of a single-V groove on a boiler pipe due to transverse shrinkage of later weld passes on the HAZ of pass 1 (root pass) (Figure 2.28) [36]. The shrinkage upon cooling in pass 2 creates a bending moment on the root of the weld, thus creating sufficient tensile strain to trigger transformation induced plasticity (TRIP). Figure 2.28 illustrates the presence of a macro crack within HAZ.
(likely SRC), which is 2 mm away from fusion zone boundary, after five months in service at 595 °C. Hence, the presence of α’ martensite and Nb(C,N) may suggest a higher susceptibility to cracking in 347 SS during service at elevated temperatures by strengthening the grain interiors and complementing the PFZ mechanism [4]. Understanding the reversion of α’-martensite (BCT) to γ-austenite (FCC) may affect SRC susceptibility because of a phase transformation leading to a more densely packed structure that may cause increases in dilation strain/stress during reheating [37].

Figure 2.28: Macro crack in a) HAZ, b) closeup of crack, c) region of dark bands representing α’ martensite [36]

Figure 2.29 demonstrates the fracture micrographs and SEM images of reheat crack related fracture surfaces at 700°C and 800°C with pre-load of 10% plastic strain in HAZ of 347H SS [6]. There are a significant number of secondary cracks that exist behind the primary fracture surface when analyzing cross sections, which indicate low ductility along grain boundaries. The fracture surfaces, via scanning electron microscope (SEM), displays an intergranular failure with small micro voids existing on the grain facets. All these metallurgical observations are evident both of SRC and reheat crack type characteristics, except SRC cracks contain oxides due to very slow progressive cracking over time at elevated temperature.
Figure 2.29: 347H SS Secondary intergranular cracks ahead of fracture surface a) under 800°C and 10% plastic strain and b) under 700°C and 10% plastic strain, and fractography analysis using SEM for 700°C and 10% plastic strain case: c) mixed-mode fracture surface showing micro voids on surface with grain facets, and d) void along grain boundaries [6]

2.4.5 Gleeble Testing Methodologies for SRC Simulation and Contributing Factors

A general way of evaluating the cracking tendency for SRC and weldability are using ‘c-curves’ or ‘crack curves’ as a function of temperature and time under constant displacement. For creep rupture tests under constant load, information is usually displayed on a stress vs. time plot with different temperature dependent lines. For 347H weld microstructures specifically, there are only a few references pertaining to SRC susceptibility in weld metal, simulated HAZ, and a record of time to failure for various examples of failure in service and various weldability tests (Figure 2.30 [4, 6, 9, 38]). The green line represents an overall inclusive curve on minimum time to failure for various SRC tests and service failures. SRC has been reported to be a significant issue typically at 700-900°C in relatively short service time [6, 39]. Some studies only provide a certain time range about when failure took place. An example of such a test where the time to failure or start of cracking isn’t reported is a three-point bend relaxation test of a 347 SS weld [4]. After heating to a temperature range between 600 and 650 °C for 347H, they would displace the center of the specimen to about 10% strain in the center of the specimen and watch load relax for a duration of 96 hrs. After 96 hours, the sample contained evidence of microcracks on the
surface bent in tension and in the middle of the specimen at the CGHAZ with a slightly higher degree of cracking at 650 °C. These tests are used to mainly evaluate SRC susceptibility during service.

For the case of Gleeble specific simulations of SRC on weld microstructures, as shown in Figure 2.30, three sets of data are shown, including data reported by Lippold [9] and Kant [6]. The curves represent a minimum time to failure for situations in which constant displacement (i.e., stress relaxation) conditions were implemented. A higher initial stress up to 100% of WM yield strength reduces the minimum time to failure and increases susceptibility to reheat cracking. A weld with relatively higher residual stresses will generally have an increased susceptibility to cracking during PWHT. Based on comparative purposes, the data from Kant [6], which is a simulated HAZ with an applied true strain of 21% (10% cold work), indicate similar time to failure with WM samples. While there are SRC tests that confirmed cracking occurs, there is a lack in understanding of temperature-dependent critical stresses and strains to failure. When designing the PWHT, such knowledge is important in order to prevent reheat cracking during PWHT.

Figure 2.30: Literature survey of SRC susceptibility curves reported as temperature vs. time from various SRC tests, and service failures [6, 9, 38]
The most recent methodology from literature was developed by Lehigh University for simulating stress relief cracking (seen in Figure 2.31 [6]). The test consists of four steps: 1) thermal cycle representative of what CGHAZ experiences, 2) applied tensile plastic strain at room temperature. 3) Heat to a PWHT temperature under zero stress, and 4) stress relaxation under constant displacement with an applied stress equal to the 0.2% offset yield strength. The current method developed by CSM applies a similar idea to simulate reheat cracking, except with a stress present during heating to the designated PWHT temperature (step 3) instead of pulling to a stress value at temperature. Ohio State University (OSU) has also another modification where they pull at room temperature and allow for thermal expansion to reduce while heating up and then pull to an additional stress representative of the current temperature 0.2% offset yield strength [40]. Their results of a E347-347H cross welded sample show no failure with an approximated pre-stress that is 50% of the WM yield strength with a temperature of 900°C.

Based on the stress relaxation curves for 347H SS with a 10% cold work (CW) condition (Figure 2.32), failure occurs at all conditions, except the 900°C condition. Some tests with no pre-plastic strain (no step 2) exhibited no failure for the 347H SS samples. An additional plastic strain was needed for cracking to occur in 347H SS HAZ, but there is no indication what minimum plastic strain is needed to allow for failure to occur within a reasonable PWHT time. The intention of this project is to determine a comprehensive understanding of the interaction between stress/strain, temperature, and time to failure.

As a comparison with other alloys, a SRC susceptibility statistical ranking was developed by Kant et al. The six parameters that influence the SRC susceptibility are: 1) ductility, 2) % stress relaxed at temperature, 3) hardness near fracture surfaces, 4) failure time, 5) fracture mode, and 6) presence of intergranular secondary cracks. Based on the Gleeble SRC tests, the precipitation strengthened alloys ranked the most susceptible to cracking (including 347H, IN 740H and Haynes 282 alloys) (Figure 2.33[6]). Type I cracking represent intergranular fracture surfaces with smooth grain facets, while type II cracking represent intergranular fracture surfaces with micro void coalescence present on grain facets. The 347H samples with applied plastic strain displayed primarily type II failure (see Figure 2.29c).
Figure 2.31: SRC Gleeble methodology developed by Lehigh University [6]

Figure 2.32: Stress relief cracking results for 347H with 10% CW condition [6]

Figure 2.33: SRC susceptibility for various materials, stress, and temperature conditions [6]
2.5 Mitigation Techniques for SRC of 347H SS Welds

SRC might not be the only cracking mechanism attributed to failure. But the mitigation approaches could be generally effective in controlling the mechanically and metallurgically related cracking mechanisms by: 1) stress control in the weld metal (FZ) and HAZ by PWHT and weld procedure design, and 2) composition control (e.g., alternative weld consumables). The three specific potential solutions for mitigating SRC are:

1. PWHT: Develop guidelines for identifying optimal PWHT condition for 347/347H SS similar welds as a function of plate thickness to reduce residual stress and to provide a stabilized HAZ and FZ microstructure through a single or multi-step process.

2. Weld Procedure Design: Identify proper Welding Procedure Specifications (WPS) for better control of weld-induced thermal profile, which, in turn, determines the weld residual-stress distribution and HAZ microstructure. Optimization of the welding process include weld geometry design (e.g., using a J-groove or double-V groove instead of a single-V groove) and heat input control. There is limited literature for this approach.

3. Alternative materials: Use alternative welding consumables to prevent SRC in the weld metal, and/or use softer consumables to lower the residual stress in HAZ and thus mitigate strain-induced reprecipitation (e.g., E16.8.2). Or use alternative base metals with similar high-temperature properties but less susceptible HAZ microstructure such as NUCL 167 SPH (i.e., 316L with boron additions and lower carbon concentration).

The two main mitigation techniques observed in this project are 1) PWHT and use of 2) alternative weld consumables and substrates, for reducing residual stress prior to service and reducing susceptibility of the microstructure, respectively.

2.5.1 Effects of PWHT

Post weld heat treatment (PWHT) has a dual purpose and benefits for components prior to elevated temperature service: 1) reduce residual stress, and 2) stabilization of microstructure [2, 3]. The effect of temperature on stress reduction is illustrated in Figure 2.34 with a single step temperature. As the temperature increases, there is an increased benefit of stress reduction. A 900°C/2hr PWHT reduced the peak longitudinal residual stress in the ID weld centerline of a 347H SS pipe from nearly 350 MPa to 75 MPa (~79%) [38, 41]. Some literature suggests a
single PWHT of 875°C for a few hours as being sufficient for reducing SRC susceptibility [4]. However, a PWHT near the solutionizing temperature (>1050°C) are needed for time-efficient complete stress relief [38, 41-43] (Figure 2.34) and is recommended to retain ductility (see Figure 2.19) in a highly restrained weld.

Therefore, two potential PWHT treatments from literature are 1) one-step isothermal 875°C for three hours, and 2) multi-step procedure with a stress relief, solutionize, and stabilization (optional) heat treatment. A multi-step process PWHT could be the best option for preventing SRC for 347/347H thick weldments, but the procedure must be carefully engineered to prevent relaxation cracking during PWHT. Parameters such as the heating rates, holding times, temperatures, and quenching rates are all important parameters that influence the final stress state and microstructure. A multi-step PWHT thermal cycle, using a three-step procedure consists of 1) initial stress relief, 2) solutionize microstructure and full stress relief, and 3) stabilization of precipitates [2, 38, 44]. The initial stress relief is recommended to reduce some initial stress prior to ramping up to the solutionizing temperature, which helps dissolve intergranular carbonitrides and reduce δ-ferrite by reverse transformation in a predominantly two-phase field (γ-Nb (C, N)). Reduction of the intergranular carbonitrides helps minimize PFZs and allows for coarsening of intragranular Nb (C, N). The final step of stabilization was initially developed by Morishige [38] as a step to further stabilize the microstructure by reducing any remaining free C (i.e., tying up of C in nucleation and growth of remaining Nb (C,N) at 900°C) to prevent any M_{23}C_6 from forming during cooling. Figure 2.35 shows the overlay of PWHT temperatures and time relative to critical C-curve formation [2, 3, 38, 44]. A microstructure that has not been solutionized will have a crack-susceptibility C-curve synonymous with a TTT curve of Nb (C, N) precipitating out of γ-austenite. Hitting the coarse NbC curve and avoiding the fine NbC and M_{23}C_6 precipitation with little to no free carbon available during cooling. The three-step PWHT method has been successful in industry for decades, with an example of a furnace PWHT schedule shown in Figure 2.36 [44].
Figure 2.34: a) Effect of temperature (at one hour approximation) on stress relief of Type 347 steel [42, 43], and b) stress relaxation measurements for the weld centerline of 1” pipe 0.84 inch thick [38, 41]
Figure 2.35: Multi-step PWHT overlayed on crack and precipitation c-curves [2, 3, 38]

Figure 2.36: Example of 3-step PWHT treatment used for thick 347H SS welds [44]

Table 2.4 below details four slightly different schedules. Method 1 only includes the first two steps, while Method 3 includes only the first two steps with slightly slower rates. Method 4 uses higher heating rates at the first two steps [44]; however, heating rates are limited to a maximum of 444°C/hr divided by thickness in inches according to AWS D10.10: “Recommended Practices for Localized Heating of Welds in Piping and Tubing” [45]. Slower heating rates give time for grain boundary creep to occur along PFZs, which make it susceptible to brittle failure during stress relaxation at PWHT temperatures. Faster heating rates are argued to reduce cracking susceptibility because higher heating rates allow for the slope to miss
susceptible C-curve regions for failure [3], but control of temperature gradients are important to reduce undesired thermal stresses. Cooling rates are typically representative of air-cooling conditions and are more acceptable for PWHT having a third stabilization step, but there are maximum cooling rates needed to prevent any formation of $M_23C_6$ and minimum cooling rates needed to prevent excessive thermal gradients. The maximum cooling rate is $278^\circ\text{C/hr}$ divided by thickness in inches for localized PWHT under AWS D10.10 [45]. ASME B31.1 and Section III list similar requirements on heating and cooling rates, but the maximum allowed is the highest for ASME B31.1 at $333^\circ\text{C/hr}$. divided by half the thickness. PWHT has been vastly used to reduce service SRC issues, but reheat cracking (the short time, high temperature failure term used for cracking during PWHT) needs to be prevented for these PWHT schedules to have success in industry.

Table 2.4: Summary of a variety of PWHT schedules using multiple steps from literature

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Step 1a</strong></td>
<td>Stress Relief</td>
<td>Ramp up to 593 $^\circ\text{C}$ at 167 $^\circ\text{C/hr.}$ (2.8 $^\circ\text{C/min}$)</td>
<td>Ramp up to 593 $^\circ\text{C}$ at 167 $^\circ\text{C/hr.}$ (2.8 $^\circ\text{C/min}$)</td>
<td>Ramp up to 600 $^\circ\text{C}$ at 150 $^\circ\text{C/hr.}$ (2.5 $^\circ\text{C/min}$)</td>
<td>Ramp up to 595 $^\circ\text{C}$ at a rate of 840-1500 $^\circ\text{C/hr}$ (14-25 $^\circ\text{C/min}$)</td>
</tr>
<tr>
<td><strong>Step 1b</strong></td>
<td>Stress Relief</td>
<td>Hold at 593 $^\circ\text{C}$ for 2 hrs.</td>
<td>Hold at 593 $^\circ\text{C}$ for 2 hrs.</td>
<td>Hold at 600 $^\circ\text{C}$ for 2 hrs.</td>
<td>Hold at 595 $^\circ\text{C}$ for 2 hrs./in. (4 hrs. for 2 in. thick)</td>
</tr>
<tr>
<td><strong>Step 2a</strong></td>
<td>Solutionize</td>
<td>Ramp up to 1052 $^\circ\text{C}$ at 333 $^\circ\text{C/hr.}$ (5.55 $^\circ\text{C/min}$)</td>
<td>Ramp up to 1052 $^\circ\text{C}$ at 333 $^\circ\text{C/hr.}$ (5.55 $^\circ\text{C/min}$)</td>
<td>Ramp up to 1050 $^\circ\text{C}$ at 315 $^\circ\text{C/hr.}$ (5.25 $^\circ\text{C/min}$)</td>
<td>Ramp up to 1050 $^\circ\text{C}$ at 18-30 $^\circ\text{C/min}$.</td>
</tr>
<tr>
<td><strong>Step 2b</strong></td>
<td>Solutionize</td>
<td>Hold at 1052 $^\circ\text{C}$ for 2 hrs.</td>
<td>Hold at 1052 $^\circ\text{C}$ for 2 hrs.</td>
<td>Hold at 1052 $^\circ\text{C}$ for 2 hrs.</td>
<td>Hold at 1050 $^\circ\text{C}$ for 2 hrs./in.</td>
</tr>
<tr>
<td><strong>Step 3a</strong></td>
<td>Stabilize</td>
<td>none</td>
<td>Air Cool to 900 $^\circ\text{C}$</td>
<td>none</td>
<td>Ramp down to 954 $^\circ\text{C}$ at 1.5-3 $^\circ\text{C/min}$</td>
</tr>
<tr>
<td><strong>Step 3b</strong></td>
<td>Stabilize</td>
<td>none</td>
<td>Hold at 900 $^\circ\text{C}$ (no time specified)</td>
<td>none</td>
<td>Hold at 954 $^\circ\text{C}$ for 1 hr/in.</td>
</tr>
<tr>
<td><strong>Step 3c</strong></td>
<td>Finish PWHT</td>
<td>Air cool to room temperature</td>
<td>Air cool to room temperature</td>
<td>Air cool to room temperature</td>
<td>Air cool to room temperature at a minimum rate of 20 $^\circ\text{C/min}$</td>
</tr>
</tbody>
</table>

2.5.2 Alternative Materials: Weld Consumables

An in-depth review into an alternative wire, 16Cr-8Ni-2Mo, shows that it can be used to weld thick sections of 347H SS, with a particular need for repair welding of 347H SS welds that cracked [3]. While providing enough solidification cracking resistance with only a ferrite number
(FN) of 2, the 16-8-2 weld consumable provides embrittlement resistance for elevated temperature service conditions [46, 47]. The 16-8-2 consumable provides a higher stress-rupture ductility than 347H SS as a matching consumable. With a lower resistance to embrittlement (formation of sigma, chi, or Laves phase) due to a low amount of weld metal ferrite, 16-8-2 is more resistant to micro fissuring and loss in ductility [46]. The creep ductility of E16.8.2 is consistently higher than E347 weld metal for elevated temperature applications, thereby proving better thermal stability [47]. A mentioned earlier, micro fissuring is defined as the formation of intergranular cracks due to grain boundary migration both during cooling after solidification and during reheating, which could act as initiation sites for stress relaxation cracks [32]. In addition, the mean coefficient of thermal expansion for 16-8-2 is lower than 347H SS. Thus, upon cooling, there is less transverse weld shrinkage (i.e., less residual stress and yielding of the weld metal) in a 2” thick 347H SS plate. In all, the use of 16-8-2 could reduce SRC susceptibility in the weldment by providing a less susceptible microstructure in the weld metal as well as potentially less welding-induced residual stress by means of a lower mean coefficient of thermal expansion [46].

Another potential weld consumable in place of E347H would be E308H for elevated temperature applications. The composition comparison between three weld metal candidates is displayed in Table 2.5. The main composition differences between the three is lower Ni and Cr content and more Mo in E16.8.2, and elimination of Nb in both E16.8.2 and E308H. E308H acts as the matching filler to E304H SS and has strength sufficient for a 565°C. However, E16.8.2 still has better thermal stability.

Table 2.5: Composition comparison in all weld metal of three electrodes that are candidates for welding of 347H SS substrates

<table>
<thead>
<tr>
<th>wt %</th>
<th>C</th>
<th>N</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Mn</th>
<th>Nb</th>
<th>Si</th>
</tr>
</thead>
<tbody>
<tr>
<td>E 16.8.2</td>
<td>0.05</td>
<td>0.03</td>
<td>8.6</td>
<td>16</td>
<td>1.2</td>
<td>1.7</td>
<td>-</td>
<td>0.3</td>
</tr>
<tr>
<td>E 347</td>
<td>0.03</td>
<td>0.03</td>
<td>10.1</td>
<td>19.5</td>
<td>0.2</td>
<td>1.5</td>
<td>0.36</td>
<td>0.5</td>
</tr>
<tr>
<td>Typical E308H [40]</td>
<td>0.06</td>
<td>0.03</td>
<td>10</td>
<td>18.7</td>
<td>0.03</td>
<td>0.6</td>
<td>-</td>
<td>0.7</td>
</tr>
</tbody>
</table>

The Schaeffler Delong diagram can be used to compare the solidification microstructure and an approximation of how much ferrite content would be present based off Cr and Ni equivalence values (see Figure 2.37 [46]). E16.8.2 WM may develop very small amount of
martensite since the martensite start temperature is close to and slightly above room temperature, but is very unlikely due to dilution effects with the substrate [40]. An instance of PWHT of E16.8.2 WM increased the magnetic response of the weld metal by four times because of the formation of more BCT martensite upon cooling from a PWHT of 705°C within a range of one to five hours [40]. However, the main benefit of E16.8.2 WM is reduced δ-ferrite, which is prone to transforming to embrittling σ-phase as in E347 WM. The formation of martensite does not negatively influence the Charpy toughness of E16.8.2 WM compared to E347 WM (Figure 2.39 [40]). The E16.8.2 WM outperformed the 347H WM toughness with a 347H SS substrate. Also, the E16.8.2 WM was shown to have higher retained Charpy toughness than E308H after 168 hours of aging at 705°C. Additionally, SMAW electrodes of E16.8.2 WM showed better low temperature (-196°C) Charpy toughness and lateral expansion values than E316L and E308L welds using the SMAW process (Figure 2.39 [46]). As a more stable electrode at aging temperatures than E308H and E316H, E16.8.2 WM would be the preferable alternative to replace E347.

Figure 2.37: Schaefer Delong diagram with Etsy Ni<sub>eq</sub> equation and experimental composition points and boxes representative of “acceptable” composition window for E16.8.2 [46]
Figure 2.38: Charpy impact toughness in AW condition, HT-4 hrs., and HT-168 hours for four different cross welded sets. The 347H welds were aged at 1650°F (900°C) [40]

Figure 2.39: Relationship between Charpy impact energy and lateral expansion of E16-8-2T, E308LT, E316LT, and E316LMn T FCAW weld metals at -196°C [46]

2.5.3 Alternative Materials: Substrate

An alternative 316L with B additions (a nuclear grade developed by ArcelorMittal NUCL 167 SPH) has been an alloy of interest in the case of new hot tanks that may consider incorporating another alloy with similar mechanical properties to 347H SS. The low carbon content restricts its use at temperatures of 565°C according to ASME Section II-D allowable stress tables [11]. However, content shared by ArcelorMittal claims that NUCL 167 SPH contains similar creep and yield strength properties to 347H SS in the solution annealed condition with additional boron [48]. The equilibrium pseudo-binary phase diagram for the modified 316L is displayed as a function of Boron content as shown in Figure 2.40.
Boron lowers the solubility of carbon in austenite and thus increases the propensity for carbonitrides forming, which increases the creep strength, but too much boron addition could contribute to weldability issues since boron affects the segregation of elements and wetting capability of grain boundaries [3, 49-51]. To be more specific, the borides (M₃B₂) that form upon cooling may be detrimental to weldability (e.g., increase the susceptibility to liquation cracking). Therefore, B content needs to be controlled to a low value to ensure weldability while being kept sufficient to provide additional creep resistance. Low carbon content allows for improved resistance to stress corrosion cracking (SCC) and intergranular corrosion while boron additions (up to 20 ppm) allow for increased high temperature creep strength and ductility and inhibits wedge cracks and creep cavities [49-51]. Composition used for experiments shown in Table 3.1.

Molybdenum allows for more stable carbide formation while allowing for good substitutional strengthening in absence of Nb (C, N) in NUCL 167 SPH. As a primarily solid solution alloy, NUCL 167 SPH may not need PWHT and is very likely to be less susceptible to SRC than 347H SS. As shown in Figure 2.40, there are a few phase constituents that could be detrimental: M₂₃C₆ (leading to loss of corrosion resistance), sigma and Laves Phase (leading to embrittlement). However, these phases might not directly affect SRC susceptibility.

Figure 2.40: Pseudo-binary phase diagram of modified 316L predicted from CALPHAD simulations using Thermocalc© software (B~ 0.0008 wt%)
CHAPTER 3: EXPERIMENTAL AND MODEL METHODS

The overall objective of this project is to determine the susceptibility of 347H weld and alternative microstructures relative to a range of PWHT temperatures and then determine the best PWHT schedule that would provide the least susceptible SRC microstructure while preventing reheat cracking during PWHT. The list of materials and corresponding alloying chemical composition is shown in Table 3.1. The approach for completion of the overarching goals and answering the research questions is to investigate impacts of welding and PWHT procedures on SRC susceptibility using a variety of laboratory scale experiments and simulations including:

- Welding experiments on 1 and 2” thick 347 H SS plates;
- Gleeble reheat crack testing and thermo-mechanical testing on simulated HAZ and extracted WM to determine when reheat cracking would take place as a function of microstructure, temperature, and stress and strain;
- Finite element analysis (FEA) and modeling (FEM) of welding process to determine residual stresses and effect of PWHT on stress reduction in welds and Gleeble testing for validation of creep parameters and critical strains to failure;
- Thermodynamic calculations using Thermocalc © (results are shown in the Background section on composition and base microstructure of 347H SS)
- Metallurgical characterizations using light optical microscopy (LOM) and scanning electron microscopy (SEM).

Table 3.1 Chemical composition of main alloying elements of all experimental materials (including substrates and consumables)

<table>
<thead>
<tr>
<th>Material</th>
<th>wt pct.</th>
<th>C</th>
<th>N</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Mn</th>
<th>Nb</th>
<th>Co</th>
<th>Si</th>
<th>B</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>ASTM A240</td>
<td>0.04-0.1</td>
<td>-</td>
<td>9-13</td>
<td>17-19</td>
<td>-</td>
<td>2</td>
<td>8 x (C+N)</td>
<td>-</td>
<td>0.75</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Gleeble Experimental</td>
<td>0.05</td>
<td>0.031</td>
<td>9.11</td>
<td>17.29</td>
<td>0.32</td>
<td>1.01</td>
<td>0.58</td>
<td>-</td>
<td>0.51</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>347H 2” plate</td>
<td>0.049</td>
<td>0.025</td>
<td>9.19</td>
<td>17.29</td>
<td>0.11</td>
<td>1.06</td>
<td>0.59</td>
<td>0.22</td>
<td>0.5</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>347H 1” plate batch 1</td>
<td>0.043</td>
<td>0.025</td>
<td>9.19</td>
<td>17.6</td>
<td>0.11</td>
<td>1.06</td>
<td>0.54</td>
<td>0.28</td>
<td>0.48</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>347H 1” plate batch 2</td>
<td>0.052</td>
<td>0.03</td>
<td>9.17</td>
<td>17.58</td>
<td>0.14</td>
<td>1.06</td>
<td>0.57</td>
<td>0.53</td>
<td>0.53</td>
<td>Bal.</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>NUCL 167 SPH Gleeble Experimental</td>
<td>0.025</td>
<td>0.074</td>
<td>12.16</td>
<td>17.27</td>
<td>2.42</td>
<td>1.7</td>
<td>0.004</td>
<td>0.03</td>
<td>0.34</td>
<td>0.0008</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>E347-16 All weld metal</td>
<td>0.03</td>
<td>0.03</td>
<td>10.1</td>
<td>19.5</td>
<td>0.2</td>
<td>1.5</td>
<td>0.36</td>
<td>0.5</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>E16.8.2-15 All weld metal</td>
<td>0.041</td>
<td>0.03</td>
<td>8.5</td>
<td>16</td>
<td>1.17</td>
<td>1.78</td>
<td>-</td>
<td>0.22</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>
The path of completion of experiments involved a critical path, as seen in Figure 3.1. The welding experiments were first completed based on welding parameters given from a manufacturer of the hot tanks (details in Appendix A). The welding experiments were inputs for the weld FEM, including parameters and material properties. The welds completed in the lab were used to generate weld samples for neutron diffraction residual stress experiments (details in Appendix B) and extract cross welded samples for Gleeble testing. The weld FEM provided guidance for the Gleeble reheat crack experimental design and the Gleeble FEM. Finally, a combination of Gleeble results, FEM stress results and metallurgical characterization were used to develop the critical stress/strain maps as a function of temperature and recommendations for weld and PWHT design.

It should be noted that a lot of help was received from a post-doc (Dr. Yu Hong) to gather the weld and Gleeble FEM results with the input of welding parameters and material properties gathered from the author. Without his help, the FEM results would be very limited. His guidance and effort in this project helped provide a complete story. His assistance is greatly appreciated.

Figure 3.1 Critical path of experimental and modeling tasks for answering research questions. Welding experiments were first completed to help complete the other tasks in the project, including the Gleeble experiments, needed to gather the critical stress/strain maps for PWHT and weld design.
3.1 Welding methodology

A variety of welding experiments were completed using 347H SS as the substrate plate, with two different thicknesses and two different filler metals (see Table 3.2). W.2 was originally planned to include a third filler (E309L), since this filler was considered as an alternative for reducing SRC susceptibility. However, due to a low carbon content (~0.03 wt%), the service temperature of 565°C is higher than allowed based on ASME code. W.1 welds (2” thick) were completed primarily for metallurgical analysis of the cross section and the initial 2” neutron diffraction experiments. All of the WM Gleeble samples were extracted from two 1” thick welds (two of W.4 and W.3). As a note, the 1” plate batch 1 composition (listed in Table 3.1) was used for the first three welds of W.4, while batch 2 plates were used for making the final W.3 and W.4 welds. Two of the W.4 welds and two of W.1 are used for neutron diffraction bulk residual stress measurements, which one from each being in as-welded condition and the other having a PWHT of 875°C/3 hr. The ref ND in the “purpose” column designates that the weld samples were used to extract reference stress-relieved samples for neutron diffraction measurements.

Table 3.2 Welding experimental matrix (MA=metallurgical analysis; GE=Gleeble extraction; ND=neutron diffraction; ref ND=reference sample extraction for neutron diffraction)

<table>
<thead>
<tr>
<th>Weld ID#</th>
<th>Base Metal</th>
<th>Batch #</th>
<th>Weld Sample</th>
<th>Weld Metal</th>
<th>Thickness (in.)</th>
<th>Heat Input (kJ/in.)</th>
<th>Passes</th>
<th>PWHT</th>
<th>Purpose</th>
</tr>
</thead>
<tbody>
<tr>
<td>W.1</td>
<td>347H</td>
<td>1</td>
<td>1</td>
<td>E347-16</td>
<td>2</td>
<td>Max from WPS</td>
<td>31</td>
<td>-</td>
<td>ref ND, MA</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2</td>
<td></td>
<td></td>
<td>2</td>
<td>40</td>
<td>-</td>
<td>ND</td>
<td>ND</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3</td>
<td></td>
<td></td>
<td>2</td>
<td>40</td>
<td>875°C/3 hr</td>
<td>MA, ND</td>
<td></td>
</tr>
<tr>
<td>W.2</td>
<td>347H</td>
<td>NA</td>
<td>E309L</td>
<td>1</td>
<td>1</td>
<td>Same as W.1</td>
<td>16</td>
<td>-</td>
<td>MA, GE</td>
</tr>
<tr>
<td>W.3</td>
<td>347H</td>
<td>2</td>
<td>1</td>
<td>E16.8.2</td>
<td>1</td>
<td>Same as W.1</td>
<td>16</td>
<td>-</td>
<td>MA, GE</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1</td>
<td>1</td>
<td>E347-16</td>
<td>1</td>
<td>Same as W.1</td>
<td>16</td>
<td>-</td>
<td>ref ND, ND</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1</td>
<td>2</td>
<td></td>
<td>1</td>
<td>-</td>
<td>-</td>
<td>ND</td>
<td>ND</td>
</tr>
<tr>
<td></td>
<td></td>
<td>1</td>
<td>3</td>
<td></td>
<td>1</td>
<td>875°C/3 hr</td>
<td>ND</td>
<td>-</td>
<td>GE</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2</td>
<td>4</td>
<td></td>
<td>1</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
3.1.1 Weld Setup

The weld setup consisted of multiple components, which includes run off and run-on tabs, a copper backing bar with a gas line and water lines, and weld length of 12 inches as shown in Figure 3.2. The copper backing bar was machined at CSM. The cross section of the backing bar with a single-V 50° 1/8” root gap groove is also shown in Figure 3.3. The backing bar, including Argon back purging gas, was used for the open root and hot passes (the first two passes) with the gas tungsten arc welding (GTAW) process. After completion of the first two passes, the backing bar was removed for the remainder of the passes that used shielded metal arc welding (SMAW) process. Argon backing gas was used to prevent heavy high-temperature oxidation (i.e., “sugaring”) on the backside unprotected surface.

Figure 3.2 SolidWorks Assembly of the experimental setup of a 2” thick weld

Figure 3.3 Closeup SolidWorks cross section of weld and backing bar showing machined grooves for water lines and gas lines for back purging with Argon gas

3.1.2 Weld parameters for all welds

The weld parameters used for the weld experiments are summarized in Table 3.3 (for 31-pass weld-W1.1) and Table 3.4 (used for all remaining welds with 2” thick 40-pass and 1”-16
pass welds-W1.2-3, W2 and W3). The first two passes are the root and hot (notice higher currents) passes completed with GTAW followed by SMAW for the remaining passes. The welding process was adopted from the welding procedure specification (WPS) and corresponding pre-qualification records (PQRs) used by Brahma, Inc. for welding of the tank seam welds. The records are displayed in Appendix A. The 2” thick-31-pass weld (W1.1) requires a high deposition rate and heat input, and thus slow travel speeds (minimum allowed by the WPS) were achieved along with a weaving technique to achieve max deposition with the specific weld geometry. However, the 2” thick-40-pass welds (W1.2-3) uses a milder heat input via faster travel speeds with max currents of 155 amps using a stringer deposition method. The same weld parameters used for the 2” thick 40-pass weld were implemented for the 1” thick 16-pass welds, including weld geometry. A maximum interpass temperature of 176°C (350°F) is specified in the given WPS and was followed for all welding experiments, with the highest interpass temperature measured at 93°C using color temperature sticks in order to minimize total weld time. The size of the electrode used for all SMAW welds is 5/32” diameter, while the GTAW wires were 3/32” diameter.

Table 3.3 Weld parameters for 31-pass W.1-1

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>GTAW</td>
<td>Root (1)</td>
<td>11.5</td>
<td>128</td>
<td>1.6</td>
<td>56.4</td>
<td>RT</td>
<td>45</td>
<td>40</td>
<td>6</td>
</tr>
<tr>
<td>GTAW</td>
<td>Hot (2)</td>
<td>13</td>
<td>150</td>
<td>1.9</td>
<td>60.8</td>
<td>RT</td>
<td>45</td>
<td>N/A</td>
<td>7</td>
</tr>
<tr>
<td>SMAW</td>
<td>3</td>
<td>24</td>
<td>150</td>
<td>4.9</td>
<td>44.0</td>
<td>52°C</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>SMAW</td>
<td>4-11</td>
<td>25</td>
<td>150</td>
<td>3.2-4.2</td>
<td>54.4-71.4</td>
<td>52-66°C</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>SMAW</td>
<td>12-15</td>
<td>25</td>
<td>140</td>
<td>3.2-3.6</td>
<td>58.9-66.4</td>
<td>79-93°C</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>SMAW</td>
<td>16-28</td>
<td>25</td>
<td>150/145*</td>
<td>2.7-3.7</td>
<td>60.6-83.0</td>
<td>21-93°C</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>SMAW</td>
<td>29-31</td>
<td>25</td>
<td>145</td>
<td>2.9-3.2</td>
<td>67.1-75.1</td>
<td>52-79°C</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

Table 3.4 Welding parameters for 2”-40 pass welds W1.2-3 and parameters used for 1”-16 pass welds W.3-W.4

<table>
<thead>
<tr>
<th>Process</th>
<th>Pass #</th>
<th>Amp.</th>
<th>Volts</th>
<th>Travel Speed</th>
<th>Arc Energy (kJ/in.)</th>
<th>Deposition Rate (lbs/min.)</th>
<th>Interpass Temp. Range (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GTAW</td>
<td>1</td>
<td>115</td>
<td>11.5</td>
<td>2.2</td>
<td>36.7</td>
<td>0.0096</td>
<td>RT</td>
</tr>
<tr>
<td>GTAW</td>
<td>2</td>
<td>160</td>
<td>13</td>
<td>2.7</td>
<td>46.2</td>
<td>0.0076</td>
<td>RT</td>
</tr>
<tr>
<td>SMAW</td>
<td>3-40</td>
<td>155</td>
<td>23</td>
<td>4.5±0.6</td>
<td>49.1±7.7</td>
<td>0.0541±0.0046</td>
<td>50-135</td>
</tr>
</tbody>
</table>
3.2 Finite element modeling (FEM) of weld simulations

In this study, two types of models are established using ABAQUS: 1) simulation of weld induced stress before and after PWHT, and 2) simulation of Gleeble thermomechanical test. The second model will be discussed after Gleeble experiment methodology. FEM provides comprehensive information of the strain and stress evolution as a function of testing stage, which is critical for understanding the dependence of threshold stress/strain to cracking as a function of temperature and thus the optimization of PWHT parameters.

3.2.1 Model setup, including mesh and boundary conditions

ABAQUS software was used to build the 3D model for the simulation of a 40-pass two-inch single-V groove multi-pass weld and PWHT, as shown in Figure 3.4. Two base plates of 6 inches wide and 12 inches long (152.4 mm × 304.8 mm) with a thickness of 2 inches (50.8 mm) were welded together. The size of the meshes gradually increase from the weld centerline to the edge of the plate to make sure the important information in the weld area will be captured, while minimizing the computation time. In total, there are 72160 elements, with an 8-node linear brick type. The minimum element size is 0.5 mm.

Figure 3.4 (a) FE model mesh of whole 12” x 12” model, (b) meshes of weld zone separated by pass number, and (c) cross section of weld zone of 40-pass experimental sample
Additionally, four other models with different joint geometries and thicknesses were examined to evaluate the effect of plate thickness on residual stress distribution. The conditions include: 1) single-V joint with one-inch thickness, 2) double-V with one-inch thickness, 3) single-V joint with one-half inch thickness, and 4) J-groove with one-inch thickness.

Two boundary conditions are applied for the weld (see Figure 3.5). The clamped boundary conditions include approximately three inches wide area with x, y, and z fixed on the edge of each side of the weld (Figure 3.5a). This boundary condition is maintained for the duration of the welding procedure. After completion of the weld process and cooling back to room temperature, the model is “unclamped” by fixing x, y, and z motion on one corner, fixing y, z motion on another corner and fixing z motion on the third corner and fourth corners (two corners on the other side of the weld as seen in Figure 3.5b) in order to simulate the actual experimental procedure of unclamping after welding completion and complete cooling to ambient temperature. The unclamped condition is maintained for the PWHT simulation as well.

In the models, nonlinear transient heat conduction analysis was performed first to get the thermal history during each welding pass with thermal 8-node DC3D8 element. The geometry of the weld pool and the sequence of each pass used in the model are depicted in Figure 3.4, with different color representing the element sets of each pass. The model change option was used to simulate the weld metal deposition, which means during the first weld pass, the other weld pass elements were killed. After the first weld was finished, the elements for the second pass were made active to simulate the weld metal deposition into the groove. The calculated thermal profile was employed as a thermal body load in the subsequent non-linear mechanical elastic-plastic calculation to obtain the residual stress distribution with different element type C3D8R. Clamped condition was considered in this simulation by adding boundary condition to fix the clamping area during the welding simulation. Then the clamping boundary condition was removed after the welding simulation to determine the effect of unclamping on stress redistribution.
Double-ellipsoidal heat source model developed by Goldak [52] was used to simulate the weld heat input. Figure 3.6 demonstrates the Goldak model and example of temperature distribution during weld pass 5 during a weld thermal cycle in the 40-pass weld model. The front and rear of the heat flux are described by Eqs. (3.1) and (3.2), respectively:

\[
q_f(x, y, z) = \frac{6\sqrt{3}f_f \eta Q}{a_f b c \sqrt{\pi}} \exp\left(-\frac{3x^2}{a_f^2} - \frac{3y^2}{b^2} - \frac{3z^2}{c^2}\right) \tag{3.1}
\]

\[
q_r(x, y, z) = \frac{6\sqrt{3}f_r \eta Q}{a_r b c \sqrt{\pi}} \exp\left(-\frac{3x^2}{a_r^2} - \frac{3y^2}{b^2} - \frac{3z^2}{c^2}\right) \tag{3.2}
\]

where the front and rear quadrant fractions of the ellipsoids, \(f_f\) and \(f_r\), are set as 0.6, 1.4 respectively. The heat input \(Q = V \times I \times \eta\), where \(\eta = 0.8\), and voltage (V) and current (I) are dependent on the pass number (see Table 3.4). The travel rate of the heat source is dependent on the weld speed specific to each pass. \(a_f, a_r, b\) and \(c\) are dependent on the specific pass number so that the region of solidification pertains to the width and depth of the corresponding elements. The interpass temperatures were maintained similarly as the experiments (~80°C), which included roughly a 20-minute cool time after the completion of each pass prior to each succeeding pass. Convective cooling was allowed with an ambient temperature boundary condition of 20°C.
Another example of thermal calculation is demonstrated in Figure 3.7, which records the temperature profiles at three nodes during pass 31 of a 40-pass weld. The three nodes monitored are located: 1) in the WM (peak temperature of 1843°C) that experienced melting ($T_m \approx 1425-1450$°C), 2) in the HAZ that experienced a peak temperature of 1354°C, and 3) the unaffected BM with a peak temperature of 774°C. The temperature profiles demonstrate heating and cooling rates commonly seen in weld thermal cycles.
3.2.3 Material properties

The temperature dependent material properties are tabulated in Table 3.5, which includes density [53], specific heat [54], thermal conductivity [54], Young’ Modulus and Poisson’s ratio [54], thermal expansion coefficient [54], and electrical conductivity [55]. Note that the material properties were assumed to be the same for both E347 weld and 347H base metal since the weld model simulates the case with a matching filler metal.

Table 3.5 Material temperature dependent mechanical and thermal properties [53-55]

<table>
<thead>
<tr>
<th>Temp. (°C)</th>
<th>Density (E-9 kg/mm^3)</th>
<th>Specific heat (E+8 mJ/(K*kg))</th>
<th>Conductivity (mW/(mm*K))</th>
<th>Young’s modulus (GPa)</th>
<th>Thermal expansion coefficient (E-6/s)</th>
<th>Poisson’s ratio</th>
<th>Electrical conductivity (Ω-mm^-1)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>7.9</td>
<td>4.42</td>
<td>15</td>
<td>200</td>
<td>16.5</td>
<td>0.278</td>
<td>1389</td>
</tr>
<tr>
<td>100</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>17.5</td>
<td>-</td>
<td>1282</td>
</tr>
<tr>
<td>200</td>
<td>7.8</td>
<td>5.15</td>
<td>17.5</td>
<td>185</td>
<td>18</td>
<td>0.288</td>
<td>1163</td>
</tr>
<tr>
<td>300</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>400</td>
<td>7.7</td>
<td>5.63</td>
<td>20</td>
<td>170</td>
<td>18.5</td>
<td>0.298</td>
<td>1000</td>
</tr>
<tr>
<td>500</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>19</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>538</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>156</td>
<td>0.298</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>600</td>
<td>7.6</td>
<td>5.81</td>
<td>22.5</td>
<td>153</td>
<td>19</td>
<td>0.313</td>
<td>901</td>
</tr>
<tr>
<td>700</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>19.5</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>800</td>
<td>7.5</td>
<td>6.09</td>
<td>25.5</td>
<td>135</td>
<td>20</td>
<td>0.327</td>
<td>826</td>
</tr>
<tr>
<td>850</td>
<td>7.45</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>900</td>
<td>7.4</td>
<td>-</td>
<td>-</td>
<td>20</td>
<td>-</td>
<td>-</td>
<td>794</td>
</tr>
<tr>
<td>1000</td>
<td>-</td>
<td>6.31</td>
<td>28.3</td>
<td>96</td>
<td>20.5</td>
<td>0.342</td>
<td>-</td>
</tr>
<tr>
<td>1100</td>
<td>7.3</td>
<td>-</td>
<td>-</td>
<td>70</td>
<td>21</td>
<td>0.342</td>
<td>-</td>
</tr>
<tr>
<td>1200</td>
<td>-</td>
<td>6.54</td>
<td>31.1</td>
<td>50</td>
<td>21</td>
<td>0.35</td>
<td>-</td>
</tr>
<tr>
<td>1300</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>10</td>
<td>22</td>
<td>0.351</td>
<td>-</td>
</tr>
<tr>
<td>1340</td>
<td>-</td>
<td>6.69</td>
<td>33.1</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>1400</td>
<td>-</td>
<td>6.75</td>
<td>-</td>
<td>22.5</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>1500</td>
<td>7.2</td>
<td>-</td>
<td>-</td>
<td>22.5</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

In addition to the properties already listed, the temperature dependent flow stress/strain data was extracted from Gleeble thermomechanical experiment results and NIMS data sheets [14] from 20-1050°C (Figure 3.8). As will be discussed later, the stress upon reaching the temperature and pre-strain level (step 6 in the methodology) in the 347H HAZ Gleeble reheat crack tests was used to construct the 600-1050°C stress strain curves, while the true stress strain curve was extracted from room temperature (20°C) step 3 data. Notice there is no strain hardening effect at 1050°C, which is a reasonable assumption as there is no resistance to multiple cross slip at early strains.
3.2.4 Setup of PWHT model

As mentioned in the former report, Norton’s creep law model was employed in this study to investigate the effect of PWHT on residual stress. The Norton’s creep law can be represented by Eq. 3.3:

$$\dot{\varepsilon} = K \sigma^n$$

(3.3)

where $\dot{\varepsilon}$ is the creep strain rate, $\sigma$ is the applied stress (residual stress in this study) and $K$ and $n$ are material constants for each temperature [56, 57]. There are only a few literature studies focusing on the creep test of 347H SS based on the Norton creep law equation [58-60], which could provide useful data as inputs to build the numerical model for simulating the stress relief behavior during the PWHT process. In this work, PWHT simulation was carried out using a combination of literature and experimentally collected creep parameters (see Table 3.7). A user-defined subroutine was developed for the PWHT model, and the schematic of a calculation loop of stress and creep strain update is shown in Figure 3.9. The temperature used for PWHT analysis is primarily a single step at 950°C with a heating rate on the surface equivalent to the heating rate used for the Gleeble experiments at 58°C/min.
Figure 3.9 Flow chart of stress and strain calculation during the PWHT simulation using creep law in ABAQUS

### 3.3 Gleeble SRC test methodology

The objective of the Gleeble reheat cracking weldability test is to determine the reheat cracking susceptibility during PWHT for four sets of samples:

1. 347H plate with simulated HAZ thermal cycles
2. NUCL 167 SPH plate (modified 316L with Boron additions) with simulated HAZ thermal cycles (alternative base alloy)
3. Cross welded E347-347H weld samples for determining E347 WM susceptibility
The intention of this test methodology was to determine critical stresses and strains (minimum values where microcracks begin to form) as a function of temperature and rank the SRC susceptibility of the four sets. The most recent study that ranks alloys to their SRC susceptibility comes from Lehigh University including Haynes 282 and 230, Inconel 740H and 617, and 347H [6]. 347H, Haynes 282 and 740H ranked the highest in SRC susceptibility using a specific methodology shown in Background section. This test methodology did not account for stress reduction during heating, which was incorporated in this Gleeble reheat crack test at Colorado School of Mines (CSM). In addition, our tests incorporated larger variations of stress/strain and temperature for fewer sets of materials.

3.3.1 Sample Geometries

A close-up of the Gleeble 347H SS tensile specimen (shown in the Gleeble grips) displays a c-gauge clamped in the center of the reduced gauge section where the thermocouples are placed, which is the geometry used for three material sets (347H HAZ, E347, and E16.8.2) (Figure 3.10). The grips used are stainless steel that allow for a more-steady, shallower temperature gradient than copper grips. The c-gauge displacement during tensile testing is used to measure the true diametral strain when pulling to a desired strain. This is used as an alternative to a longitudinal extensometer to measure longitudinal strain. The true diametral strain (Eq. 3.4) is calculated with inputted change in the diameter (assuming uniform plastic strain with volume conservation):

$$\varepsilon = 2 \ln \left(\frac{d_o}{d_o + \Delta d}\right)$$  \hspace{1cm} (3.4)

With $d_o$ being the initial diameter (mm) of the gauge section measured and $\Delta d$ as the in-situ change in diameter due to thermal strain, plastic strain, and creep strain.

The true stress (Eq. 3.5) can be calculated with the change in the diameter and the measurement of force (F, kN) from the load cell:

$$\sigma = \frac{F}{\pi \left(\frac{(d_o + \Delta d)^2}{4}\right)}$$  \hspace{1cm} (3.5)

As temperature varies, the true stress will be calculated since the diameter is measured in-situ.
The 347H HAZ samples were extracted from 5/8” thick solution annealed plates (1080°C anneal after hot rolling followed by water quenching). The 347H HAZ tensile samples were machined with the uniaxial stress applied perpendicular to the rolling direction (RD), i.e., parallel to transverse direction (TD) of the plates for all samples. The manufacturer reported bulk chemical composition of the substrates are tabulated in Table 3.1 and compared to the standardized limitations of 347H according to ASTM A240 [10]. The carbon content in wt% is 0.05, and the Nb content is 0.58 wt%.

The cross welded samples were all extracted from one-inch-thick welds perpendicular to the weld direction to maximize number of samples to be extracted from the 16 pass-1” thick welds (W.3 and W.4). The gauge section includes both the WM and HAZ on both sides of the fusion boundary (~20 mm width) so that these represent cross-welded samples (see Figure 3.11). The samples were extracted at roughly the ¼ thickness position (6 mm) from the top side of the joint. Thus, the gauge section includes passes 6-13 in the 16-pass 1” thick weld, with most of the gauge section making up of passes 8-11. The gauge section exists within the top subsurface region of the weld, which is the most desired region due to significant residual stresses existing in this region. The width of the WM is generally on average 18-20 mm located at the center of the 32 mm (~1.25 in.) long gauge section. It should be noted that the E16.8.2 samples had a slightly off-center gauge section by 2 mm; however, the center of the gauge section is mostly WM. The temperature gradient, inherent due to the resistance heating nature of the Gleeble, ensures failure within the WM for SRC tests. Another representation of the sample’s extraction
geometry relative to the bulk weld and welding direction is illustrated in Figure 4.6. All the samples extracted from the welds were taken within the center 9-10 inches of the weld along the welding direction, which were enough to provide 18-20 samples per multi-pass weld, while providing samples within a reasonable steady state portion of the 12-inch weld.

![Diagram of weld geometry](image)

Figure 3.11 Gleeble sample extracted from 1” thick weld in transverse direction

The second set (NUCL 167 SPH HAZ) of samples were extracted from one-and-a-half-inch plate with the same pulling orientation as the 347H HAZ samples (stress applied along the TD normal to the RD). A different geometry (flat dog-bone) was used to reduce machining time and costs since the samples were extracted from 1.5” thick plate as provided by ArcelorMittal (see Figure 3.12). However, the same gauge length as the round tensile samples were used (1.25 inches) and the c-gauge was used to measure the in-situ change of the gauge width (w). The strain and cross section area were calculated using the measured width (w), and the rate of change was used to determine the change in the thickness direction (t) for a final area calculation. Simpler versions of the strain (Eq. 3.6) and stress (Eq. 3.7) equations are used as follows:

\[ \varepsilon = \ln \left( \frac{A_o}{A_o + \Delta A} \right) \quad (3.6) \]

and

\[ \sigma = \frac{F}{A_o + \Delta A} \quad (3.7) \]
with $A_0$ representing the initial area and $\Delta A$ representing the change in area in-situ. A tolerance of 20 thousandths was used for the samples; however, the initial area values for each sample were recorded separately.

Figure 3.12 Dimensions of the dog-bone flat tensile sample for NUCL 167 SPH samples (inches)

3.3.2 Reheat cracking Gleeble test procedure

To physically simulate SRC in the HAZ/partially-melted-zone (PMZ) samples, multiple steps are needed, as illustrated in Figure 3.13, which includes stress, stroke, and temperature as a function of time.

1) Apply thermal cycle based on FEM results with a heating rate of 66°C/s to the designated peak temperature and held at peak temperature for 1 second while “Force” control is equal to 0 using an inert atmosphere (<30 ppm O$_2$).
2) Control cooling to room temperature (with an initial cooling rate of 16 °C/s down to 1200°C, 23°C/s from 1200°C to 800°C, 9°C/s from 800°C to 500°C, 2.5°C/s from 500°C to 300°C, followed by air cooling to room temperature).
3) Pull to desired stress/strain representative of the welding condition at room temperature (RT) with a 0.001 s$^{-1}$ strain rate.
4) Then, switch to “Stroke” (i.e., displacement) control and set the current stroke value as the “0” point.
5) Heat up to designated temperature at 58 °C/min related to in-service conditions or PWHT temperatures (e.g., 600 - 1050 °C) while pulling the stroke to account for thermal expansion (Stroke value depends on the reheat temperature.).
6) While still in “stroke” control, hold at temperature until failure or after 24 h of non-failure.

7) Cool to ambient temperature if no failure after 24 h. If load drops to near 0, the “force level heat mute” command will be triggered, and the sample will return to room temperature. Failure may occur during cooling, since samples are constrained.

8) Repeat for another 24 h if failure does not occur the first 24 h:
   a. Heat up to the same temperature under a 0kN force.
   b. Pull to the ending load before the first 24-h test finished.
   c. Constrain the stroke from moving.
   d. Wait for 24 h or until failure.

Figure 3.13 Six step Gleeble reheat crack test methodology displayed with stress, stroke, and temperature as a function of time for HAZ simulations (1335°C HAZ peak temperature, 0.174 pre-strain, and 800°C reheat temperature condition shown). (NOTE: WM tests use the same methodology, except steps 1-2 (HAZ thermal cycle))

The cross welded WM samples did not include steps 1-2, since the microstructure in the machined, as-received condition already contains the desired starting microstructure. The remaining steps were applied similarly to the HAZ tests. Note that the mechanical properties of the two regions are different, particularly the 0.2% offset yield strength.
3.3.3 Steps 1-2: HAZ thermal cycles

The first two steps of the reheat crack test for the 347H HAZ samples include a weld HAZ thermal cycle with one of three peak temperatures (1150, 1275, and 1335°C) under a zero load (Figure 3.14). The thermal cycle was applied ensuring a chamber oxygen content less than 30 parts per million (ppm) to prevent heavy surface oxidation, since the shielding gas during the welding process helps protect the FZ and surrounding HAZ from oxygen contamination. Thus, a combination of a rough vacuum and purging the chamber with Argon is used to provide a near oxygen (air) free atmosphere with 20 ppm as the most accurate value. The heating and cooling rates are the same for every simulation. There is noticeable liquation for the 1335°C samples, as will be shown later.

![Figure 3.14 Thermal cycles used for generating HAZ microstructures in 347H with 1150, 1275, and 1335 °C peak temperature (details listed on steps 1-2 for reheat crack test)](image)

Thermocalc simulations, using PRISMA interface, were implemented to determine the effect of cooling rate on Nb (C, N) precipitation kinetics during the weld thermal cycle for two heterogenous nucleation sites: 1) grain boundaries, and 2) dislocation cores. Figure 3.15 shows a 0.001 volume fraction detection limit (about a sixth of the equilibrium phase fraction amount of Nb (C, N) in γ-austenite) to show the smallest amount during cooling, assuming cooling from 1335°C with zero Nb (C, N) in solution at time = 0. Based on the simulation predictions, grain
boundary precipitation is most likely to occur during the HAZ cooling cycle, while a much lower nucleation rate is present at the dislocation cores (assuming a dislocation density of $10^{12}$ m$^{-2}$). A minimum $9^\circ$C/s cooling rate would be needed to reach 0.001 volume fraction of Nb (C, N) present on dislocation cores, while a minimum of $27^\circ$C/s is needed to reach a 0.001 volume fraction of Nb (C, N) precipitation on grain boundaries. The main conclusion from this analysis is that the majority of the Nb (C, N) precipitation occurs along grain boundaries, while there is less precipitation along dislocation cores. Upon reheating during steps 5-6, there will be a driving force for continued nucleation and growth of Nb (C, N) precipitates while under a stress. Since step 3 leads to an increasing plastic strain prior to reheating, the time to formation of the dislocation Nb (C, N) is proportional to the amount of pre-strain applied. This mechanism helps explain the influence on intragranular precipitation on the cracking mechanisms present on grain boundaries.

Figure 3.15 Continuous cooling transformation (CCT) curve calculations, using Thermocalc©, showing three different cooling rates, including the 347H HAZ cooling rate of 23°C/s with respect to a 0.001 volume fraction detection of grain boundary (GB) Nb (C,N) and dislocation Nb (C,N) precipitation in $\gamma$-austenite using 347H experimental composition.

The 167 SPH samples had the same peak temperature of 1335°C only and same heating rate of 66°C/s; however, the cooling rates were slightly slower since the area is larger than the 347H HAZ samples. The generated microstructure will be discussed in detail in later experimental result sections.
3.3.4 Steps 3-4: Apply stress at room temperature

Steps three and four follow the completion of the HAZ thermal cycle (or first step for Gleeble WM tests). Room temperature tensile tests were performed for E347-347H, E16.8.2-347H, and NUCL 167 SPH HAZ (peak 1335°C) samples, as summarized in Figure 3.16, to generate the true stress-strain curves. These data are used to determine the pre-applied plastic strains for desired stress levels, especially for the 0.2% offset yield strength. As a comparison, room temperature tensile test was not performed on 347H HAZ material, and therefore two stress-strain curves with strains up to 0.1 and 0.174, respectively, from SRC tests of the 347H HAZ samples (which is generated with a heat treatment temperature of 1335°C) are included to demonstrate the yielding behavior, as shown in Figure 3.16. Here are some of the results observed from the data:

- The 0.2% offset yield strength for WM is approximately two times higher than the HAZ generated with a 1335°C peak temperature.
- The E347 WM is slightly stronger than the E16.8.2 WM. It should be noted that failure in the E16.8.2 WM -347H cross weld sample resulted in necking in the HAZ/base metal of the 347H, which means the E16.8.2 WM may have higher toughness than the 347H HAZ and BM. This behavior was not observed with the E347-347H samples, for which failure occurred in the WM.
- The true stress at 10% true strain of 347H HAZ is equivalent to the yield strength of the E347 WM (~450 MPa). Since the yield strength is different between WM and HAZ, the room temperature stress values, rather than strain, will be used for comparisons of SRC susceptibility between the HAZ and WM samples.
- One test with a known void in the gauge section (W.4-1) greatly reduced the ductility of the E347 WM sample, but still exhibited the same yield strength as a defect free sample from the second weld (W.4-4). The presence of already existing defects greatly increases crack susceptibility.
- The yielding behavior of 347H HAZ and NUCL 167 SPH HAZ generated from 1335°C peak temperatures Gleeble heat treatment behaves similarly at room temperature.
Figure 3.16 Room temperature true stress vs. true strain results for all four sets of materials

3.3.5 Step 5: Stroke calibration for heating up step at 58°C/minute

After switching to stroke mode in step four, the sample heats up to temperature while under zero stress for step five. All samples undergoing a SRC test require a stroke calibration curves, as illustrated in Figure 3.17, with a heating rate of 58 °C/min. The data was extracted from the thermomechanical tests that ramped up to 1050°C. To avoid stress reduction caused by thermal expansion, additional stroke during heating was applied and the value depends on the designated temperature. The designated stroke value (see Table 3.6) for all tests was ramped in the same heating step at a linear rate. The Gleeble FEM provides comprehensive information of the thermomechanical profile experienced by the samples, such as additional plastic strain potentially introduced during this step, that the Gleeble experiment won’t be able to provide. Generally, the cylindrical samples all behaved similarly in terms of thermal expansion rates, but the flat dog-bone sample used for NUCL 167 SPH had more stroke compared to the cylindrical sample data set due to a larger cross-sectional area (i.e., more material undergoing thermal expansion). Specific stroke values were collected independently from the calibration curves for each material and was applied consistently for all the same target temperatures but with variations of pre-strain/stress.
3.3.6 Experimental Variables

The three main variables for the HAZ tests are: 1) peak temperature during heat HAZ thermal cycle, 2) pre-strain/stress during start of SRC test and after HAZ thermal cycle, and 3) the reheat temperature during the reheat crack tests. Each of the variables has a variation range of parameters listed below for both 347H HAZ and 167 SPH HAZ. Note that the conditions underlined are for both 347H and 167 SPH, while the others are for 347H only:

1) Peak CGHAZ temperature at 1150, 1275, 1335 °C;
2) Pre-strain/stress of 275 MPa, 0.01 strain (250 MPa), 0.025 strain (300 MPa), 0.04 strain (335 MPa), 0.05 strain (350 MPa), 0.08 strain (415 MPa), 0.1 true strain (~450 MPa) and 0.174 true strain (~600 MPa);

3) Reheat Temperature: 600, 750, 800, 900, 950, 1000, and 1050°C

Two-step PWHT tests were conducted to get a better understanding of potential two-step PWHT. 550, 600 and 750°C were the first step (held for a couple hours) followed by heating to 950 or 1050°C. These tests did not show dramatic differences from the single step experiments in terms of time to failure. Therefore, only five tests were completed with a two-step method. The single step PWHT tests were the primary focus of these experiments. The summary table of variables and test results are tabulated in Table 5.1 and Table 5.5.

Some of the low strain tests did not result in failure during isothermal hold due to stress relaxation to near zero, and during the subsequent cooling stage while constrained no failure occurred either. The results are categorized based on whether failure occurred during cooling or while at the designated holding temperature.

The two main variables for the WM SRC tests are 1) initial room temperature stress and 2) temperature. The specific variable values for E347 and E16.8.2 samples are listed below with conditions underlined for both E347 and E16.8.2. Those in italics are for E16.8.2 only and the others for E347 only:

1) Stress: 155, 220, 280, 325, 400, 475-520 (1% strain) and 550-590 (6% strain)
2) Temperature: 800, 850, 900, 950, 1000, and 1050°C

Table 5.6 has the specific values listed along with the specific weld each test sample was extracted from.

3.4 Gleeble FEM

Finite element analysis (FEA) of the Gleeble tests was established to gain a comprehensive understanding of the strain and stress evolution during reheat test and generate the creep database necessary for PWHT simulation. ABAQUS software was used to build the 3D model for the Gleeble test, and the mesh model and boundary conditions are shown in Figure 3.18. The total specimen length is 119.6 mm, the gage length is 31.75 mm, and radius of the gage section is 3.175 mm. The element size has a minimum value of 0.59 mm, and a maximum value
of 1 mm. In total, there are 15360 elements, with an 8-node linear brick type. In the model, a thermal-electrical-structural analysis was performed with element type Q3D8. This fully coupled analysis provided temperature distribution and non-linear mechanical elastic-plastic calculation to obtain the stress and strain distribution. Concentrated current was loaded on grip area to heat the sample. Displacement loading were applied on the side area to simulate the strokes in the Gleeble test. The model accounts for a boundary condition of 10°C on the surface where the stainless-steel grips are.

![Figure 3.18 FEA simulation model of Gleeble test sample](image)

The same material and mechanical properties used for the weld FEM in section 3.2.6 were used for this model. In addition, the creep parameters were determined based on Gleeble experiment results for 347H SS in the HAZ condition. The measured data depends on the c-gauge to measure creep strain and strain rate and the change in stress during step 6 (isothermal hold). An example of creep strain and strain rate for the 750°C with 0.174 pre-strain (sample 6) is illustrated in Figure 3.19. The creep data extracted was estimated to be secondary creep, in which a minimum creep rate (as observed in steady state creep) was achieved with increasing time. Since stress relaxation occurs with decreasing strain rate and increasing time in contrast to constant load/stress creep tests, the parameters were generally extracted from steady state creep rate regions from the extracted creep strain and strain rate curves. The general creep rate equation (Eq. 3.8) is expanded into the format in Eq. 11:

$$\dot{\varepsilon} = K \sigma^n = A^* \sigma^n \exp \left( -\frac{Q}{RT} \right)$$

(3.7)

where $A^*$ is a creep constant, $Q$ is the activation energy (kJ/mol), and $R$ is the universal gas constant of 8.314J/mol*K. $K$ is a creep constant that includes the temperature dependent exponential term and $A^*$. 68
The creep data is plotted in ln(\(\dot{\varepsilon}\)) vs ln(\(\sigma\)), so that the slope of the line is n (creep parameter), and ln(K) (constant) is the intercept (Figure 3.20) due to the natural log form of the creep rate equation (Eq. 3.9):

\[
\ln(\dot{\varepsilon}) = \ln(K) + n \ln(\sigma)
\] (3.8)

K and n were collected from 750, 800, 850, 900, 950, 1000, and 1050°C tests that had sufficient steady state creep and time to collect information before cracking (lower pre-strain and stress ranges for higher temperature tests). The creep data was broken down for multiple temperature and stress ranges (Table 3.7) so that smooth transitions in creep strain were observed during heating and isothermal temperature steps in the Gleeble FEM (steps 5-6 of Gleeble tests). Creep behavior was assumed to be negligible below 625°C as the creep rates are very small to be effective. Thus, no creep strains were calculated below 625°C.

![Graph](image.png)

Figure 3.19 Example of creep strain and strain rate collected from c-gauge during steady state creep at isothermal holding step for 750°C/0.174 strain condition (sample 5)
Figure 3.20 Creep data from Gleeble experiments used for FEM

Table 3.7 Creep parameters obtained from Gleeble experiments for temperatures ranging from 625-1075°C (grey-interpolated; white-experimentally fitted). Q-activation energy (assumed constant), R-gas constant

<table>
<thead>
<tr>
<th>Temp range (°C)</th>
<th>n</th>
<th>A*</th>
<th>K</th>
<th>Q (J/mol)</th>
<th>R (J/mol. K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(+∞, 625)</td>
<td>0.00</td>
<td>0</td>
<td>0</td>
<td>556400</td>
<td>8.314</td>
</tr>
<tr>
<td>[625,675)</td>
<td>5.45</td>
<td>6.66E+09</td>
<td>2.18E-22</td>
<td>556400</td>
<td>8.314</td>
</tr>
<tr>
<td>[675,725)</td>
<td>5.45</td>
<td>6.66E+09</td>
<td>9.06E-21</td>
<td>556400</td>
<td>8.314</td>
</tr>
<tr>
<td>[725,775)</td>
<td>5.45</td>
<td>6.66E+09</td>
<td>2.61E-19</td>
<td>556400</td>
<td>8.314</td>
</tr>
<tr>
<td>[775,825)</td>
<td>5.41</td>
<td>3.66E+09</td>
<td>3.02E-18</td>
<td>556400</td>
<td>8.314</td>
</tr>
<tr>
<td>[825,875)</td>
<td>4.83</td>
<td>2.36E+10</td>
<td>3.12E-16</td>
<td>556400</td>
<td>8.314</td>
</tr>
<tr>
<td>[875,925)(^1)</td>
<td>4.06</td>
<td>7.06E+10</td>
<td>1.19E-14</td>
<td>556400</td>
<td>8.314</td>
</tr>
<tr>
<td>[925,975)(^2)</td>
<td>1.77</td>
<td>2.18E+14</td>
<td>3.23E-09</td>
<td>556400</td>
<td>8.314</td>
</tr>
<tr>
<td>[975,1025)(^3)</td>
<td>1.46</td>
<td>2.81E+14</td>
<td>3.03E-08</td>
<td>556400</td>
<td>8.314</td>
</tr>
</tbody>
</table>

\(^1\) The parameter is adjusted based on creep data (derived from 80-120MPa within the total range of 53-112 MPa)
\(^2\) The parameter is adjusted based on creep data (derived from 40-47MPa within the total range of 8-55 MPa)
\(^3\) The parameter is adjusted based on creep data (derived from 42-51MPa within the total range of 42-57 MPa)

3.5 Gleeble Thermomechanical Testing at 600°C with and without PWHT

All four sets of microstructures were tensile tested at 600°C after a thermomechanical and heat treatment process. Both 347H and NUCL 167 SPH substrates were given a 1335°C peak temperature thermal cycle followed by a 10% plastic strain at room temperature to simulate a HAZ with accumulated plastic strain (like the first three steps of the SRC test methodology).
Then, the load dropped to 0 prior to the heat treatment. The WM sets did not include these thermomechanical steps as the microstructure in its given state is the desired starting microstructure. Each set underwent at least three types of treatments with the following conditions prior to pulling to failure at 600°C:

1. No PWHT and 4 hr. service age at 600°C
2. 950°C-2 hr. PWHT followed by 4 hr. age at 600°C
3. 1050°C-30 min PWHT followed by 4 hr. age at 600°C

The 347H HAZ data set included two more tests with the same service age condition: 1) 2-step PWHT at 750°C-2hr with a second step of 1050°C for 30 minutes, and 2) an overheated sample to 1200°C for approximately 30 seconds during a 950°C heat treatment for 2 hr. The profiles for four heat treatments are summarized in Figure 3.21. The strain rate applied was 0.001/s (approximate displacement rate of 3 mm per minute). The same heating rate and cooling conditions as the SRC tests are applied for thermomechanical tests.

Figure 3.21 Thermomechanical test heat treatments prior to tensile test at 600°C
CHAPTER 4: WELD EXPERIMENTAL AND FINITE ELEMENT MODEL RESULTS

The welding experimental characterization results and weld FEM stress results are shown in this chapter. The welds developed in the lab were used to develop the weld FEM. Some initial neutron diffraction measurements of elastic strain are shown in Appendix B for the 2” thick FEM validation. There are additional neutron diffraction experiments that were completed for 1” thick welds that were intended to validate the 1” thick FEM. However, those results have not yet been collected. Thus, the FEM results here are assumed to be accurate based on the trends seen in the 2” thick weld using one strain direction (transverse direction). Those are shown here, with the specific information shown in Appendix B.

4.1 Weld Experiment Characterization

4.1.1 2” thick-31 pass weld (W1.1)

Figure 4.1 summarizes the microstructure characteristics observed in W1.1 weldment, including the weld sequence. In all the weld metal micrographs, ferrite etches dark, while austenite etches white. Figure 4.1b shows a typical single pass weld metal microstructure in pass 30, which consists of acicular and skeletal ferrite dendrites. Figure 4.1c reveals a “re-heated” region between pass 30 and 26, which shows very minimal ferrite content (small equiaxed ferrite) at the base of pass 30. Figure 4.1d shows the fusion boundary of pass 26, which contains a ferrite gradient when transiting from the HAZ into the FZ. Figure 4.1e shows fine equiaxed microstructure in the root pass as a result of the fast-cooling rate, indicating a fast growth rate at the root, which is associated with the water-cooled Cu backing plate. It should be noted that solidification rates (known as growth rate, R) and temperature gradients (G) greatly affect the FZ grain morphology (e.g., columnar dendritic to equiaxed dendritic transition). In W1.1 weld specimen, a relatively higher G/R ratio leads to more columnar dendritic microstructure in Figure 4.1b-d while a lower G/R ratio leads to an equiaxed dendritic microstructure in Figure 4.1e.

Ferrite measurements in the WM and HAZ are of significance in terms of potential embrittlement for high temperature applications. Little amounts of ferrite in the weld metal (~2-3% ferrite) is desirable for mitigating hot cracking (aka solidification cracking). On the other hand, ferrite may transform to sigma phase during service temperatures based on a diffusion-based phase transformation where ferrite dissolves at the expense of sigma phase nucleation and
growth [12, 62], which can lead to high temperature embrittlement. An understanding of the ferrite content in the as-welded condition may be necessary for mitigating high temperature embrittlement.

**Figure 4.1** Microstructure representation of 2”- 31-pass weld: (a) macro cross section of weld, (b) FZ microstructure of pass 30 indicating columnar morphology, (c) microstructure of intersection between pass 26 and 30 showing reduction of ferrite in pass 30 due to reheating effects, (c) transition microstructure of FZ and HAZ of pass 26, and (d) FZ microstructure of root pass showing equiaxed microstructure.

Ferrite scans across the top layer of the 31-pass weld reveal the ferrite behavior across the weld (see Figure 4.2). Gradients of ferrite begin in the HAZ and increase rapidly into the WM (~2 → 8% ferrite). There are dips in ferrite content that exist within the weld metal region. These dips may represent the intersectional zones between the weld passes. There is a dip from 12 to 10% near 0.45 on the x-axis, which is the intersection between passes 29 and 31, and another dip from 12 to 10% ferrite within 0.6-0.7 on the x-axis, which is the intersection between passes 30 and 31 (see Figure 4.1a where the red boundaries represent the intersection between passes). The top passes had relatively higher reading of ferrite (~12-14%), while the root pass measured about ~8.5% ferrite (not shown in figure). Thus, the middle passes in the weld closer to the top surface (most undiluted weld metal) are most susceptible to sigma embrittlement.
Figure 4.2 Ferrite scan across the top layer of 31pass-2” thick weld

The top surface CGHAZ are regions of specific interest due to the highest Von Mises residual stress existing sub surface near the top (shown in later sections). Figure 4.3 below shows a scan of micrographs in the HAZ and BM adjacent to pass 29 (top left pass in Figure 4.1a with an average arc energy per unit length of 70.9 kJ/inch). The grains in the CGHAZ near the FZ showed sizes up to 83 µm in diameter, which is significantly greater than the ASTM grain size of 6 (~45 µm) that is labeled on the material test report (MTR) for the received 347H SS weld plates. Image 1 to 10 represents the microstructures transiting from CGHAZ, fine grained HAZ (FGHAZ), and base metal. BM has an approximate grain size closer to 45 µm. The peak temperature and cooling rate experienced during the weld cycle of pass 29 cause the difference in grain sizes in the different regions, e.g., CGHAZ experiences temperatures above 1200°C. With a avg. arc energy per unit length of 70.9 kJ/in, a relatively small cooling rate occurs which then allows for coarse grain growth. It should be noted that pass 29 represents the maximum allowed arc energy per unit length specified in the Brahma WPS. Thus, slower cooling rates and high heat inputs create a CGHAZ with large, coarse grains.
4.1.2 2” thick-40 pass weld (W1.2-3)

The 40-pass weld has 18 vertical layers, which is slightly different from the 31-weld pass. After pass 7, each succeeding layer has 2 passes and so on to accommodate a maximum fusion zone width of a half-inch (recommendation from Table 3.5 in AWS D1.6-Structural Welding Code-Stainless Steel [63]). Note that the very top layer has five passes. An example of a 40-pass weld (W.1-2) shows significant distortion even before unclamping, indicating significant residual distortion developing during the welding process (Figure 4.4), which is like the 31-pass welds and 1” thick welds as well. More clamping force is needed to prevent distortion, which would ultimately increase residual stresses.

Metallurgical characterization was not emphasized as much on the 40-pass 2” thick welds, since all of the Gleeble samples were extracted from the 1” thick welds. However, similar characteristics can be seen between the two different thicknesses since the same welding parameters and weld consumables were used. The effect of PWHT (875°C/3 hours) on microstructure can be seen in the W.1-3 weld (Figure 4.5). Electrolytic etching was performed on the polished surface of the welds for 20 seconds with roughly 30% nitric acid solution in water and 2-3 V. A mixture of coarse columnar morphology of the solidification grains and finer columnar solidification grains was revealed. The finer grains are representative of “cold areas” that exist along 1) fusion boundary or 2) reheated weld metal undergoing recovery and recrystallization during welding reheating or during PWHT. The preceding weld metal and base metal microstructure (texture orientation and grain size) and temperature field determines the
evolution of succeeding weld metal microstructures. The epitaxial growth initiates instantaneously from the fusion boundary followed by grain growth towards the weld center following the maximum temperature gradient. The relatively slow cooling rate away from the fusion boundary leads to formation of coarser columnar grains in comparison to the finer grains forming at the fusion boundary as shown in Figure 4.5. Further grain coarsening could take place during PWHT at 875°C. The grain structure, including morphology, tortuosity and orientation of stress relative to long, straight grain boundaries, influences the susceptibility to reheat cracking. The coarse columnar grains would have lower strength, so that cracking, with sufficient stress, would initiate and propagate most likely along coarser solidification grain boundaries. It should be noted that no microcracking was detected visually when inspecting the cross section after PWHT.

Figure 4.4 Example of weld (W.1-2) a) after completion of root pass from top view, b) top view after completion of pass 40, and c) side view after completion of 40 passes showing angular deformation (measured to be 14°)
4.1.3 1" thick-16 pass welds (W.3 and W.4)

The one-inch thick 16-pass welds were used primarily for the purpose of obtaining Gleeble extraction samples. The weld parameters used were like the two-inch thick 40-pass welds but only up to 16 passes, and the parameters are listed in Table 3.4. The only difference other than the thickness is the sequence placement after pass 5. After the completion of pass 5 with all passes centered on the weld centerline, the next layer includes two passes and so on until the top layer with three passes (see Figure 4.6 for E347-347H SS weld and Figure 4.7 for E16.8.2-347H SS weld). The extraction location of the Gleeble cross weld samples were primarily centered on passes 7-12, with a gauge section length of 32 mm which includes a 18-20 mm wide FZ. Furthermore, the gauge section includes the HAZ on both sides of the FZ. Figure 3.10 displays the dimensions of the cylindrical round tensile samples. The weld bead width for the E16.8.2 welds are generally smaller than the E347 weld passes when using the same weld parameters, which may be caused by the differences in the electrode coating flux. For E16.8.2-15, -15 designates limestone flux which is a faster freezing flux and leads to a more convex bead shape than the E347-16 welds. For E347-16, -16 designates titania-based flux which leads to a smoother arc transfer behavior and wider weld puddle during welding.
A higher magnification of pass 15 (most undiluted pass) using LOM shows a higher amount of δ-ferrite present in the E347-16 microstructure (Figure 4.8) than E16.8.2. The E347
and E16.8.2 microstructures are mostly representative of a FA solidification mode, meaning ferrite forms first in the melt followed by austenite. After completion of solidification and cooling, the γ-austenite content increases as δ-ferrite becomes unstable. The amount of retained δ-content depends on the composition (e.g., \( \text{Cr}_{eq}/\text{Ni}_{eq} \) ratio) and cooling rates. Slower cooling rates and lower \( \text{Cr}_{eq}/\text{Ni}_{eq} \) ratios generally reduce the amount of δ-ferrite retained in the weld metal microstructure. The weld morphology consists of a mix of lathy and skeletal ferrite in both cases, with the tendency for more lathy ferrite to exist in the E347 WM due to a higher \( \text{Cr}_{eq}/\text{Ni}_{eq} \) ratio (assuming similar cooling rates between the two welds).

![Figure 4.8](image-url) Weld morphology of center of weld metal in pass 15 (representative of the higher ferrite regions) in both (a) W.4-4 (E347) weld and (b) W.3-1 (E16.8.2). The δ-ferrite etches dark and γ-austenite is the light phase.

The WRC-1992 diagram illustrates the range of solidification modes (A, AF, FA, and F) and amount of ferrite predicted based on the \( \text{Cr}_{eq}/\text{Ni}_{eq} \) ratio only for austenitic and ferritic stainless steels (Figure 4.9 [64]). The corresponding weld metal composition (undiluted) and one-inch-thick plate (347H-batch #2) are labeled using points on the diagram (composition used from Table 3.1). As predicted, the solidification mode is FA, with low ferrite amounts in the E16.8.2 WM versus the E347 WM. An average ferrite content from the center of the gauge section based on Feritescope measurements in the weld metals for the Gleeble samples are 3.7±0.7 % volume for E16.8.2 WM and 10.8±1.5 % volume for E347 WM. A ferrite gradient across the transverse direction is very minimal for the E16.8.2-347H weld since the 347H base plate has similar predictions of ferrite content to E16.8.2, in contrast to the E347-347H welds. It
should be noted that localized differences in chemical composition may change the solidification mode slightly, e.g., AF for E16.8.2 WM since the composition lies close to the AF/FA demarcation line.

Higher ferrite content generally leads to a more rapid diffusion transformation for sigma phase (σ) during elevated temperature service between 500-800°C, which reduces the toughness and ductility of the weld metal as service time progresses. The benefit of E16.8.2 versus E308H and E347 WM is the tendency for reduced σ content, leading to improved thermal stability.

Figure 4.9 WRC 1992 diagram [64] with predicted points for undiluted weld metal and 347H base metal (batch #2)

Microhardness measurements in the welds are observed to have a depreciable drop in hardness in the coarse-grained heat affected zone (CGHAZ) of the as-welded condition (Figure 4.10). From a mechanical strength perspective, the CGHAZ is weaker than the PMZ, BM, and FZ. There is not a distinct difference in microhardness between the E16.8.2 and E347 WM; however, the CGHAZ microhardness for the E347 weld is lower than the CGHAZ of the E16.8.2 weld. A steeper hardness gradient from the CGHAZ to the FZ may increase chances of crack susceptibility in the CGHAZ. Since ductility and strength is lower in the CGHAZ, this region would generally be more susceptible to reheat cracking than the FGHAZ and BM. However, there are many factors that affect SRC susceptibility, such as dislocation density (proportional to applied strains) and weld morphology, in which straight, non-tortuous columnar solidification grain boundaries may be more susceptible than more tortuous equiaxed grain boundaries (such as
seen in the root pass in Figure 4.1e. The 347H CGHAZ is a primary region of interest in addition to the sub-subsurface E347 FZ.

Figure 4.10 Average hardness profile (five data points) from BM to FZ (pass 15) for both E347 (W.4-4) and E16.8.2 (W.3-1) welds

To represent the microstructure where the Gleeble cross welded samples were extracted from, microhardness measurements and micrographs were taken across passes 9 and 11 in E347 FZ before and after Gleeble testing as illustrated in Figure 4.11. Three columns of indents were taken across pass 9 and 11 as seen in Figure 4.11a, while the corresponding microhardness results are shown in Figure 4.11b. A Vickers’s hardness load of 300 g was used with a spacing of approximately 150µm apart from each other (3x the average indent size). It seems apparent that the center of the weld passes is weaker relative (<200 HV) to the transition region between pass 9 and 11, which reached peak values of 240V. Reheated regions may experience more local strain hardening due to cooling strains imposed by the weld thermal cycle. The reheated region of pass 9 and the lower δ-ferrite regions (less dark etchant response) are the highest hardness areas (outlined in red box). The microhardness in these regions is generally harder than the surface microhardness, which is generally below 200 HV in the top layer -pass 15 as seen in Figure 4.10. These results indicate that sub-surface regions (where the Gleeble gauge section is allocated) contain a much higher hardness than the top surface regions, likely due to more material constraint. The effect of reheating in multi-pass welds can cause changes in local mechanical properties. During reheating, pass 9 and 11 are area of interest for cracking, as the ductility may be less than the surrounding regions with lower hardness. SRC has been reported to be in regions of hardness exceeding 250 HV [4], likely because there is more resistance to grain
deformation that leads to localized cracking along solidification grain boundaries (i.e., ductility “exhaustion”).

Figure 4.11 Effect of E347 multi-pass microstructure on microhardness in passes 9 and 11 (gauge section of Gleeble samples): (a) Multi-pass microstructure of W4.4 passes 9 and 11 showing three diamond Vickers’s indent rows spaced 150μm from each other, and (b) microhardness contour map corresponding to measurements using a 300 g load. Microhardness reached peak hardness values of ~240 HV in upper regions of pass 9 and lower δ-ferrite regions of reheated pass 9 and pass 10 (outlined by red box).

4.2 Weld Finite Element Modeling Results

The FEM results are separated into three sets: 1) as-welded residual stress/strain, 2) validation of TD elastic strain using neutron diffraction on 2” thick welds, 3) stress profiles after PWHT, and 4) effect of thickness and joint geometry on residual stress.
4.2.1 As-Welded 2” FEM Stress Results

This section describes the results from the first set, i.e., as-welded condition, which provides inputs on the temperature profile and stress level for the Gleeble reheat crack tests for both the HAZ and WM.

For 2” thick 40-pass weld, von Mises stress as high as ~450 MPa (approximately the 0.2% offset yield strength of the E347 WM) was observed near the top of the weld surface in the unclamped condition (Figure 4.12). Stresses in all three principal directions are calculated from the weld process to calculate a 3-D stress state, which accounts for all three-strain direction, elastic modulus (E) and Poisson’s ratio (ν). The stress calculated for the 11 direction, as an example is shown in Eq. 4.1:

\[
\sigma_{11} = \frac{E}{1+\nu} \left( \varepsilon_{11} + \left( \frac{\nu}{1-2\nu} \left( \varepsilon_{11} + \varepsilon_{22} + \varepsilon_{33} \right) \right) \right)
\]  

(4.1)

The von Mises stress equation (Eq. 4.2) accounts for an effective combination of stress for all three principal directions (11,22,33), which is:

\[
\sigma_{eff} = \sqrt{\frac{(\sigma_{11} - \sigma_{22})^2 + (\sigma_{22} - \sigma_{33})^2 + (\sigma_{33} - \sigma_{11})^2}{2}}
\]  

(4.2)

Figure 4.12 summarizes and compares stress contours for all three principal directions and the von Mises contour plots within the rear left quarter of the weld model, where x, y and z axis represent transverse (TD), longitudinal (LD) and normal direction (ND), respectively. y axis is parallel to the welding direction.

The effect of unclamping is evidenced with reduction in stresses along both TD and LD. In the unclamped condition, along TD highest tensile stress is observed in the root (>400MPa), near-zero and slightly compressive stresses are present in the mid-thickness, followed by a slight increase in tensile stress near the top subsurface (~200 MPa). Along LD, the highest stress, approximately 450 MPa, is located underneath the top surface. Along ND, low stress < 40MPa is observed throughout the weld and slightly compressive stresses are present along the FZ boundary. In all, there is a peak von Mises stress of ~450 MPa near the top sub-surface regions of the weld in the unclamped condition and up to ~550 MPa in the clamped condition.
Figure 4.12 As-welded von Mises residual stress (MPa) and three principal stress directions in clamped (a-d) and unclamped (e-h) condition for the rear left quarter of the model. x (i.e., 11), y (i.e., 22) and z (i.e., 33) axis represents transverse, longitudinal and normal direction, respectively.

In addition, high plastic strain develops in the root of the 40-pass weld. Based on analysis of the unclamped and clamped conditions, there is no change in plastic strain since most of the plastic strain development occurs during the welding process. Thus, only the unclamped plastic strain condition is shown in Figure 4.13. The peak plastic strain in the weld metal (WM) is approximately ~0.22 in the root/hot passes, while the peak plastic strain in the HAZ is approximately ~0.09 in the as-welded condition. The plastic strain near the root increases due to reheating and subsequent yielding with an increasing number of weld passes, where the elements experiencing high enough stresses upon thermal contraction led to an increase in plastic strain. The strain in the ND (33) contributes to majority of the tensile strain seen with the effective plastic strain contours. A saturated tensile strain occurs in the root after approximately seven passes since it experiences fewer thermal strains when subsequent welds centered further away from the root with the broadening of the single-V weld joint. Therefore, weld regions closer to
the top side of the weld experience less plastic strain than the root, since they do not experience as much thermal contraction strains caused by reheating cycles.

Figure 4.13 Effective plastic strain and three principal strains in the as-welded condition in unclamped (a-d) condition for the rear left quarter of the model. x (i.e., 11), y (i.e., 22) and z (i.e., 33) axis represents TD, LD, and ND, respectively.

4.2.2 Transverse direction neutron diffraction measurements on 2” thick welds

Neutron diffraction mapping of residual strain in these welds have been performed for the purpose of providing validation for the models. All experimental details are explained in Appendix B. A contour plot accounting for the three lines of TD data in the AW condition is compared to the TD elastic strain field in the 40-pass FEM, which is shown in Figure 4.14. There is a reasonable agreement and validation of the FEM based on the TD elastic strain results, with the orders of strain magnitude having a similar value for near the top surface (z=11.5mm), mid-thickness (z=0) and root of the weld (z=-20mm). Based on this validation of TD strains in the FEM with experimental neutron diffraction results, it is assumed that the LD and ND elastic strains in the FEM would be valid to complete the stress calculation. In the unclamped condition, along TD as shown in Figure 4.12f, highest tensile stress is observed in the root (>400MPa), while near-zero and slightly compressive stresses are present in the mid-thickness, followed by a slight increase in tensile stress near the top subsurface (~200 MPa). These TD stress results correspond well to the elastic strain results determined from neutron diffraction and FEM.
Figure 4.14 TD elastic strain comparison for the TD ($\varepsilon_{11}$) in the (a) FEM and (b) neutron diffraction experiments. Orders of strain are comparable, with peak tensile strains near root and compressive strain in the mid-thickness.

4.2.3 PWHT effect on stress in weld FEM

PWHT process was simulated based on the as weld model and updated material properties from Gleeble test and simulation. During heating, constant temperature boundary condition was applied on both top and bottom surface of the plate model with a linear heating rate of 60°C/min (see Figure 4.15a-c), then keep at the target temperature of 950°C for 4 hours, and finally linearly cool down to room temperature within 2 hours (see Figure 4.15d-e). Heating reduces the stress significantly in the 2” thick weld, compared to the as-welded condition in Figure 4.12. For example, the highest stress regions experience a reduction from 485 to 95 MPa from heating only. Over time, the stress relaxes during the 4-hour hold time, followed by slight increases in stress when cooling to ambient temperatures (Figure 4.15d). Note that the PWHT conditions used here are not final recommendations. The two main mechanisms for reducing tensile residual stress during PWHT is by yielding inelastically (increasing temperature to reduce the yield strength, T-dependent) and creep (T & time -dependent). Creep strain increases at the expense of elastic strain (Figure 4.15e). Therefore, higher temperatures and times (where creep can take place) is desired to reduce the tensile residual stresses. The development of creep strain and stress during a simulated PWHT of 950°C for 4 hours reveal the effect of both temperature and time on stress relief.
Figure 4.15 (a) Temperature distribution upon reaching 950°C on surface, (b) von Mises stress contour upon reaching 950°C (MPa), (c) overall effective plastic strain contour upon reaching 950°C, (d) von Mises stress history of high stress area (circled location in contours) for the duration of 4 hour PWHT at 950°C and cooling and (d) strain history of the high stress area for the duration of the whole PWHT

4.2.4 Effect of joint geometry and plate thickness on residual stress

The effect of plate thickness and joint geometry on residual stress distribution was also evaluated through FEM. The plate thickness varies from 2”, 1”, to 0.5”) and the joint geometries evaluated include single-V, double-V, and J groves for the 1” thick weld. The weld sequence is the same for all the single-V and J joints, which goes from left to right for each weld layer. The double-V has an oscillating sequence, where one layer on one side is welded (left to right) first, followed by welding a layer with the same weld pass progressions on the opposite side until the joint is complete. As shown in Figure 4.16, in the single-V joints, the peak stress values are similar in all the three thicknesses (>450 MPa), although the 0.5” case exhibits a very small area of high stress near the middle thickness of the plate, while the 1” and 2” thick plates have the high stress appearing underneath the top surface. It suggests that thickness lower than 0.5” has much lower susceptibility to reheat cracking due to the very limited high stress area size.

Furthermore, by changing the joint geometry from single- to double-V for 1” thick plate, the
high stress area is significantly reduced and confined to the middle thickness. In addition, a J-bevel could help reduce residual stresses and weld time by reducing the weld volume nearly in half. Management of residual stress can therefore be achieved by optimizing the weld process and geometry, which in turn assist to control SRC during service and during PWHT. The joint geometry is dependent on feasibility and versatility of welding position, but a J-groove design may be desired if feasible for joints greater than 0.5 inches.

Figure 4.16 2D stress contours of welds with different conditions: (a) 2 inch thick plate with a single-V groove; (b) 0.5 inch plate with a single-V groove; and 1 inch plate with a (c) single-V groove, (d) double-V groove, and (e) J groove
CHAPTER 5: STRESS RELAXATION/REHEAT CRACK GLEEBLE RESULTS AND DISCUSSION

This chapter summarizes the results from the Gleeble 3500© stress relaxation/reheat crack tests, including the four sets of materials and microstructures. First, the 347H HAZ SRC tests are shown first, followed by the Gleeble FEM results. Then, the E347-347H SS cross welded transverse sample results will be discussed, followed by the modified 316L SS results and the E16.8.2-347H SS welded transverse sample results.

5.1 347H HAZ SRC results

5.1.1 HAZ microstructures prior to SRC testing

Evolution of microstructure after the CGHAZ thermal cycle is examined first prior to the discussion on the microstructure and results of the SRC test. Constitutional liquation along equiaxed grain boundaries occurs from segregation of Nb or from dissolution of pre-existing coarse Nb (C, N) at grain boundaries (parallel to the rolling direction of the solution annealed hot rolled plate) (see Figure 5.1a) at the high temperature of 1335°C during the CGHAZ thermal cycle and after 9 hours at 600°C (see Figure 5.1b). A closeup (higher magnification) of the liquated microstructures are shown in Figure 5.2, with just the 1335°C CGHAZ thermal cycle applied (Figure 5.2a) and a closeup of Figure 5.1b (seen in Figure 5.2b). A combination of eutectics consisting of Nb rich phases, likely Nb(C,N), and austenite forms in the PMZ along with continuous grain boundary Nb (C,N) [19]. Based on a value of 0.1 for Cullen and Freeman parameter in this 347H grade, the liquation temperature is predicted to be around 1335-1340°C (see Figure 2.16). δ-ferrite stringers in the base metal may be sources for a lower liquation temperature, since δ-ferrite is rich in Nb. There seems to be a presence of finer grains in regions of liquation, as seen in Figure 5.2b. Solute depletion of Nb, C and N from the surrounding grains may occur, and also melting back of those grains may lead to the observation of finer grains in the 1335°C CGHAZ thermal cycle condition. There is perhaps a slightly reduced susceptibility, as will be discussed later, with a PMZ 1335°C peak temperature due to the presence of finer grains (assuming there are no pre-existing liquation cracks prior to steps 3-6 of the reheat crack test).

However, the 1275 °C thermal cycle did not reveal any liquation based on observations using light optical microscopy (LOM). During this thermal cycle, slight grain growth is expected
at temperature below the liqation temperature (based on Figure 2.12 [18]). Evaluating three peak temperatures would assist to determine what region adjacent to the weld fusion zone is more susceptible to SRC, such as the PMZ (1335°C), CGHAZ (1275°C), and FGHAZ (1150°C), and provide guidance for future welding practice.

Figure 5.1 (a) 347H SS base metal of location with Nb-rich oriented precipitates (unetched-SEI) (b) Lication evidence after 1335°C peak HAZ thermal cycle and 600 °C for 9 hours (electro etched-SEI)

Figure 5.2 Lication evidence during the HAZ thermal cycle (a) After a 1335°C peak temperature CGHAZ simulation only (un-etched polarized LOM), and (b) Specimen 3 (see Table 5.1) after completion of SRC test for 9 h at 600°C (center of specimen (electro etched)-FESEM SEI. The microstructures are representative of an eutectic Nb (C,N)/γ microstructure due to grain boundary liqation, with light phase being Nb (C,N) and surrounding darker grains being γ-austenite.

Since the electro etchant procedure may cause precipitates to be flushed out and can contribute to a misunderstanding of the EDS results, an unetched specimen with a 1 µm polish was scanned in a map mode (22 counts) to understand more about the cellular precipitation seen on GB. As shown in Figure 5.3, the brighter phase shows depressions in Fe, Cr, and Ni while Nb
increases in intensity in the brighter cellular phase. Results indicate Nb segregation relative to Cr, Fe, and Ni on the brighter phase. A line scan across the layers of the bright phase and matrix showed the same behavior. This indicates that the brighter phase is most likely Nb (C, N). More in-depth metallurgical analysis prior to SRC testing would provide insights for both the reference base metal and the Gleeble specimens to determine what phase constituents pre-exist before applying a strain, particularly during the CGHAZ thermal cycle.

Figure 5.3 EDS map of Nb(C,N)/γ-austenite liquation microstructure due to 1335°C HAZ, (a) SEI image, (b) Fe map, (c) Nb map, (d) Cr map, (e) Ni map (need to include in results as the first results to show)

5.1.2 Reheat crack susceptibility of 347H HAZ

The stress evolution profiles at 950 and 1050°C with various pre-strains are displayed in Figure 5.4 and Figure 5.5, respectively. For the samples that did not fail, the “force level heat mute” command was triggered (meaning load was very low, almost 0 kN). This safety command is used to prevent arcing and melting of samples if the specimen fractures while at elevated temperatures. While this command is triggered, the heat power will cease, and the temperature will drop to room temperature. Therefore, the load (stress) will increase since the stroke is constrained during contraction, as illustrated in the case of 0.05 pre-strain at 950°C in Figure 5.4. Another good indication of susceptibility to cracking is failing while cooling under constraint even if failure does not occur while at temperature. If failure occurs soon after temperature drops with small load increases, then the strain is considered an unsafe condition for the peak temperature applied during PWHT. For instance, at 950 °C with pre-strain of 0.08, the sample failed shortly after cooling from 950°C after the “force level heat mute” command was triggered.
However, the 0.05 pre-strain sample did not fail during cooling. Similarly, Figure 5.5 shows that all the 1050°C tests with pre-strains of 0.05 and lower did not fail during cooling.

Figure 5.4 Comparison of different pre-strains for 950°C Gleeble tests, showing failure at 0.08 (S35) and 0.1 strains (S12), with no failure during cooling from 0.05 strain test (S34).

Figure 5.5 Comparison of different pre-strains for 1050°C Gleeble tests, showing failure at 0.05 (S28) and 0.1 strains (S13), with no failure during cooling from 0.04 (S33), 0.025 (S32), and 0.01 (S31) strain tests.
The stress relaxation curves of the 347H HAZ tests reveal the effect of temperature and pre-strain/stress on starting stress at target temperature and stress relaxation during holding at temperature. Figure 5.6 summarizes all the stress relaxation curves of the 347H HAZ tests, which reveals the effect of temperature and pre-strain/stress on starting stress at target temperature and stress relaxation during holding at temperature. A higher initial pre-strain/stress typically leads to a higher starting stress at the same step 6 temperature. Also, increasing the reheat temperature typically leads to a lower stress when reaching isothermal temperatures, i.e., a higher extent of stress relaxation (step 6). Note that thermal expansion was offset by extra pulling during heating stage to avoid stress reduction from thermal expansion. With a relatively fast heating rate and extra pulling for thermal expansion offset, it is assumed that the starting stress at temperature corresponds to the yield point under a specific pre-strain condition, and therefore is used to develop the stress strain curves labeled “Gleeble” in Figure 3.8. For example, the 750°C tests are labeled with two pre-strain conditions, 0.174 and 0.1 with starting stresses at temperature being 260 and 195 MPa, respectively.

Figure 5.6 Stress relaxation curves of the 347H HAZ samples to show the effect of temperature and pre-strain on stress evolution during step 6 for 550-1050°C temperature conditions
SRC results, i.e., time to cracking, from Gleeble tests for 347H SS HAZ are summarized in Table 5.1 and Figure 5.7, including all the tests such as one- and two-step PWHT tests. In Figure 5.7, open symbols indicate no failure occurred during test, asterisk symbols represent failure during cooling, and solid symbols indicate failure isothermally. The main objective for this set of tests is to identify the critical strain/stress in which failure may occur for each designated temperature. The main conclusions determined from the tests are:

- Higher pre-strains/stress (prior to and during PWHT) generally led to more rapid failure. A higher residual stress after the completion of welding leads to more rapid failure at higher temperatures. Careful control of stress during PWHT, e.g., through a slow heating rate or potentially the use of a multi-step PWHT process, is needed to prevent reheat cracking in the 347H SS HAZ.

- Different peak HAZ temperatures may slightly impact the resulting microstructure. However, based on the Gleeble HAZ SRC testing results, the 1275°C tests failed sooner than the 1335°C samples using the same stress conditions at 900 and 850°C, which indicates that the CGHAZ (1275°C) is slightly more susceptible than the PMZ (1335°C) and fine-grain HAZ (1150°C) in terms of faster time to failure.

- Some two step tests were experimented, including samples 17-20 and 24 with a peak temperature of 950 and 1050°C, respectively. A first step of 550 and 600°C had been used. Based on the results, there is not a statistical difference in the results between the first step and two step tests. The additional stroke was applied the same. However, the effect of using slower heating rates may change these results. Mainly the fast-heating rate of 58°C/minute was used for both the first and second step heating.
Table 5.1 Summary of Gleeble 347H SS HAZ SRC test variables and results (samples not listed were used for thermomechanical tensile tests)

<table>
<thead>
<tr>
<th>Sample#</th>
<th>Peak HAZ temp. (°C)</th>
<th>Pre-strain/stress (MPa)</th>
<th>SRC test temperature (°C)</th>
<th>Time to failure</th>
<th>Failed during Heating, Isothermal or Cooling</th>
<th>Type of test</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Dummy samples to calibrate the testing method</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>1335</td>
<td>275 MPa</td>
<td>600</td>
<td>No failure after 9 h</td>
<td>N/A</td>
<td>1 step PWHT, Cu chucks, no c-gauge</td>
</tr>
<tr>
<td>3</td>
<td>1335</td>
<td>0.174 / 600</td>
<td>600</td>
<td>No failure after 8 h</td>
<td>N/A</td>
<td>Isothermal</td>
</tr>
<tr>
<td>4</td>
<td>1335</td>
<td>0.174 / 600</td>
<td>800</td>
<td>2.9 h</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>1335</td>
<td>0.174 / 600</td>
<td>750</td>
<td>22.2 h (8.2 h into 2nd try)</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>1335</td>
<td>0.174 / 600</td>
<td>850</td>
<td>20.3 min.</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>1335</td>
<td>0.174 / 600</td>
<td>900</td>
<td>5.8 min.</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>1335</td>
<td>0.1 / 450</td>
<td>900</td>
<td>31 min.</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>1335</td>
<td>0.1 / 450</td>
<td>800</td>
<td>No failure after 48 h</td>
<td>N/A</td>
<td>One-step PWHT, SS chucks, c-gauge</td>
</tr>
<tr>
<td>10</td>
<td>1335</td>
<td>0.1 / 450</td>
<td>850</td>
<td>1.1 h</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>1335</td>
<td>0.1 / 450</td>
<td>950</td>
<td>7.1 min.</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>1335</td>
<td>0.1 / 450</td>
<td>1050</td>
<td>3 sec</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>13</td>
<td>1275</td>
<td>0.1 / 450</td>
<td>900</td>
<td>13.5 min.</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>14</td>
<td>1275</td>
<td>0.1 / 450</td>
<td>850</td>
<td>48.5 min.</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>15</td>
<td>1275</td>
<td>0.1 / 450</td>
<td>800</td>
<td>No failure after 3.8 h</td>
<td>N/A</td>
<td>Two-step PWHT, SS chucks, c-gauge</td>
</tr>
<tr>
<td>16</td>
<td>1150</td>
<td>0.1 / 450</td>
<td>950</td>
<td>2.5 h</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>17</td>
<td>1335</td>
<td>0.1 / 450</td>
<td>550 (2 hr), 950</td>
<td>38.4 min.</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>18</td>
<td>1335</td>
<td>0.1 / 450</td>
<td>600 (2 hr), 950</td>
<td>13.9 min.</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>19</td>
<td>1335</td>
<td>0.1 / 450</td>
<td>550 (2 hr), 950</td>
<td>18.7 min.</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>1335</td>
<td>0.1 / 450</td>
<td>550 (4 hr), 950</td>
<td>18.3 min.</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>21</td>
<td>1335</td>
<td>0.1 / 450</td>
<td>900</td>
<td>23.3 min.</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>22</td>
<td>1335</td>
<td>0.1 / 450</td>
<td>900</td>
<td>24.1 min.</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>23</td>
<td>1335</td>
<td>0.1 / 450</td>
<td>595</td>
<td>No failure after 10.9 h</td>
<td>N/A</td>
<td>Two-step PWHT, SS chucks, c-gauge</td>
</tr>
<tr>
<td>24</td>
<td>1335</td>
<td>0.1 / 450</td>
<td>595 (3 hr) /1050</td>
<td>Failed upon heating to 1050 °C at 1020 °C</td>
<td>Heating</td>
<td></td>
</tr>
<tr>
<td>25</td>
<td>1335</td>
<td>0.1 / 450</td>
<td>1050</td>
<td>2.4 min.</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>26</td>
<td>1335</td>
<td>0.05 / 350</td>
<td>1050</td>
<td>No failure after 3.8 h</td>
<td>N/A</td>
<td>One-step PWHT, SS chucks, c-gauge</td>
</tr>
<tr>
<td>27</td>
<td>1335</td>
<td>0.01 / 250</td>
<td>1050</td>
<td>No failure after 3.8 h</td>
<td>N/A</td>
<td>One-step PWHT, SS chucks, c-gauge</td>
</tr>
<tr>
<td>28</td>
<td>1335</td>
<td>0.025 / 300</td>
<td>1050</td>
<td>No failure after 3.6 h</td>
<td>N/A</td>
<td>One-step PWHT, SS chucks, c-gauge</td>
</tr>
<tr>
<td>29</td>
<td>1335</td>
<td>0.04 / 335</td>
<td>1050</td>
<td>No failure after 1.94 h</td>
<td>N/A</td>
<td>One-step PWHT, SS chucks, c-gauge</td>
</tr>
<tr>
<td>30</td>
<td>1335</td>
<td>0.05 / 350</td>
<td>950</td>
<td>No failure after 10.9 h</td>
<td>N/A</td>
<td>One-step PWHT, SS chucks, c-gauge</td>
</tr>
<tr>
<td>31</td>
<td>1335</td>
<td>0.08 / 415</td>
<td>950</td>
<td>6.4 h</td>
<td>Cooling</td>
<td></td>
</tr>
<tr>
<td>32</td>
<td>1335</td>
<td>0.08 / 415</td>
<td>900</td>
<td>4.9 h</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>33</td>
<td>1335</td>
<td>0.06 / 350</td>
<td>900</td>
<td>1.03 h</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>34</td>
<td>1335</td>
<td>0.04 / 335</td>
<td>900</td>
<td>No failure after 24 h</td>
<td>N/A</td>
<td>Cooling</td>
</tr>
<tr>
<td>35</td>
<td>1335</td>
<td>0.08 / 415</td>
<td>850</td>
<td>24 h</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>36</td>
<td>1335</td>
<td>0.08 / 415</td>
<td>800</td>
<td>24 h</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>37</td>
<td>1335</td>
<td>0.13 / 500</td>
<td>1000</td>
<td>1.2 h</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>38</td>
<td>1335</td>
<td>0.05 / 350</td>
<td>1000</td>
<td>4.7 h</td>
<td>Cooling</td>
<td></td>
</tr>
<tr>
<td>39</td>
<td>1335</td>
<td>0.04 / 335</td>
<td>1000</td>
<td>1.2 h</td>
<td>Isothermal</td>
<td></td>
</tr>
<tr>
<td>40</td>
<td>1335</td>
<td>0.06 / 350</td>
<td>950</td>
<td>1.2 h</td>
<td>Isothermal</td>
<td></td>
</tr>
</tbody>
</table>
Another method for describing the stress relaxation behavior in the 347H HAZ is the susceptibility map as a function of starting stress at room temperature in step 3 and testing temperature, as shown in Figure 5.8. With increasing temperature, there is a lower tolerance of stress needed to cause failure. An additional susceptibility map as a function of starting stress at testing temperature in step 6 and the testing temperature, Figure 5.9, reveals a clearer trend of crack susceptibility. For example, even though sample 45 (0.06 pre-strain/365 MPa at 950°C) failed sooner than sample 35 (0.08 pre-strain/415 MPa at 950°C) with a lower pre-strain/stress, the starting stress at 950°C was higher for sample 45 (80 MPa versus 77 MPa). The 0.08 pre-strain test failed upon cooling (grey data point) after 6.4 hours at 950°C, while the 0.06 pre-strain sample failed at temperature after 1.2 hours (black data point). These plots of critical stresses may be used to determine whether the stress during heating is safe to prevent cracking during PWHT. It should be noted that tests indicating no failure during the reheat crack test still experienced formation of a small amount of microcracking along grain boundaries, as demonstrated by 800, 950, and 1050°C stress tests in Figure 5.9. It suggests that a complete
stress relief is desired to avoid further propagation of the microcracks; however, stress relief is minimized by grain interior strengthening.

Figure 5.8 Susceptibility map as a function of initial stress at room temperature in step 3 and testing temperature for 347H HAZ samples

Figure 5.9 Susceptibility map as a function of starting stress at temperature in step 6 and testing temperature for 347H HAZ samples with corresponding critical samples at 800, 950 and 1050°C showing presence of microcracks and void formation along grain boundaries
5.1.3 Characterization of 347H HAZ reheat cracks

Some of the samples that did not fail during the test showed a progression of microcracks that develop to form a primary fracture surface as shown in Figure 5.10 at 800°C and 0.1 strain. A significant amount of grain boundary voids (as small as ~ 1µm) was present at the center of the gauge length, as shown in Figure 5.10a. The coherency of the Nb (C, N) and austenite matrix exists at very small Nb (C, N) sizes (<10 nm in diameter). The Nb (C,N) precipitates lose coherency when coarsened above 10 nm since there is approximately an Nb (C,N)/γ interface mismatch of 20% [65]. Thus, incoherent precipitates at grain boundaries may lead to creep voids, when under sufficient stress during the isothermal period of the SRC test.

![Figure 5.10 Presence of micro voids on GB (a) in the center of the gauge section of specimen 10 (800°C/0.1 strain). FESM SEI image (20 kV and 8.5mm WD) and (b) voids along with polarized image of microcracks on GB with TRIP α’ martensite surrounding the microcracks and presence of liquation (Nb (C, N)/ γ eutectic microstructures) within vicinity of the microcracks. Tensile axis is shown to be perpendicular to the crack growth direction.](image)

In addition, α’ martensite seems to be oriented perpendicular to the microcracks seen in Figure 5.10.b with a strain of 0.1. The α’ martensitic laths (habit planes) are oriented perpendicular to the microcracks. Martensite formed potentially by the transformation induced plasticity (TRIP) mechanism with a mechanical load applied to metastable γ-austenite at room temperature during step 3 and perhaps there was not 100% reversion of the martensite upon reheating to 800°C or TRIP occurred during the polishing process where stress relief may occur.

Additional strengthening of the matrix can be achieved by cold work, which allows for transformation induced plasticity (TRIP) α’ martensite to form. Meanwhile, cold work will also facilitate formation of fine Nb (C, N) precipitations to further strengthen the material. An array of 40 microhardness points with a 1-kg load were taken on the radial cross section in the center
of the gauge length. Figure 5.11 summarizes and compares the average and standard deviation error bars for five conditions: 347H SS base metal, after 1275°C CGHAZ thermal cycle, after 1335°C thermal cycle, after specimen 3 test (0.002 strain and 600°C SRC temperature), and after specimen 4 test (0.174 strain and 600°C SRC temperature). The base metal and 1275°C CGHAZ thermal cycle seem to have a similar hardness (140-150 HV), but the hardness increases slightly (avg. of 167 HV) after the 1335°C thermal cycle potentially due to a higher fraction of reprecipitated fine Nb (C, N). The hardness measurements were taken mostly away from the liquation areas, so that the hardness indicate a change in hardness in the general bulk microstructure. Specimen 3 shows a similar hardness to the other 1335°C sample, because specimen 3 had a 1335°C thermal cycle prior to pulling to the 0.002 strain and heating to 600°C. However, specimen 4 shows a sharp increase in hardness from an average of 165 (specimen 3) to 255 HV (specimen 4), which is attributed to the formation of α’ as evidenced by the optical micrograph in Figure 5.11. As mentioned in previous reports related to SRC related characteristics [8], the typical hardness near SRC fracture surfaces are above 250 HV. The reported cases of 250 HV and above do not appear to reveal the specific strengthening mechanism for the reported cracking issues, but α’ martensite and strain induced aging of Nb (C, N) may both contribute to strengthening of regions surrounding PFZs and facilitate SRC to occur. TRIP α’ martensite was reported to be present in the CGHAZ of a two-pass pipe weld that had macro cracks on the inside of the pipe after 150 days of service at 595°C [36].

![Figure 5.11 Comparison of microhardness in a variety of microstructures of 347H SS](image-url)
All the specimens failed in a brittle manner based on fractography examination of samples 5-9 and 11-20. For the surfaces that did not oxidize during the process of cracking, a shiny, brittle crack surface was present, while the oxidized surfaces show a darker, radial crack surface, as seen in Figure 5.12a. No shear lips were present on all of the 347H HAZ samples observed. The crack direction was noticeable in a couple specimens. For specimen 5, in Figure 5.12a, the darker region was where the crack started because there was enough time for cracks to be exposed to the air at 800 °C and form a dark oxide layer before the whole crack propagated across the whole cross section.

A closeup examination of the fracture surfaces of high strain samples (0.174) within 750-850°C using an electron scanning microscope (ESEM) illustrates intergranular fracture. Note that secondary electron (SE) mode was used for gathering surface topography. The ESEM fracture surfaces show evidence of intergranular cracking due to accumulation of GB microcracks across the whole surface and a brittle cleavage appearance along grain boundaries as seen in Figure 5.12b-d. In some cases, particularly for the 750°C case (see Figure 5.12d), the grain size seems to be finer and the cracks seem to be oriented along the Nb-rich particles and or liquation bands as seen in Figure 5.1. It should be noted that while no shear lips or primary ductile fracture were observed, there are some instances where the sample does not fail completely across the cross section before the load drops below the “force level heat mute” command. However, the analyzed regions were in the center of the fracture samples, where no remnants of secondary, final ductile fracture are analyzed. When the load drops quickly, the fracture accumulates quickly in a “catastrophic” manner, which is a characteristic of brittle failure. Therefore, the fracture surfaces mainly are a mixed mode of micro void coalescence on grain boundaries in combination with a brittle grain boundary decohesion. In other words, the fracture surfaces are not purely one mode of fracture but would be described as mixed mode failure.
Figure 5.12 Fracture surfaces in center of cross section of 0.174 strain conditions for (a-b) 800°C, (c) 750°C (fracture parallel to Nb-rich oriented particles), and (d) 850°C samples

Two sub-surface secondary cracks for specimen 5 are shown in Figure 5.13, which implicate the presence of coarse Nb (C, N) based EDS point scans that showed enrichment of Nb and depletion of Fe, Ni, and Cr. In both locations, the incoherency of the carbonitrides and GB seem to allow for micro voids to form during the SRC test. Based on the up-close images in Figure 5.13.d, a PFZ may be present along the darker region (<500nm) between the grain and intergranular carbonitrides but a transmission electron microscope would be needed to reach nm resolution to confirm this mechanism in these samples. Dislocation pileups, which may develop near grain boundaries in step 3, influence work hardening within the microstructure. Upon reheating to elevated temperatures there is an increased nucleation of Nb (C, N) intragranularly, assuming there is negligible recovery and recrystallization that would results in dislocation annihilation. Ultimately there is a competition between recovery, recrystallization and Nb (C, N) precipitation that influence the mobility of dislocations and therefore the reduction of stress. The resistance of grain deformation due to the presence of nucleated Nb (C, N) on dislocation pileups near grain boundaries (and in the center of the grains) that may lead to strain localization along the PFZs on the grain boundaries. Decohesion of intergranular precipitates on grain boundaries contributes to crack propagation.
Secondary features lying near the primary fracture surfaces indicate certain metallurgical interactions that influence the specific fracture mechanisms of reheat cracking, particularly secondary cracks. Figure 5.14 illustrates a list of phenomena that is critical for reheat cracking to develop for two samples of 0.174 and 0.1 strain with a 1335°C peak temperature thermal cycle and reheat temperature of 900°C. The effect of strain on grain morphology and TRIP martensite formation and nucleation and growth of intragranular Nb (C,N) on dislocation forests [12] and dislocation pileups, peak HAZ temperature on grain growth, and high volume fraction of grain boundary Nb (C,N) all influence the susceptibility to intergranular fracture.

Figure 5.13 Representation of unetched secondary cracks of S5 (800°C-0.174 strain) showing coarse intergranular GB Nb (C, N) along crack path ahead of primary fracture surfaces for sub-crack 1 (a-b) and sub-crack 2 (c-d).

Further characterization of failed Gleeble samples continue to support the hypothesis that failure occurs along grain boundaries, in which ductility is “exhausted” (Figure 5.14). TRIP α’ martensite seems to be present within coarse grains in the center of Figure 5.14b at a high strain of 0.174 and 900°C. It’s possible that polishing procedures may have caused TRIP to occur; however, it may explain extra grain hardening if it does in fact exist still at elevated temperatures. The light areas depict coarse, continuous Nb (C, N) along grain boundaries. An EDS line scan on sample 8 (900°C/0.174 pre-strain) depicts a high intensity of Nb along the
continuous particle with drops in Nb surrounding the coarse Nb (C, N) (Figure 5.15), indicating the potential presence of a soft narrow region, i.e., PFZ. A PFZ microstructure has been observed at the crack tips of 347H SS welds from a high resolution STEM-EDS mapping at EPRI (see Figure 2.27 [35]). A closeup of the fracture surfaces on the grain facets (Figure 5.12) shows micro ductile voids, which is also seen in Kant’s work in Figure 2.29[6]), where regions of decohesion occur around the incoherent GB carbonitrides along the grain boundaries. These observations in this work and literature support the PFZ hypothesis as a metallurgical mechanism for SRC failure. Figure 5.14a reveals wedge or w-type cracks in the 0.174 initial strain samples where the cracks initiate at the grain boundaries aligned for maximum shear (i.e., 45° relative to the tensile axis). Wedge cracks are indicative of grain boundary sliding, which is a common creep mechanism that can lead to intergranular failure at elevated temperature conditions with applied stress [66].

Figure 5.14 900°C with 0.174 strain. (a) edge of sample, (b) center of sample. Failed at 5.8 min after reaching 900 °C. FESEM, electroetched with 50% nitric acid solution: 900°C with 0.1 strain. (c) edge of sample, (d) center of sample failed at 31 min after reaching 900 °C.SEM-SEI, electroetched with 50% nitric acid solution.
Figure 5.15 EDS Line scan across Nb (C, N) of on grain boundary close to primary fracture surface of Sample 8 (900°C/0.174 pre-strain)

5.2 Gleeble FEM Results and Comparison with Experimental Results

The sequence of steps in the model, as demonstrated in Figure 5.16 (0.01 pre-strain and 1050°C), was set up to duplicate exactly the Gleeble testing procedures as seen in Figure 3.13. For the Gleeble FEM simulation, steps 1-2 are neglected since the model does not account for microstructural changes associated with a HAZ thermal cycle. The change in the gauge length and additional stroke during heating is labeled in Figure 5.16. The gauge length increases with the applied strain/stress during step 3 (4 mm in this case). An additional stroke of 1 mm is shown during steps 3 and 4 to account for thermal expansion effects on stress drop. The gauge length increases with the applied strain/stress, which are 2 mm for 0.05 strain, 3.2 mm for 0.08 strain and 4 mm for 0.1 strain for all simulations. The second stroke applied during heating was approximately 0.85 mm up to 900°C, 0.9 mm up to 950°C and 1 mm up to 1050°C. The
intention of Figure 5.16 is to demonstrate 1) the progression of temperature and stress (particularly stress relaxation) during the duration of the Gleeble SRC test and 2) the additional stroke applied in the experiment during step 5 to counteract the effect of thermal expansion.

Figure 5.16 Stress and temperature contours for an example of a 0.1 pre-strained sample heated up to 1050°C

As shown in Figure 5.17 with the 0.08 pre-strain and 900°C condition, the stress evolution predicted by the Gleeble model and experimental measurement show a good agreement both during heating and for a long time after reaching temperature. Half of the gauge section contours are shown for initial stress at room temperature and the temperature and stress contours once reaching step 6, with the plots representing the mid-gauge cross section behavior. As observed, a temperature gradient exists in the sample across the gauge section, which was observed to be comparable to experimental temperature gradients. The stress gradient across the sample gauge section seems to be consistent once reaching temperature; however, the creep damage occurs mostly where temperature is highest. The failure location for all the samples usually is located within a few mm of the middle cross section and very close to where the c-
The creep parameters extracted from the Gleeble experiments (Table 3.7) determine the Gleeble FEM stress relaxation behavior via creep law during the final step at temperature, while a combination of creep and inelastic plastic strain leads to a reduction in elastic strain during step 5 heating. The other eight simulations show similar stress relaxation validation with the experimental data, which are important for verifying the material data base of 347H SS used for future and existing weld models.

![Stress and temperature evolution in center of gauge section during Gleeble test with a pre-strain of 0.08 (~415 MPa) and peak temperature of 900 °C, including FEM stress contours and stress validation with experiment](image)

Also, extra information was extracted from the validated Gleeble model, such as the values of each component of the total strain. During the initial test conditions, there was concern that the additional stroke applied during heating was causing additional mechanical strain that did not properly compensate to the effect of thermal expansion on stress reduction. The importance of each strain component for each strain component of a uniaxial test, assuming isotropic properties, is important considering the complexity of the reheat cracking testing. The total strain consists of the following components:

- Elastic strain ($\varepsilon_{el}$)-due to an elastic load (reversible),
Plastic strain ($\varepsilon_{pl}$)-due to an inelastic load above yielding (irreversible, except for metallurgical recovery and recrystallization),

- Thermal strain ($\varepsilon_{th}$)- due to thermal expansion and contraction (reversible),
- Creep strain ($\varepsilon_{cr}$)-inelastic strain due to creep mechanism at high temperature (irreversible),
- Metallurgical strain ($\varepsilon_{ph}$)-strain induced by phase transformations, which is neglected in this model since it does not account for phase transformations and volume non-conservation, such as athermal martensite transformation upon cooling. Austenitic stainless steels typically have a martensite start temperature well below ambient temperatures since austenite is metastable at room temperatures.

Therefore, the total strain (Eq. 5.1) can be written in the form:

$$ \varepsilon_{tot} = \varepsilon_{el} + \varepsilon_{pl} + \varepsilon_{th} + \varepsilon_{cr} \quad (5.1) $$

The FEM can assist to determine the additional plastic strains applied during steps 4-5, in addition to creep and thermal strains, so that the critical strain to failure can be more accurate for each Gleeble test. Step 3 of the Gleeble experiment, i.e., pulling at room temperature, contain only plastic and elastic strain, with plastic strain being dominant. During step 4-5, the stroke is pulled at a linear rate with increasing temperature to account for thermal expansion and therefore contains all four component strains. Step 6 includes only elastic and creep strain changes since no additional plastic strain or thermal strain is applied. While there would be reversion of martensite to austenite at elevated temperatures, the model assumes no transformation strains for simplicity.

Elastic, plastic, thermal, creep, and total strain for sample 36 are shown in Figure 5.18 and Figure 5.19. While plastic strain is approximately 0.081 before steps 4-5, an additional plastic strain of 0.024 applied during pulling for offsetting thermal expansion in step 5 brings the plastic strain to ~0.105 prior to step 6. Total thermal strain during heating is approximately 0.018, while elastic and creep strains are very low (0.002 and below). The total strain is approximately 0.1236 before reaching step 6, which is driven primarily by plastic (0.102) and thermal strain (0.018). The total plastic strain (including steps 3-5) will be defined as the “true plastic strain” and will be used for determining the critical strain to failure for 347H HAZ tests. While thermal strain is significant, it is reversible and would not be the rate limiting factor for...
failure. The elastic and creep strains are very low (0.004 and below), but they heavily influence the stress relaxation behavior while constrained. Creep strain increases at the expense of a drop in elastic strain, which is shown to have a strong validation between the experiment and model. Reduction of elastic strain to zero would mean complete stress relief, which depends on the temperature and time at temperature. While creep strain would intuitively increase with increasing temperatures, a zero elastic strain would not allow for more creep strain to develop. Therefore, soak times needed to provide complete stress relief (during PWHT would depend on temperature and corresponding creep mechanisms to lead to stress relief). At lower temperatures, Coble creep (grain boundary diffusion) dominates the creep mechanisms. At higher testing temperatures, creep rates are much faster and are usually associated with dislocation creep (movement of dislocations past barriers i.e., Nb (C, N)) and or Nabarro-Herring creep (diffusional flow of vacancies and atoms) that is evident in elongation of the deformed grains.

Figure 5.18 Original mesh and example of stress and temperature evolution during Gleeble test with a pre-strain of 0.08 (~415 MPa) and peak temperature of 900 °C (sample 36) with stress contours at each step
Figure 5.19 Plastic, thermal, and creep strain development from steps 3-6 for 900°C and 0.08 pre-strain (sample 36): (a) plastic strain contours at each step and (b) calculation of strain components of sample in center of gauge section as a function of time.

Another example is given for stress and strain evolution under 950°C with 0.08 pre-strain (sample 35) in Figure 5.20. Again, good agreement was achieved in the stress evolution between Gleeble testing results and FE calculations. A total of seven additional simulations (total of nine) with changing pre-strains and temperatures were completed for stress comparison and strain calculations (0.1, 0.08, and 0.05 pre-strain with 1050, 950, and 900°C as the variables). All the simulations showed good stress agreement between the experiment and model. The total strain and its component strains are tabulated at the end of step 3 (after pulling to designated strain at room temperature) and after two hours of annealing at each temperature in Table 5.2 and Table 5.3, respectively. Total strain (LE) is listed as 100%, in which plastic strain makes up most of the total strain (~80%). Thermal strain (THE) is the highest for 1050°C. Elastic strain is zero at 1050°C after 2 hours, meaning complete stress relief. Creep strain increases at the expense of elastic strain. Reduction of elastic strain to zero means complete stress relief, which depends on the temperature and time at temperature. While creep strain would intuitively increase with increasing temperatures, a zero elastic strain would not allow for more creep strain to develop.
Therefore, a PWHT at 1050°C more than 2 hours may be unnecessary. For lower temperatures, longer times are needed for complete stress relief (i.e., zero elastic strain).

Figure 5.20 (a) Comparison of stress evolution between FE prediction and Gleeble experimental results and (b) calculation of strain components in center of gauge section as a function of time for 950°C 0.08 pre-strain condition (sample 35)
Table 5.2 Strain components for three pre-strains of 0.1, 0.08, and 0.05 and testing temperatures at 1050, 950, and 900°C after step 3. LE=total strain; PE=plastic strain; EE=elastic strain; THE=thermal strain; CE=creep strain

<table>
<thead>
<tr>
<th>Aim Pre-strain</th>
<th>0.1</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature (°C)</td>
<td>1050</td>
<td>950</td>
<td>900</td>
</tr>
<tr>
<td>LE</td>
<td>0.1018</td>
<td>100.0%</td>
<td>0.1018</td>
</tr>
<tr>
<td>PE</td>
<td>0.0996</td>
<td>97.8%</td>
<td>0.0996</td>
</tr>
<tr>
<td>EE</td>
<td>0.0022</td>
<td>2.2%</td>
<td>0.0022</td>
</tr>
<tr>
<td>THE</td>
<td>0.0000</td>
<td>0.0%</td>
<td>0.0000</td>
</tr>
<tr>
<td>CE</td>
<td>0.0000</td>
<td>0.0%</td>
<td>0.0000</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Aim Pre-strain</th>
<th>0.08</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature (°C)</td>
<td>1050</td>
<td>950</td>
<td>900</td>
</tr>
<tr>
<td>LE</td>
<td>0.0829</td>
<td>100.0%</td>
<td>0.0829</td>
</tr>
<tr>
<td>PE</td>
<td>0.0809</td>
<td>97.6%</td>
<td>0.0809</td>
</tr>
<tr>
<td>EE</td>
<td>0.0020</td>
<td>2.4%</td>
<td>0.0020</td>
</tr>
<tr>
<td>THE</td>
<td>0.0000</td>
<td>0.0%</td>
<td>0.0000</td>
</tr>
<tr>
<td>CE</td>
<td>0.0000</td>
<td>0.0%</td>
<td>0.0000</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Aim Pre-strain</th>
<th>0.05</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature (°C)</td>
<td>1050</td>
<td>950</td>
<td>900</td>
</tr>
<tr>
<td>LE</td>
<td>0.0535</td>
<td>100.0%</td>
<td>0.0535</td>
</tr>
<tr>
<td>PE</td>
<td>0.0518</td>
<td>96.8%</td>
<td>0.0518</td>
</tr>
<tr>
<td>EE</td>
<td>0.0017</td>
<td>3.2%</td>
<td>0.0017</td>
</tr>
<tr>
<td>THE</td>
<td>0.0000</td>
<td>0.0%</td>
<td>0.0000</td>
</tr>
<tr>
<td>CE</td>
<td>0.0000</td>
<td>0.0%</td>
<td>0.0000</td>
</tr>
</tbody>
</table>

Table 5.3 Strain components for three pre-strains of 0.1, 0.08, and 0.05 and testing temperatures at 1050, 950, and 900°C after 2 hours at step 6 (LE=total strain; PE=plastic strain; EE=elastic strain; THE=thermal strain; CE=creep strain)

<table>
<thead>
<tr>
<th>0.1 Pre-strain</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature (°C)</td>
<td>1050</td>
<td>950</td>
<td>900</td>
</tr>
<tr>
<td>LE</td>
<td>0.1548</td>
<td>100.0%</td>
<td>0.1484</td>
</tr>
<tr>
<td>PE</td>
<td>0.1277</td>
<td>82.5%</td>
<td>0.1233</td>
</tr>
<tr>
<td>EE</td>
<td>0.0000</td>
<td>0.0%</td>
<td>0.0020</td>
</tr>
<tr>
<td>THE</td>
<td>0.0216</td>
<td>14.0%</td>
<td>0.0190</td>
</tr>
<tr>
<td>CE</td>
<td>0.0055</td>
<td>3.6%</td>
<td>0.0058</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>0.08 Pre-strain</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature (°C)</td>
<td>1050</td>
<td>950</td>
<td>900</td>
</tr>
<tr>
<td>LE</td>
<td>0.1329</td>
<td>100.0%</td>
<td>0.1286</td>
</tr>
<tr>
<td>PE</td>
<td>0.1063</td>
<td>80.0%</td>
<td>0.1047</td>
</tr>
<tr>
<td>EE</td>
<td>0.0000</td>
<td>0.0%</td>
<td>0.0002</td>
</tr>
<tr>
<td>THE</td>
<td>0.0216</td>
<td>16.3%</td>
<td>0.0191</td>
</tr>
<tr>
<td>CE</td>
<td>0.0050</td>
<td>3.8%</td>
<td>0.0046</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>0.05 Pre-strain</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature (°C)</td>
<td>1050</td>
<td>950</td>
<td>900</td>
</tr>
<tr>
<td>LE</td>
<td>0.1001</td>
<td>100.0%</td>
<td>0.0960</td>
</tr>
<tr>
<td>PE</td>
<td>0.0737</td>
<td>73.6%</td>
<td>0.0730</td>
</tr>
</tbody>
</table>
For all experimental test conditions, the critical strains (minimum strain to failure) will be determined from the initially applied pre-strain (Real_PE) in step 3 of the Gleeble experiment, and the FE calculated additional plastic strains after step 3 (ΔPE). Table 5.4 is an example for the plastic strain before and after heating steps for nine cases. Generally, the plastic strain (PE) increase is approximated at ~0.02-0.03 for all conditions, which include mechanical yielding due to the additional stroke offsetting thermal strain (labeled as “second stroke”) during heating.

Table 5.4 True plastic strain increase after heating step for all nine simulations

<table>
<thead>
<tr>
<th>Temperature(°C)</th>
<th>Aim_PE</th>
<th>First stroke (mm)</th>
<th>Real_PE</th>
<th>Second stroke (mm)</th>
<th>PE after heating</th>
<th>ΔPE</th>
</tr>
</thead>
<tbody>
<tr>
<td>1050</td>
<td>0.1</td>
<td>4</td>
<td>0.096</td>
<td>0.95</td>
<td>0.1277</td>
<td>0.0281</td>
</tr>
<tr>
<td></td>
<td>0.08</td>
<td>3.2</td>
<td>0.0809</td>
<td>1</td>
<td>0.1063</td>
<td>0.0254</td>
</tr>
<tr>
<td></td>
<td>0.05</td>
<td>2.0</td>
<td>0.0518</td>
<td>1</td>
<td>0.0737</td>
<td>0.0219</td>
</tr>
<tr>
<td>950</td>
<td>0.1</td>
<td>4</td>
<td>0.096</td>
<td>0.9</td>
<td>0.1233</td>
<td>0.0237</td>
</tr>
<tr>
<td></td>
<td>0.08</td>
<td>3.2</td>
<td>0.0809</td>
<td>0.9</td>
<td>0.1047</td>
<td>0.0238</td>
</tr>
<tr>
<td></td>
<td>0.05</td>
<td>2.0</td>
<td>0.0518</td>
<td>0.9</td>
<td>0.0730</td>
<td>0.0212</td>
</tr>
<tr>
<td>900</td>
<td>0.1</td>
<td>4</td>
<td>0.096</td>
<td>0.85</td>
<td>0.1221</td>
<td>0.0225</td>
</tr>
<tr>
<td></td>
<td>0.08</td>
<td>3.2</td>
<td>0.0809</td>
<td>0.85</td>
<td>0.1022</td>
<td>0.0213</td>
</tr>
<tr>
<td></td>
<td>0.05</td>
<td>2.0</td>
<td>0.0518</td>
<td>0.85</td>
<td>0.0709</td>
<td>0.0191</td>
</tr>
</tbody>
</table>

Table 5.5 summarized the SRC test results with updated true plastic strain from Gleeble model for 347H SS HAZ. In addition, the starting stress at temperature and failure information are added. The susceptibility map as a function of updated true pre-strain (based on the Gleeble FEM) and temperature is shown in Figure 5.21. This figure includes the three separate data points of failure, failure upon cooling and no failure. The objective of the susceptibility maps in Figure 5.21 and Figure 5.9 is to determine when and if failure would take place during PWHT in the 347H HAZ based on the plastic strain plots generated from the weld FEM. Since the peak plastic strain in the HAZ is near 0.09 strain, PWHT temperatures of 850 °C and below would be safe since the critical strain is 0.1. However, plastic strain alone does not contribute to cracking but rather contributes to the susceptible microstructure because of grain interior strengthening. An elastic stress must be present to open any microcracks that develop along PFZs, and thus the
critical stress plots, particularly starting stress at temperature shows a steadier monotonically
decreasing trend with increasing temperature (see Figure 5.9). Note that some samples with a
higher pre-strain had a faster stress relaxation rate than those with lower pre-strains. For
example, a pre-strain of 0.06 with testing temperature of 950°C (sample 45) failed at temperature
sooner than sample 35 (0.08 strain) at same testing temperature, but the extent of stress
relaxation of the higher pre-strain sample 35 was greater than the lower pre-strain. Thus, the
stress at target temperature was greater in sample 45, leading to a faster failure than sample 35.

Table 5.5 Summary of 347H SS SRC HAZ Gleeble results with updated true plastic strain from
Gleeble model (blue highlights represent critical strain/stress)

<table>
<thead>
<tr>
<th>Temp. (°C)</th>
<th>HAZ Peak Temp. (°C)</th>
<th>Set strain</th>
<th>True plastic strain</th>
<th>Starting Stress at Temp. (MPa)</th>
<th>Failure Criteria</th>
<th>Time to failure/cool</th>
</tr>
</thead>
<tbody>
<tr>
<td>750</td>
<td>1335</td>
<td>0.174</td>
<td>0.194</td>
<td>260</td>
<td>Yes, isothermally</td>
<td>22.2 h</td>
</tr>
<tr>
<td>800</td>
<td>1335</td>
<td>0.1</td>
<td>0.12</td>
<td>184</td>
<td>No</td>
<td>48 h</td>
</tr>
<tr>
<td>850</td>
<td>1335</td>
<td>0.13</td>
<td>0.15</td>
<td>184</td>
<td>Yes, cooling</td>
<td>24 h</td>
</tr>
<tr>
<td>850</td>
<td>1335</td>
<td>0.174</td>
<td>0.194</td>
<td>213</td>
<td>Yes, isothermally</td>
<td>2.9 h</td>
</tr>
<tr>
<td>850</td>
<td>1335</td>
<td>0.08</td>
<td>0.1</td>
<td>144</td>
<td>Yes, cooling</td>
<td>24 h</td>
</tr>
<tr>
<td>850</td>
<td>1275</td>
<td>0.1</td>
<td>0.12</td>
<td>152</td>
<td>Yes, isothermally</td>
<td>48.5 min</td>
</tr>
<tr>
<td>850</td>
<td>1335</td>
<td>0.1</td>
<td>0.12</td>
<td>158</td>
<td>Yes, isothermally</td>
<td>1.1 h</td>
</tr>
<tr>
<td>850</td>
<td>1335</td>
<td>0.174</td>
<td>0.194</td>
<td>174</td>
<td>Yes, isothermally</td>
<td>20.3 min</td>
</tr>
<tr>
<td>850</td>
<td>1335</td>
<td>0.04</td>
<td>0.06</td>
<td>89</td>
<td>No</td>
<td>24 h</td>
</tr>
<tr>
<td>850</td>
<td>1335</td>
<td>0.06</td>
<td>0.08</td>
<td>111</td>
<td>Yes, isothermally</td>
<td>1.03 h</td>
</tr>
<tr>
<td>900</td>
<td>1335</td>
<td>0.08</td>
<td>0.1</td>
<td>107</td>
<td>Yes, isothermally</td>
<td>4.9 h</td>
</tr>
<tr>
<td>900</td>
<td>1150</td>
<td>0.1</td>
<td>0.12</td>
<td>111</td>
<td>Yes, isothermally</td>
<td>1 h</td>
</tr>
<tr>
<td>900</td>
<td>1150</td>
<td>0.1</td>
<td>0.12</td>
<td>111</td>
<td>Yes, isothermally</td>
<td>24.1 min</td>
</tr>
<tr>
<td>900</td>
<td>1275</td>
<td>0.1</td>
<td>0.12</td>
<td>117</td>
<td>Yes, isothermally</td>
<td>13.5 min</td>
</tr>
<tr>
<td>900</td>
<td>1335</td>
<td>0.1</td>
<td>0.12</td>
<td>119</td>
<td>Yes, isothermally</td>
<td>31 min</td>
</tr>
<tr>
<td>900</td>
<td>1335</td>
<td>0.1</td>
<td>0.12</td>
<td>113</td>
<td>Yes, isothermally</td>
<td>18.3 min</td>
</tr>
<tr>
<td>Temperature</td>
<td>Corrected Pre-strain</td>
<td>Failure Mode</td>
<td>Time</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>-------------</td>
<td>----------------------</td>
<td>--------------</td>
<td>------</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1335</td>
<td>0.1 0.12 114</td>
<td>Yes, isothermally</td>
<td>23.3 min</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1335</td>
<td>0.174 0.194 128</td>
<td>Yes, isothermally</td>
<td>5.8 min</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>950 1335</td>
<td>0.05 0.07 69</td>
<td>No</td>
<td>10.9 h</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>950 1335</td>
<td>0.08 0.1 77</td>
<td>Yes, cooling</td>
<td>6.4 h</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>950 1335</td>
<td>0.1 0.12 97</td>
<td>Yes, isothermally</td>
<td>7.1 min</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1000 1335</td>
<td>0.04 0.06 56</td>
<td>Yes, cooling</td>
<td>4.7 h</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1000 1335</td>
<td>0.05 0.07 62</td>
<td>Yes, isothermally</td>
<td>1.2 h</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1000 1335</td>
<td>0.01 0.03 56</td>
<td>No</td>
<td>3.8 h</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1000 1335</td>
<td>0.025 0.045 35</td>
<td>No</td>
<td>3.6 h</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1000 1335</td>
<td>0.04 0.06 43</td>
<td>No</td>
<td>1.94 h</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1050 1335</td>
<td>0.05 0.07 70</td>
<td>Yes, isothermally</td>
<td>2.4 min</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1050 1335</td>
<td>0.1 0.12 60</td>
<td>Yes, isothermally</td>
<td>3 s (could have started during heating)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Figure 5.21** Susceptibility map as a function of corrected pre-strain from Gleeble FEM predictions and temperature
In conclusion, the 347H HAZ Gleeble simulation results match experimental results reasonably well, which indicated the material properties including stress strain relation with elevated temperature and creep parameters for SS347H is validated. This enables FE modeling of PWHT for future parameter optimization. On the other hand, the FE modeling of Gleeble experiment assisted to correct the true plastic strain in the susceptibility map by providing the plastic strain introduced by the stroke for thermal expansion offset during heating.

5.3 E347-347H cross welded SRC results

5.3.1 Gleeble reheat cracking results

Out of the 18 samples machined from the first set of E347-16 SS weld, 15 samples were used for SRC tests (Table 5.6) with one test being faulty (G.W.13). 11 additional tests were completed using the second batch of E347-16 SS weld. As mentioned earlier, the yield strength of weld specimens is significantly higher than the 347H HAZ SS samples, so that the first step involved pulling to an elastic strain mostly within 0.2% offset yield strength rather than introducing target plastic strain (few tests up to 1% strain). Like 347H HAZ SS samples, the starting stress at temperature for E347-16 WM samples is equally important to the starting stress at room temperature in terms of defining time to failure. The critical stresses (both step 3 and step 6 stresses) are highlighted in blue in Table 5.6 to indicate below which, fracture may not occur in the weld metal samples.

Table 5.6 Summary of E347-16 WM Gleeble SRC test results (blue highlights represent critical stress values for those temperatures)

<table>
<thead>
<tr>
<th>Sample #</th>
<th>Weld Batch #</th>
<th>Temp( °C)</th>
<th>Designated Stress @ RT (MPa)</th>
<th>Starting Stress @ Temp (MPa)</th>
<th>Failure</th>
<th>Time to failure/cool</th>
</tr>
</thead>
<tbody>
<tr>
<td>G.W.31</td>
<td>2</td>
<td>800</td>
<td>155</td>
<td>95</td>
<td>No</td>
<td>24 hr</td>
</tr>
<tr>
<td>G.W.29</td>
<td>2</td>
<td></td>
<td>217</td>
<td>126</td>
<td>Yes, isotherm</td>
<td>4 hr</td>
</tr>
<tr>
<td>G.W.28</td>
<td>2</td>
<td></td>
<td>218</td>
<td>120</td>
<td>Yes, isotherm</td>
<td>7.1 hr</td>
</tr>
<tr>
<td>G.W.27</td>
<td>2</td>
<td></td>
<td>401</td>
<td>175</td>
<td>Yes, isotherm</td>
<td>17.3 min</td>
</tr>
<tr>
<td>G.W.6</td>
<td>1</td>
<td></td>
<td>523</td>
<td>144</td>
<td>Yes, isotherm</td>
<td></td>
</tr>
<tr>
<td>G.W.23</td>
<td>2</td>
<td>850</td>
<td>156</td>
<td>81</td>
<td>No</td>
<td>9.2 hr</td>
</tr>
<tr>
<td>G.W.22</td>
<td>2</td>
<td></td>
<td>219</td>
<td>105</td>
<td>Yes, cool</td>
<td>4.8 hr</td>
</tr>
<tr>
<td>G.W.21</td>
<td>2</td>
<td></td>
<td>265</td>
<td>125</td>
<td>Yes, cool</td>
<td>2.2 hr</td>
</tr>
<tr>
<td>G.W.19</td>
<td>1</td>
<td></td>
<td>325</td>
<td>117</td>
<td>Yes, isotherm</td>
<td>5.4 hr</td>
</tr>
<tr>
<td>G.W.7</td>
<td>1</td>
<td></td>
<td>477</td>
<td>112</td>
<td>Yes, isotherm</td>
<td>3 hr</td>
</tr>
<tr>
<td>G.W.18</td>
<td>1</td>
<td></td>
<td>219</td>
<td>77</td>
<td>No</td>
<td>8.4 hr</td>
</tr>
<tr>
<td>G.W.16</td>
<td>1</td>
<td>900</td>
<td>262</td>
<td>85</td>
<td>Yes, isotherm</td>
<td>70 min</td>
</tr>
</tbody>
</table>
The susceptibility maps in Figure 5.22 illustrate similar behavior to the 347H HAZ samples. Note that these are the first batch of samples (see composition in Table 3.1) in order to confirm consistency in the material set used, particularly dilution of E347 WM composition from the first batch 347H substrate composition. Unfortunately, the composition for all 347H substrates used for the welding experiments was not the same since more plates were needed to complete testing, and the supplier did not have the same heat in stock. The carbon content for batch one was 0.043 wt% in contrast to batch 2 that had a carbon content of 0.052 wt%. The time to failure strongly depends on the temperature and the initial pre-stress condition. The range of stress points within the legend represent starting stresses at step 3, while the specific stress values associated with each data point correspond to the starting stress value at temperature when reaching step 6. Since the WM samples have a higher propensity for discontinuities and defects present in the weld, there is a possibility for higher data fluctuation (e.g., overlap between failure at temperature and cooling) in the WM results in contrast to the 347H HAZ results. However, the time to failure generally increases with decreasing temperature and decreasing residual stress. The threshold stress for failure decreases with increasing temperature, primarily due to creep and precipitation kinetics dominating at higher temperatures.
Figure 5.22 SRC susceptibility curves of E347-347H cross welded samples (all failures in WM) with various starting stresses (legend) and stress upon reaching temperature (labeled) for first batch of welds

5.3.2 Comparison between 347H HAZ and E347 WM

A comparison between the susceptibility of 347H HAZ and E347 WM (see Figure 5.23 and Figure 5.24) shows that the E347 WM would be more susceptible to reheat cracking due to the lower critical stresses in both susceptibility maps as a function of the starting stress and stress at temperature, respectively. If PWHT would be requested to prevent SRC in service, the parameters must be carefully designed to prevent reheat cracking during PWHT. One potential solution for future tank manufacturing would be stress control via design optimization of weld procedure and joint geometries (reduction of WM volume) in order to suppress susceptibility of SRC during PWHT. Techniques for gathering stress relaxation data during PWHT would be optimal, but there are no in-situ methods to collect this information on bulk welds in the field. Therefore, the validated weld FEM in combination with the susceptibility maps generated from Gleeble testing must be used to assist understanding of the stress relaxation behavior during PWHT to prevent cracking upon heating. For welds thicker than 1” made with E347 filler, direct heating to above 950°C would not be recommended since the threshold stress for cracking decreases as temperature increases (see Figure 5.24).
Figure 5.23 Starting stress at room temperature (step 3) vs. temperature for all samples, including failure at temp., failure upon cooling, and no failure.

Figure 5.24 Stress upon reaching temperature (step 6) vs. temperature for all samples, including failure at temp., failure upon cooling, and no failure.
Additionally, a combination of temperature and time at temperature are incorporated also to compare susceptibility between the 347H HAZ and E347 WM. The Larson Miller Parameter (LMP) is a tempering formula (Eq. 5.2) used to account for temperature and time for creep rupture tests and can be used for analysis of stress relaxation tests as well. The LMP (assuming $C=20$ [67]) is again written in the form:

$$LMP = \left( \frac{\tau(\kappa) \times (C + \log(t(hr))))}{1000} \right)$$

(5.2)

The initial stress at room temperature (Figure 5.25) and starting stress at temperature (Figure 5.26) are plotted versus the LMP. The HAZ shows a comparable trend to creep rupture data for both sets of stress and shows better trends than the WM at step 3. However, the WM only shows a reasonable trend for the starting stress at temperature vs. LMP, where with increasing LMP, the starting stress to failure reduces. The 1050°C tests are labeled to show high susceptibility (very short time to failure) with low critical stresses in Figure 5.26. The main conclusion from these figures is that the E347 WM fails at a much shorter time than the HAZ with a similar starting stress value, which therefore requires careful design of PWHT.

![Figure 5.25 Initial stress (step 3) vs. LMP for E347 WM and 347H HAZ samples](image-url)
Another important measurement for SRC susceptibility is the percentage of stress relaxation during heating to the designated test temperature (representative of PWHT). As shown in the Background section, the percentage of stress relief depends on the temperature (inelastic yielding) and time at temperature (creep) (see Figure 2.34). A percentage of stress relaxation for all the 347H HAZ and E347 WM samples was collected in Figure 5.27, which accounts the relative stress change from room temperature to target test temperature before holding starts (step 5 in Figure 3.13). From a stress relief point of view, longer times at higher temperatures would lead to complete stress relief during PWHT, e.g., the 1050°C simulations from the Gleeble FEM shows complete stress relief after two hours at temperature. As the temperature increases to beyond 950°C, the window of stress relaxation % increases to a narrower range. Note that 950°C was able to achieve a similar level of stress relaxation to 1050°C under a fast-heating rate and shorter heating time (due to less time needed to achieve the target temperature), which suggests 950°C would be a good candidate PWHT temperature. The stress relaxation for the WM samples contains a broader range than the HAZ samples, indicating more uncertainty in the SRC behavior of the WM with the presence of inhomogeneous microstructure and potential defects.
Metallurgical characterization of E347 WM samples

As mentioned previously, presence of any discontinuities (e.g., lack of penetration or slag inclusions) or pre-existing cracks could increase reheat cracking susceptibility by increasing local stress intensities. In addition, any cracks that exist prior to reheating, particularly ductility dip cracks (DDC) that develop in multi-pass welds, may be the initiation sites for reheat cracks to propagate under sufficient stress at elevated temperatures. All the WM samples that did not show any evidence of cracking during the Gleeble tests were cross sectioned to visually inspect the presence of microcracks. One sample particularly contained some DDC in low δ-ferrite containing reheated weld metal (Figure 5.28), which is very similar to what is observed in multi-pass Ni-Cr welds in literature [7, 68]. DDC is classified as a “warm” weldability crack type, which occurs during subsequent weld passes in WM of multi-pass welds due to reheating. For example, pass 11 induces a lateral tensile strain on pass 10 during solidification and cooling, so that the stresses combined with re-solutionizing of δ-ferrite (“micro fissuring”) leads to cracking during cooling. A high population of segregants (i.e., S, P, Nb, etc.) and coarse, incoherent,
precipitates along solidification grain boundaries is present when δ-ferrite is re-solutionized in the weld metal. A combination of cooling tensile stresses perpendicular to straight, columnar, low tortuous primary solidification grain boundaries, high population of segregation elements or coarse precipitates, and elevated temperature contributes to DDC. The presence of DDC could be initiation sites for reheat cracking during PWHT. Minimizing DDC by utilizing low heat input weld conditions and reducing stress could reduce SRC susceptibility.

Figure 5.28 Example of ductility dip crack (DDC) in reheated weld metal in 1000°C test with lowest stress condition (G.W.30)

The fracture surfaces for all the WM samples show a “woody” type fracture, as seen in Figure 5.29. The woody type of fracture indicates an orientation relationship with the columnar solidification morphology generally seen in the WM microstructures, which typically has a columnar ferritic-austenitic (FA) lathy solidification mode. In contrast to the HAZ samples, which consist of an equiaxed grain structure, the WM microstructure in it’s as-welded condition contains columnar solidification grain boundaries (SGB) with less tortuosity than the HAZ. Boundaries with an orientation perpendicular to maximum stresses would be more susceptible, particularly when grain boundary mobility (Coble creep) is highest with higher temperatures. The combination of stress relief combined with phase transformations at straight columnar SGB (e.g., reversion of δ-ferrite at temperatures above 800°C or sigma formation at lower temperatures at 500-800°C) may be a more susceptible microstructure than simply the PFZs mechanisms along equiaxed GB in the HAZ samples. However, the PFZs should still be present along SGB since Nb wants to diffuse from retained δ-ferrite into the γ-austenite or form as Nb (C, N) at temperature of 800-1050°C. The δ/γ interfaces in the WM microstructure are likely
regions where there would be a depletion of Nb (C, N) and significant PFZ formation. Examples of γ’ depletion in In 740H WM microstructures would support a similar PFZ mechanism for E347 WM microstructures [33] but at a much finer scale.

Figure 5.29 Fracture surfaces of 950°C sample with 518 MPa initial stress and 71 MPa stress at temperature (G.W.3) relative to the columnar orientation of the weld morphology and applied stress.

The cracks seem to mostly occur in interdendritic boundaries of the original solidification grain boundaries (SGB) (Figure 5.30 and Figure 5.31). While it seems SGB is the most susceptible crack boundary, MGB may be crack sensitive since they form more low tortuous grain boundaries during cooling from welding or during reheating in the Gleeble test. In the E347-16 WM, the microstructure seems to have mostly a FA solidification mode, where the first letter signifies what phase first develops a solid in the melt. The solidification rates and local composition (Cr/Ni eq ratio) determine which mode and amount of ferrite (~ 10% volume ferrite) develops in certain regions. For the 950°C sample that failed at 8.5 minutes (Figure 5.30), there seems to be very little change in the microstructure from the as-welded condition. However, the 800°C that failed upon cooling after 24 hours at temperature showed evidence of
sigma (σ) phase in the etched condition (Figure 5.31), which is typically seen as a darker etch
darker (due to richer Cr content) than δ-ferrite as seen in other similar microstructures confirmed
in literature [40]. However, EBSD might be needed to confirm whether or not sigma (σ) phase
The ferrite to sigma transformation has been reported to take place most rapidly between 750-
875°C with a minimum aging time of two hours [2]. The presence of sigma phase can greatly
reduce the WM ductility for service temperatures. Thus, re-solutionizing δ-ferrite during PWHT
is desired to prevent service embrittlement.

Figure 5.30 Microcracks in interdinderitic regions of columnar solidification grains for 950°C
sample with 437/75 MPa stress values (G.W.9) that failed at temperature.
Figure 5.31 Microcracks in interdendritic regions of columnar solidification grains for 800°C sample with 523/144 MPa stress values (G.W.6) that failed on cooling with some indication of darker phase near the microcracks. The arrows are pointing to a darker etched phase (darker than δ-ferrite), which is likely sigma.

Additional characterization was performed on samples with only microcracks and no complete failure during testing using electron back scatter diffraction (EBSD). Figure 5.32 demonstrates cracks along the main ferritic-austenitic solidification grain boundaries for the 1050°C condition. A very coarse scan (with a 2 µm resolution) mainly shows FCC austenite as the primary phase, with remnants of BCC ferrite within the bulk solidification grains. A neighbor grain correlation filtering using a minimum confidence index of 0.1 was used to help clean up the EBSD IPF figure. δ-ferrite would be noticeable with finer resolutions and lower magnification images. The inverse pole figure (IPF) reveals the orientation relationships between the main solidification and sub solidification grains. A histogram was developed (see Figure
by measuring the misorientation angle between the grain boundaries of the cracks and other grain boundaries with no cracks in the EBSD IPF maps (see Figure 5.32b). The distribution of misorientation angles between the crack solidification grain boundaries are mostly between 30-40°, which would be indicative of high angle grain boundaries. The no crack boundaries seem to have a much wider distribution ranging from ~6°-60°, with the majority of rotation angles between grains being 30-40°. The cracks occur primarily along (111) and (101) type solidification grains, which is typically the preferred solidification orientation of most FCC welds. Increasing grain boundary tortuosity would redirect the cracks and help prevent crack propagation in contrast to the low tortuous, straight solidification grain boundaries that are oriented perpendicular to the tensile axis (as seen in Figure 5.32 with the tensile axis being left to right of the images).

Figure 5.32 EBSD representation of macro cracks in WM of GW24 (1050°C): (a) micrograph of microcrack, (b) inverse pole figure (IPF) map, (c) phase identification map, and (d) grain color map
Certain methods for increasing tortuosity and grain refinement in the fusion zone (FZ) have been shown to be promising for arc welding applications by increasing strength and elevated temperature ductility. Arc oscillation, particularly transverse arc oscillation, has been a method to 1) increase temperature gradients with shallower weld pool widths and 2) produce faster travel speeds. Grain refinement is achieved by disturbing the max temperature gradient direction in the melt during initial stages of solidification with an oscillating arc (using alternating currents). Arc pulsation is another method to achieve similar microstructures. There is greater control of the microstructure with an automated, continuous process like gas metal arc welding (GMAW) or gas tungsten arc welding (GTAW). Shielded metal arc welding (SMAW) is limited to arc oscillation and pulsing methods due to its manual limitations even though it provides flexibility for field welding.

5.4 FE Analysis of Failure Susceptibility

For a single-V joint, the fusion zone (FZ) near the top surface and root area (see Figure 5.34) seems to be the most susceptible region (or area of crack initiation) to reheat cracking or SRC with the highest degree of tensile residuals stresses and with a lower stress threshold compared to HAZ. However, there is not one specific location within the high stress regions at
top subsurface that would fail first, but rather a general area for SRC susceptibility. There is literature verification to support the top subsurface region of a single-V joint as highly susceptible to SRC (see Figure 5.35). Stress contours with another literature FEM [69] verify similar degrees of longitudinal stress (parallel to weld direction in the WM). An example of cracking near the top sub-surface regions of a dissimilar weld between stainless steel and P91 with ErNiCrFe-3 WM after 125,000 hours at service at 565°C indicate cracking along the fusion boundary due to a combination of residual stresses and carbide free regions [70]. Another technique for reducing crack susceptibility would be to grind out existing E347 WM in the top half of the single-V grooves and weld using an alternative filler that is more SRC resistant (e.g., E16.8.2). This method would help reduce reheat cracking during PWHT, which may be needed for thick, constrained welds above a half-inch.

Figure 5.34 (a) 2-D von Mises and (b) plastic strain contours for high stress region area from FE simulation of 2” weld in unclamped condition combined with (c) initial stress vs. temperature susceptibility map of E347 WM and 347H HAZ
Figure 5.35 Comparison of stress in (a) FEM simulation with (b) literature FEM [69] and (c) example of service failure in dissimilar weld (DSW) of stainless steel and P91 in similar location [70].

The stress contours upon reaching 950°C during FEM PWHT simulation are shown in Figure 5.36 with the highest stress located near the top subsurface and around the root area. Based on the crack susceptibility map as a function of starting stress and temperature, a stress of 93 MPa (peak von Mises in the FEM) would be higher than the WM critical stress for cracking to occur, even though the peak stresses would be close to the no-failure conditions for the 347H HAZ. The top sub-subsurface region would be the most susceptible region during PWHT. A multi-step PWHT is hence necessary in this case to reduce the stress at temperature below the threshold value of 65 MPa at 950°C. Using a double-V groove (see Figure 4.16) would move the susceptible crack region to the mid-thickness region but with smaller regions of peak stresses. J groove has been recommended by Worley Parsons (one of our industrial partners) for residual stress control, which is proven effective in a one-inch weld as shown in Figure 4.16. While future tanks should implement using less susceptible weld consumables for the completion of the whole joint, current tanks using a single-V joint and E347 filler may experience formation of cracks in highly stressed regions during PWHT, especially when a one-step PWHT is applied, and thereafter need to undergo repair welding (particularly the top half of the joints with thicknesses of a half-inch and above). During and after PWHT, careful non-destructive evaluation (NDE) of these high residual stress areas would be needed prior to installments of other components within the tank and entering service.
Three reheat crack tests were completed at 950°C with NUCL 167 SPH simulated HAZ with a peak temperature of 1335°C. The three strains used were 0.165, 0.085, and 0.07, using the c-gauge to measure displacement for calculating true strain. There were no indications of failure for all three tests based on the stress relaxation curves shown in Figure 5.37. The creep rates are rapid above 600°C during heating for NUCL 167 SPH relative to 347H (see Figure 5.38). Therefore, stress relaxes during heating at a much faster rate than 347H HAZ due to faster creep strain rates, and the stress at elevated temperatures are much lower than 347H HAZ. NUCL 167 SPH HAZ would then have lesser high temperature strength in comparison to 347H HAZ. Therefore, the creep ductility, and crack resistance of NUCL 167 SPH are expected to outperform 347H. The NUCL 167 SPH results should be comparable to the E16.8.2 WM since they have very similar chemical compositions, except for some differences in Cr content.

A cross sectional analysis of the samples for the three SRC tests indicate no microcracks of any kind. However, there seemed to be indications of grain boundary liquation that nucleate along rolling bands in the hot rolled plate during the 1340°C peak temperature HAZ thermal cycle (Figure 5.39). In addition, abnormal grain growth seems to occur during the high temperature weld thermal cycle due to relatively coarser pre-existing grains and grain boundaries with higher mobility (less grain boundary pinning from secondary particles (e.g., borides)).
The next section shows some microcracking in the E16.8.2 WM with a 6% strain (560 MPa) and 950°C condition. Since no cracks developed up to 16.5% (~580 MPa) plastic strain for a temperature of 950°C in the NUCL 167 SPH samples, this data set represents the lowest susceptibility compared to the other three material sets. In all, NUCL 167 SPH would be a less SRC susceptible alloy. However, the ASME BP & V codes limit the use of an alloy with low C contents for a service condition of 565°C.

Figure 5.37 Stress evolution as a function of temperature and time for three pre-strains of 0.165, 0.085, and 0.07 at 950°C for NUCL 167 SPH samples (no cracking)
Figure 5.38 Comparison of stress evolution as a function of temperature and time among HAZ of 347H and NUCL 167 SPH simulated with 1335°C peak temp and at a few similar starting stresses at room temperature (step 3)

Figure 5.39 Polarized LOM of electroetch (2V) with 40% nitric acid solution of 0.085 strain sample at 950°C (half width, center of gauge section) with signs of varying grain sizes and liquation along the rolling bands (due to HAZ thermal cycle)
5.5 E16.8.2-347H cross welded SRC results

5.5.1 Gleeble reheat crack results and characterization

A summary of the SRC test results and failure characteristics are outlined in Table 5.7. No obvious cracks were observed in samples with 1% strain up to 950°C (e.g., Sample Mo5 and Sample Mo6). Additional plastic strain (more extreme condition) is needed to induce cracking in the lower temperature samples. Some microcracks in the E16.8.2 WM were discovered for the 1 and 6% strain at 1050°C and higher strain (6%) for 950°C as shown in Figure 5.40. Some microcracking was seen in the 347H HAZ of the Mo10 sample in Figure 5.40. Figure 5.41 compares a few cases under 6% strain demonstrating a level of inconsistency in the weld metal SRC testing results, which agrees with the unclear trend of time to failure for WM samples observed in Figure 5.25. Two cases at 900 and 950°C exhibited complete failure in the HAZ of the cross welded samples, while two other cases at 950°C and 1050°C only exhibited surface microcracks in the weld metal without failure for the 6% strain tests. It should be noted that temperature of HAZ is approximately 50°C lower than peak temperature in center of gauge sample. Therefore, the E16.8.2 WM would be less susceptible to reheat cracking and SRC than 347H HAZ.

Table 5.7 Summarized results of E16.8.2-15-347H cross welded SRC Gleeble samples

<table>
<thead>
<tr>
<th>Sample ID #</th>
<th>Strain/stress (°C)</th>
<th>Temperature (°C)</th>
<th>Time at T</th>
<th>Cracks</th>
<th>Failure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mo6</td>
<td>0.01/ 453 MPa</td>
<td>850</td>
<td>24 h</td>
<td>None</td>
<td>No</td>
</tr>
<tr>
<td>Mo5</td>
<td>0.01/ 455 MPa</td>
<td>950</td>
<td>1.94 h</td>
<td>None</td>
<td>No</td>
</tr>
<tr>
<td>Mo7</td>
<td>0.01/ 471 MPa</td>
<td>1050</td>
<td>5 min</td>
<td>Macro surface cracks in E16.8.2 WM</td>
<td>No</td>
</tr>
<tr>
<td>Mo9</td>
<td>0.06/ 577 MPa</td>
<td>900</td>
<td>5.6 h</td>
<td>Failed in 347H HAZ (850°C)</td>
<td>Yes</td>
</tr>
<tr>
<td>Mo8</td>
<td>0.06/ 590 MPa</td>
<td>950</td>
<td>0.51 h</td>
<td>Failed in 347H HAZ (900°C)</td>
<td>Yes</td>
</tr>
<tr>
<td>Mo11</td>
<td>0.06/ 560 MPa</td>
<td>950</td>
<td>1.35 h</td>
<td>Macro surface cracks in E16.8.2 WM, Micro crack in 347H HAZ (900°C)</td>
<td>No</td>
</tr>
<tr>
<td>Mo10</td>
<td>0.06/ 551 MPa</td>
<td>1050</td>
<td>3.6 min</td>
<td>Macro surface cracks in E16.8.2 WM, Micro crack in 347H HAZ (1000°C)</td>
<td>No</td>
</tr>
</tbody>
</table>
Figure 5.40 Representation of cracks in E16.8.-347H cross welded samples for Mo7, Mo11 and Mo10, including HAZ microcracks

Figure 5.41 SRC testing results of E16.8.2-347H cross welded SRC samples with 6% strain showing failure in HAZ under 900 and 950°C, but surface cracks only in WM under 950 and 1050°C (NOTE: temperature of 347H HAZ is approximately 50°C lower than peak temperature in center of gauge sample)
A brief fracture analysis of the cross-welded sample under 950°C and 6% strain (sample Mo8) is shown in Figure 5.42, where fracture occurred in HAZ of the base metal. Similar fracture characteristics are observed to the 347H HAZ simulated samples (see Figure 5.12), which validates the HAZ simulation tests. The fracture surfaces seem to represent intergranular cracking with the presence of micro-ductile features on grain boundaries, so that failure occurs along grain boundaries or the interface between grain boundaries and the intergranular continuous Nb (C, N). The incoherency between the γ-austenite/Nb (C, N) precipitate surface at grain boundaries provide a weak interface for where failure to propagate through. There is no significant difference in the main fracture characteristics across the whole surface, except for the oxide color developed during the cracking process. The fracture initiation starts on the surface where the oxide layer is a dark grey while final fracture occurs where the lower temperature brownish, orange oxide exists as observed in Figure 5.42. The fracture surfaces do not reveal any clear WM reheat cracking fracture surface (i.e., woody fracture), with linear void indicative of failure parallel to a columnar solidification microstructure in WM. A comparison of some of the fracture surfaces for the E347-347H samples and E16.8.2-347H samples is shown in Figure 5.43. A columnar orientation is observed in the E347 WM fracture surfaces versus “shiny” brittle fractures surfaces in the 347H HAZ of the E16.8.2-347H samples.

Figure 5.42 Fracture surface analysis of Mo8 sample (950°C/6% strain) that failed in 347H HAZ where Top represents area closer to top surface of weld and Bottom represents more subsurface region.
Austenitic stainless steel weld microstructures, unlike most low C steel grades, preserves solidification microstructures at room temperature. For solidification morphologies, the three important free surfaces are: 1) solidification grain boundaries (SGB) 2) solidification sub-grain boundaries (SSGB), and 3) migrated grain boundaries (MGB). MGB are essentially “migrated” boundaries from the primary, tortuous SGB. Based on a rough analysis of one of the E16.8.2 samples, the cracks seem to propagate along the SGB which exhibits a less tortuous path in Figure 5.44. Also, according to Lippold, MGB may essentially develops during reheating because there is a driving force for migration, to lower the boundary energy of the original SGB [9]. This behavior can be observed in mainly A, sometimes AF, solidification modes (A meaning negligible δ-ferrite during solidification; AF meaning austenite forming first followed by ferrite in the melt). Based on the analysis earlier, AF solidification morphologies are very likely present in the E16.8.2 microstructures (see Figure 4.9).
5.5.2 Comparison of E347 and E16.8.2 SRC susceptibility

While there is microcracking within the E16.8.2 1% strain samples due to reheating under stress, the degree of cracking in the E347 WM exceeds all four sets of samples investigated since its critical stresses to failure are the lowest. Figure 5.45 and Figure 5.46 compares stress relaxation curves under 1% strain conditions for the E16.8.2 and E347 samples at 850°C and 950°C, respectively. No failure was observed in E16.8.2 welds within testing duration of 24 hours at temperature and during cooling, while E347 welds failed all failed at temperature within a few hours. It seems that the stress relaxation and creep rates are similar for both weld fillers, but creep ductility is much higher in the E16.8.2 WM since these samples need more applied strain to induce microcracking. Therefore, E16.8.2 as an alternative filler may be used for repair welding of 347H welds or as the primary choice of filler for newly developed weld joints.
Figure 5.45 Comparison of stress relaxation between E16.8.2 (Mo6) and E347 (GW7) cross-welded samples during 850°C, 1% strain test. Failure occurs in E347, but not in E16.8.2.

Figure 5.46 Comparison of stress relaxation between E16.8.2 (Mo5) and E347 (GW3) cross-welded samples during 950°C, 1% strain test. Failure occurs in E347, but not in E16.8.2 after 2 hours and during cooling.
CHAPTER 6: ELEVATED TEMPERATURE TESTS AT 600°C OF 347H WELDS AND ALTERNATIVES WITH CONSIDERATION OF PWHT

Thermomechanical tests were performed on all four sets of materials at 600°C with and without PWHT. The thermomechanical data, including 0.2% offset yield strength, engineering ultimate tensile strength (UTS), peak uniform true strength, true uniform plastic strain, and calculated uniform engineering elongation, are tabulated in Table 6.1.

Table 6.1 Thermomechanical data of sample with and without PWHT pulled at 600°C after 4 hours service age (grey boxes represent two-step PWHT tests)

<table>
<thead>
<tr>
<th>Group</th>
<th>Sample #</th>
<th>PWHT</th>
<th>Yield strength (MPa)</th>
<th>Engineering UTS (MPa)</th>
<th>Peak true stress (MPa)</th>
<th>True uniform strain</th>
<th>Uniform engineering strain</th>
</tr>
</thead>
<tbody>
<tr>
<td>347H HAZ</td>
<td>26</td>
<td>none</td>
<td>282</td>
<td>411</td>
<td>510</td>
<td>0.221</td>
<td>0.247</td>
</tr>
<tr>
<td></td>
<td>27</td>
<td>none</td>
<td>278</td>
<td>409</td>
<td>518</td>
<td>0.237</td>
<td>0.267</td>
</tr>
<tr>
<td></td>
<td>29</td>
<td>750°C/2 hr, 1050°C/30 min</td>
<td>200</td>
<td>347</td>
<td>405</td>
<td>0.16</td>
<td>0.174</td>
</tr>
<tr>
<td></td>
<td>43</td>
<td>950°C/2 hr</td>
<td>223</td>
<td>352</td>
<td>416</td>
<td>0.167</td>
<td>0.181</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>1050°C/30 min</td>
<td>193</td>
<td>347</td>
<td>415</td>
<td>0.179</td>
<td>0.196</td>
</tr>
<tr>
<td></td>
<td>42</td>
<td>950°C/2 hr- overheated to 1200°C</td>
<td>119</td>
<td>301</td>
<td>372</td>
<td>0.217</td>
<td>0.24</td>
</tr>
<tr>
<td>NUCL 167 SPH (316L) HAZ</td>
<td>NUC-6</td>
<td>none</td>
<td>260</td>
<td>404</td>
<td>526</td>
<td>0.263</td>
<td>0.301</td>
</tr>
<tr>
<td></td>
<td>NUC-8</td>
<td>950°C/2 hr</td>
<td>127</td>
<td>380</td>
<td>504</td>
<td>0.293</td>
<td>0.342</td>
</tr>
<tr>
<td></td>
<td>NUC-7</td>
<td>1050°C/30 min</td>
<td>85</td>
<td>360</td>
<td>513</td>
<td>0.356</td>
<td>0.428</td>
</tr>
<tr>
<td>E347</td>
<td>GW11</td>
<td>none</td>
<td>302</td>
<td>382</td>
<td>447</td>
<td>0.159</td>
<td>0.172</td>
</tr>
<tr>
<td></td>
<td>GW12</td>
<td>950°C/2 hr</td>
<td>210</td>
<td>340</td>
<td>392</td>
<td>0.145</td>
<td>0.156</td>
</tr>
<tr>
<td></td>
<td>GW1</td>
<td>1050°C/30 min</td>
<td>170</td>
<td>330</td>
<td>406</td>
<td>0.209</td>
<td>0.233</td>
</tr>
<tr>
<td>E16.8.2</td>
<td>Mo2</td>
<td>none</td>
<td>260</td>
<td>372</td>
<td>427</td>
<td>0.14</td>
<td>0.15</td>
</tr>
<tr>
<td></td>
<td>Mo3</td>
<td>950°C/2 hr</td>
<td>144</td>
<td>335</td>
<td>437</td>
<td>0.269</td>
<td>0.309</td>
</tr>
<tr>
<td></td>
<td>Mo4</td>
<td>1050°C/30 min</td>
<td>85</td>
<td>336</td>
<td>430</td>
<td>0.246</td>
<td>0.280</td>
</tr>
</tbody>
</table>

The weld strength reduction factor (WSRF) must be considered when planning to calculate the thickness of a tank or pipeline with a PWHT using ASME BP&V code. The
strength may vary depending on the temperature and time profile of a PWHT. 950°C and 1050°C were both chosen as options for PWHT of 347H in this evaluation process due to recommendations from literature [2, 3], which are expected to reduce residual stress and provide a less susceptible microstructure before service while maintaining sufficient strength. For all four sets of samples that underwent a PWHT, there is a drop in yield strength with increasing PWHT temperature for both HAZ and WM.

The maximum prediction of strain development in the FEM as-welded condition in the HAZ approaches approximately ~0.09 near the root. Testing the thermomechanical behavior with an experimental 0.1 strain prior to PWHT or tensile testing was deemed necessary to see the effect of recovery and recrystallization of a HAZ with a high plastic strain. The WM samples did not have an applied strain prior to testing as the received microstructure is the desired initial condition. True stress strain was plotted to compare yield strength and uniform elongation in all the samples. Comparisons of the thermomechanical behavior in true stress/strain are made between the pre-deformed HAZ at 0.1 strain (450 MPa) and E347-16 WM at service temperatures of 600°C (Figure 6.1):

- With no PWHT, WM appears to have a slightly higher yield strength (302 MPa) than HAZ (~280 MPa), but with the expense of ductility (0.16 v. 0.22 -0.24 for HAZ)
- 950 °C heat treatment leads to a slightly higher yield strength (223MPa) and ductility (0.17) in HAZ than WM (210MPa and 0.145) at 600 °C
- 1050 °C heat treatment seems to lead to a distinctive difference between the yield strength for WM (170 MPa) and HAZ (193 MPa), while the ductility was better in the WM (0.21) than the HAZ (0.18)

The thermomechanical behavior of the 347H HAZ and both WM sets (E16.8.2 WM and E347) at service temperature and with and without PWHT can be compared through Figure 6.1 and Figure 6.2. The yield strength in the no PWHT condition for both WM sets is very similar with a 10% pre-strained 347H HAZ. However, both 950°C and 1050°C PWHT greatly reduce the yield strength in the E16.8.2 WM. The drop in yield strength after PWHT is likely due to recovery (annihilation of dislocations) at 950 and 1050°C from the pre-deformed, as-welded microstructure. However, Nb (C, N) provide a grain boundary pinning effect and recrystallization delay in the E347 solidification grain boundaries and the 347H HAZ equiaxed
grain boundaries, thereby retaining a better strength than E16.8.2 WM. For structural design, a PWHT may not be needed when considering E16.8.2 WM by itself since it has low SRC susceptibility and great creep ductility. The limiting factor would then be the 347H HAZ as it is more susceptible to SRC than E16.8.2 WM.

Figure 6.1 Comparison of 347H HAZ and E347-16 WM tensile test behavior at 600°C with and without PWHT (two conditions including 950°C/2 hr. and 1050°C/30 min). Two-step PWHT (750°C-2hr/1050°C-30min) and one test with an overheating PWHT to 1200°C is included for 347H HAZ.

On the other hand, E16.8.2 WM after PWHT exhibits better ductility than both 347 HAZ and E347 WM samples. A one-step PWHT at 950°C reduced the uniform elongation the most for both the 347H simulated HAZ and E347 WM, possibly because it falls within the active Nb (C, N) precipitation temperature range for two hours. A higher number of Nb (C, N) present after aging within a high intragranular nucleation rate temperature range contributes to greater pinning of dislocation movement during deformation. However, the E16.8.2 WM achieved almost twice as much ductility as the E347 WM after a 950°C PWHT. Samples heat treated at 1050°C showed better ductility than those heat treated at 950°C for both HAZ and WM samples. Generally, PWHT provides a better ductility in E16.8.2 WM with a reduction in strength. It should be noted that PWHT temperature for E16.8.2 may not need to be as high as 950°C due to reduced cracking susceptibility.
It was found out that for 347H HAZ, with the same peak temperature during PWHT and duration, e.g., 1050°C for 30min, similar mechanical behavior was observed in a two-step (S29) and one-step PWHT (S30) as highlighted in grey in Table 6.1. However, multi-step PWHT may still be preferred to avoid high starting stress at temperature, which leads to high susceptibility to cracking.

![Graph showing tensile test behavior at 600°C with and without PWHT](image)

Figure 6.2 Comparison of E347-16 and E16.8.2-15 WM tensile test behavior at 600°C with and without PWHT (two conditions including 950°C/2 hr. and 1050°C/30 min)

Another example of a weld filler and plate combination would be NUCL 167 SPH HAZ and E16.8.2. A comparison of the thermomechanical data is illustrated in Figure 6.3. The yield strength is very similar for all conditions for these two data sets. However, the substrate maintains better ductility than the E16.8.2 WM for all cases but particularly in the as-welded condition. A 950°C PWHT may be useful for increasing the ductility of the E16.8.2 WM. However, PWHT may not be necessary for NUCL 167 SPH welded with a matching filler or E16.8.2 due to the low cracking susceptibility.

The last comparison of thermomechanical data pertains to the 347H HAZ and 167 SPH HAZ, which is displayed in Figure 6.4. In the as-welded condition (with 10% applied strain), the yield strength is similar for both sets with 167 SPH maintaining better ductility for all cases. However, the yield strength drops significantly in the 167 SPH HAZ versus the 347H HAZ after PWHT. As 167 SPH may not require PWHT as 347H does, 167 SPH HAZ without PWHT
provides a comparable strength to 347H HAZ with PWHT but with a better ductility. Essentially, PWHT parameters must be accounted for during mechanical design for current and future tanks to ensure the minimum yield strength (dependent on thickness) is maintained prior to service to prevent issues of mechanical overloading (while also preventing SRC during service).

Figure 6.3 Comparison of NUCL 167 SPH (noted as 316L in the plot) HAZ and E16.8.2-15 WM tensile test behavior at 600°C with and without PWHT (both 950°C/2 hr. and 1050°C/30 min)

Figure 6.4 Comparison of 347H HAZ and NUCL 167 SPH (316L) HAZ tensile test behavior at 600°C with and without PWHT (both 950°C/2 hr. and 1050°C/30 min). Two-step PWHT (750°C-2hr/1050°C-30min) and one test with an overheating PWHT to 1200°C is included for 347H HAZ
CHAPTER 7: CONCLUSIONS AND KEY TAKEAWAYS

7.1 Conclusions

Based on the results discussed in this report, the following summary of work and conclusions can be made:

1. Gleeble reheat cracking tests were completed on four sets of samples: 1) 347H HAZ, 2) NUCL 167 SPH (modified 316L), 3) cross welded samples of E347-347H, 4) cross welded samples of E16.8.2-347H WM, and the following conclusions can be made:
   a. The pre-strain/stress level, which is directly related to residual stress, is critical for determination of safe PWHT temperatures for 347H SS HAZ and E347-16 WM
   b. SRC susceptibility can be ranked as E347 WM > 347H HAZ > E16.8.2 WM > NUCL 167 SPH HAZ
   c. E16.8.2 WM is recommended as the filler consumable for 347H SS welds.

2. Effect of plate thickness on residual stress distribution was evaluated through three thicknesses through FEM including 0.5, 1, and 2”:
   a. 1” and 2” thick welds generated similar peak stresses in both thicknesses, while 0.5” thick weld showed slightly lower peak stress and much smaller high-stress area size.
   b. Single-V joint is not recommended for plates thicker than 0.5” due to introduction of high residual stresses. FE analysis in combination with the Gleeble results indicated failure preferentially occur in the subsurface region near top passes with high tensile stress/strain.
   c. Adoption of a J-groove effectively reduced residual stresses in the WM and is recommended for joints greater than 0.5 inches where feasible.

3. PWHT could be a viable solution for reducing stress.
   a. 950°C is the recommended peak temperature for PWHT, due to the relatively high critical strain/stress to fracture, and for the purpose of retaining strength and controlling oxidation.
   b. A multi-step PWHT is recommended for thick welds with high tensile stresses to prevent SRC during PWHT.
4. Thermomechanical experiments (elevated temperature tensile tests) were completed on all four sets of samples to reveal the effect of PWHT on yield strength reduction.
   a. A higher PWHT temperature reduces the yield strength of all four sets of samples, but the degree of strength reduction depends on the material. E347 WM and 347H samples retained the highest yield strength compared to the E16.8.2 WM and NUCL 167 SPH HAZ samples.
   b. E16.8.2 WM and NUCL 167 SPH HAZ (w/ 10% strain) behave similarly before and after PWHT.
   c. PWHT may not be needed for alternative alloys but may be needed for a E16.8.2-347H SS weld due to susceptibility of the HAZ to cracking.

7.2 Key Takeaways with Respect to Generation 2 TES Fabrication

Based on these conclusions, there are some practical key takeaways corresponding to welding fabrication of existing and future tanks for Generation 2 CSP TES hot tanks experiencing a service temperature of 565°C. The three scenarios that are accounted for are: 1) existing tanks of 347H SS, 2) future tanks being manufactured with 347H SS as the shell material, and 3) future tanks with alternative base alloy options.

1. Existing tanks of 347H SS
   a. Localized PWHT should be incorporated prior to further service conditions. Stress reduction through PWHT (2-step PWHT with 950°C peak temperature) while avoiding SRC and formation of Cr-carbides during cooling. AWS D10.10 ("Recommended Practices for Local Heating of Welds in Piping and Tubing") provides details on heating and cooling rates as well as ceramic pad and induction heating method parameters for various weld geometries.
   b. E16.8.2 weld consumables are recommended for repair welding of high stressed regions prior to PWHT. Grinding out old E347 weld metal may be necessary and replacing I with E16.8.2 filler for high stressed regions to prevent reheat cracking during PWHT.

2. Future tanks with 347H SS as the shell material
   a. Use alternative, less SRC susceptible filler metals for the whole welding joint (e.g., E16.8.2).
b. Control residual stress by optimizing joint geometry design and welding procedure (e.g., J groove and/or double-V groove) for easily accessible joints.

c. Use high deposition-rate low heat-input processes (e.g., arc pulsation/oscillation methods) to 1) provide a finer, equiaxed, more tortuous weld metal microstructure, 2) reduce HAZ width and 3) reduce residual stress.

d. Localized PWHT is recommended if 347H SS is still the shell material.

3. Future tanks with alternative options

a. Alternative base metals and filler wires that are not SRC susceptible would be preferred. However, the temperature limitation (454°C) for 316L alloys according to ASEM Section II-D for Section VIII applications would hinder the ability to fabricate tanks using NUCL 167 SPH (316L w/ B)) [11]. The other concern for weaker alloys would be an increase in thickness. An increase in thickness increases production time but alternative alloys might be cheaper if no PWHT is needed. An update to the ASME code for max stress allowances of NUCL 167 SPH might be needed to qualify this new alloy for CSP hot tanks.

b. Advanced manufacturing technologies using cladding. Using a thin layer 347H clad (for sensitization resistance) on a 304H SS backer may be an option. However, there has been evidence that 304H has experienced SRC in literature, too [8]. Therefore, a PWHT may still be needed for thick 304H SS welds. Another option would include cladding of 347H on a Gr. 91/92 backer, which is a creep enhanced ferritic steel (CEFS).
CHAPTER 8: FUTURE WORK

The work developed from this project has been extended to a one-year lab call. The recommended protocols developed here are used to perform localized PWHT procedures on a lab-scale weldments using ceramic and induction heating methods in conjunction with non-destructive testing and evaluation methods to detect potential cracks before and after PWHT. Industry driven heating and cooling rates for localized heating, e.g. AWS D10.10 [45], are being investigated, such as 111°C/hr. and 222°C/hr.

An example of the effect of heating rate, using ceramic heating on the top side of the 2” thick weld FEM, combined with the E347 critical threshold values are shown in Figure 8.1. The temperature and stress profiles at a node within the region with the highest stress, the most susceptible region to SRC, are shown in Figure 8.1a (black circle). Heat treatment reduces the stress significantly in the 2” thick weld in comparison to the as-welded condition. For example, the highest stress regions experience a reduction from 481 to 53 MPa during the heating stage. The temperature-stress failure criteria obtained from the E347 WM SRC experiments are used to identify the appropriate heating rate range. For instance, in Figure 8.1(c), the open triangle, square and diamond sign show no failure at their plotted stress and temperature after 24 hours (95 MPa at 800°C, 81 MPa at 850 °C and 77 MPa at 900 °C), while the solid symbols (120 MPa at 800 °C after 7.1 hrs., 105 MPa at 850 °C after 4.8 hrs., and 85 MPa at 900 °C after 70 min.) show failure in experiment. During PWHT, the stress as a function of temperature and time should be at least located underneath the solid symbols, or more conservatively, close-to or below the open symbols, to avoid formation of cracks or micro-voids.

As mentioned earlier, faster heating rates (within the maximum allowable heating rate due to temperature gradient concerns) have been recommended to avoid meeting the crack c-susceptible curves. However, too fast of a heating rate (e.g., 333°C/h.) may lead to fast cracking or formation of micro-voids based on Gleeble SRC susceptibility tests since the stress did not relax sufficiently in time to fall below the critical stress values up to 900°C. It is obvious that 111 °C/h is a safer condition to use than 333 °C/h case based on a stress relaxation that falls below the critical stress values for 850°C and above. Since time to failure at 120 MPa and 800°C (solid symbol) condition was 7.1 hrs, there would be little to no concern for cracking to occur since the stress drops below the critical stresses for temperatures above 800 °C during heating and there is not that much time spent near 800°C during heating up. While slow enough heating rates are
recommended to allow for stress relaxation to occur during heating before reaching susceptible temperatures (>800°C), too slow heating rates will allow for extended times at temperature during heating that may hit a c-crack susceptibility time range and lead to undesired microstructural changes. Thus, an appropriate heating rate range needs to be established to make sure not too much time is spent during heating, while at the same time allowing for stress to reduce below a critical stress threshold determined from the Gleeble experiments. A finer step size will be used to further confirm the appropriate heating rate range between 222 and 111°C/h.

Figure 8.1 (a) Von Mises stress contour under as-welded condition after unclamping; (b) Temperature field after 4-hour holding at 950°C, (c) Stress relief along the heating with different heating rate. Solid and open symbols show failure and no failure at certain temperatures and stresses in experiment, respectively.
Also, analysis of the 1” thick neutron diffraction residual strain measurements will take place within the next couple months to further validate the weld FEM. For this case, there were better diffraction peaks and many more data points with half the thickness of the first initial experiments. The as-welded and PWHT (870°C/3 hours) bulk welds were both investigated with two sets of reference samples to have better calibration of the stress-free d-spacing values in the as-welded and PWHT conditions. All three principal directions were investigated. This work will be placed in the form of a publication.

Additionally, collaboration with OSU and ORNL regarding the stress relaxation creep failure model could be useful for gathering additional information regarding validation of SRC mechanisms and contributing factors. For example, time to failure predictions using a combination of multiple metallurgical factors (e.g., grain size, void formation, etc.) for both WM and HAZ would benefit industry when considering PWHT.
REFERENCES


R. L. Phebus, "Weldability of Heavy Sections of AISI Type 347 Stainless Steel," U.S. Navy Engineering Experiment Station 1957.


APPENDIX A: WELD PARAMETERS

The welding procedure specification (WPS) given by Brahma, Inc. shows weld details used for a hot tank joint (Figure A.1). It should be noted that the design is a 75° single-V bevel joint with GTAW used for root and hot passes, while SMAW is used for all filling passes. The pre-qualification records (PQR-numbered 81 and 82 in the WPS) used to qualify this WPS were 3/8 (Figure A.2) and 1” (Figure A.3) thick welds. As observed from the PQRs, all the transverse face and side bend tests passed the acceptance criteria according to ASME Section IX. Tensile tests and corrosion tests were also used to qualify the weld procedures. The thicker welds were qualified to use higher heat input values (higher amps for the 1” thick SMAW passes compared to the 3/8” thick weld) to maximize deposition rate of the weld consumable. The limit of 150 Amps was considered as the high heat input condition and was used for all SMAW weld passes, where a higher heat input will likely coarsen the FZ solidification grains and HAZ equiaxed grains. A coarser grain size typically reduces the creep ductility of the weld samples and would likely be more SRC susceptible.
Figure A.1 Welding Procedure Specification (WPS) developed by Brahma Inc. used for hot tank seam/circumferential welds
Figure A.2 PQR_81 used for developing WPS (3/8" weld)
Figure A.3 PQR_82 used for developing WPS (1" weld)
APPENDIX B: NEUTRON DIFFRACTION EXPERIMENTS ON 2” THICK WELDS

Two bulk welds with 2” thickness, with and without PWHT (870°C/3 hours), were investigated for stress measurements using neutron diffraction methods at ORNL. There have been examples where neutron diffraction successfully measured stress in 2” thick 347H SS plates within the base metal [71]. Strain measurements along all three principal directions of a weld were performed to calculate residual stresses along LD (parallel to the welding direction), TD, and ND as seen in the setup used at ORNL (Figure B.1).

The elastic strain of a particular crystal plane can be calculated by the following expression:

$$\varepsilon_{311} = \frac{d_{311} - d_{0,311}}{d_{0,311}}$$  \hspace{1cm} (B.1)

where \(d_{0,311}\) represents the interplanar spacing between (311) Fe planes in the stress-free microstructure, while \(d_{311}\) represents the interplanar spacing measurements taken from the bulk welds. By determining \(\varepsilon_{311}\) for all three principal directions relative to the weld, the elastic residual strain can be calculated in all three principal directions for comparison to the FEM stress predictions. By knowing the elastic modulus (E) of 347H SS at room temperature (~200 GPa) and Poisson’s ratio (~0.28), the residual stress map can be calculated for all three principal directions. The stress formula for LD can be expressed by:

$$\sigma_{x,311} = \frac{E}{1+v} \left( \varepsilon_{x,311} + \left( \frac{v}{1-2v} \left( \varepsilon_{x,311} + \varepsilon_{y,311} + \varepsilon_{z,311} \right) \right) \right)$$  \hspace{1cm} (B.2)

The reference comb specimens were machined from an actual welded plate which provides the \(d_{0,211}\) distribution as a function of location within the welds. Three sets of reference samples were cut out of the first weld replicate in the middle of the 12-inch weld (31 pass, W.1) using electrical discharge machining (EDM), as shown in Figure B.2 and Figure B.3. The stress-free reference samples (comb shaped as shown in Figure B.2c) were designed with 4 mm x 4 mm x 4 mm thick cubes with a 3 mm wide comb handle. In addition to the comb specimens, three smaller cubes are extracted farther into the base metal to obtain the base metal interplanar spacing. Lines 1 and 3 are machined so that the measurement location is approximately 9 mm from the surface, while Line 2 lies directly in the mid-thickness of the specimen (as seen in Figure B.3). The length of the combs is dependent on the width of the fusion zone. Line 1 has the longest comb specimens, followed by Line 2 and 3.
The \( d_0 \)-spacing, determined using Bragg’s law based on the diffraction peak from Fe (311) planes, shows raw peak values in the weld metal (centerline with \( x=0 \)) in Figure B.4. The weld metal morphology, including grain size, affects the peak \( d_0 \)-spacing values and is proportional to the amount of retained \( \delta \)-ferrite that exists in E347 WM. Based on estimations from the cube locations seen in Figure B.3, each cube represents a location with one unique scan location to calculate one \( d_0 \) value with a volume of 4x4x4 mm\(^3\). The cubes were designed to be located specifically in one of three regions of the BM, HAZ, and WM, in which an average \( d_0 \) value was calculated for each region. Assuming isotropic properties, a single measurement from one orientation is used for all strain measurements. Only the as-welded condition is used for the reference samples for both analysis of the AW and PWHT bulk welds.

![Setup of 2” thick welds for HB-2B beamline at ORNL](image)

**Figure B.1** Setup of 2” thick welds for HB-2B beamline at ORNL
Figure B.2 A) 4 mm thick slice cut from bulk weld plate, b) 4mm thin sliver showing the relative 3 lines of interest, and C) comb shape assembly (x 2 combs) from each line

Figure B.3 A) weld cross section showing the three sets of stress-free comb shapes from the 31-pass weld with the center of the cubes being the center of the beam locations

Figure B.4 do-spacing of reference samples for all 3 lines as calculated from Bragg’s law based on the measured peak θ
The main limitations from measuring stresses on 2” thick welds are: 1) time it takes for the neutrons to penetrate the metal and collect in the detector, and 2) accuracy of each data point due to the large gauge volume needed (7.5 x 7.5 x 7.5 mm$^3$ for LD and 7.5 x 7.5 x 28 mm$^3$ for TD and ND). The LD measurements require four times the time needed to get a diffraction peak intensity comparable to the TD and ND. The accuracy with using larger gauge volumes decreases since the interaction of the neutrons with the material is over a larger volume. An example of a diffraction peak with a low signal-to-noise ratio for the TD direction is illustrated in Figure B.5. The LD and ND results are not shown here.

![Diffraction peak](image)

Figure B.5 Best case diffraction peak for TD showing low signal-to-noise ratios and fit to determine peak d-spacing value

Therefore, the results shown here are the d-spacing and corresponding strains for the TD direction only. The d-spacing values for Lines 1, 2, and 3 data (including ref (raw d$_0$ values), AW, and PWHT conditions) across the weld are displayed in Figure B.6, Figure B.7, and Figure B.8, respectively. Lines 2 and 3 have the most distinct differences between tensile and compressive values relative to the reference, as seen in Figure B.9. Line 2 (mid-thickness) indicates all compressive strains in the AW condition across the weld joint, while line 3 (near root) shows all tensile strains in the AW condition across the weld joint. Line 1 has a mixture of compressive and tensile strains but are generally close to zero for the AW condition. These results are very comparable to the results in the BM/HAZ regarding a 2” thick double v-joint, where the inner diameter had the highest axial tensile strains with the mid-thickness regions having compressive strains [41, 71]. The PWHT results have less transverse strain gradients through the thickness of the weld compared to the AW condition. Most of the WM-PWHT
results have compressive or near zero strains in all three lines of data, while the BM contains some tensile TD strain in the PWHT condition.

Figure B.6 d-spacing measurements of TD orientation (311 planes) for Line 1- top of weld

Figure B.7 d-spacing measurements of TD orientation (311 planes) for Line 2-mid thickness
Figure B.8 d-spacing measurements of TD orientation (311 planes) for Line 3-root

Figure B.9 Calculated $\epsilon_{311}$, TD for AW and PWHT conditions for all three lines of data. The reference $d_o$ values are averaged for three regions each in WM, HAZ, and BM, with each line representing unique values dependent on values from Figure B.4
Table C.1 contains the list of figures that have received a publication permission. The permissions were granted through Copyright Clearance Center (CCC) or through email approval. The permissions are submitted as supplemental files with the thesis in ProQuest.

Table C.1 Publication permissions given for figures in text from external sources (CCC-Copyright Clearance Center) and associated supplemental file names submitted in ProQuest

<table>
<thead>
<tr>
<th>Figure #</th>
<th>Type of Permission</th>
<th>Supplemental File Name</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.1</td>
<td>Email</td>
<td>Email Permissions-Figure 1.1 (NREL), Figure 2.23 (XCEL) and Figure 2.27 (EPRI)</td>
</tr>
<tr>
<td>2.7</td>
<td>CCC/RightsLink</td>
<td>Figure 2.7 permission_Springer_Metallurgical and Materials Transactions A</td>
</tr>
<tr>
<td>2.10</td>
<td>CCC/RightsLink</td>
<td>Figure 2.10 and 2.11 permission_Elsevier_Material Science and Engineering A</td>
</tr>
<tr>
<td>2.11</td>
<td>CCC/RightsLink</td>
<td>Figures 2.12 and 2.13 permission_Materials Science Forum</td>
</tr>
<tr>
<td>2.12</td>
<td>CCC/RightsLink</td>
<td>Figure 2.15 and 2.18 permission_Taylor and Francis_publication permission</td>
</tr>
<tr>
<td>2.13</td>
<td>CCC/RightsLink</td>
<td>Figure 2.22 permission_John Wiley and Sons_Weldability of Stainless steel_Lippold</td>
</tr>
<tr>
<td>2.22</td>
<td>CCC/RightsLink</td>
<td>Figure 2.24 and 2.36 permissions_Corrosion</td>
</tr>
<tr>
<td>2.23</td>
<td>Email</td>
<td>Email Permissions-Figure 1.1 (NREL), Figure 2.23 (XCEL) and Figure 2.27 (EPRI)</td>
</tr>
<tr>
<td>2.24</td>
<td>CCC/RightsLink</td>
<td>Figure 2.24 and 2.36 permissions_Corrosion</td>
</tr>
<tr>
<td>2.26</td>
<td>CCC/RightsLink</td>
<td>Figure 2.26 permission_Taylor and Francis_publication permission</td>
</tr>
<tr>
<td>2.27</td>
<td>Email</td>
<td>Email Permissions-Figure 1.1 (NREL), Figure 2.23 (XCEL) and Figure 2.27 (EPRI)</td>
</tr>
<tr>
<td>2.28</td>
<td>CCC/RightsLink</td>
<td>Figure 2.28 permission_Elsevier_Engineering Failure Analysis</td>
</tr>
<tr>
<td>2.36</td>
<td>CCC/RightsLink</td>
<td>Figure 2.24 and 2.36 permissions_Corrosion</td>
</tr>
<tr>
<td>5.35</td>
<td>CCC/RightsLink</td>
<td>Figure 5.35 permissions_combined_Springer</td>
</tr>
</tbody>
</table>