

**STRESS RELAXATION CRACKING IN 347H AUSTENITIC STEEL
WELDMENTS UNDER VARIOUS HEAT TREATMENTS: EXPERIMENTS AND
MODELING**

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ABSTRACT

347H austenitic stainless steel exhibits exceptional creep and corrosion resistance, rendering it an exemplary candidate for pipeline materials, particularly in mid- to high-temperature working conditions. However, due to constraints in component dimensions, welding has been chosen as the preferred method for joining pipeline systems extensively employed in nuclear power plants, fossil fuel plants, and petrochemical companies. The welding process entails the accumulation of residual stress during the cooling stage, along with the introduction of microstructure evolution. Moreover, the residual stress field and microstructure continuously evolve under service conditions, thereby intensifying the susceptibility of crack initiation and propagation. The initial residual stress attains its maximum value but subsequently undergoes relaxation, while the microstructure gradient and uneven distribution of residual stress give rise to stress concentration, ultimately promoting premature failure known as stress relaxation cracking (SRC).

Prior investigations pertaining to the SRC issue have typically separate simulation and experimentation, leading to unreliable predictions. Concurrently, the lack of appropriate methods to integrate material thermo-mechanical properties into simulation models exacerbates the inaccuracies in predicting failure as the service time increases. Additionally, SRC tests necessitate the application of external loads to accelerate the testing process, thereby introducing additional uncertainties into the experiments and widening the disparity between simulation predictions and the actual failure time. Therefore, this report introduces a methodology that combines both simulation and experimentation to forecast the lifespan of 347H weldments, whereby the simulation model and experimental measurements can mutually corroborate each other.

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CHAPTER ONE

INTRODUCTION AND GENERAL INFORMATION

1.1 Introduction of SRC

The pipeline system plays a crucial role in energy generation industries, including petrochemical, nuclear power plants, and fossil fuel plants, and has been in use for several decades. Apart from serving as a means of material transportation, the pipeline systems also provide the necessary working conditions, such as pressure, heat, and protective gas. To meet the temperature requirements of these service conditions, 347H austenitic stainless steel has been extensively employed in pipeline systems for these industries. This choice is made not only to its exceptional resistance to creep and corrosion but also to its cost-effectiveness compared to Ni-base superalloys. However, despite these advantages, unexpected cracks have been observed during recent field inspections. These cracks, occurring along the welding direction, are classified as Type IV failures in weldments. Stress Relaxation Cracking (SRC), a mechanism associated with Type IV failures, can occur during service, post-weld heat treatment (PWHT), and particularly during the welding process.

The welding process establishes the fundamental composition of the weldment. For example, the microstructure distribution gradient is determined after the dissipation of the heat source, and the residual stress field adopts a unique distribution based on the shape of the heat source and the welding material. During welding, the heat input dictates the shape of the molten pool, which in turn influences the size of the fusion zone (FZ) and the dimensions of the heat-affected zone (HAZ). The HAZ and FZ have a significant impact on crack initiation and propagation. The specific microstructure after welding is highly influenced by the material composition. For instance, in 347H, a small number of precipitates may coexist with the austenitic matrix, while in P91 material, intermetallic phases appear after welding, providing additional sites for microcrack formation. Microstructural variations and heat input jointly influence the distribution of residual stress fields. Particularly, during PWHT, high-temperature thermal treatment amplifies the differences in microstructure gradients and redistributes the residual stress field. As

components enter the service stage, thermal aging effects continue to contribute to microstructural changes. In the case of 347H austenitic stainless steel, specific types of precipitates emerge after a certain duration of treatment, which is a time-temperature dependent process. Meanwhile, the residual stress field gradually relaxes, but the rate of relaxation is contingent upon temperature and the level of external load [1].

1.1.1 Previous Research on SRC Problems

Premature failure in pipeline weldments, such as those made of 347H steel, can result in catastrophic accidents. Therefore, it has become imperative to investigate methods for predicting the lifetime and failure location of these weldments. Moreover, the ultimate goal is to provide guidance on how to extend their lifetime. Numerous studies have been conducted to address the issue of stress relaxation cracking (SRC), including the specific problem of SRC in 347H steel. Typically, two approaches have been employed: 1) utilizing simulation models to predict lifetime and 2) conducting experiments (often involving creep/SRC tests) to establish a connection between service conditions and lifetime. However, each method has its own advantages and disadvantages. Meanwhile verification of results from these methods is either lacking or not sufficiently convincing. In the case of simulation models, several factors are often overlooked during their development. One such factor is the evolution of the microstructure. While some models can adjust the material's thermomechanical properties based on specific working conditions, they fail to consider the evolution of precipitates and phase transformations. Neglecting these phenomena can lead to an overestimation of the lifetime, as they weaken creep resistance and accelerate failure. On the other hand, experimental methods like creep tests or SRC tests also have their limitations. For instance, creep tests require a long duration to mimic the actual failure process, which can be impractical in certain scenarios. Similarly, SRC tests often apply external loads higher than the existing residual stress levels in the service components, resulting in premature failure. Therefore, it is crucial to address these limitations and seek more robust approaches that can accurately predict the lifetime and failure location of 347H pipeline weldments. Additionally, efforts should be made to

overcome the drawbacks of existing methods to ensure reliable and comprehensive assessments [2].

1.2 Hypothesis of SRC Problem Solution Method

Consequently, combining simulation models with experimental data can enhance the credibility of predictions and provide mutual verification. In this study, we propose a methodology that integrates both approaches and incorporates input from each other. As shown in Figure 1.1, the research flowchart indicates the relationship and connections between each section. In order to build reliable simulation model, the experimental data used as input for the simulation model parameters fitting. Meanwhile, obtained simulation predictions will be verified through advanced diffraction technologies and limited industrial service data. For neutron and synchrotron measurement, they are partially used as input information for the residual stress field distribution and variation, then, will be the key experiments to verify the simulation model calculated residual stress value and precipitation information. And failure mechanisms are gathered from previous research used as input information in this project. With the prepared key input information, several simulation models were built.

1.2.1 Purpose of Simulation Models

In order to better mimic the real failure process from the beginning of the component making procedure, the simulation is divided into three parts. The first step of the simulation aims to replicate the welding process, encompassing the heating and cooling stages. During the heating step, the shape of the melting pool determines the dimensions of the HAZ and FZ, as well as the microstructure within these regions. Transitioning to the cooling step, residual stresses gradually accumulate. By simulating this process, the temperature field can be obtained, which serves as input for the second step: thermo-mechanical analysis. In the second step, the residual stresses generated by the welding process are calculated. Additionally, the relaxation of residual stresses caused by post-weld heat treatment (PWHT) at different temperatures is also evaluated. Utilizing the calculated residual stress field, the final step involves predicting the lifetime using a two-dimensional model. The first two steps employ a three-dimensional model to generate the complete

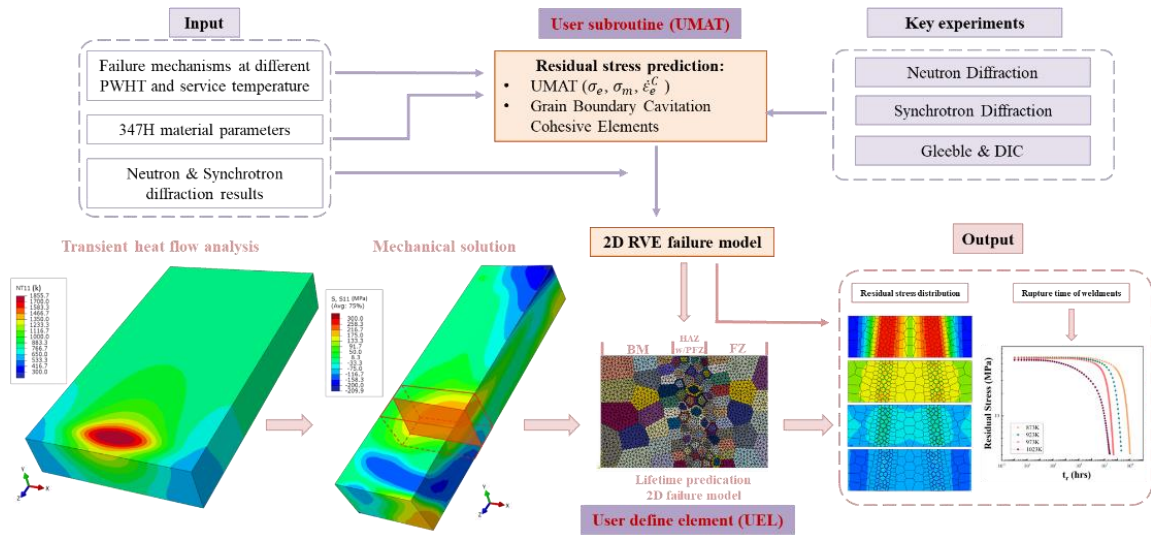


Figure 1.1. Research flowchart.

temperature and residual stress fields, allowing the extraction of random cross-section residual stress distributions for application in the final step. The choice of using a two-dimensional model in the final step instead of a three-dimensional model is based on the focus being on the failure time after applying the initial residual stress. Implementing a three-dimensional model to simulate the failure process would exponentially increase computational time. Hence, the utilization of a two-dimensional Representative Volume Element (RVE) model not only provides reliable predictions but also significantly reduces computational time compared to a three-dimensional setup [3-7,11]. The simulation models setup and results analysis will be discussed in Chapter three.

1.2.2 Neutron Diffraction Experiment

The above paragraphs are a brief summary of the flowchart, here only list three key experiments. However, enormous content includes in the experimental site. And each of them has their own purpose. In the meantime, they can combine to support certain parts of the simulation results. The following part provides an overview of each experiment and its specific purpose.

The neutron diffraction experiment aims to measure the existence of residual stress fields in laboratory-made welds and actual service pipes. This project's neutron residual stress measurement has a unique feature, which is a sequence measurement of thermally treated samples. In this case, the calculation of residual stress reduction level caused by thermal treatment can be obtained. Additionally, the experiment includes measuring thick/thin pipes that have been in service for 5 years and 30 years, which has rarely been reported. Two laboratory-made 347H welds are prepared under identical conditions following the same welding procedure. One of the welds undergoes PWHT according to the process used at Shell, where the sample is heated to 538°C for 1 hour and then increased to 900°C for 4 hours. Neutron measurements are performed on these two plates to generate the residual stress field. The level of residual stress reduction resulting from PWHT can be determined. Subsequently, after removing the plates from the neutron beamline, long-term thermal treatment is applied at 650°C for 361.5 hours to simulate the service stage. This allows for the evaluation of the effects brought by short-term thermal treatment (PWHT)

and long-term thermal treatment (thermal aging). The significant aspect of this neutron measurement is that the obtained results can verify the fidelity of the simulation model and help adjust the distribution of residual stress which will be explained in detail in Chapter two.

1.2.3 Microstructure Characterization

The utilization of atom probe tomography (APT) experiments is crucial for accurately characterizing the precipitation types and distributions in the heat-affected zone (HAZ) and fusion zone (FZ) at the micro to nano level. This is particularly important due to the occurrence of Type IV failure, which primarily takes place in the HAZ. APT has been employed in this project as the most effective method for revealing the morphology of precipitates and their precise locations which will be discussed in detailed in Chapter four. In comparison to scanning electron microscopy (SEM), APT surpasses the nano-scale level, enabling the identification of particle composition, element enrichment, grain boundaries, and their relative distributions. While transmission electron microscopy (TEM) also provides information on element distribution and the relationship with grain boundaries and particle types, the two-dimensional nature of TEM data is insufficient to fully capture the surrounding environment. APT serves as the most efficient approach to comprehensively characterize the multiple types of precipitation in 347H weldments, particularly the nano-sized NbC precipitates. Therefore, APT is employed in conjunction with SEM and TEM characterization methods [8-13]. The use of scanning electron microscopy (SEM), transmission electron microscopy (TEM), and energy-dispersive X-ray spectroscopy (EDS) is essential in studying 347H, a precipitation-strengthened material, due to the sensitivity of certain precipitates (such as nano-sized NbC, G-phase, M₂₃C₆ (carbides, M stands for metallic elements), and laves phase) to temperature and thermal treatment time. Unless the working environment meets the specific requirements of these precipitates, or unless the microstructure evolution involving these precipitates is excluded, SEM and EDS serve as widely utilized tools to illustrate microstructural morphology and elemental distribution. These techniques effectively operate at the micro-scale level. However, some precipitates exist at the nano-scale level, where SEM and EDS

are insufficient to fully characterize variations occurring at that scale. The SEM & TEM related micro-scale characterization will be listed in Chapter six.

1.2.4 Synchrotron Diffraction Experiment

The synchrotron diffraction experiment was employed to gather information regarding precipitates, including their types, phase transformations, and residual stress in sheet samples. The evolution of precipitation significantly affects the redistribution of residual stress and variations in the thermo-mechanical properties of materials, which in turn determine their lifetime. Therefore, capturing the relationship between precipitation evolution, temperature, and thermal treatment time is a crucial factor for ensuring accurate lifetime predictions. In this project, both in-situ and ex-situ synchrotron diffraction experiments were conducted to acquire in-situ information on precipitation evolution and observe variations in precipitation fraction and types resulting from long-term thermal treatment. Additionally, residual stress calculations were performed using these data. However, it should be noted that the synchrotron diffraction experiment has limitations regarding sample thickness, which means that using the measurement results to calculate residual stress may not accurately represent the actual residual stress value in the service component. The experiment setup, samples information and further data analysis will be in chapter five.

1.2.5 Material Thermomechanical Properties

The material thermomechanical properties which will be utilized in simulation model parameters fitting and further verification steps will be measured through Gleeble, DIC and tensile tests. The Gleeble thermo-mechanical testing experiment is employed to assess variations in thermomechanical properties and strain hardening rates resulting from thermal and external loading influences. Some researchers utilize the Gleeble system to simulate welding processes, taking advantage of its fast heating capability and the ability to apply boundary conditions. This allows for the recording of temperature history and the generation of the heat-affected zone (HAZ) region, enabling further characterization of the microstructure. Various mechanical properties of the HAZ metal are tested using the Gleeble system, such as yield strength, ultimate tensile strength, and creep rate at different

temperatures. Defining these properties is essential as material properties generally degrade at high temperatures. Using the same values at room temperature would introduce inaccuracies in simulation models. Another crucial factor that can be identified through Gleeble testing is the strain hardening effect. Localized temperature changes, such as those occurring during welding processes involving localized melting, result in degraded stress-strain behavior in the heated region compared to the unaffected region. Neglecting this factor would increase deviations in simulation predictions. The universal tensile test is employed in this study to determine the general mechanical properties of 347H austenitic stainless steel. Although the nominal composition of the steel is 347H, the actual material used in each case may exhibit slight variations in the fraction of constituent elements, leading to fluctuations in mechanical properties. Measuring the stress-strain curve enables the determination of the stress and strain range, which aids in better defining other measurement results within the fluctuation range or accounting for variations caused by factors such as phase transformations, concentration of microcracks, and precipitation strengthening. This part will be discussed in Chapter seven.

CHAPTER TWO

RESIDUAL STRESS MEASUREMENT THROUGH NEUTRON DIFFRACTION AND ITS EFFECT ON SRC

2.1 Feature of the Neutron Diffraction Measurement of Residual Stress in this work:

Residual stress plays a crucial role in stress relaxation cracking (SRC) process, as acknowledged in the literature. Traditionally, creep tests have been conducted to simulate the failure process and predict the lifetime of materials; however, this approach can be time-consuming and over-estimation. An alternative way is to determine the residual stress distribution and its value which is a significant factor in the failure process. Although evaluating lifetime based on residual stress values is not a straightforward technique, it is more reliable than other methods. Additionally, verification can be performed at various stages and different methods. For example, the lab-made welds at different thermal treatment conditions can be compared with the corresponding simulation results; the residual stress relaxation level can be compared in different shape samples. The novelty of this research lies in the sequence thermal treatment performed on the sample, allowing for the acquisition of residual stress evolution information. Meanwhile, the actual service components; 5-year service 32mm thick pipe and 30-year service 8mm thin pipe has been measured for residual stress field in the girth weld region, which is a pioneer work in the welding and material science field. This study includes several unique aspects, which may provide deeper insights [36-39].

2.1.1 Residual stress information in all three primary (principle) directions.

The sample collection for this research includes lab-made 1-inch-thick plates, 8mm-thin service pipe (with tee support), 1-inch-thick service pipe, and corresponding d_0 comb reference samples that can match each case. These samples are representative of the three most common cases encountered in practice for the residual stress measurement: actual serviced pipes (either thin-wall or thick-wall), laboratory-made welds capable of undergoing a range of thermal treatments, and reference samples for the absolute residual stress calculation. A key attribute of these samples is that residual stresses in all three

primary directions have been measured, enabling the calculation of residual stress using the d-spacing results from each of the three directions.

2.1.2 Sequence thermal treatment performed on the lab-made plates.

The evolution of the residual stress field is a critical determinant of stress relaxation cracking. Prior research has scarcely explored the impact of thermal treatment on residual stress evolution, and only a few studies have reported results under specific conditions. In contrast, our investigation, which involved lab-made thick plate samples and a collaborative research neutron proposal, enabled us to examine the effects of short-term and long-term thermal treatment contribution to the residual stress evolution. We began by studying the two most common cases: As-Welded and PWHT (post weld heat treatment) conditions. By comparing the results from these two sets of samples, we could assess the degree of residual stress relaxation induced by the PWHT procedure. Then we subjected both the plates and corresponding d_0 comb samples to relatively long-term thermal treatment at 650°C for 361.5 hours to simulate service conditions. By analyzing the residual stress values of the plate samples after they had undergone service conditions, we were able to obtain insights into the impact of short/long-term thermal treatment on residual stress evolution.

2.1.3 Residual stress reduction level can be determined.

In addition to comparing the residual stress values between two different states, calculating the reduction percentage of residual stress between different statuses, and comparing it with simulation results can also serve as an effective evaluation factor. For instance, using the residual stress value at a particular stage to determine the accuracy of a simulation model may not be reliable as it only contains a few data points that are insufficient to support the reliability of the entire curve. However, by calculating the residual stress reduction percentage between two stages in a single thermal treatment condition, it is possible to establish a relationship between the reduction percentage and the thermal treatment temperature. Subsequently, further predictions can be made along this thermal treatment condition with a reliable base reduction rate.

2.1.4 Cross validation though two different methods

The neutron diffraction method is currently considered one of the most reliable techniques for measuring residual stress, as it ensures the accuracy of both the measured residual stress value and its distribution. However, despite its reliability, experimental errors, such as human errors, induced errors caused by the preparation procedure (e.g., machinery-induced residual stress field changes, microstructure variation caused by cutting), and other factors, can lead to uncertainties in the results. To mitigate these uncertainties, we also adopted the plane stress assumption, which excludes the d_0 value from the calculation. By doing so, one of the uncertainty factors can be controlled. The two methods were then compared to determine the degree of variation between them [40-43].

2.2 Experiments setup and materials

2.2.1 Material

The material investigated in this study was 347H austenitic stainless steel. The pipe sample was obtained from Shell Global Solution and the lab-made welds were using commercial procurement of 347H stainless steel to make the weldments. Two service pipe samples with different wall-thickness, one is an 8mm thin wall 30-year service pipe as shown in Figure 2.1; another is a 1in' wall thickness pipe experienced 5-year service as shown in Figure 2.2. For both pipes, the exact service conditions and chemical composition still needs to be determined. The thin pipe was connected to a tee support, and a crack had developed in the weld zone of the tee support, while the girth weld zone remained its integrity. The dimensions of the pipe allowed for measurements to be taken in the axial and radial directions, but slots were required for penetration in the hoop direction. To ensure measurements were taken at the same location as the other two directions, the neutron beam needed to penetrate the wall thickness to reach the specific location. However, due to the neutron-absorbing properties of the 8mm-thick 347H stainless steel material, the resolution and intensity of the data acquired at the specific location could not be guaranteed. Therefore, a slot was cut 90° from the actual measurement location, covering the base metal (BM), heat-affected zone (HAZ), and fusion zone (FZ) with a

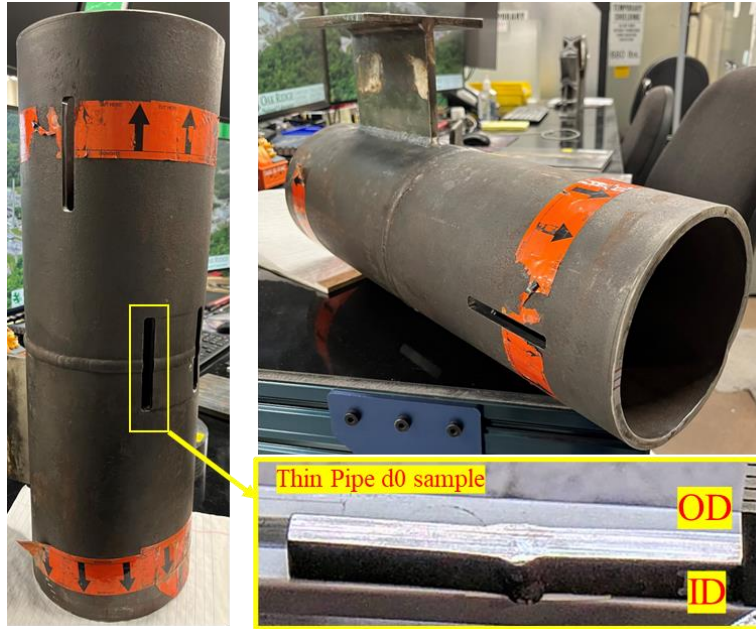


Figure 2.1. 30-year service 8mm wall thickness thin 347H pipe; yellow frame figure is the d₀ comb sample cut from the pipe.

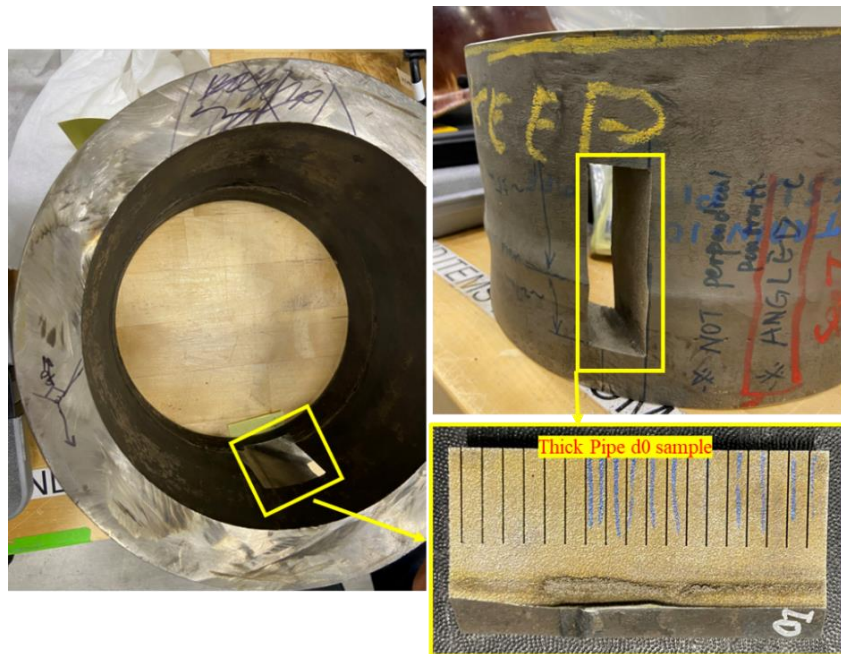


Figure 2.2. 5-year service 32mm (1 in') wall thickness thick 347H pipe; yellow frame figure is the d₀ comb sample cut from the pipe.

range of -60mm to 60mm from the weld centerline. Milling was performed at the slot location, as electrical discharge machining was not possible to perform at that specific location. The d_0 comb slot was cut after the first measurement, which can ensure the minimum damage to the residual stress field. The thin pipe, the d_0 comb and its slot location as shown in Figure 2.1.

With a similar strategy, a slot was also cut from the thick pipe. The difference is the cutting slot in the thick pipe is not perpendicular to the girth weld. Due to the thickness level in this pipe, an angled slot was cut from the pipe as shown in the Figure 2.2. The incident beam and diffracted beam both have a 45° relationship with the measuring direction. If a perpendicular slot was cut from the pipe which will cause the beam to be blocked due to the penetration requirement. Hence, this angled cutting strategy can allow the incident beam to reach the targeting location without any impede. Meanwhile, the d_0 comb was also measured to calculate the absolute residual stress in the thick pipe.

The two lab-made welds were produced by the material joining group at the Material Science and Technology Division (MSTD) of Oak Ridge National Laboratory (ORNL), using gas tungsten arc welding (GTAW) processes of both manual and machine types. A total of 32 passes were used to join two plates together, resulting in weldments with dimensions of approximately 12.25 inches by 12.25 inches by 1 inch. The clamping method used is illustrated in Figure 2.3. In order to calculate the reduction in residual stress through neutron diffraction measurements, the two plates were subjected to different thermal treatment conditions. One was left in the As-Welded condition, while the other underwent PWHT (post-weld heat treatment). The PWHT process involved heating the plate to 811K (1000°F/538°C) and holding it at that temperature for one hour, before raising the temperature to 1172K (1650°F/900°C) and holding it for an additional four hours, before finally being removed from the furnace. This PWHT process was designed to simulate the actual PWHT procedure performed at Shell, with the aim of relieving the residual stress accumulated during the welding process, and its relaxation percentage during PWHT. The temperature and time profile during the PWHT process are depicted in Figure 2.4.

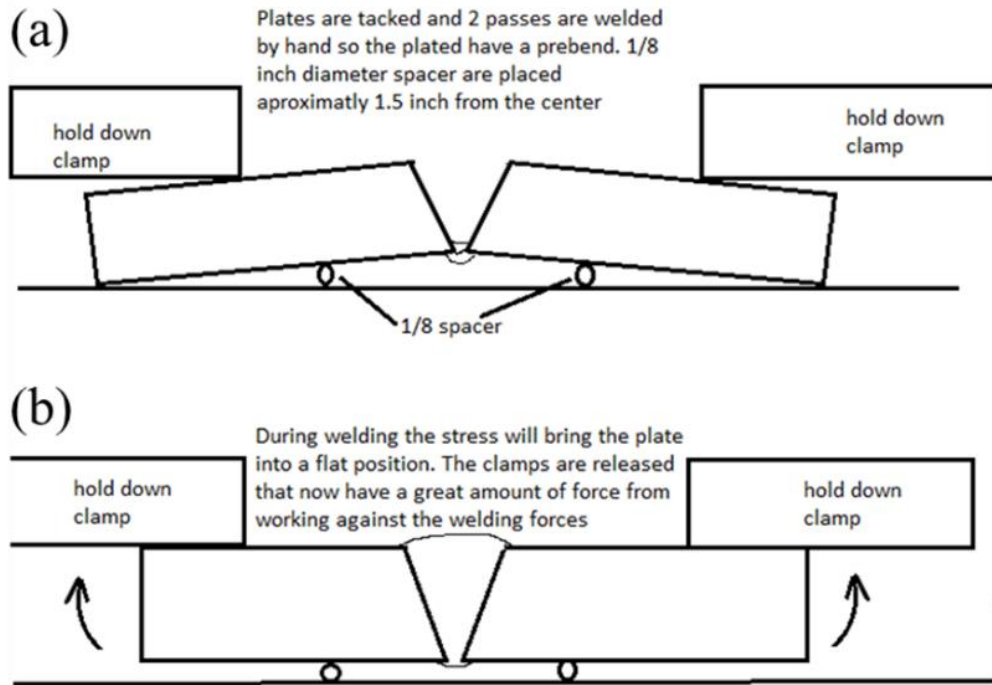


Figure 2.3. Lab-made 347H plate welds welding procedure details.

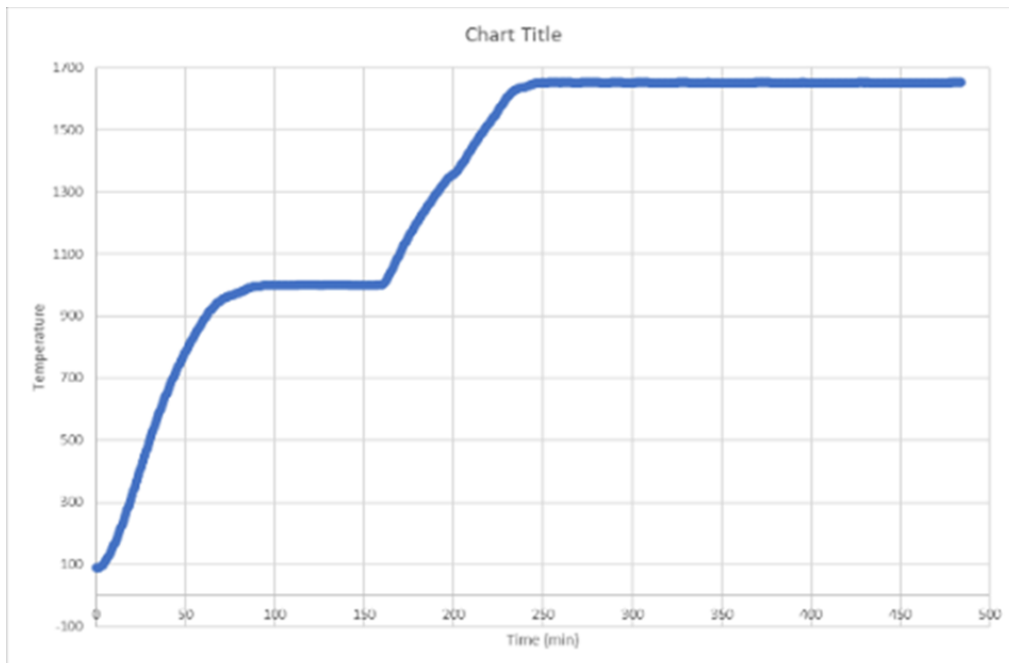


Figure 2.4. Lab-made 347H plate welds, the PWHT procedure time and temperature recording.

2.2.2 Experiment Setup

To generate the residual stress field, measurements need to be taken from all three directions, requiring the samples to be remounted and rotated each time. For plate samples, the cross-section in the mid-length of the plate is the target location for residual stress measurement. The scanning matrix, as shown in Figure 2.5, contains dense points in the HAZ and FZ and relatively loose points in the BM region. The scanning matrix covers five thickness levels: 2.2mm (close to plate bottom), 7.58mm, 12.95mm, 18.32mm, and 23.7mm (close to plate surface), (with the welding process start at the bottom and finish at the surface). This scanning matrix provides relatively reliable measurement results.

Figure 2.6 shows the As-Welded plate and PWHT plate, they both have the same dimensions of approximately 307.3mm * 309.9mm * 25.4mm (12.1in' * 12.2in' * 1in'), with a fusion zone width ranging from 20mm to 22mm. The scanning matrix can fully cover the weld plate FZ and HAZ. To calculate the absolute residual stresses in the lab-made plate welds, the d_0 comb sample is needed, especially for the weld sample. Due to significant microstructure changes that can lead to d-spacing and lattice parameter variation. Figure 2.7 shows the d_0 samples for As-Welded case and PWHT case. These two plates were measured during the first beam cycle, providing information on the As-Welded and PWHT plate. In order to calculate the absolute residual stress value, the d_0 comb are needed for each state's plate. As shown in Figure 2.7, are the d_0 comb samples for both As-Welded plate and PWHT plate. The comb teeth are in dimensions of 3mm * 3mm, which are along with longitudinal direction. Due to the d_0 sample have already machined into small comb teeth which can ensure the residual stress have been relaxed. The measured direction is along the transverse direction. By measuring the d-spacing in the d_0 sample, it can provide the d-spacing value under a stress free condition, and only microstrain/ caused by microstructure variation contains. Therefore, the absolute residual stress can be calculated by subtracting the measuring results from the plate and the results from the d_0 samples.

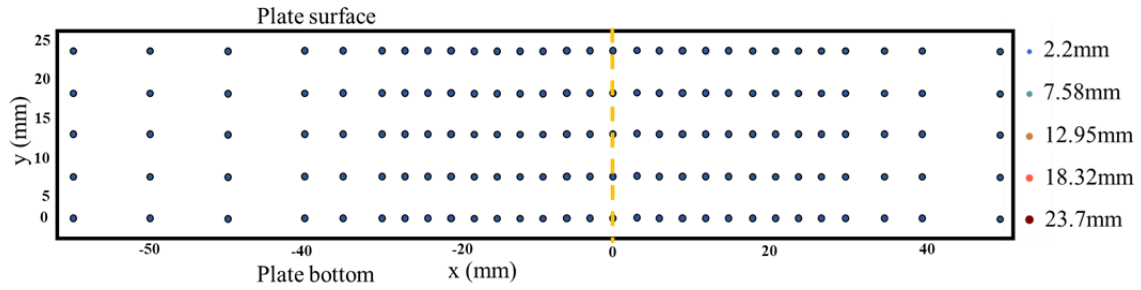


Figure 2.5. Scanning matrix for the lab-made thick plate welds. The yellow dash line marked the plate center, five scanning thickness level marked as the right side of the matrix, each has its corresponding color for clear distinction.

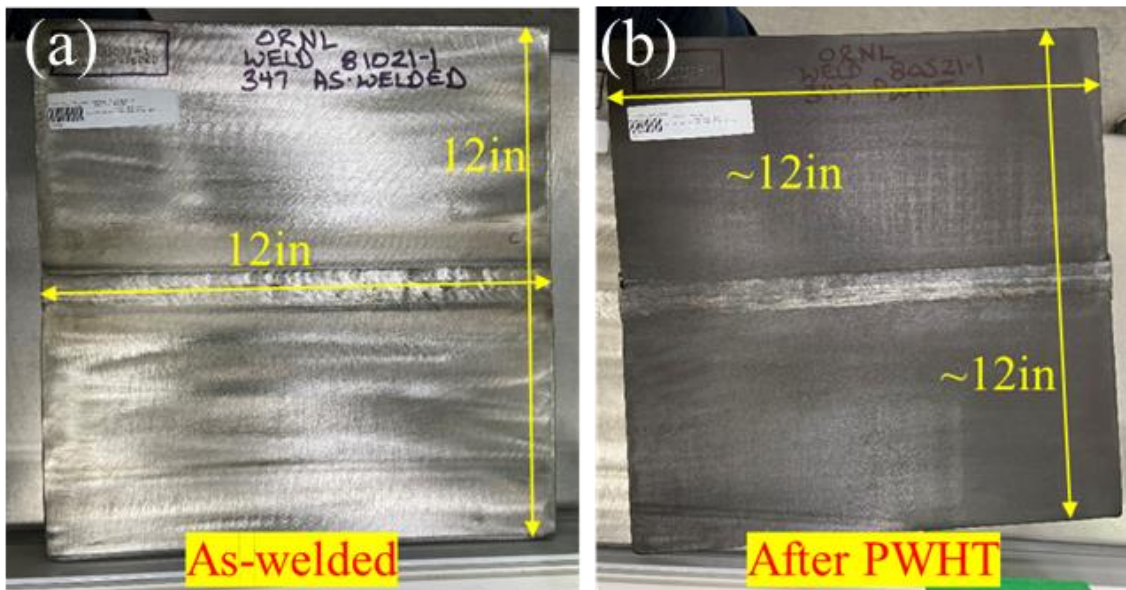


Figure 2.6. lab-made 347H 1-in thick plate. (a) is in As-Welded condition; (b) is in PWHT.

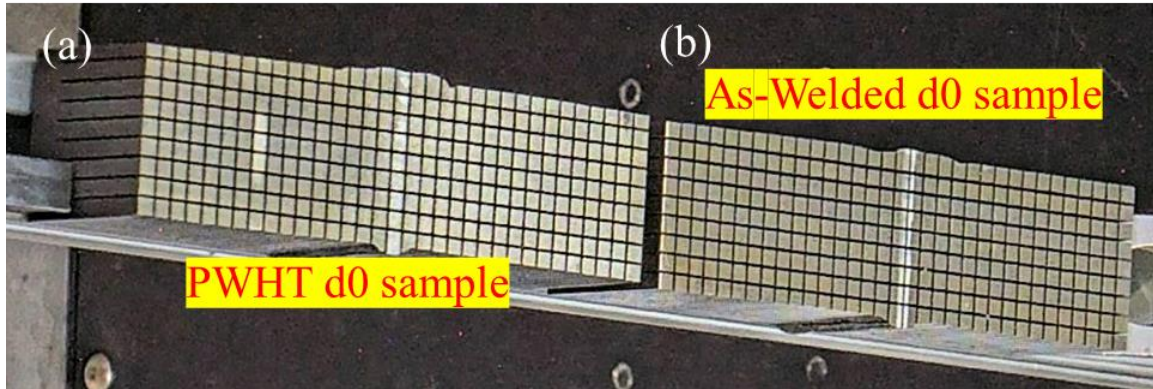


Figure 2.7. (a-b) PWHT d_0 comb sample; (c-d) As-Welded d_0 comb sample.

The aim of this research is to investigate the contribution of thermal treatment, so both plates experienced another long-term thermal treatment (thermal aging, shorted as TA) to provide more data points for the simulation results to fit with. The long-term thermal treatment involved thermal holding at 923K (650°C) for 361.5 hours. Both plates after the TA as shown in Figure 2.8, and their corresponding d_0 comb samples are shown in Figure 2.9. The thermal aging plates aim to further determine the thermal treatment effects on the residual stress reduction and compare with short term thermal treatment (PWHT).

Due to each case's plate and its corresponding d_0 comb samples were measured at different beam cycle, the error induced by beam, alignment, measurement environment, etc., can bring additional uncertainties which will further cause the calculation error. Hence, to take care of this issue, four cubes cut from the base metal region from each state also been measured during the last beam cycle with TA-As-Welded plate, TA-PWHT plate, As-Welded d_0 comb, PWHT d_0 comb and thin pipe d_0 comb. So, the adjustment can be made according to the cube sample's d-spacing value. The detail explanation shows below:

The As-Welded plate, PWHT plate, TA-As-Welded d_0 comb, TA-PWHT d_0 comb was measured before the last beam cycle and not scheduled in the same beam cycle. Using the cubes measured during the last beam cycle can reset the previous measured data set to the same range. For example, the As-Welded plate was measured in 2021 beam cycle; the As-Welded d_0 comb was measured in 2023 beam cycle with the four cubes. Using the As-Welded cube can compare with the As-Welded plate data set base metal region value and adjust the plate base metal region value in the same range as the As-Welded cube value (basically a fix value). Other state's sample can adjust accordingly. The reason and calculation detail to explain to verify this adjustment is reasonable will be presented in the following paragraphs.

The two pipe samples provided by Shell Global Solution (US) are real service pipes, one is a 30-year service thin pipe with a tee support at one end and the wall thickness is 8mm; another pipe sample is a 5-year service pipe with 32mm wall thickness. For the thin pipe sample, it consists of two welding zones: a girth weld connecting two pipes, and a tee weld supporting the pipe, as depicted in Figure 2.1. The location of the slots can also be seen in Figure 2.1. A visible crack is present in the region of the tee weld, indicating

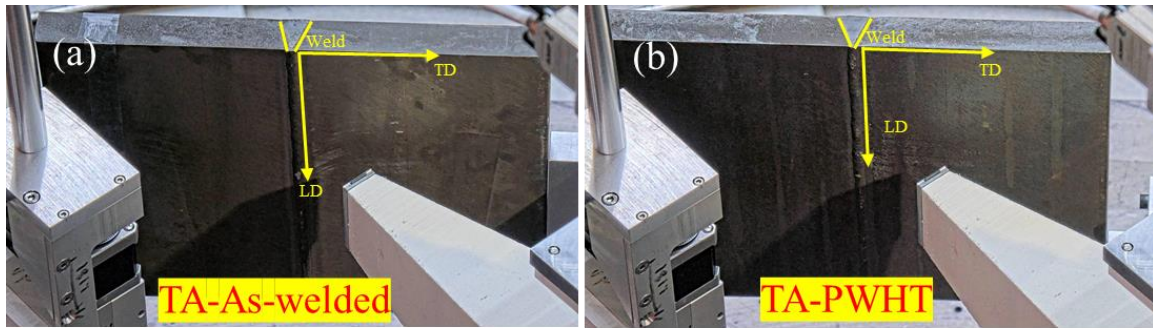


Figure 2.8. lab-made 347H 1-in thick plate. (a) is in TA-As-Welded condition; (b) is in TA-PWHT condition.

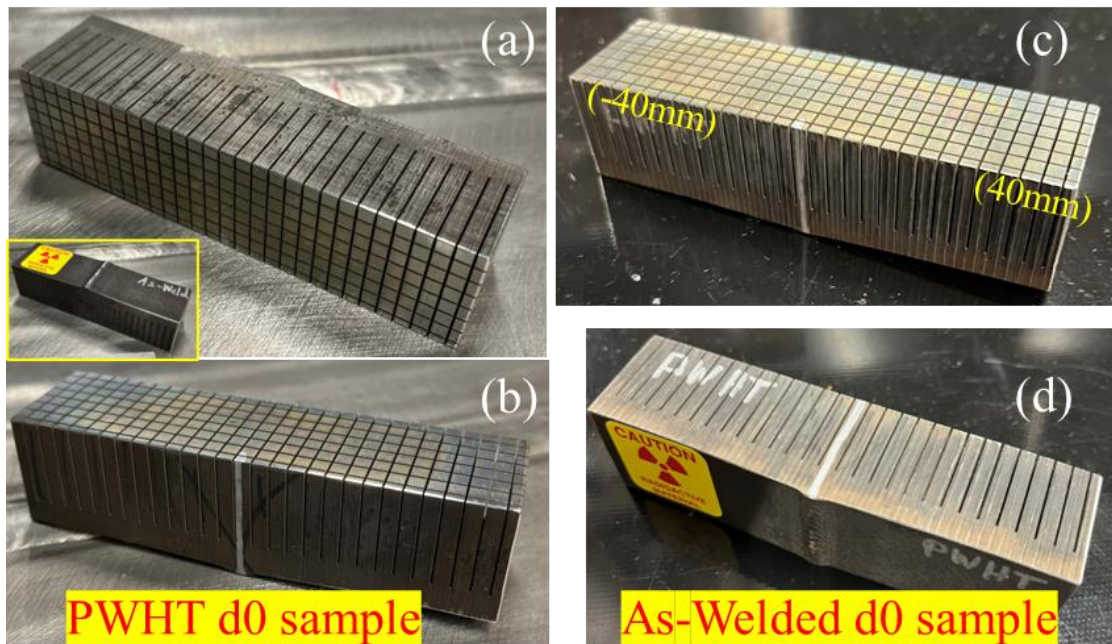


Figure 2.9. (a-b) TA-PWHT d_0 comb sample; (c-d) TA-As-Welded d_0 comb sample.

that the residual stress field there has relaxed. In contrast, the girth weld remains intact. Accordingly, by measuring the girth weld, it is possible to map the residual stress field in the girth weld region of the pipe. The scanning matrix used for measurement is illustrated in Figure 2.10 for the thin pipe, with the measuring area situated 90° from the tee support weld. Meanwhile, the scanning matrix for the thick pipe is shown in Figure 2.11. This choice of location allows the influence of the tee support weld crack to be ignored while also providing information about variations in the residual stress field when another welding zone is present nearby.

The measuring location is shown in Figure 2.12, encompassing a range of -60mm to 60mm from the girth weld centerline. Theoretical estimations suggest that residual stress will increase as the measuring location approaches the tee support (i.e., the side near 60mm). The incident beam and diffracted beam have an angle of 45 degrees with respect to the measuring direction, necessitating the plate to be rotated and flipped accordingly. However, for the d_0 comb sample, the d-spacing is the same in all three directions, thus requiring only one direction's measurement.

2.3 Results and Discussion

2.3.1 Lab-made plate welds results

2.3.1.1 Using actual d_0 comb sample to calculate the residual stress.

During two beam cycles, a total of four conditions of lab-made welds were measured. The As-Welded and PWHT conditions were measured during the 1st beam cycle which can provide the information of two base conditions. The second beam cycle measured two additional 1-year room temperature relaxation of the lab-made plate welds. The collected data consisted of d-spacing, FWHM, peak intensity, and other relevant parameters. The d-spacing dataset was used to calculate the residual strain and subsequent residual stress. However, since the samples were weldments, the d-spacing was highly position-sensitive. To obtain thermal-induced residual stress variation, it was necessary to exclude microstructure-induced stress. Therefore, in this report, the calculation of residual stress was based on the scanning points' location. The equations for calculating the residual stress are provided below:

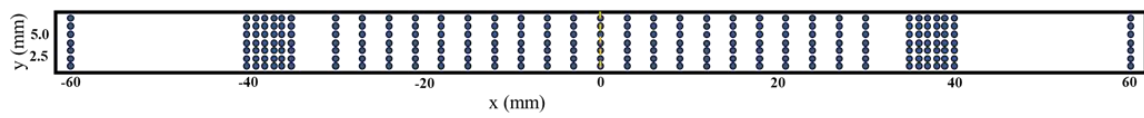


Figure 2.10. 8mm thin pipe scanning matrix.

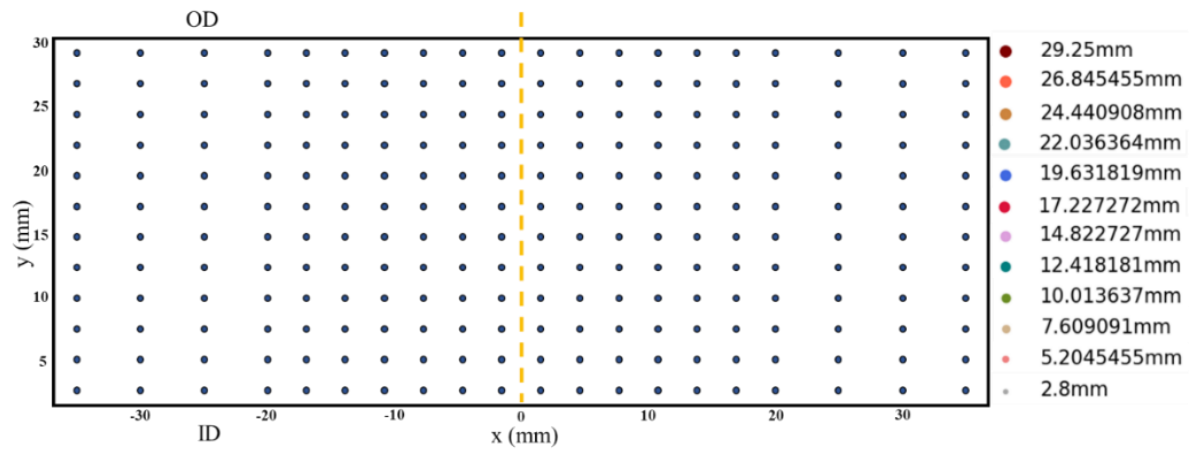


Figure 2.11. 32mm thick pipe scanning matrix.

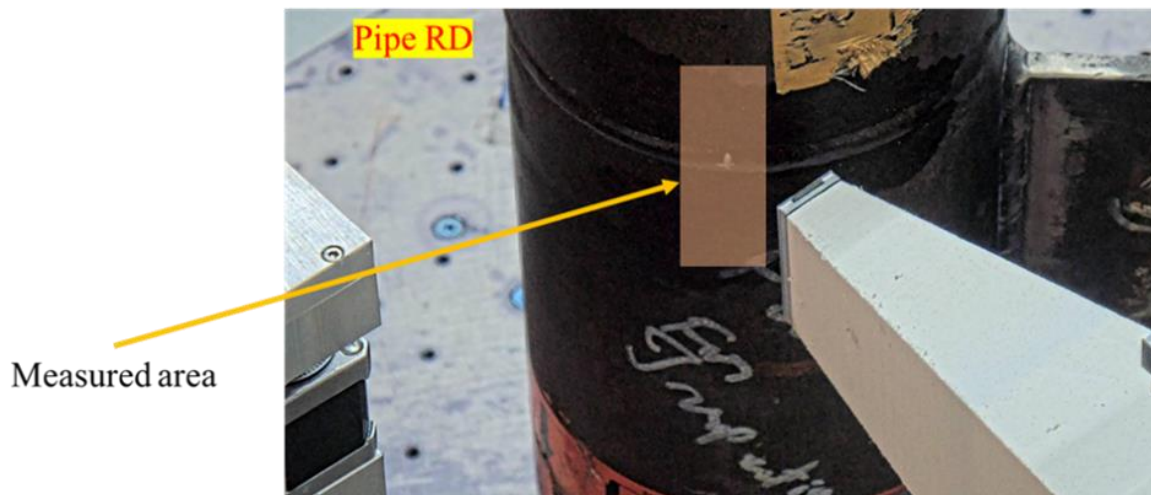


Figure 2.12. 8mm thin pipe scanning location schematic drawing on the actual pipe.

$$\varepsilon_{absolute} = \frac{d_{Plate}^{position\#} - d_{comb}^{position\#}}{d_{comb}^{position\#}}$$

$$\sigma_{ND} = \frac{E}{(1 + \nu)} \left[\varepsilon_{LD} + \frac{\nu}{(1 - 2\nu)} (\varepsilon_{TD} + \varepsilon_{ND} + \varepsilon_{LD}) \right]$$

$$\sigma_{ND} = \frac{E}{(1 + \nu)} \left[\varepsilon_{ND} + \frac{\nu}{(1 - 2\nu)} (\varepsilon_{TD} + \varepsilon_{ND} + \varepsilon_{LD}) \right]$$

$$\sigma_{TD} = \frac{E}{(1 + \nu)} \left[\varepsilon_{TD} + \frac{\nu}{(1 - 2\nu)} (\varepsilon_{TD} + \varepsilon_{ND} + \varepsilon_{LD}) \right]$$

The Young's modulus and Poisson's ratio for 347H austenitic stainless steel are: $E = 195\text{GPa}$, $\nu=0.27$

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

$$\sigma_{ii} = \frac{195}{(1 + 0.27)} \left[\varepsilon_{ii} + \frac{0.27}{(0.46)} (\varepsilon_{11} + \varepsilon_{22} + \varepsilon_{33}) \right]$$

$$\sigma_{ii} = \frac{195}{1.27} \left[\varepsilon_{ii} + 0.587 (\varepsilon_{11} + \varepsilon_{22} + \varepsilon_{33}) \right]$$

So, the method above summaries the equation will be used for the residual stress calculation based on the d_0 comb sample. Another method based on the Plane-Stress assumption is shown below.

2.3.1.2 Plane stress methodology

The plane-stress assumption is a widely used simplification technique that involves considering a two-dimensional state of stress, as mentioned in [5]. This condition is often applied in practice when one of the dimensions of a body is significantly smaller than the others. For instance, in a thin plate subjected to loading in the plane of the plate, there will be no stresses acting perpendicular to the plate. Although the plates in this study have a thickness of 1 inch, which is not ideally suited for the thin plate assumption, the normal residual stress at the near-surface level is assumed to satisfy the plane-stress condition. Furthermore, the plane-stress condition is applicable to the pipe samples, and thus, the results obtained from the plane-stress calculations have been included in this report to facilitate comparison with other methods. The relevant calculation equations are provided below.

According to the constitutive model:

$$\sigma_{xx} = \frac{E}{1 + \nu} \left[\varepsilon_{xx} + \frac{\nu}{1 - 2\nu} (\varepsilon_{xx} + \varepsilon_{yy} + \varepsilon_{zz}) \right]$$

$$\sigma_{yy} = \frac{E}{1+\nu} \left[\varepsilon_{yy} + \frac{\nu}{1-2\nu} (\varepsilon_{xx} + \varepsilon_{yy} + \varepsilon_{zz}) \right]$$

$$\sigma_{zz} = \frac{E}{1+\nu} \left[\varepsilon_{zz} + \frac{\nu}{1-2\nu} (\varepsilon_{xx} + \varepsilon_{yy} + \varepsilon_{zz}) \right]$$

For the plane stress case, assume $\sigma_{zz} = 0$,

$$\sigma_{zz} = \frac{E}{1+\nu} \left[\varepsilon_{zz} + \frac{\nu}{1-2\nu} (\varepsilon_{xx} + \varepsilon_{yy} + \varepsilon_{zz}) \right] = 0$$

$$\frac{1-2\nu+\nu}{1-2\nu} \varepsilon_{zz} + \frac{\nu}{1-2\nu} (\varepsilon_{xx} + \varepsilon_{yy}) = 0$$

$$\frac{1-\nu}{1-2\nu} \varepsilon_{zz} + \frac{\nu}{1-2\nu} (\varepsilon_{xx} + \varepsilon_{yy}) = 0$$

$$\varepsilon_{zz} = \frac{1-2\nu}{1-\nu} \frac{-\nu}{1-2\nu} (\varepsilon_{xx} + \varepsilon_{yy})$$

$$\varepsilon_{zz} = \frac{-\nu}{1-\nu} (\varepsilon_{xx} + \varepsilon_{yy}) \quad (1)$$

$$\varepsilon_{zz} = \varepsilon_{ND} \quad (2)$$

Substitute (1) into (2) gives (3)

$$\varepsilon_{xx} = \varepsilon_{LD}$$

$$\varepsilon_{yy} = \varepsilon_{TD}$$

$$\varepsilon_{ND} = \frac{d_{ND}-d_0}{d_0} = \frac{-\nu}{1-\nu} (\varepsilon_{LD} + \varepsilon_{TD}) \quad (3)$$

$$\varepsilon_{LD} = \frac{d_{LD}-d_0}{d_0} \quad (4)$$

$$\varepsilon_{TD} = \frac{d_{TD}-d_0}{d_0} \quad (5)$$

$$\frac{d_{ND}-d_0}{d_0} = \frac{-\nu}{1-\nu} (\varepsilon_{LD} + \varepsilon_{TD}) \quad (6)$$

Insert (4), (5) into (6)

$$\frac{d_{ND}-d_0}{d_0} = \frac{-\nu}{1-\nu} \left(\frac{d_{LD}-d_0}{d_0} + \frac{d_{TD}-d_0}{d_0} \right) \quad (7)$$

$$d_0 = \frac{\nu}{1+\nu} (d_{TD} + d_{LD}) + \frac{1-\nu}{1+\nu} d_{ND}$$

If $\nu = 0.27$, then

$$d_0 = \frac{27}{127} (d_{TD} + d_{LD}) + \frac{73}{127} d_{ND} \approx 0.213 * (d_{TD} + d_{LD}) + 0.575 * d_{ND} \quad (8)$$

2.3.2 Results of As-Welded plate

Figure 9 displays the residual stress distribution in the As-Welded plate using the actual d_0 distribution to calculate the residual stress. The two-dimensional image provides a clear comparison of the different thickness levels. The highest tensile residual stress is observed at the near-surface level, located at a distance of 23.7 mm from the plate bottom. The highest longitudinal residual stress occurs at a location 18 mm away from the weld centerline, with a value of 653 MPa. The residual stress gradually decreases towards the plate bottom, with the smallest average residual stress observed at the level closest to the plate bottom, which is 2.2 mm away. The longitudinal residual stress variation at the 2.2 mm level has an average value of ~100 MPa, with a noticeable decrease in stress as shown in Figure 2.13. The red dotted line represents the scanning line at a distance of 23.7 mm from the plate bottom, while the blue dotted line represents the scanning line at 2.2 mm distance from the plate bottom. As the thickness level increases towards the plate surface, the longitudinal residual stress continues to increase. This is attributed to the fact that the last pass is located on the surface, approximately 15 mm away from the plate centerline. The previous passes act as a thermal treatment to subsequent passes, resulting in residual stress relaxation. This explains why the near-surface level has the highest longitudinal residual stress, while the bottom level has the lowest longitudinal residual stress. Large residual stresses were found to occur in different thickness levels within a range of -6 mm to -9 mm away from the weld centerline, which is related to the last pass being located in this range, rather than in the plate centerline.

The residual stress in the normal direction as shown in Figure 2.14 exhibits a similar trend to the longitudinal direction. The largest tensile residual stresses are observed at the near-surface thickness level, with a value of 234 MPa. Unlike the longitudinal direction residual stress, which gradually decreases along the thickness level, the normal direction residual stress has an approximate average value of ~80 MPa for all five thickness levels. A significant change occurs in the HAZ and FZ regions, while the BM remains stable. For all five thickness levels, the normal direction residual stress ranges from -100 MPa to 240 MPa. Compared to the longitudinal and transverse directions, the normal direction residual

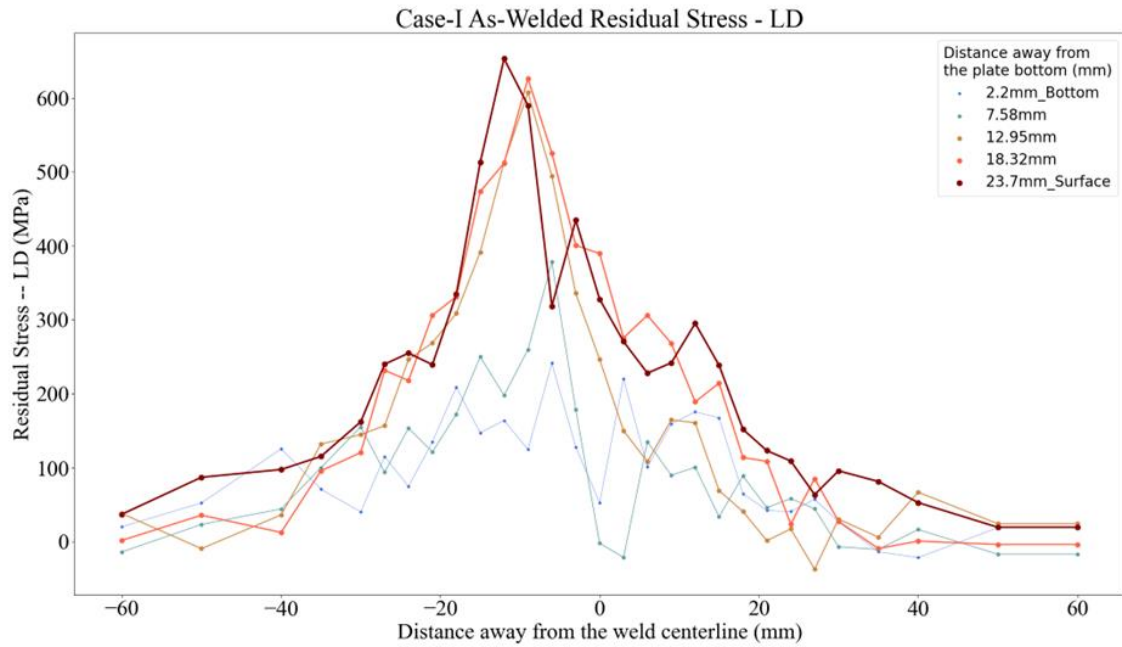


Figure 2.13. As-Welded plate longitudinal direction residual stresses distribution.

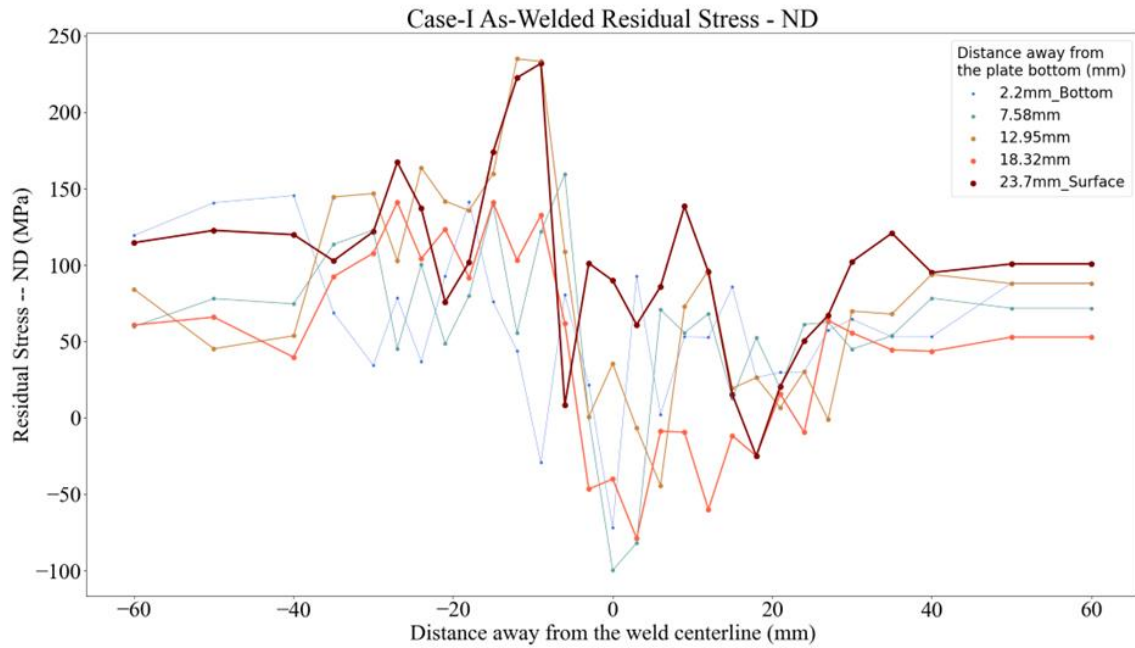


Figure 2.14. As-Welded plate normal direction residual stresses distribution.

stress has a minor impact on crack development. This is due to both the low level of residual stress and the fact that the normal direction is not the responsible direction. Previous studies by other researchers have also found that normal direction residual stresses have a minor contribution to crack initiation and propagation.

Figure 2.15 displays the distribution of residual stresses in the transverse direction, which exhibits a unique feature. Specifically, at a near surface thickness level (23.7mm from the plate bottom), the transverse residual stress reaches its maximum value of 346MPa at a location 12mm away from the weld centerline. Surprisingly, instead of gradually decreasing towards the plate bottom, the tensile transverse residual stresses in thickness levels 7.58mm and 12.95mm from the plate bottom transform into compressive transverse residual stresses. The largest compressive residual stresses occur at the weld centerline (0mm away from the weld centerline) in the thickness level 7.58mm from the plate bottom, with a magnitude of 228MPa. The average transverse residual stress across all five thickness levels ranges between 70MPa and 150MPa, with relatively larger values near the plate bottom and surface thickness level. The abrupt changes in the middle of the plate lead to local stress concentrations, which are the primary cause of crack initiation. These microcracks will continue to propagate during further thermal aging (service time) and ultimately lead to failure. Notably, opposite trends in the variation of transverse residual stresses occur in the middle thickness levels, with increasing stress observed in the 23.7mm, 18.32mm, and 2.2mm thickness levels, and decreasing stress in the 7.58mm and 12.95mm thickness levels. These changes are mainly located in the heat-affected zone (HAZ) and fusion zone (FZ) and are accompanied by microstructural evolution.

2.3.3 Results of PWHT plate

The As-Welded condition accumulates the highest residual stresses due to no relaxation procedures have been performed. The purpose of this research is to investigate the thermal effect on residual stress relaxation. Hence, results of other thermal treatment conditions are listed below. Figures 2.16-2.18 illustrate the residual stress distribution in the plate after PWHT in all three directions. Figure 2.16 shows the variation of longitudinal direction residual stress. Among all three directions, the longitudinal direction has the

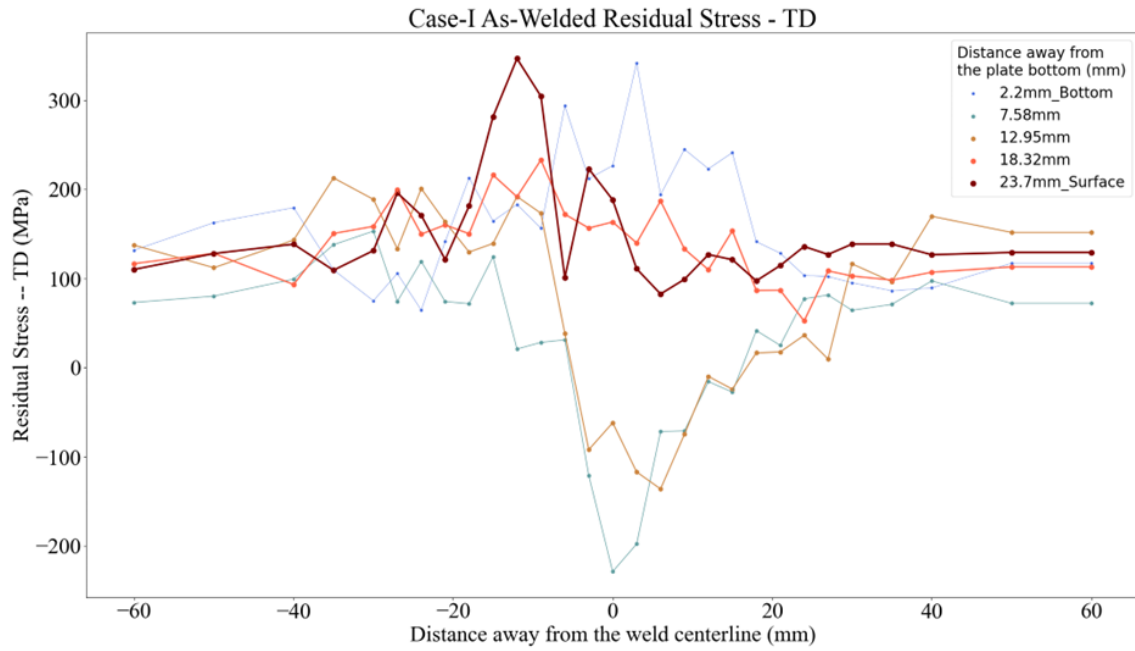


Figure 2.15. As-Welded plate transverse direction residual stresses distribution.

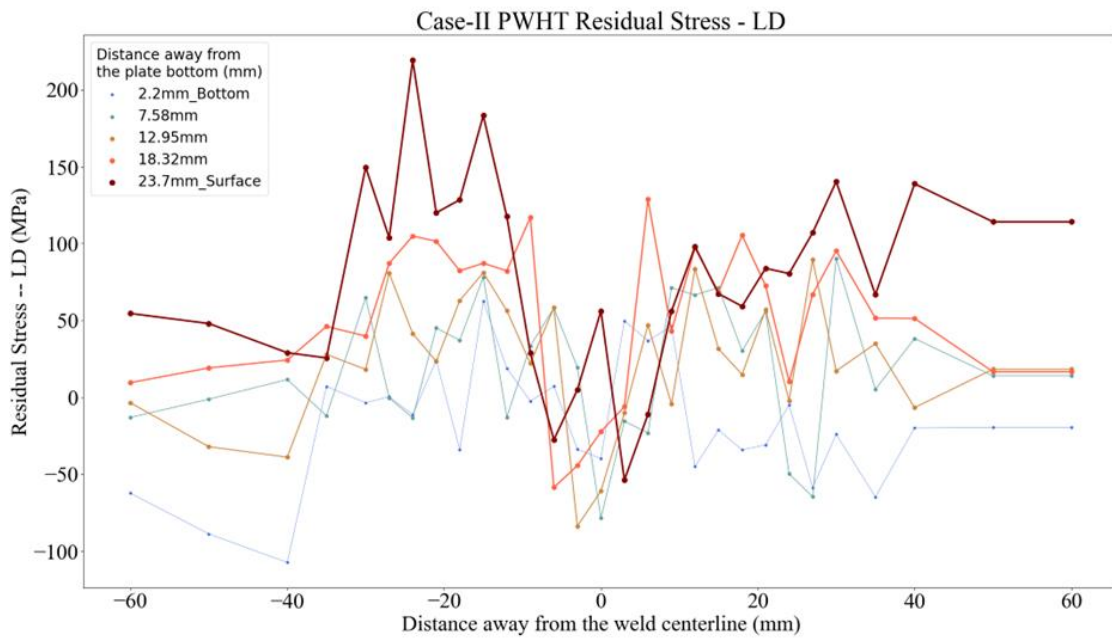


Figure 2.16. PWHT plate longitudinal direction residual stresses distribution.

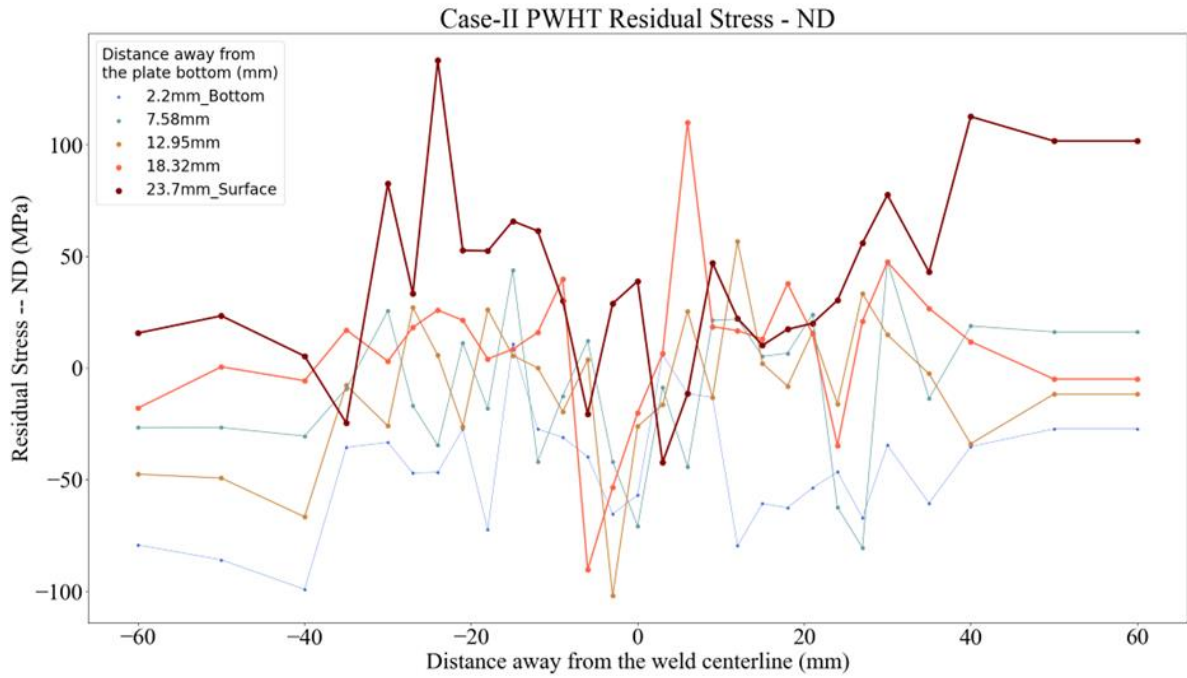


Figure 2.17. PWHT plate normal direction residual stresses distribution.

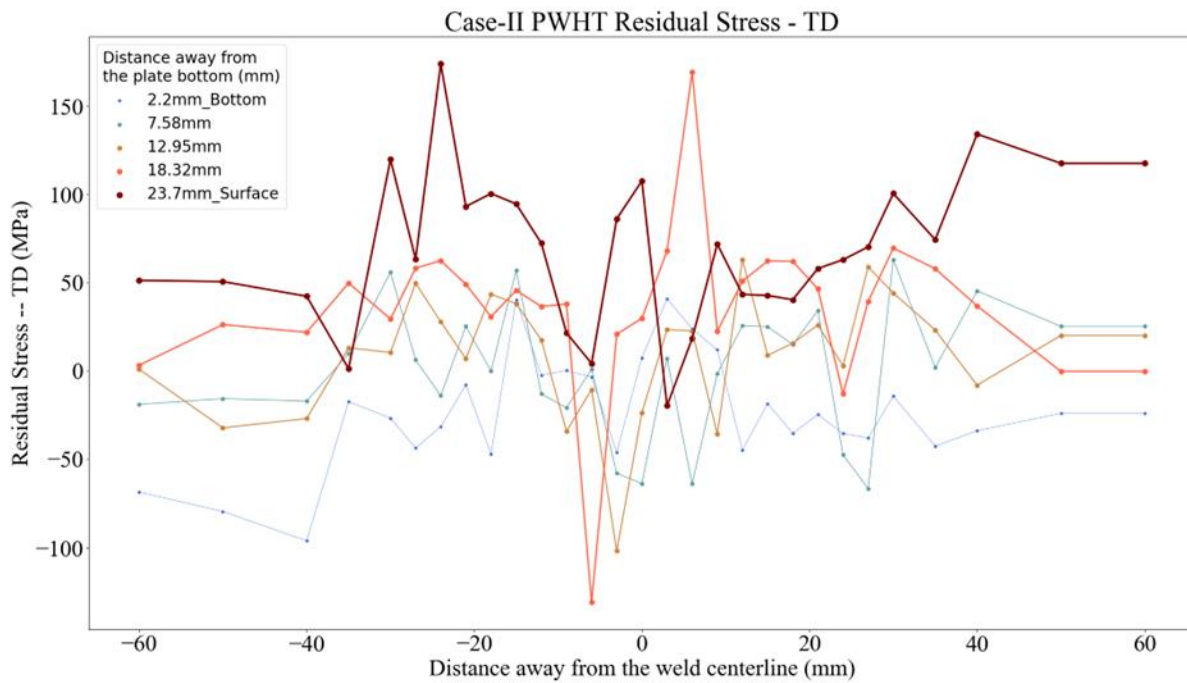


Figure 2.18. PWHT plate transverse direction residual stresses distribution.

largest average residual stress value, which is similar to the As-Welded case, although the difference is not significant. The largest longitudinal residual stress in the PWHT weldments is observed 21mm away from the weld centerline at a thickness level of 23.7mm from the plate bottom, with a value of 219MPa. A slightly smaller value of ~140MPa longitudinal residual stress is detected at a thickness level of 18.32mm away from the plate bottom and 9mm away from the weld centerline. The compressive residual stress region is mainly located 40mm away from the weld centerline on both sides, with values ranging from 0MPa to 50MPa. The difference in longitudinal residual stress distribution between the As-Welded and PWHT plates is the variation range. In the PWHT plate, the residual stresses can change from approximately 150MPa tensile residual stress to approximately 100MPa compressive residual stress, covering a wide range from -40mm to 40mm away from the weld centerline, which is wider than the HAZ + FZ. This variation confirms that the PWHT procedure is intended to relax the residual stresses and reduce the dramatic local changes. These two factors increase the susceptibility to residual stress relaxation cracking (SRC). Therefore, performing PWHT is one of the useful methods to reduce the susceptibility to SRC. Moreover, in Figure 2.16, the two-dimensional longitudinal residual stress distribution image shows that the difference between each thickness level is negligible, unlike the As-Welded case, where there is an obvious gap.

Figure 2.17 illustrates the distribution of normal direction residual stresses in the PWHT plate weld. Since the normal direction has limited impact on SRC, the analysis will not focus on it. The highest residual stresses are mainly concentrated in the middle to surface thickness levels, ranging from 50 MPa to 100 MPa, whereas the lowest peak value is ~15 MPa at the bottom level. The compressive residual stress is widely distributed throughout the entire cross-section with values ranging from 40 MPa to 80 MPa.

Figure 2.18 illustrates the distribution of residual stresses in the transverse direction, which differs from that observed in the As-Welded case and exhibits a smaller variation range. The double-peak distribution can be observed which correlates to the HAZ distribution. The surface levels exhibit relatively higher residual stress values of approximately 174MPa in the form of tensile residual stresses. The maximum values for different thickness levels compressive residual stress range from 50MPa to 100MPa. The

highest compressive residual stress appears in the middle thickness level at ~6mm from the weld centerline with a value of 131 MPa.

2.3.4 Results of TA-As-Welded and TA-PWHT plate

Once the initial residual stress range has been determined, the subsequent step involves defining the relaxation percentage of residual stress induced by long-term thermal treatment. To simulate the service condition, the plates were subjected to a thermal treatment involving isothermal holding at 650°C for 361.5 hours. Figure 2.19-2.21 shows the residual stress redistribution in the TA-As-Welded plates.

Figure 2.19 illustrates the longitudinal distribution of residual stresses for the TA-As-Welded plate. Similar to the As-Welded condition, the highest tensile residual stress was observed at the 18.32mm thickness level, with a value of 316MPa. The largest compressive residual stress was detected at the 23.7mm thickness level, with a value of 91MPa. In comparison to the As-Welded condition, the degree of changes has been mitigated, and the peak position has slightly changed to double-peak distribution and closer to the weld centerline. The long-term thermal treatment leads to the residual stress relaxation resulted from 653MPa to 316MPa, representing a reduction percentage of ~52%. Additionally, in Figure 2.19, the two-dimensional longitudinal residual stress distribution image indicates that the differences between each thickness level are minor, and they follow the same trend of variation.

Figure 2.20 illustrates the distribution of normal direction residual stresses in the TA-As-Welded plate. Upon subjecting the As-Welded plate to the thermal treatment at 650 °C for 361.5 hours which can also lead to the d-spacing data variation, a reduction in the magnitude of normal direction residual stress is observed in comparison to the As-Welded condition. The range of residual stress distribution has been diminished from -100MPa ~ 234MPa to -82MPa ~ 155MPa. . The highest value of normal direction residual stress in the TA-As-Welded plate is 155MPa, which represents a 34% decrease from the corresponding value of 234MPa observed in the As-Welded case. The overall trend is similar to that of the As-Welded case, characterized by a high concentration of compressive

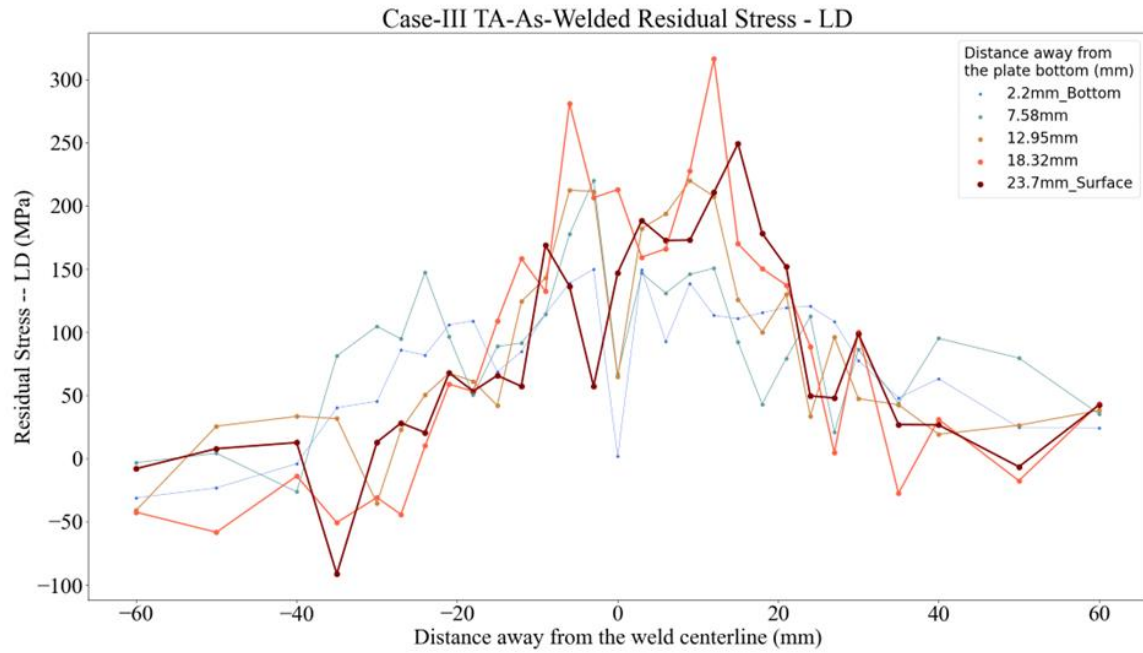


Figure 2.19. TA-As-Welded plate longitudinal direction residual stresses distribution.

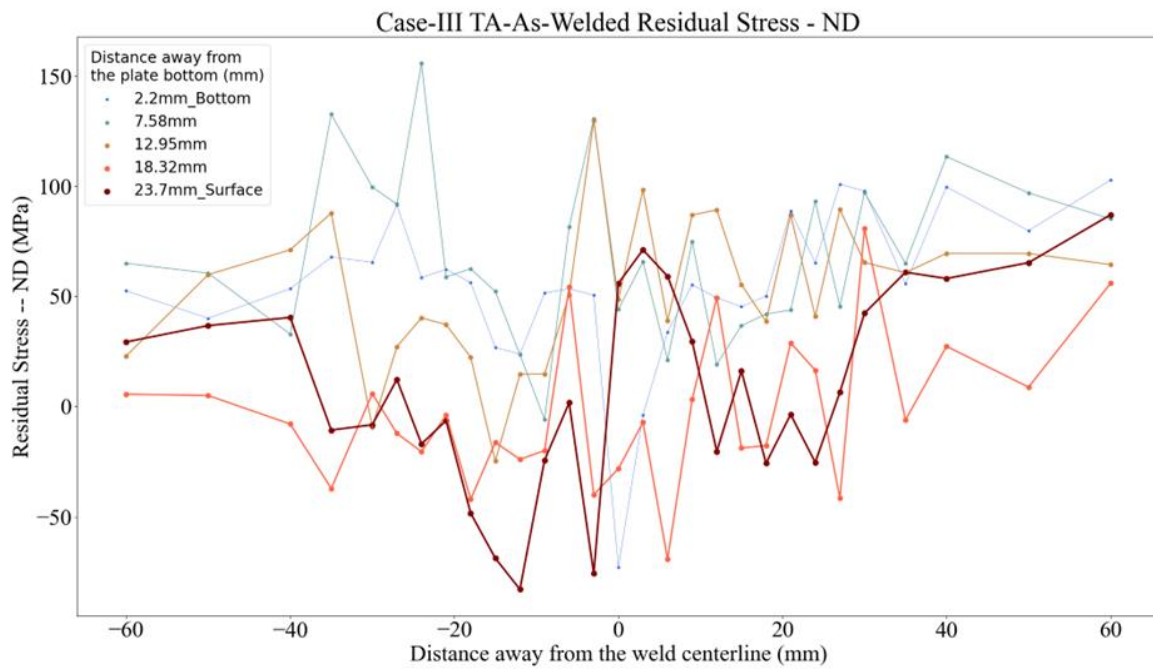


Figure 2.20. TA-As-Welded plate normal direction residual stresses distribution.

residual stress in the middle thickness region, while the entire plate experiences fluctuation around a low stress level.

Figure 2.21 displays the distribution of transverse direction residual stresses in the TA-As-Welded plate. The trend in residual stress distribution is similar to that observed in the As-Welded plate case, with the key variation being a change in the range of residual stress value distribution from -228MPa ~ 346MPa to -35MPa ~ 211MPa after the long-term thermal treatment. Either the tensile or compressive residual stress in the transverse direction have been decreased compared to the As-Welded condition after the long-term thermal treatment. However, the distribution trend is different when compared to the As-Welded plate case due to the influence of the extra thermal treatment process. The distribution trend has altered in a noticeable manner.

Figure 2.22 illustrates the redistribution of the residual stress field in the PWHT plate after thermal aging (TA-PWHT). A comparable trend can be observed between the TA-PWHT case and the PWHT case. The highest longitudinal residual stress has decreased from 219 MPa to 116 MPa. However, the changes in the compressive residual stress are small, which were 107MPa in the former and 100MPa in the latter. Additionally, the distribution trends are also changed caused by the long-term thermal treatment. The distribution in the PWHT case is a double-peak distribution and then transfers to a flatter variation.

Figure 2.23 illustrates the variation trend in normal direction residual stress in the TA-PWHT plate. The distribution is similar to that of the longitudinal residual stresses. The highest value is located 9mm away from the weld centerline, with a value of 107MPa. In comparison, the PWHT case exhibits a value of 137MPa at 21mm away from the weld centerline. The difference of normal direction residual stress in both cases is small. The largest compressive residual stress in the PWHT plate is 102MPa at a distance of 40mm from the weld centerline. However, after thermal aging, the value has decreased to 11MPa and moved to the weld centerline. This phenomenon can be explained by the same mechanism observed in the longitudinal direction. Microstructure characterization information that supports this explanation will be presented in another report.

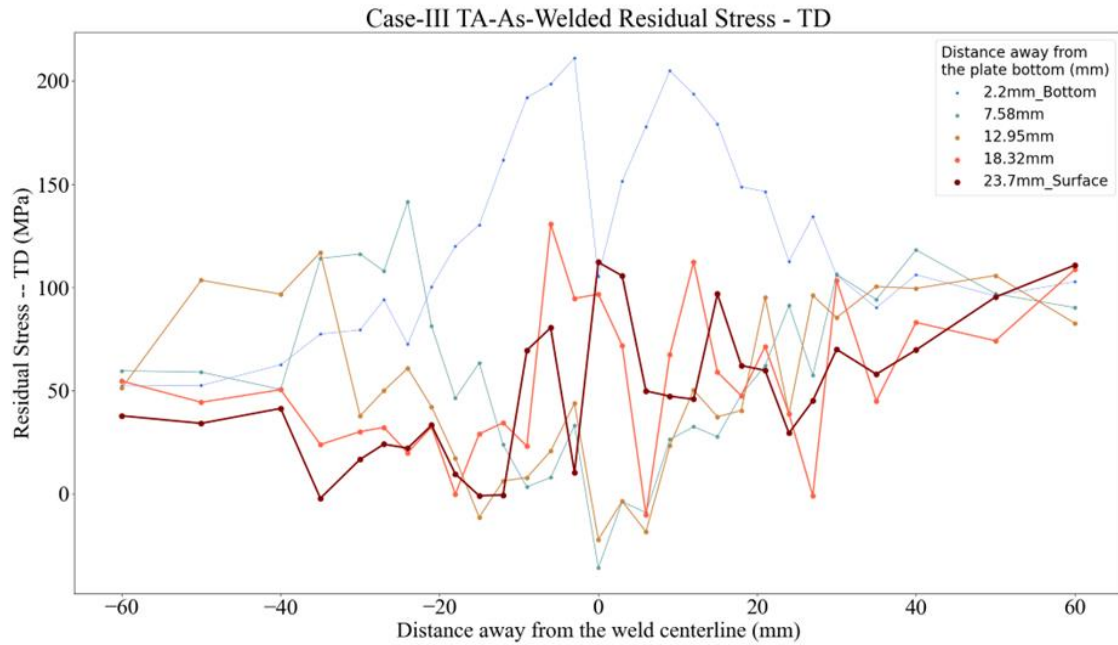


Figure 2.21. TA-As-Welded plate transverse direction residual stresses distribution.

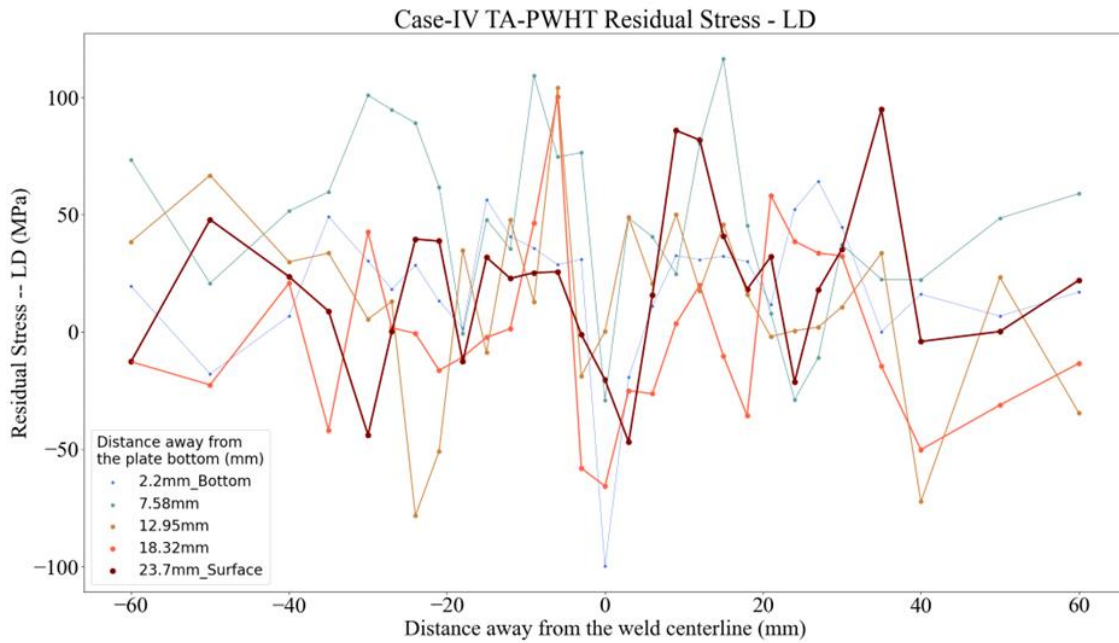


Figure 2.22. TA-PWHT plate longitudinal direction residual stresses distribution.

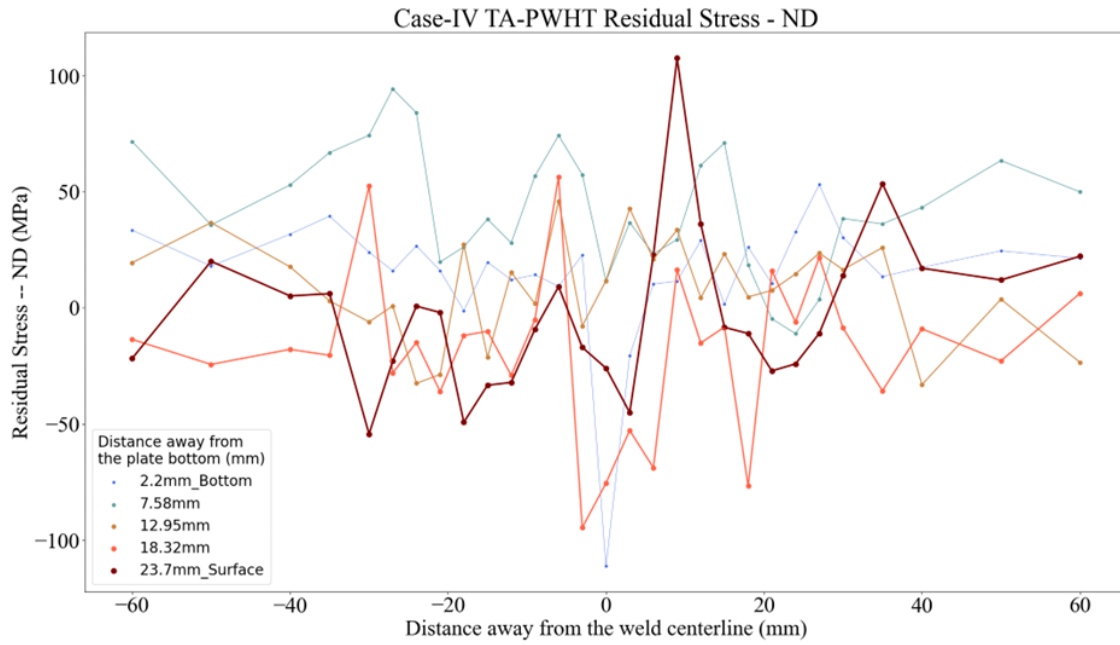


Figure 2.23. TA-PWHT plate normal direction residual stresses distribution.

Figure 2.24 illustrates the distribution of transverse direction residual stress in the TA-PWHT plate. Similar to the previous cases, the TA-PWHT plate exhibits a decreasing trend in both tensile residual stress region compressive residual stress region compared to the PWHT plate. Additionally, the TA-PWHT plate shows a milder transition. Notably, the surface thickness level (23.7mm away from the plate bottom) in the TA-PWHT plate exhibits a lower tensile residual stress value compared to the middle thickness level at 18.32mm away from the plate bottom. However, when compared to the TA-As-Welded condition, distinct differences can be observed. Firstly, the sharp opposite variation trend in the lower thickness level at 2.2mm away from the plate bottom has disappeared after the long-term thermal treatment. Therefore, this thickness level has undergone a dramatic transition from compressive residual stresses to tensile residual stresses.

The colormap for each thermal condition are shown in Figure 2.25-2.28 below:

The visual representation of residual stress fields using a three-dimensional line connecting dots may lead to confusion when attempting to differentiate between different thickness levels and their corresponding residual stress values. An alternative approach to displaying the distribution of residual stress fields in four different cases is using a colormap. In the figures above, colormaps are employed to present the distribution of all three directions in all the four cases, as well as to compare the same direction's distribution among them which will show below. This method facilitates a more straightforward understanding of the influence of thermal treatment on the evolution of residual stress. For instance, Figure 2.25 shows the As-Welded plate's residual stress distribution colormap for all three directions, enabling the identification of the corresponding residual stress value from the color bar, while also making it easy to observe the locally stress concentration in the LD and TD directions. Similarly, by following the same procedure, colormaps for the PWHT case, TA-As-Welded plate, and TA-PWHT plate are plotted in Figures 2.26-2.28. However, as these colormaps do not compare the thermal treatment effect on the residual stress distribution by analyzing the same residual stress direction, it is necessary to set the same upper and lower limits to generate colormaps for the same direction residual stress in all four cases. This process can be useful in evaluating the residual stress evolution under different thermal treatment conditions, as demonstrated in Figure 2.29, where the evolution

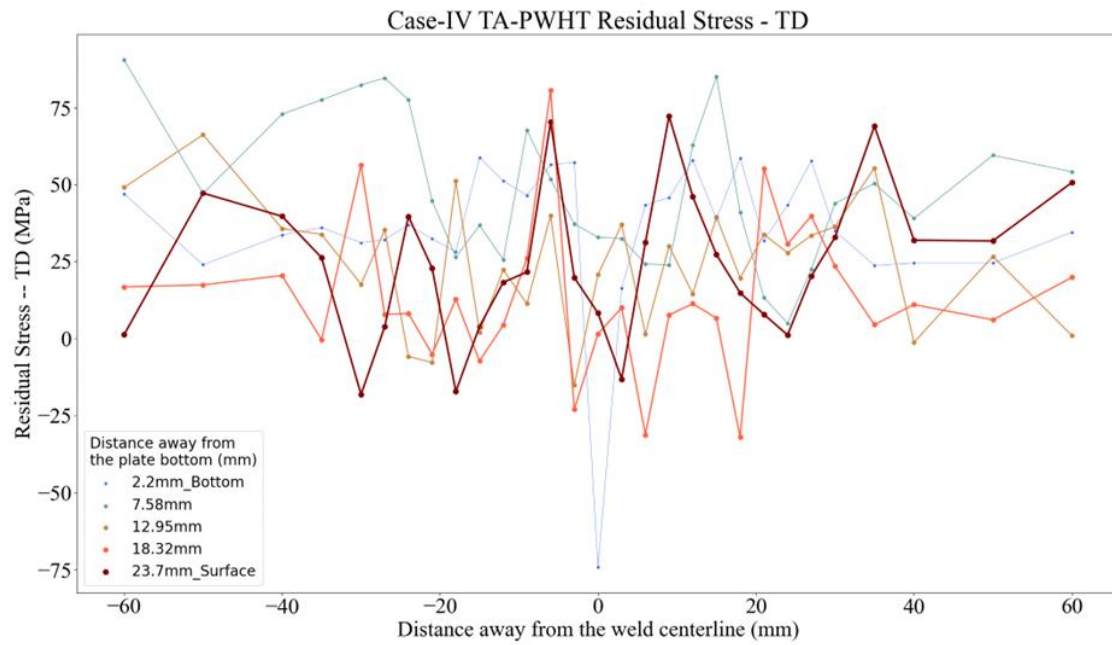


Figure 2.24. TA-PWHT plate transverse direction residual stresses distribution.

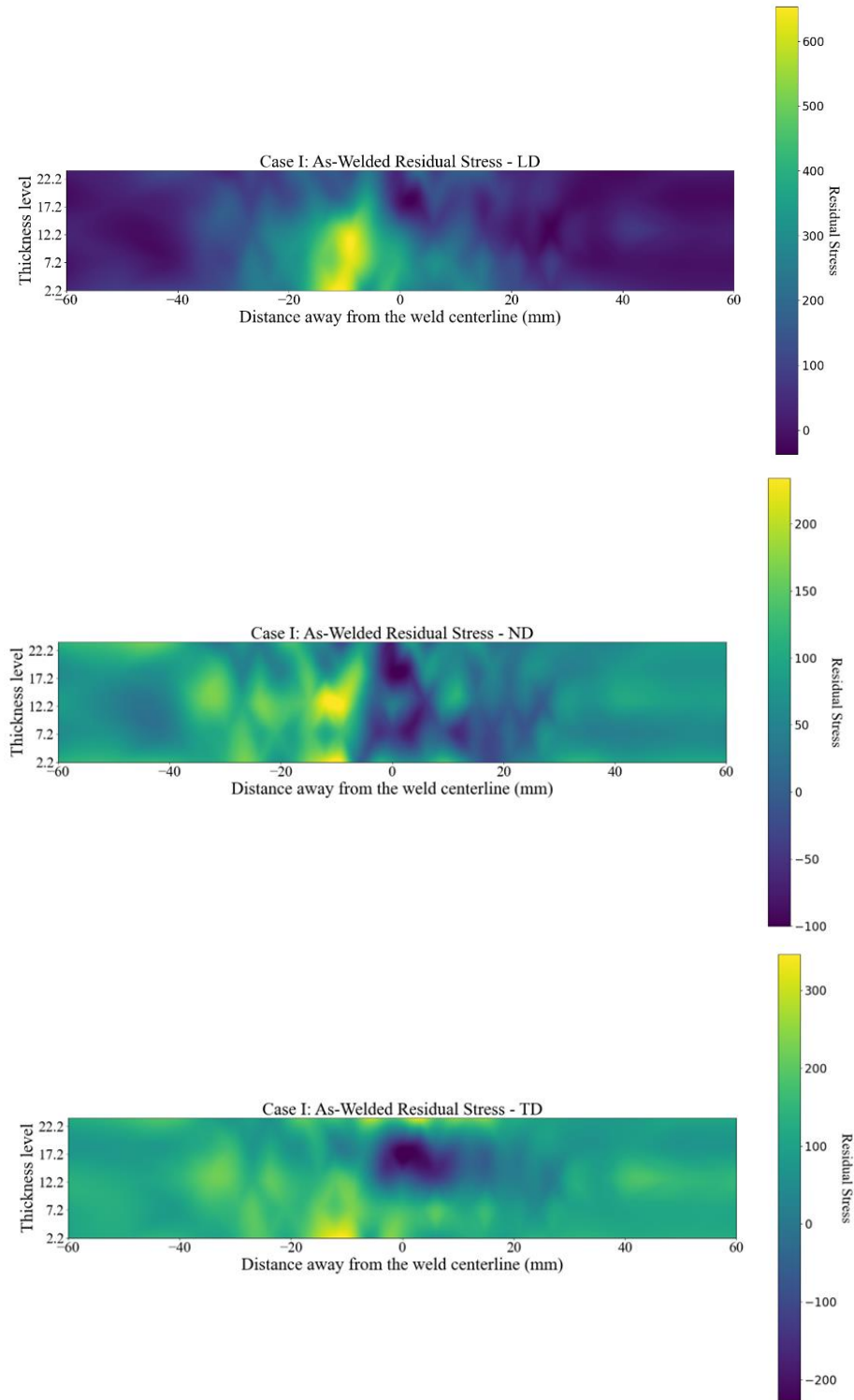


Figure 2.25. As-Welded plate residual stress distribution colormap for all three directions.

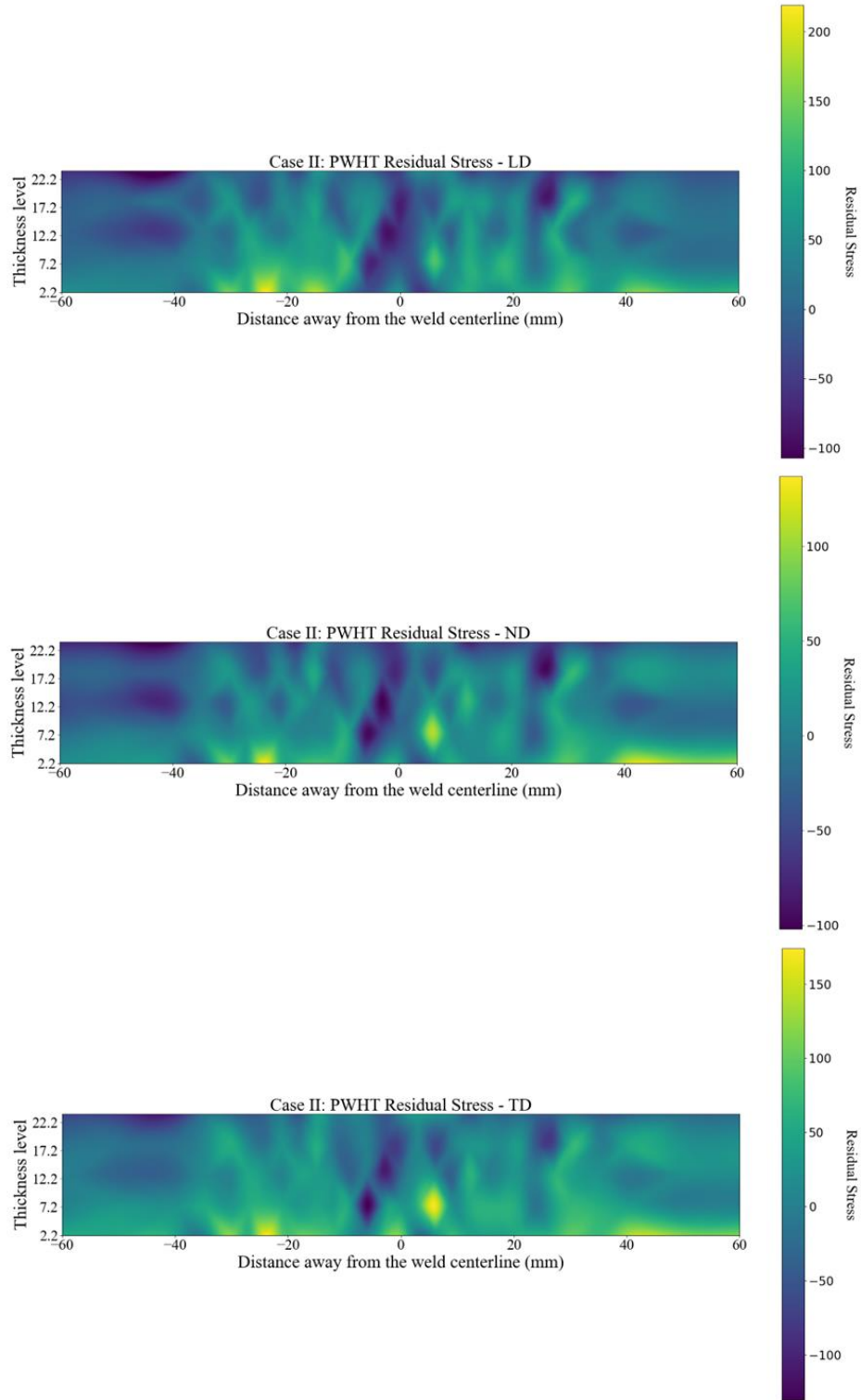


Figure 2.26. PWHT plate residual stress distribution colormap for all three directions.

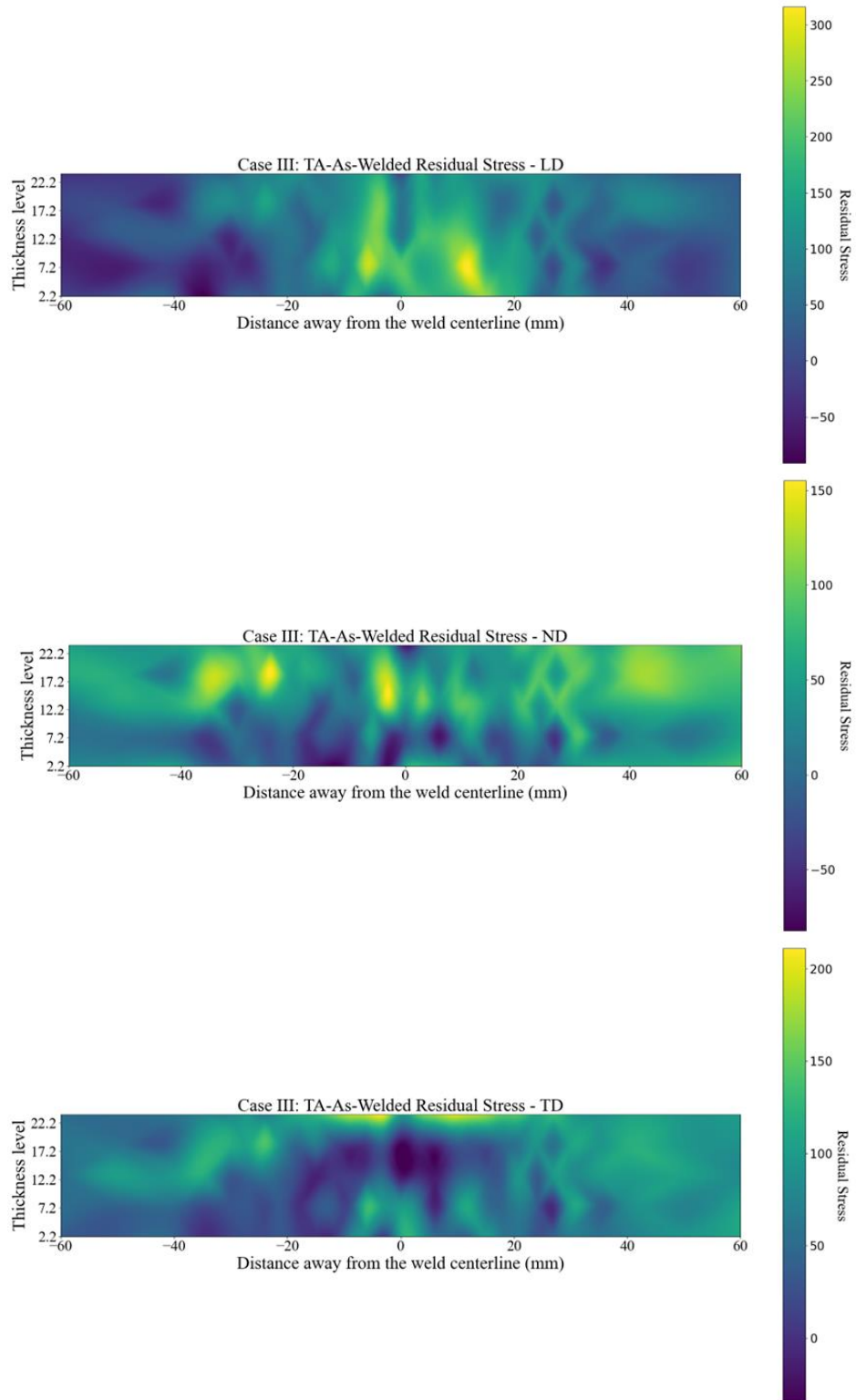


Figure 2.27.TA-As-Welded plate residual stress distribution colormap for all three directions.

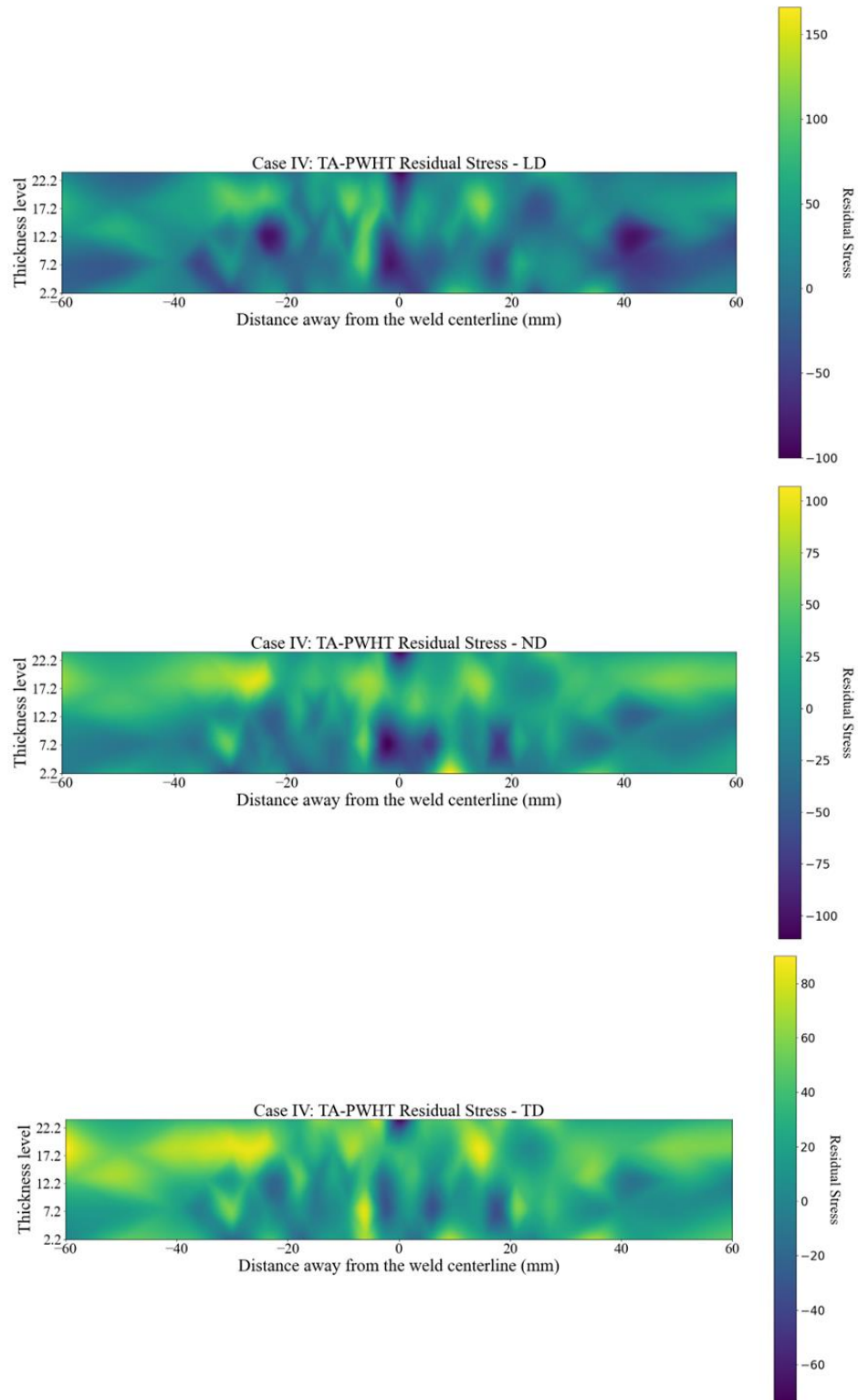


Figure 2.28. TA-PWHT plate residual stress distribution colormap for all three directions.

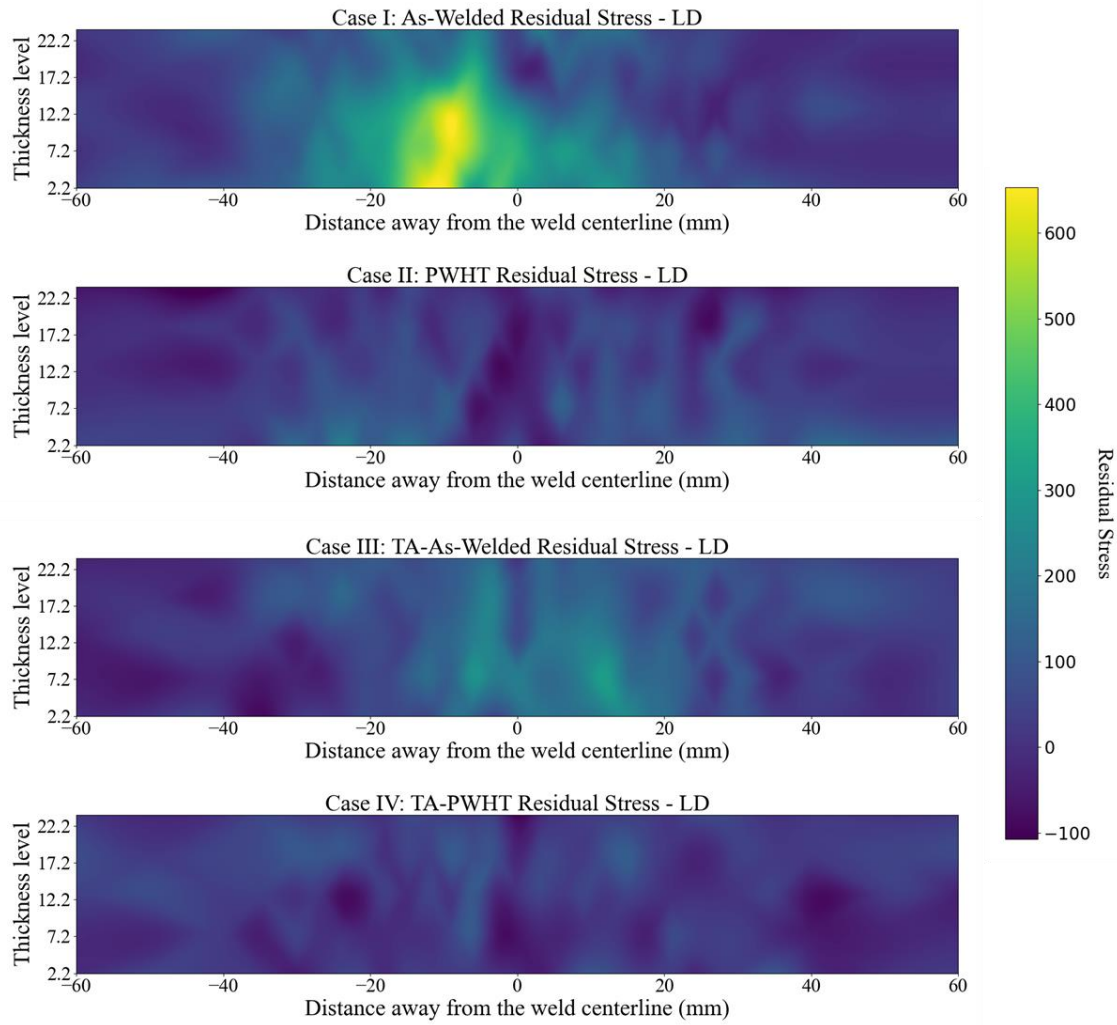


Figure 2.29. Colormap of longitudinal residual stress distribution in all four different thermal treatment cases.

is clearly visible when the same upper and lower boundary is applied. Specifically, the largest tensile residual stress is observed in the As-Welded plate, and its fast relaxation is achieved through PWHT. Without performing PWHT, the relaxation level is limited.

A similar trend is also evident in the normal direction and transverse direction colormap comparison, as shown in Figures 2.30 and 2.31.

2.3.5 d_0 Selection Explanation

The calculation of the absolute residual stress in each thermal treatment case requires the exclusion of the influence of microstructure evolution, which is a crucial factor. Furthermore, each thermal treatment case has its specific microstructure setting that is determined by the degree of microstructure evolution. Thus, four d_0 data sets are needed to correlate with the corresponding thermal treatment condition.

Due to all the d_0 comb samples and their related conditions' plate is not measured in the same beam cycle, so the adjustment is needed to exclude the error bring by the beam, measurement environment, machinery caused error, etc. Four cubes cut from each conditions' base metal region has been measured during the last beam cycle accompanied by the TA-As-Welded plate, TA-PWHT plate, As-Welded d_0 comb, PWHT d_0 comb, and thin pipe d_0 comb. The following explanation will be used As-Welded condition as an example.

The As-Welded plate was measured in the first beam cycle, which gives an average base metal d-spacing value in ~ 1.0849 . However, the As-Welded d_0 comb was measured in the last beam cycle which gives an average value of s-spacing in the base metal region ~ 1.0935 . Meanwhile, the As-Welded cube was also measured in the last beam cycle and gives a value of 1.09295. Hence, the As-Welded plate data set needs to add a value (~ 0.0074 , the value is different for each direction) to ensure the base metal region's d-spacing value can match. Using this method, the sum of normal direction residual stress is $\sim 6\text{MPa}$, which is reasonable theoretically.

$$\sum_{\text{position from } -60\text{mm}}^{\text{position to } 60\text{mm}} \sigma_{ND} \approx 6\text{MPa} \quad (\text{As} - \text{Welded case})$$

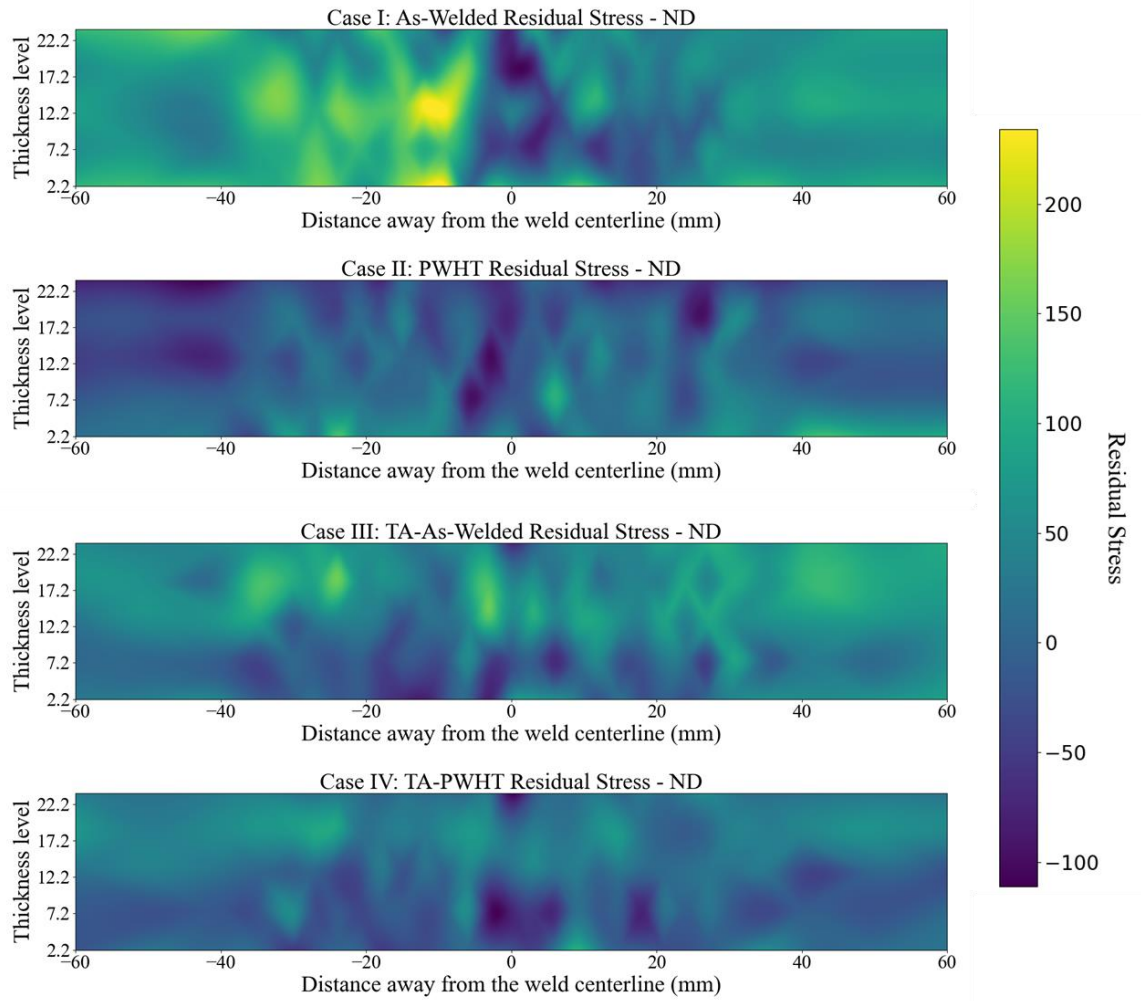


Figure 2.30. Colormap of normal residual stress distribution in all four different thermal treatment cases.

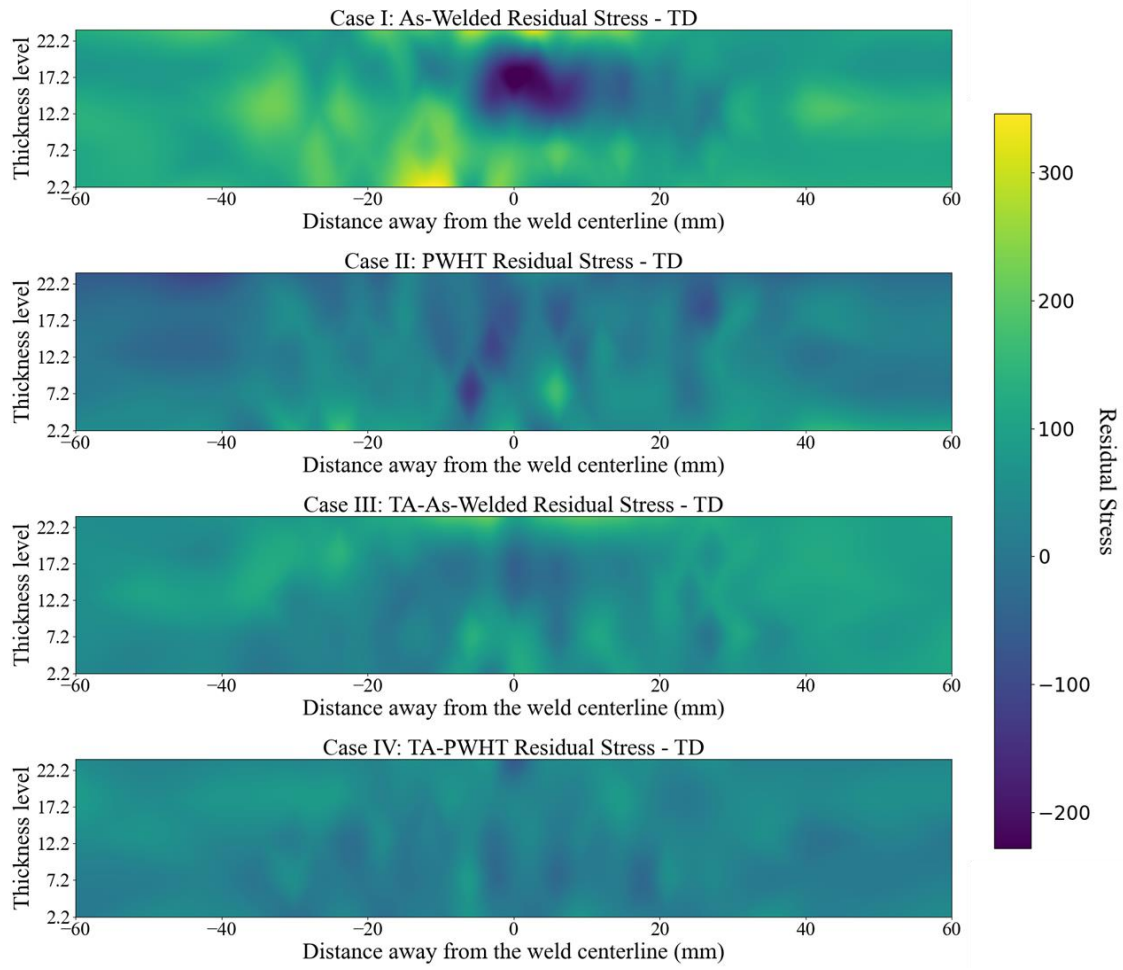


Figure 2.31. Colormap of transverse residual stress distribution in all four different thermal treatment cases.

2.3.6 Results calculated through plane stress methodology

Utilizing a reference comb sample (d_0 comb) to estimate residual stress presents a beneficial method for calculation, but also introduces an element of uncertainty. The selection location for the comb sample is typically at the edge of the target sample (in this case, at the edge of each plate), which raises two major concerns. Firstly, there may be slight differences in the microstructure between the scanning location of the plate (i.e., at the plate's center) and the actual location of the comb sample. Secondly, cutting the edge of the plate for the comb sample using EDM or saw may induce a small amount of residual stress, which is something that one seeks to avoid in the comb sample.

To ensure the accuracy of the d_0 method, it is beneficial to verify it using an alternative approach to calculate residual stress. In this study, we employed the plane stress assumption to generate the residual stress field. The plane stress method is based on the assumption that the normal direction residual stress is equal to zero. This simplifies many problems by considering a two-dimensional state of stress, which is often used in practice when one dimension of the body is small relative to the others, such as in thin plates [5-7]. Under the plane stress assumption, the stress system consists of two normal stresses (σ_x and σ_y) and a shear stress (τ_{xy}), with only one primary direction having a zero value. By applying the equations in Section 2.3.1.2, the influence from the microstructure site can be excluded. Therefore, a residual stress field can be obtained through a plane stress calculation without the need for d_0 development. The resulting plane stress case is depicted in the following figures.

Figure 2.32 displays the residual stress distribution in the longitudinal and transverse directions of the As-Welded and PWHT plates. The highest tensile longitudinal residual stress is located at a distance of 9 mm from the weld centerline and 18.32 mm from the plate bottom, with a value of 497MPa. This value follows the similar trend as the d_0 comb sample calculation method. The surface and near-surface levels of the plate exhibit high levels of tensile residual stress accumulation, which gradually decrease to a level near 100MPa compressive residual stress. Although the highest value is approximately 100MPa less than that obtained from the comb sample method, the base

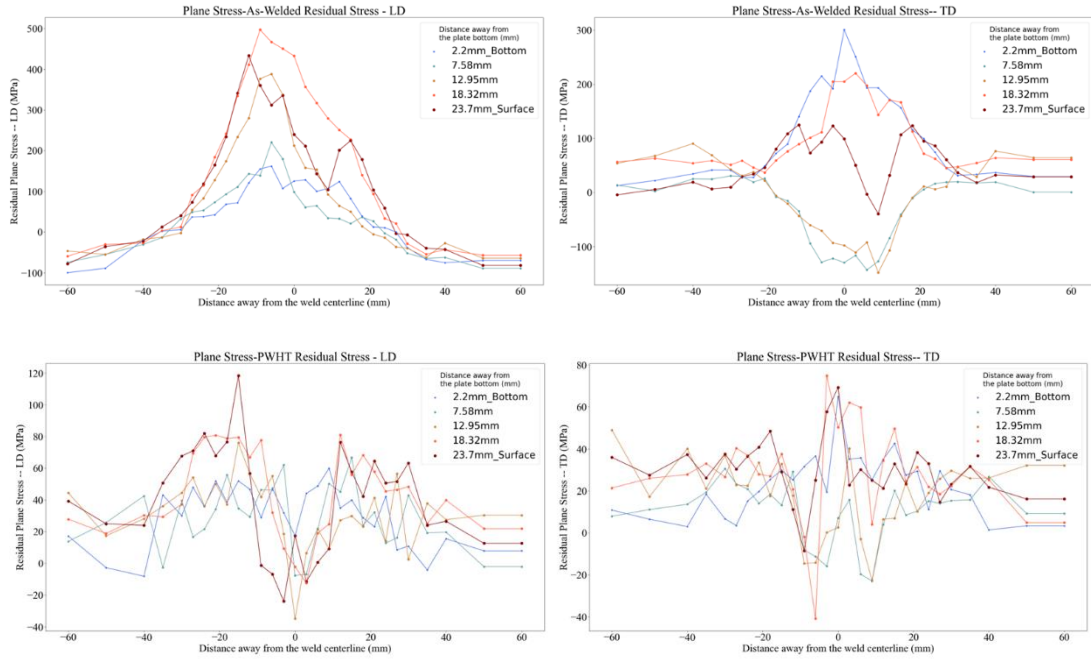


Figure 2.32. As-Welded & PWHT plates longitudinal and transverse direction residual stresses distribution calculated by using plane stress method.

metal region exhibits approximately 100 MPa compressive residual stress. Therefore, a shift has occurred in the entire plot compared to the comb method. Another notable feature is a sharp downward trend in the comb calculation method in the near-bottom thickness levels in the middle of the plate, while the plane stress case displays a gradually decreasing trend. This phenomenon may be related to the relaxation process of residual stress. The thickness level near the plate bottom experiences thermal treatment from the upper-level heat source, which induces residual stresses that are subsequently relaxed to some degree. For the transverse direction residual stress in the plane stress case, the near-bottom thickness level shows the highest tensile residual stress at the middle of the plate, with a value of 300 MPa, which differs from the other method. However, a similar trend is observed in the middle thickness levels, which are 7.58 mm and 12.95 mm away from the plate bottom, respectively; both exhibit compressive residual stress in the middle of the plate. The peak position in the plane stress case is mainly focused on the middle of the plate, unlike the comb method, which displays a slight shift to the left.

After undergoing PWHT, the longitudinal residual stress values were observed to range from 118MPa tensile to 34MPa compressive residual stress. This range is narrower than that observed in the As-welded condition. The residual stress variation in both longitudinal and transverse directions was found to be flatter in the PWHT condition than in the As-welded condition. The PWHT procedure is responsible for this change. In the longitudinal direction, the residual stress starts increasing from both base metal side ends at the HAZ region and then changes sharply to a decreasing trend towards the weld centerline. In contrast, the transverse residual stress variation across the entire cross-section is small in the PWHT condition, with only a ~40MPa variation observed in the HAZ and FZ regions.

Two additional cases, namely TA-As-Welded and TA-PWHT, were investigated and presented in Figure 2.33, respectively. The relaxation process in these cases occurred at 650°C without external load. Nevertheless, a comparison between these cases and the As-Welded and PWHT plates revealed a shift in the peak position. In the TA-As-Welded case, the maximum tensile residual stress of 264MPa was found at a thickness level of 2.2mm away from the plate bottom and 3mm from the weld centerline. The peak position

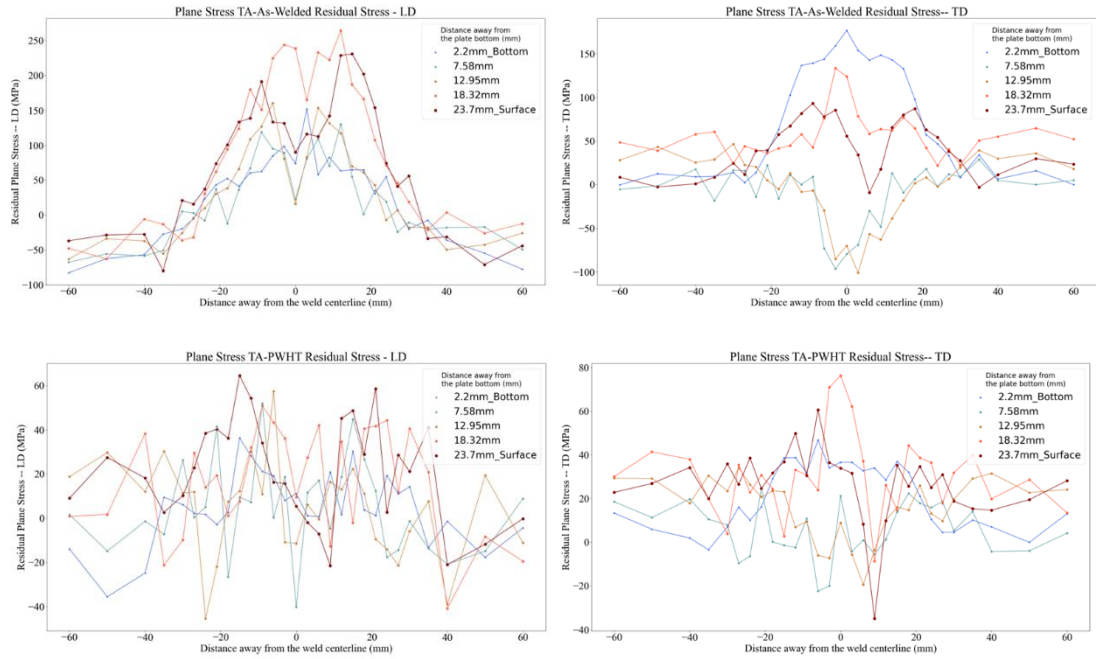


Figure 2.33. TA-As-Welded & TA-PWHT plates longitudinal and transverse direction residual stresses distribution calculated by using plane stress method.

was observed to be closer to the welded centerline compared to the As-Welded plate. The entire plot became more flattened and was observed in the TA-As-Welded plate transverse direction. In comparison with the As-Welded plate, the tensile residual stress decreased by nearly 130MPa, while the compressive residual stress was also decreased by approximately 40MPa. Meanwhile, the variation across the BM to FZ did not change significantly. In the TA-PWHT case, only a minor shift down was observed in the longitudinal direction. However, the line figures alone did not provide a straightforward understanding of the residual stress field variation. Therefore, colormap was utilized to facilitate this comparison.

Figure 2.34 depicts the colormap of the As-Welded plate condition. It shows a high concentration of tensile residual stress in the middle thickness level of the LD, while a slightly higher thickness level exhibits compressive residual stress concentrated in the weld centerline of the TD. The BM presents a relatively uniform distribution of residual stress, with the exception of the FZ and the HAZ.

Figure 2.35 displays the colormap of the plate in the PWHT conditions of LD and TD. The PWHT procedure was effective in significantly reducing the residual stress concentration, especially in the HAZ and FZ in the middle thickness level. However, some locations still show noticeable stress concentrations, which may be attributed to the fact that complete residual stress relaxation is not achievable in practice or that the scanning matrix used caused some fluctuations.

Comparing these results with those obtained after further thermal aging case TA-As-Welded plate and TA-PWHT plate, it can be observed from Figures 2.34 and 2.36 that long-term residual stress relaxation at RT caused a minimal reduction in stress concentration. Nevertheless, in both figures, the location of stress concentration remains unchanged, even though the magnitude has shifted. The same trend is also observed in the comparison figures of Figures 2.35 and 2.37.

In order to facilitate a clear comparison of the residual stress variation among four different heat treatment cases in the longitudinal and transverse directions, Figure 2.38 and Figure 2.39 respectively depict the colormap of longitudinal and transverse residual stress variations. These figures provide a similar comparison to that obtained by the comb

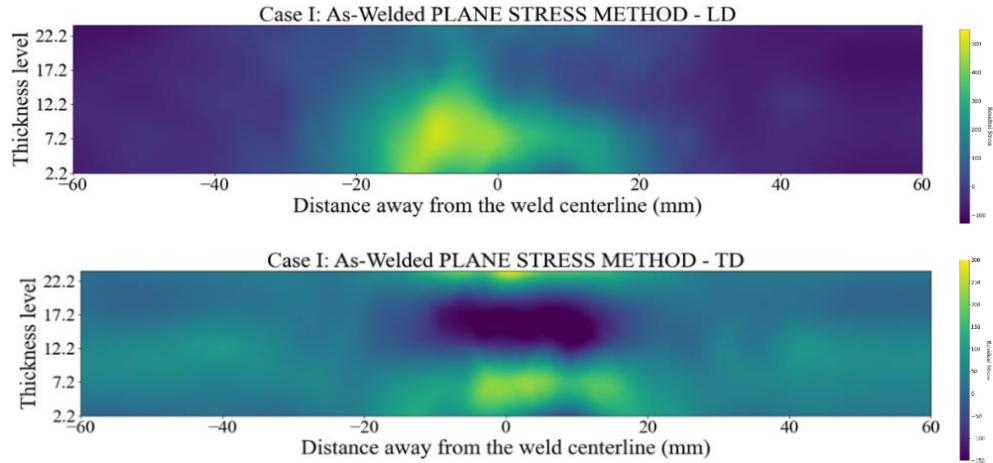


Figure 2.34. As-Welded plate LD & TD colormap.

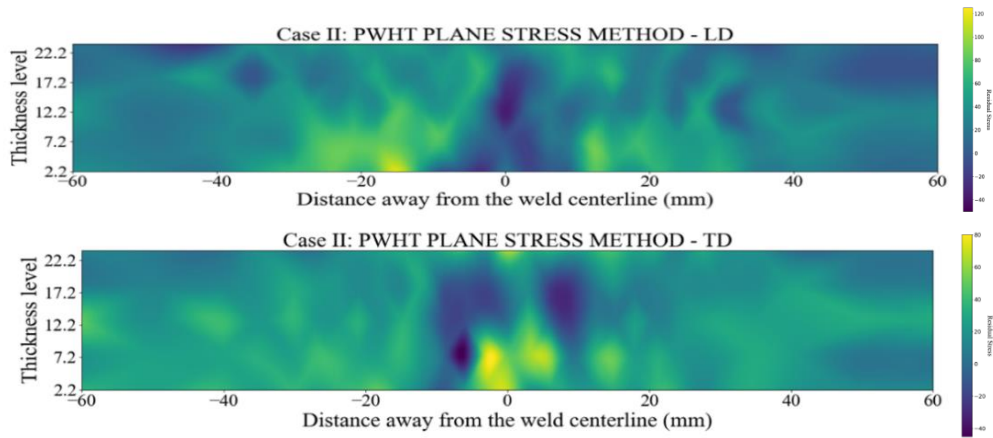


Figure 2.35. PWHT plate LD & TD colormap.

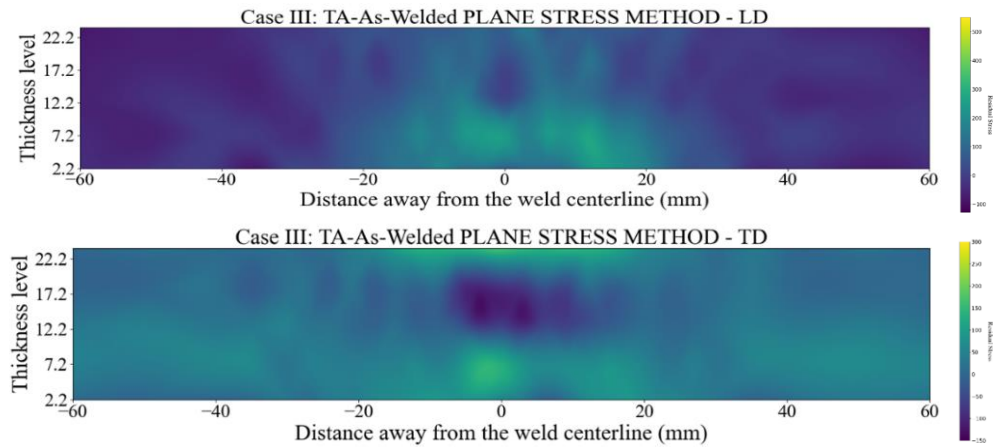


Figure 2.36. TA-As-Welded plate LD & TD colormap.

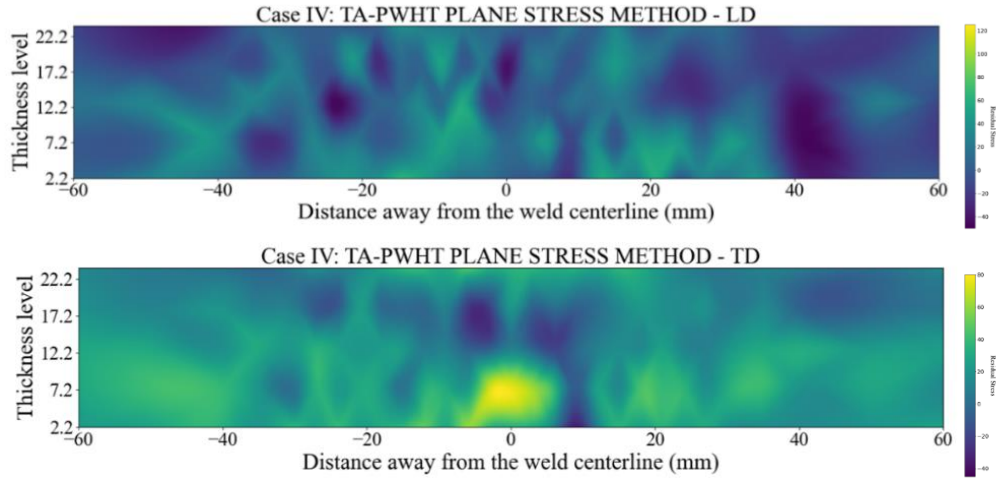


Figure 2.37. TA-PWHT plate LD & TD colormap.

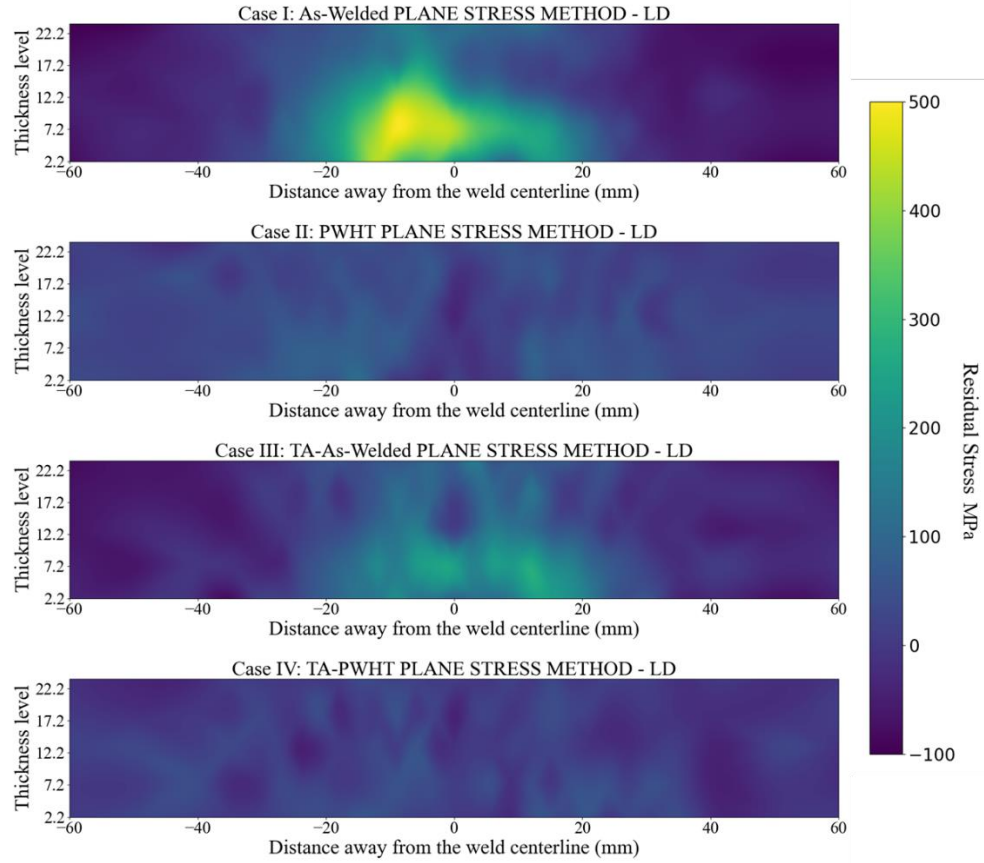


Figure 2.38. Longitudinal direction (LD) residual stress colormap variation among four different heat treatment cases.

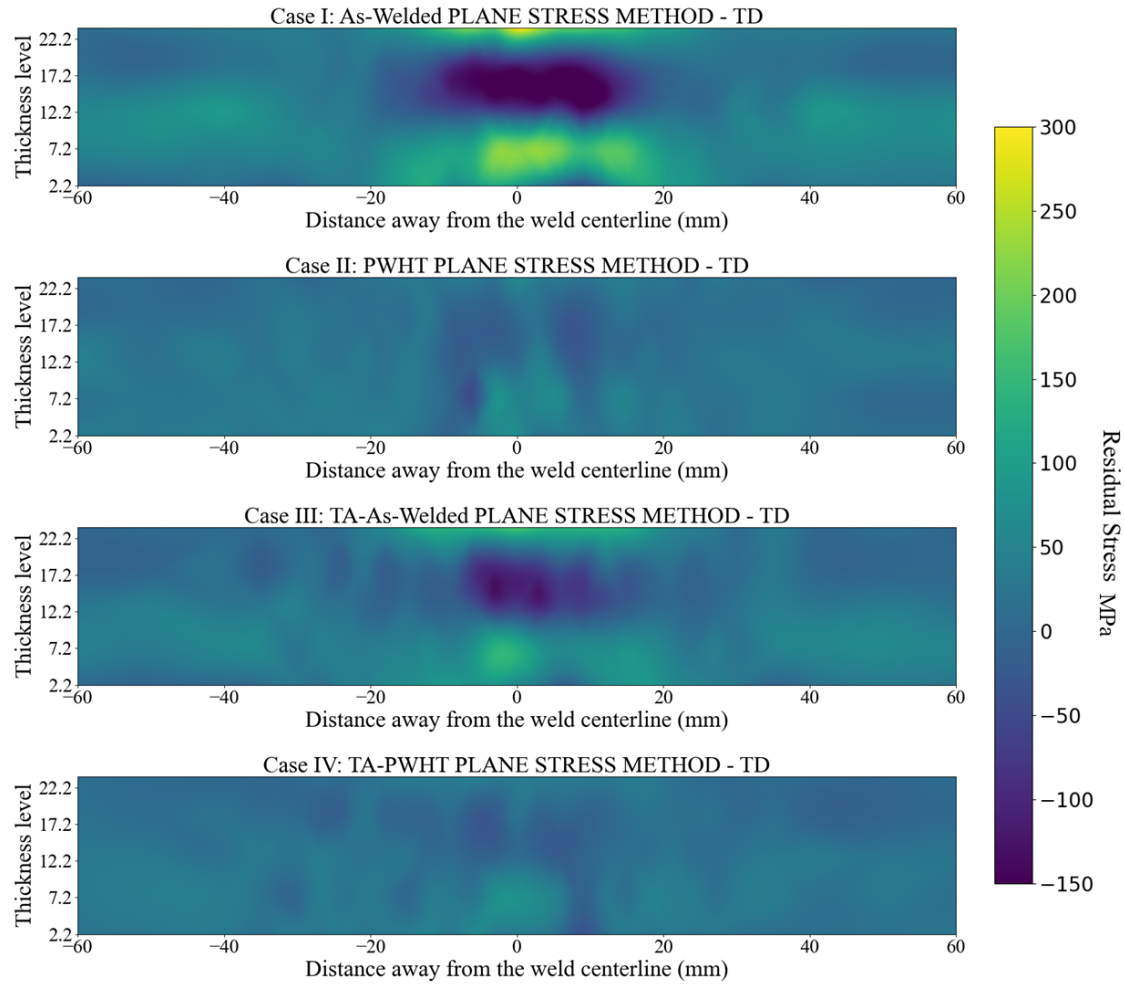


Figure 2.39. Transverse direction (TD) residual stress colormap variation among four different heat treatment cases.

method. The effect of room temperature relaxation is negligible as it causes only a slight change in the position and magnitude of the highest residual stress (either tensile or compressive), which falls within the data fluctuation range. The same variation can be observed in the transverse direction, as shown in Figure 2.39.

CHAPTER THREE

APPLY SIMULATION MODELS TO PREDICT 347H WELDMENTS LIFETIME

3.1 Introduction of 347 SRC simulation models

The abnormal failures observed in pipeline systems subjected to thermal cyclic loading have garnered close attention from welding community and material scientists. A critical task in this area is to develop a method that can simulate the entire failure process and provide a reasonable estimation of the failure lifetime. A critical task in this area is to develop a method that can simulate the entire failure process and provide a reasonable estimation of the failure lifetime. Three methods are commonly used for lifetime prediction: (1) designing a series of creep tests to simulate the failure process, (2) using simulation models to calculate the failure time, and (3) combining experimental and numerical models to support and verify each other. In this study, the third method is employed with some modifications. The target material for this study is 347H austenitic stainless steel, which poses several challenges due to the variations in thermal-mechanical properties and microstructure evolution throughout the entire thermal treatment procedure. Therefore, three main challenges need to be considered when building the simulation model.

The first challenge in this study involves determining the optimal temperature range for PWHT. A higher PWHT temperature can result in a more uniform and lower residual stress field which can prolong the lifetime of the pipeline system[13]. However, if the residual stress relaxation rate is too fast, the susceptibility of crack initiation and propagation will increase due to the stress variation and microstructure evolution. PWHT is typically applied after the welding process to reduce residual stress level. The percentage of residual stress reduction depends on the PWHT temperature, with higher temperatures leading to more rapid relaxation of residual stress. While this can be beneficial for reducing residual stress, it can also increase the susceptibility of strain age cracking (SRC) by creating more sites for voids nucleation and microcrack connection. However, PWHT can also reduce the dramatic residual stress variation in adjacent regions and promote a decrease in microstructure gradient. Therefore, a balance between a larger ratio of stress

relaxation and lower susceptibility of SRC needs to be achieved to determine the proper PWHT temperature range.

The second challenge concern in this study is the controlling failure mechanism. Previous research [6, 14] indicates that creep rupture, or a coupled mechanism including diffusion and creep failure are typical modes of failure control. The formation of voids at grain boundaries and their accumulation under thermal-mechanical field are the root cause, with precipitation evolution playing a key role in void coalescence, crack initiation and propagation, especially in the case of precipitation preferentially formed at grain boundaries. The above behaviors can be explained through an appropriate failure mechanism or their combination. Diffusion-controlled failure is more likely to occur at high-temperature service conditions. For the service temperature range considered in this study, failure is first controlled by creep, then transfer to diffusion control as the applied stress (residual stress) decreases and the service temperature increases. The broad range of thermal treatment temperatures from mid- to high- temperature adds further complexity to determining the controlling mechanism of failure. Another contributing factor is the variation in thermal-mechanical properties caused by microstructure evolution, which will be discussed in detail in the third challenge. Previous research has used simulation to predict lifetime, for example, D. R. Hayhurst [15] used a continuous damage method to predict the lifetime of high-temperature, low-alloy ferritic steel weldments. Hayhurst's model was successful in identifying the failure location, although the prediction error was noticeable when comparing 2D to 3D results, and even more obvious to compare with experimental data. V. Gaffard focused on revealing the relationship between microstructure state and low creep strength in weldments, and correlated experiments and model were developed [16]. However, the limitations of the sample shape and the ability to extrapolate to other conditions must be acknowledged.

Wei Zhang's work provided a detailed explanation of the effect of microstructure evolution and the transition of failure mechanisms. Moreover, comparison was made with NIMS creep data [11]. In order to address the failure process more precisely, the simulation model in this study is enhanced with a grain boundary cavity nucleation feature. The growth of cavities under different governing mechanisms is incorporated to fit different

working conditions. In the meantime, viscous grain boundary sliding and the emergence of microcracks caused by coalescence are included in the constitutive law.

The final and most distinct challenge is assessing the impact of microstructure evolution. The intricate relationship between precipitations generation and annihilation, as well as changes in thermal-mechanical properties, can dictate the failure process. For example, the working environment significantly affects the thermal-mechanical properties variation of 347H austenitic stainless steel, which is a high alloy-type precipitation strengthening material. Examples of such variation include an increase in creep rate, a decrease in yield strength, and an increase in thermal expansion coefficient, among other factors. Moreover, the gradual evolution of precipitation over time during service also influences pipe performance. Several types of precipitates exist in 347H SS at different stages of service, including NbC, Cr₂₃C₆, Fe-rich α , Cr-rich α' , sigma phase, G-phase, Z-phase (NbCrN), and Laves phase. The appearance sequence of different precipitates determines the level of crack initiation and propagation. For example, during the stabilizing annealing stage, NbC has a high correlation with reducing the susceptibility to intergranular cracking of the material. During the welding and PWHT stage, a synergistic process occurs wherein Cr₂₃C₆ formation in the HAZ takes place, along with the annihilation of NbC due to the significant temperature gradient present in the local area. As transferring to the service stage with in temperature range of approximately 400°C, the formation of Fe-rich α , Cr-rich α' , and G-phase occurs due to ferrite spinodal decomposition [10]. The effect of each phase is different; for example, the formation of Cr₂₃C₆ and sigma phase has no contribution to strengthening because they accumulate along the grain boundaries, providing nucleation sites for voids, serving as a driving force for crack initiation, and contributing to the carbon-free process [17, 18] (for example, the carbon cannot combine with Nb during solidification or stabilizing annealing). Conversely, the effects of other types of precipitation have not been clearly defined. The precipitation size and distribution significantly affect microstructure evolution, and differences in grain size, precipitation fraction, and precipitation type variation dictate the degradation of mechanical properties, which can be located in a specific region identified as the HAZ.

In addition to the aforementioned challenges, it is imperative to assess the impact of residual stress measurement on the overall evaluation process. There are several methods available for residual stress measurement, including non-destructive, semi-destructive, and destructive methods. However, the choice of method is limited by the component size and degree of destructiveness that can be allowed [19]. (The Non-Destructive methods includes: Barkhausen noise method, X-ray diffraction method, Neutron diffraction method and Ultrasonic method; Semi-Destructive methods includes: Hold-drilling technique, Ring-core method and Deep-hole method; the last category is Destructive which includes Sectioning technique and Contour method [19]). Even using the same component, two different residual stress levels can be obtained by measuring through two different methods, which can cause fluctuations in the data. In addition, the inaccurate results can be caused by calculation method or human error. Due to failure time usually can reach more than several thousand hours, experiments that exactly mimic the failure process are time-consuming and not feasible. Therefore, accelerated experiments are often employed for SRC lab tests, where a 100% or 90% yield stress level is usually used as the applied stress level to shorten the experiment time. However, serious problems can generate due to this, like shown in Figure. Simulation-1. After adding applied force to accelerate the progress, the lifetime time range is in hours level or even shorter. Despite the challenges outlined earlier, the data presented in Figure 3.1 demonstrates a C-curve trend, which is a key objective of this study. Figure 3.1 illustrates the relationship between service temperature, failure time, and applied stress levels in various alloy systems. Instead of a linear relationship between working temperature and lifetime, the C-curve trend reveals a minimum lifetime that can be achieved within the map. The turning point for each line, also known as the C-curve nose, provides the lower limit of the failure time across a broad range of service temperatures. This phenomenon is also observed in some long-term creep experiments. This phenomenon is also observed in some long-term creep experiments. The reason that C-curve map has become a significant research goal and is being pursued through simulation models is because it covers a wide temperature range, a variety of applied stress levels, and different alloy systems in a single map. Consequently, guidance

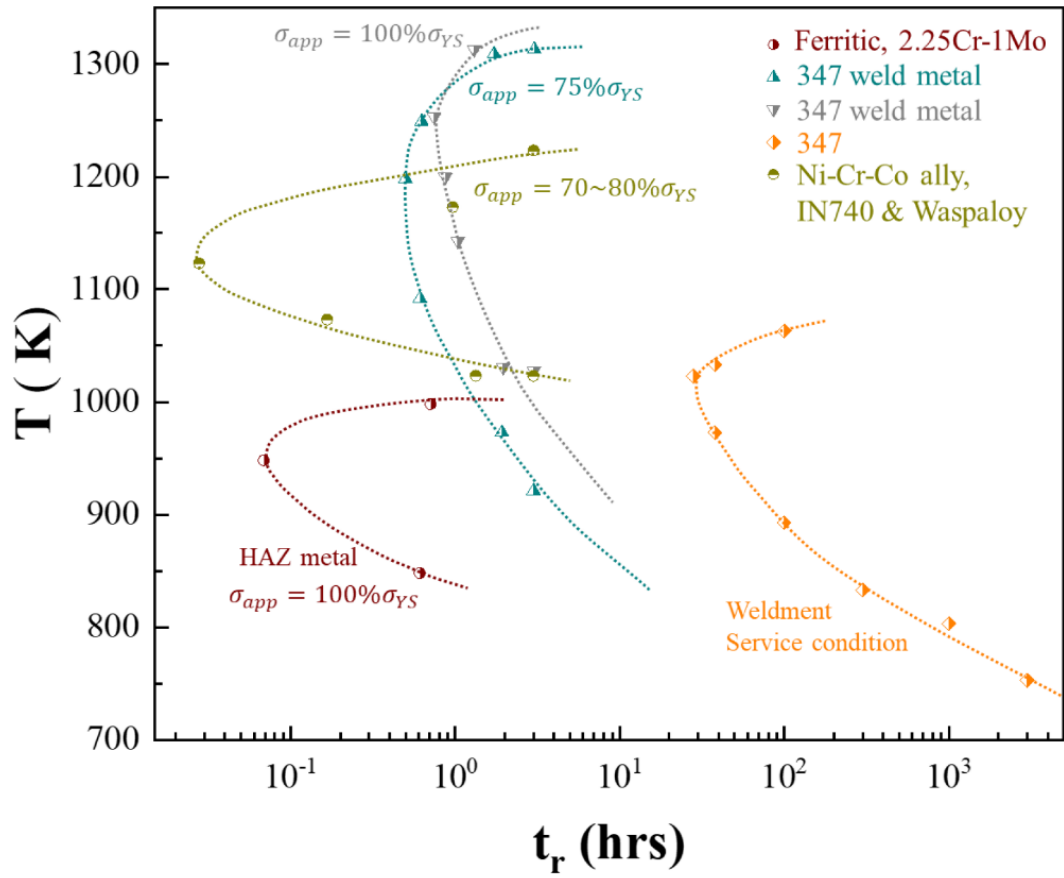


Figure 3.1. C-curve map for different materials of relationship between lifetime and temperature at different applied stress.

can be provided based on this C-curve map to select the optimal working conditions for a specific material.

Figure 3.2 provides a comprehensive estimation of the residual stress relaxation and lifetime distribution of a component during different stages. The thermal history of the component is shown in (a), where four different temperatures are selected to generate the initial relationship of influence on the residual stress reduction. The estimation of residual stress reduction during PWHT is shown in (b), and the comparison can be made with (c), which shows the failure time under different service temperature. The estimation indicates an inverse relationship between service temperature and lifetime, where high temperature preferentially leads to a longer lifetime. Finally, the predicted microstructure evolution is shown in (d), where the emergence of the precipitation-free zone (PFZ) makes an influence on the failure path. By comparing the simulation results with experimental data at each step, the unrevealed behavior and underlying mechanisms can be investigated. This "master curve" can be used as a predictive tool for optimizing the working conditions of the component, and further experiments can be conducted to validate the simulation results.

3.2 Modeling methods

The failure process in austenitic stainless steel weldments, specifically in 347H steel, is a complex phenomenon influenced by multiple factors that work synergistically. Although several mechanisms may contribute to the failure process, only two dominant mechanisms - creep failure and diffusional creep failure - are significant in specific working conditions. Weldments service cracking can manifest in four types of failure, as illustrated in Figure A.1. [11, 20]. Type I and II failures occur in the weld metal, with the former arrested in the weld metal and the latter preferentially propagating into the HAZ and BM. Type III failure forms in the coarse-grained heat-affected zone (CGHAZ) and may propagate into the base metal, whereas Type IV occurs in the fine-grained heat-affected zone (FGHAZ) or intercritical heat-affected zone (ICHAZ). The failure type observed in 347H weldments is Type IV, indicating that intergranular creep fracture is the dominant failure mechanism. Moreover, diffusional creep failure may prevail when the working temperature is high, and the applied stress is low [21]. However, unexpected

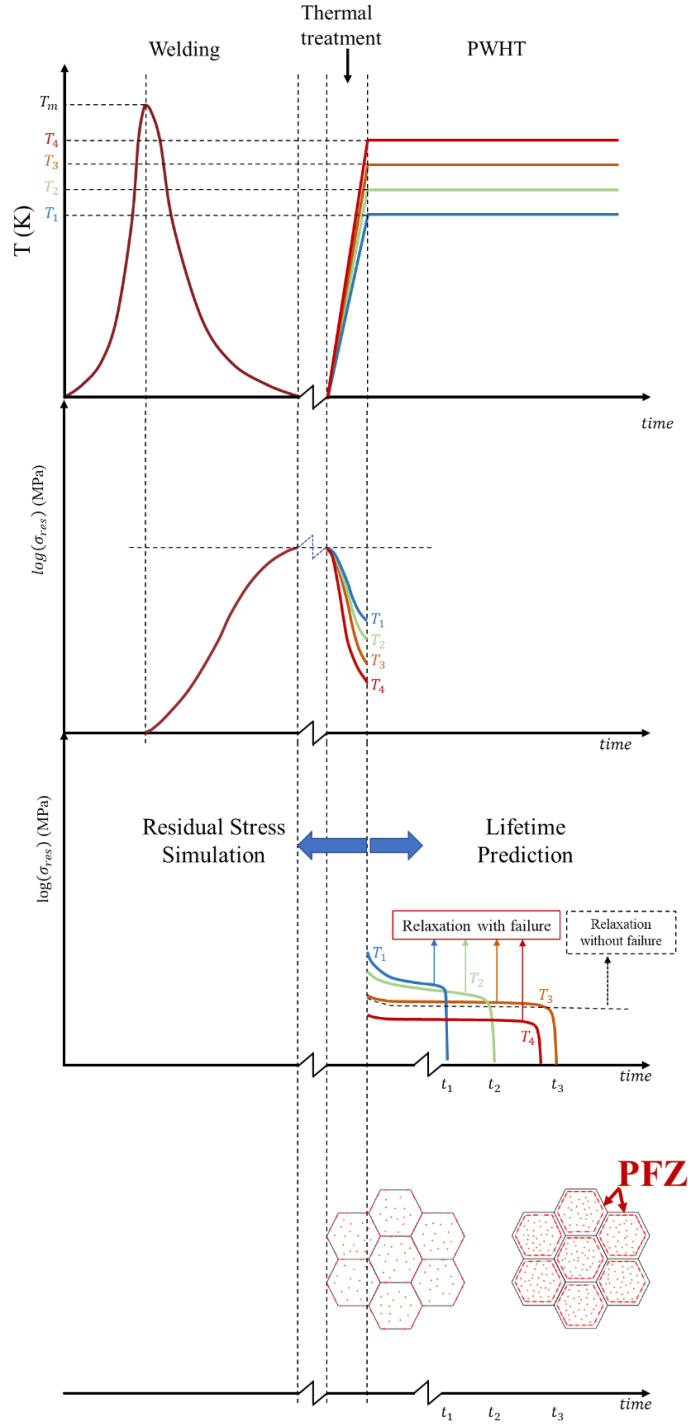


Figure 3.2. a) thermal history of component; (b) residual stress generation during welding procedure and reduction during reheating process (or PWHT heating stage); (c) residual stress variation during aging (or serving condition); (d) microstructure change at different steps.

changes in the working environment can alter the working conditions. Therefore, to develop a reliable simulation model that considers the main contributors to the failure process is necessary. The entire process should be divided into three steps: welding simulation to generate the temperature field, thermal-mechanical analysis to obtain the residual stress field, and failure model to predict lifetime with different microstructure setups. This approach can reduce fluctuations and errors that may arise when applying a single model.

3.2.1 Heat transfer analysis

As an initial step, the objective of the heat transfer analysis is to determine the temperature distribution within a three-dimensional model. This is critical in understanding the residual stress in weldments, which is induced by localized and dramatic temperature changes during the welding process. However, accurately capturing the temperature field is challenging due to the difficulty in selecting a heat source model that can precisely describe the melting pool shape. The accuracy of the temperature field distribution is closely related to the heat source model, which affects the temperature gradient in adjacent elements. Therefore, the investigation of melting pool shape is crucial, and previous researchers have conducted studies in this area. Rosenthal developed a quasi-stationary heat transfer model in the 1930s using the following equation [22]:

$$\frac{\delta^2 T}{\delta X^2} + \frac{\delta^2 T}{\delta Y^2} + \frac{\delta^2 T}{\delta Z^2} = \rho C \frac{dT}{dt} \quad (1)$$

However, it has been demonstrated that Rosenthal's model contains significant errors in the temperature distribution in or near the FZ and HAZ, with the error rate gradually increasing for infinite temperature and material thermal properties around the heat source. To address these issues, Pavelic developed a new Gaussian model based on Rosenthal's model, known as the "disc" model, which improves the accuracy of the heat source model by distributing heat input over a source area rather than a point, resulting in a significant improvement compared to Rosenthal's model. Another heat source model developed by Goldak et al. is the double ellipsoid heat source model, capable of simulating cases lacking radial symmetry [23, 24]. This model can be easily adjusted for size and shape to match different welding methods, allowing it to simulate both shallow and deep

penetration cases. The two ellipsoidal sources can be divided into a front half and a rear half, each corresponding to two different quadrants of an ellipsoid. The schematic drawing of the double ellipsoidal model is shown in Figure A.2.

The front and rear quadrant power density distribution can be expressed as follows:

$$q(x, y, z, t) = \frac{6\sqrt{3}f_f Q}{abc\pi\sqrt{\pi}} e^{-3x^2/a^2} e^{-3y^2/b^2} e^{-3[z+v(\tau-t)]^2/c^2} \quad (2)$$

$$q(x, y, z, t) = \frac{6\sqrt{3}f_r Q}{abc\pi\sqrt{\pi}} e^{-3x^2/a^2} e^{-3y^2/b^2} e^{-3[x+v(\tau-t)]^2/c^2} \quad (3)$$

f_f : the fraction of the heat deposited in the front.

f_r : the fraction of the heat deposited in the rear.

a, b, c can have different values for both front and rear quadrant.

Using a reliable heat source model, the temperature field can be generated in the three-dimensional plate model with dimensions of 100mm*15mm *60mm as shown in Figure S-c. This model not only simulates the welding process but also able to mimic the post-weld heat treatment (PWHT) procedure, enabling the calculation of residual stress reduction accordingly.

3.2.2 Thermomechanical analysis

The temperature field obtained from the heat transfer model serves as input for generating the residual stress field. The essence of using simulation model to predict the lifetime in weldments is to obtain an accurate residual stress magnitude and its distribution. Residual stress is generated due to localized mismatch of thermal expansion coefficient. Although phase transformation and microstrain variation caused by lattice parameter changes occur during thermal treatment, their effects can be ignored due to the magnitude difference compared with that of the thermal expansion coefficient mismatch. The residual stress value in each location can be obtained through the thermal histories from each element, which also indicates the level of residual stress reduction. As mentioned earlier, the PWHT temperature has a significant impact on residual stress relaxation, both in terms of magnitude and distribution. The PWHT procedure aims to regenerate a residual stress field, reducing the dramatic changes in the local area and creating a more homogeneous microstructure. The importance of residual stress calculation lies in employing the

corresponding thermal-mechanical parameters in the simulation model. Table 1 lists the thermal-mechanical related parameters used in the simulation. The equations for calculating the relationship between temperature variation and residual stress reduction percentage are listed below:

$$\frac{\Delta T}{T} = \frac{T_{PWHT} - RT}{T_{PWHT}} \quad (4)$$

$$\sigma_{reduction} = \frac{-(\sigma_{PWHT} - \sigma_{As-welded})}{\sigma_{As-welded}} \quad (5)$$

3.3 Two-dimensional failure model

The failure mechanism observed in this study is Type IV intergranular creep fracture. This type of failure typically occurs in the heat-affected zone (HAZ) at grain boundaries where different length scale precipitations serve as nucleation sites for voids, as confirmed through microstructure examination. The detailed failure control mechanism and the process of void nucleation, cavity growth, and coalescence are demonstrated as follows.

Plastic deformation in the grain interior is primarily dominated by dislocation (power-law) creep, while elastic deformation is also involved. Hence, the total strain can be calculated from two separate components, as shown in the equations below:

$$\dot{\epsilon}_{ij} = \dot{\epsilon}_{ij}^e + \dot{\epsilon}_{ij}^p \quad (6)$$

$$\dot{\epsilon}_{ij}^e = \frac{1+\nu}{E} \left(\dot{\sigma}_{ij} - \frac{\nu}{1+\nu} \dot{\sigma}_{kk} \delta_{ij} \right) \quad (7)$$

$$\dot{\epsilon}_{ij}^p = A_{dis} \frac{EbD_1}{k_B T} \left(\frac{\sigma_e}{\sigma_0} \right)^n \frac{3S_{ij}}{2\sigma_e} \bar{M}^I \quad (8)$$

ν and E are Poisson's ratio and Young's modulus respectively, δ_{ij} represents the Kronecker delta; k_B is the Boltzmann's constant; b is the Burgers vector; T is the absolute temperature; $\sigma_e = \sqrt{3S_{ij}S_{ij}/2}$ is the von Mises stress; n is the stress exponent; σ_0 is the reference stress; A_{dis} is the material constant; $D_1 = D_{01} \exp(-Q_1/RT)$ is the diffusivity coefficient for the dislocation creep. And the normalized micromechanical Taylor factor for the i_{th} grain $\bar{M}^I = M^I / \max(M^1, M^2, \dots, M^I, \dots, M^N)$, where N is the total number of grains within the polycrystalline model. $\bar{M}^I$ here is introduced by Raabe et al. to serve as

Table 1. (a). Material parameters used for heat transfer analyzing model; (b). 347H stainless steel Young's modulus and Poisson's ration variation with temperature; (c). 347H stainless steel thermal expansion coefficient variation with temperature; (d). 347H stainless steel yield strength and plastic strain variation as temperature changes.

Heat Transfer analyzing model input parameters	
Mass Density (g/mm ³)	7.96E-06
Melting Temperature (K)	1719
Latent Heat (J/mol)	40000
Solidus Temperature (K)	1671
Temperature (K)	Conductivity (W/(mK))
300	0.0134
700	0.0208
1200	0.0335
1600	0.0381
1700	0.0762
Temperature (K)	Specific Heat (J/kg•K)
300	372.2
700	397.3
800	409.8
1200	476.7
1400	493.5

(a)

Table 1. Continued

Young's Modulus (GPa)	Poisson's Ratio	Temperature (K)
195.1	0.27	294
190.3	0.27	366
186.2	0.29	422
182.7	0.31	477
177.9	0.32	533
174.4	0.31	589
171.0	0.30	644
166.2	0.28	700
162.0	0.26	755
153.1	0.24	866
143.6	0.23	977
131.7	0.22	1089
116.5	0.22	1173
100.0	0.22	1273
80.0	0.22	1373
57.0	0.22	1473
30.0	0.22	1573
2.0	0.22	1673

(b)

Table 1. Continued

Expansion coefficient α ($10^{-6}/\text{K}$)	Temperature (K)
15.4	311
15.6	339
15.8	366
16.0	394
16.1	422
16.4	450
16.6	477
16.8	505
17.0	533
17.1	561
17.3	589
17.4	616
17.6	644
17.7	672
17.8	700
18.2	873
18.5	973
18.8	1073
19.1	1173
19.4	1273
19.7	1373
20.0	1473
20.7	1693
20.7	1733

(c)

Table 1. Continued

Yield Strength (MPa)	Plastic Strain	Temperature (K)
241.0	0.00	294
290.0	0.01	294
393.0	0.05	294
483.0	0.10	294
621.0	0.20	294
827.0	0.40	294
827.0	0.80	294
117.0	0.00	693
193.0	0.01	693
276.0	0.05	693
359.0	0.10	693
483.0	0.20	693
621.0	0.40	693
724.0	0.80	693
96.5	0.00	923
145.0	0.01	923
214.0	0.05	923
283.0	0.10	923
379.0	0.20	923
448.0	0.40	923
483.0	0.80	923
68.9	0.00	1089
110.0	0.01	1089
145.0	0.05	1089
172.0	0.10	1089
207.0	0.20	1089
241.0	0.40	1089
241.0	0.80	1089
27.6	0.00	1366
38.6	0.01	1366
41.4	0.05	1366
41.4	0.10	1366
41.4	0.20	1366
41.4	0.40	1366
41.4	0.80	1366
5.0	0.00	1550
5.0	0.01	1550
5.0	0.05	1550
5.0	0.10	1550
5.0	0.20	1550
5.0	0.40	1550
5.0	0.80	1550

(d)

the slip anisotropy provoked by crystallographic orientation of each grain, which is defined as the ratio of the local accumulated shear $\sum \gamma^{local}$ and local von Mises strain $\langle \varepsilon_{VM}^{local} \rangle$ [25]. A simplified spherical-caps model is employed to represent the cavities, with a radius of a , half-spacing of b and cavity tip angle $\varphi = 75^\circ$ [26]. The cavity volume V and spherical-caps shape parameters h is calculated using following equations:

$$V = \frac{4}{3} \pi a^3 h(\varphi) \quad (9)$$

$$h(\varphi) = \frac{[(1+\cos\varphi)^{-2} - \frac{1}{2}\cos\varphi]}{\sin\varphi} \quad (10)$$

Due to the difficulty of modeling each individual cavity on the grain boundaries, Rice proposed a smeared-out representation to simplify the problem [27]. This enables the replacement of discrete distributed cavities with continuous varying separation in the grain boundary. The separation and separation rate equations are listed below:

$$\mu_n = V/(\pi b^2) \quad (11)$$

$$\dot{\mu}_n = \frac{\dot{V}}{\pi b^2} - \frac{2V\dot{b}}{\pi b^3} \quad (12)$$

Based on the pioneering work of Needleman and Rice [28], the contribution to the volumetric cavity growth rate $\dot{V}$ is decoupled into two parts including the contribution of grain boundary diffusion $\dot{V}_1$ and the effects of creep deformation $\dot{V}_2$ which are defined respectively as followed:

$$\dot{V}_1 = 4\pi D \frac{\sigma_n - (1-f)\sigma_s}{\ln\left(\frac{1}{f}\right) - \frac{1}{2}(3-f)(1-f)} \quad (13)$$

$$\dot{V}_2 = \begin{cases} \pm 2\pi \dot{\varepsilon}_e^c a^3 h(\Psi) [\alpha_n \left| \frac{\sigma_m}{\sigma_e} \right| + \beta_n]^n, & \text{for } \left| \frac{\sigma_m}{\sigma_e} \right| > 1 \\ 2\pi \dot{\varepsilon}_e^c a^3 h(\Psi) [\alpha_n + \beta_n]^n \frac{\sigma_m}{\sigma_e}, & \text{for } \left| \frac{\sigma_m}{\sigma_e} \right| < 1 \end{cases} \quad (14)$$

Here σ_s represents the sintering stress, $\alpha_n = 3/(2n)$ and $\beta_n = (n-1)(n+0.4319)/n^2$, f is the effective cavity volume fraction given by

$$f = \max \left[\left(\frac{a}{b} \right)^2, \left(\frac{a}{a+1.5L} \right)^2 \right] \quad (15)$$

L is a material length scale dependent on stress and temperature able to demonstrate the combination of diffusion and creep deformation to the cavity growth [29] which can be described as follow:

$$L = \left[D \frac{\sigma_e}{\dot{\epsilon}_e^c} \right]^{1/3} \quad (16)$$

When L is much larger than a , the effect of creep deformation became trivial. In comparison, the impact of grain boundary diffusion on the cavity growth is decreasing as L turns smaller. In equation (16), D is the grain boundary diffusion parameters defined as follows:

$$D = \frac{D_{b0}\delta_b\Omega}{k_B T} \exp\left(\frac{-Q_b}{RT}\right) \quad (17)$$

Q_b is the activation energy in grain boundary diffusion, $D_{b0}\delta_b$ is the grain boundary diffusion coefficient, Ω is the atom volume. Hence, the cavity growth rate can be derived as

$$\dot{a} = V/(4\pi a^2 h(\Psi)) \quad (18)$$

As one of the competing mechanisms on creep deformation and damage of ferritic steel weldments [20, 30], grain boundary sliding behaves in a Newtonian viscous manner [31] and is essentially a thermally activated process, governed by

$$\dot{\mu}_t = \frac{\Omega \eta_0 \exp(-Q_{gbs}/RT)}{k_B T} \sigma_t \quad (19)$$

Here $\dot{\mu}_t$ is the relative sliding velocity of adjoining grains initiated by the shear stress σ_t in the grain boundary, η_0 is a characteristic sliding velocity, Q_{gbs} is the activation energy for the grain boundary sliding.

Based on the constitutive law outlined above, Wei et al., proposed a mechanism-based finite element method to model high temperature deformation behavior [11]. This behavior was simulated separately for the grain interior and grain boundary through two subroutines. Specifically, the elastic deformation and dislocation creep in the grain interior were defined using a user-defined material (UMAT) subroutine implemented in ABAQUS. Meanwhile, the grain boundary cavitation and grain boundary sliding (GBS) were modeled using a rate-dependent cohesive element through a user-defined element (UEL) subroutine. Building upon their previous work, a two-dimensional digital model containing microstructure features was created to compare various microstructure settings with the actual microstructure evolution. Three different cases were simulated by gradually adding microstructure features, beginning with only the base metal case. This allowed the

simulation results to be fitted to NIMS experimental data. Subsequently, the fusion zone and HAZ were included to decrease the gap between the simulation model and actual morphology, due to the magnitudes of thermal-mechanical properties differing among the BM, HZA, and FZ. However, experimental data for the HAZ and FZ, which govern the lifetime of service components, is rarely available. Therefore, an experienced estimation was introduced to generate initial parameters for the FZ and HAZ, with the lifetime in the BM being 10 times longer than that in the HAZ.

Finally, the precipitation free zone (PFZ) was incorporated into the model, which is related to the microstructure feature of the late service stage of the service component. Precipitation concentration leads to strengthening of the grain interior, while the dissipation of particles at grain boundaries results in the formation of PFZ and weakening of grain boundaries. This behavior plays a crucial role in the initiation and propagation of cracks during the late service stage. To simulate these cases, digital microstructures with 50, 166, and 750 grains were established separately with random crystallographic orientations and material properties assigned through UMAT subroutine, as illustrated in Figure 3.3. The boundary conditions of the simulation model were established to compare with the actual service situation, with fixed left and right edges and free expansion at the bottom and top surfaces. The simulation case of the 50-grain base metal is shown in (b), with different colors representing different grains. Subsequently, the HAZ was symmetrically added to the model, as shown in (c), with the PFZ highlighted in yellow in (d), showing the grain boundary PFZ and grain interior with different colors representing distinct material properties. One of the crucial steps in the 2D-failure model is to create different microstructure setups, while another step is to apply the residual stress field correctly. By employing Goldak's double ellipsoidal heat source model, the temperature and melting pool shape can be replicated with high fidelity compared to real welding processes. The temperature field obtained through this method can then be used to generate the residual stress field after the welding procedure and after exposure to different PWHT temperatures. The thermal-mechanical properties, failure mechanisms, and microstructure are highly dependent on temperature and stress field variation. Therefore, it is important to determine

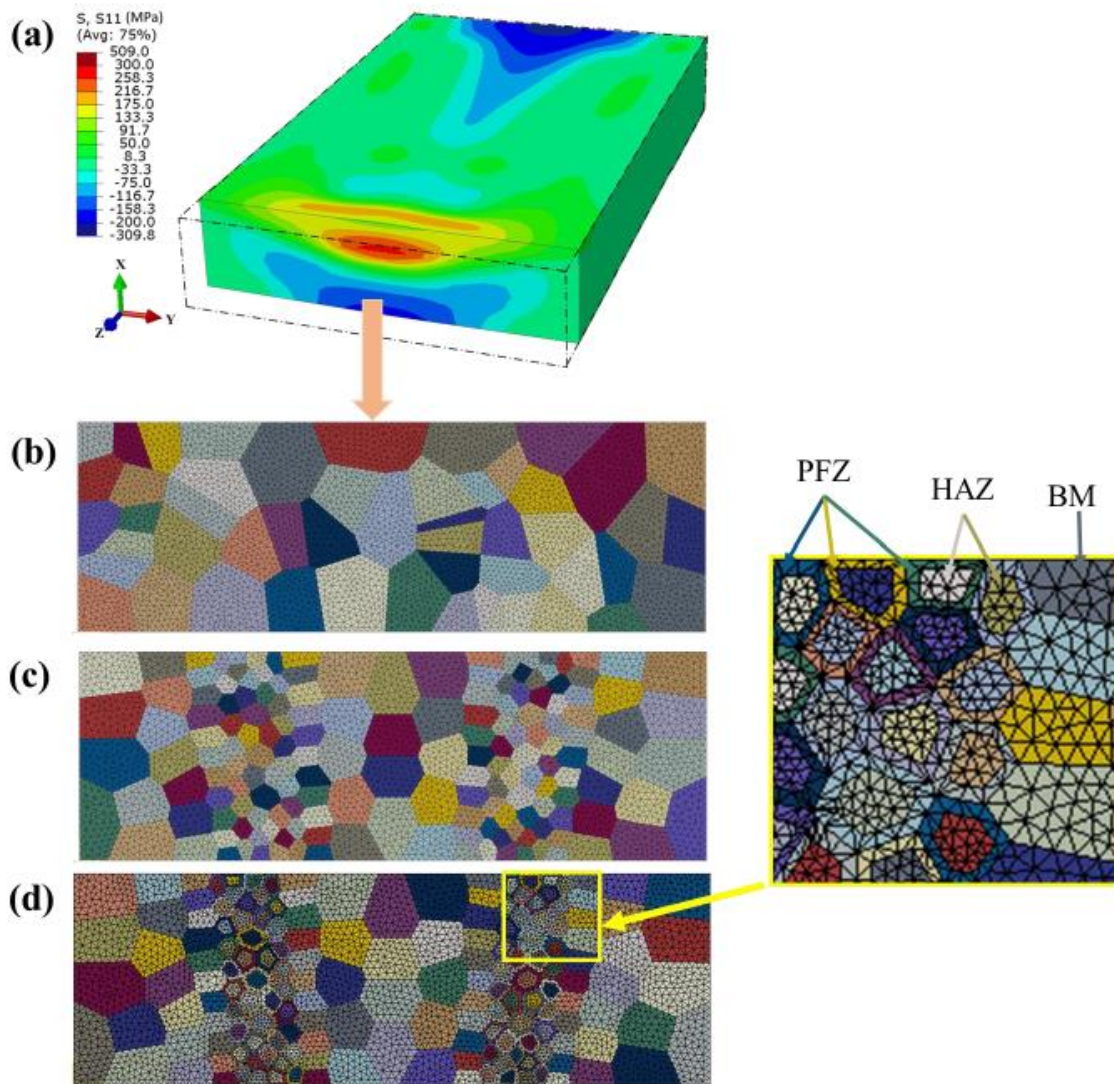


Figure 3.3. (a) Two-dimensional cross-section selection for failure analysis; (b) Base metal two-dimensional failure model setup; (c) Two-dimensional failure model contains base metal (BM), heat-affected zone (HAZ) and fusion zone (FZ); (d) Two-dimensional failure model add precipitation free zone (PFZ) based on (c).

the distribution of the residual stress field and the influence brought by PWHT temperatures.

A cross-section located at the midpoint of the three-dimensional plate was selected to extract the residual stress field, perpendicular to the longitudinal direction which is also the heat-source moving direction. A quarter section of the 3D model is shown in Figure 3.4. (a) to demonstrate the melting pool and the relationship of each direction. Figure 3.4. (b)-(i) shows the residual stress field under different thermal treatments, with (b) as the after welding case, and PWHT temperatures ranging from 723K to 1023K in 50K intervals. The residual stress values can be read from the legend color bar. As the PWHT temperature increases, the residual stress gradually decreases from the highest value of 450MPa in the after welding case to ~160MPa in the case exposed to PWHT at 1023K. The highest compressive residual stress is found in the after welding case at -340MPa.

The residual stress reduction percentage in each simulated temperature can be calculated and plotted, as shown in Figure 3.5. The fitting curve indicates a linear relationship, suggesting that as the PWHT temperature increases, the residual stress reduction percentage increases accordingly. A slope can be obtained from the fitting results, and the residual stress reduction percentage at other temperature variation rates can be predicted approximately. The governing reason for residual stress generation is raised from thermal expansion coefficient mismatch. The prediction on the residual stress field distribution for both welding process and different PWHT procedure were calculated based on this.

The 2D-failure model aims to simulate the failure process of components in the service stage after PWHT. To achieve this, the microstructure setup needs to combine the residual stress field to perform the prediction. The three different microstructure setups have varying grain sizes and distributions. Previous research has shown that HAZ accumulates the highest residual stress under various working conditions. Therefore, in the 2D-failure model, it is crucial to ensure that the highest residual stress is located in the HAZ. During welding, the weld zone is typically in a V-shape or X-shape to avoid bending or warpage behavior, which can cause further stress concentrations. Consequently, the FZ and HAZ are not perfectly perpendicular to the plate top and bottom surface, and a specific

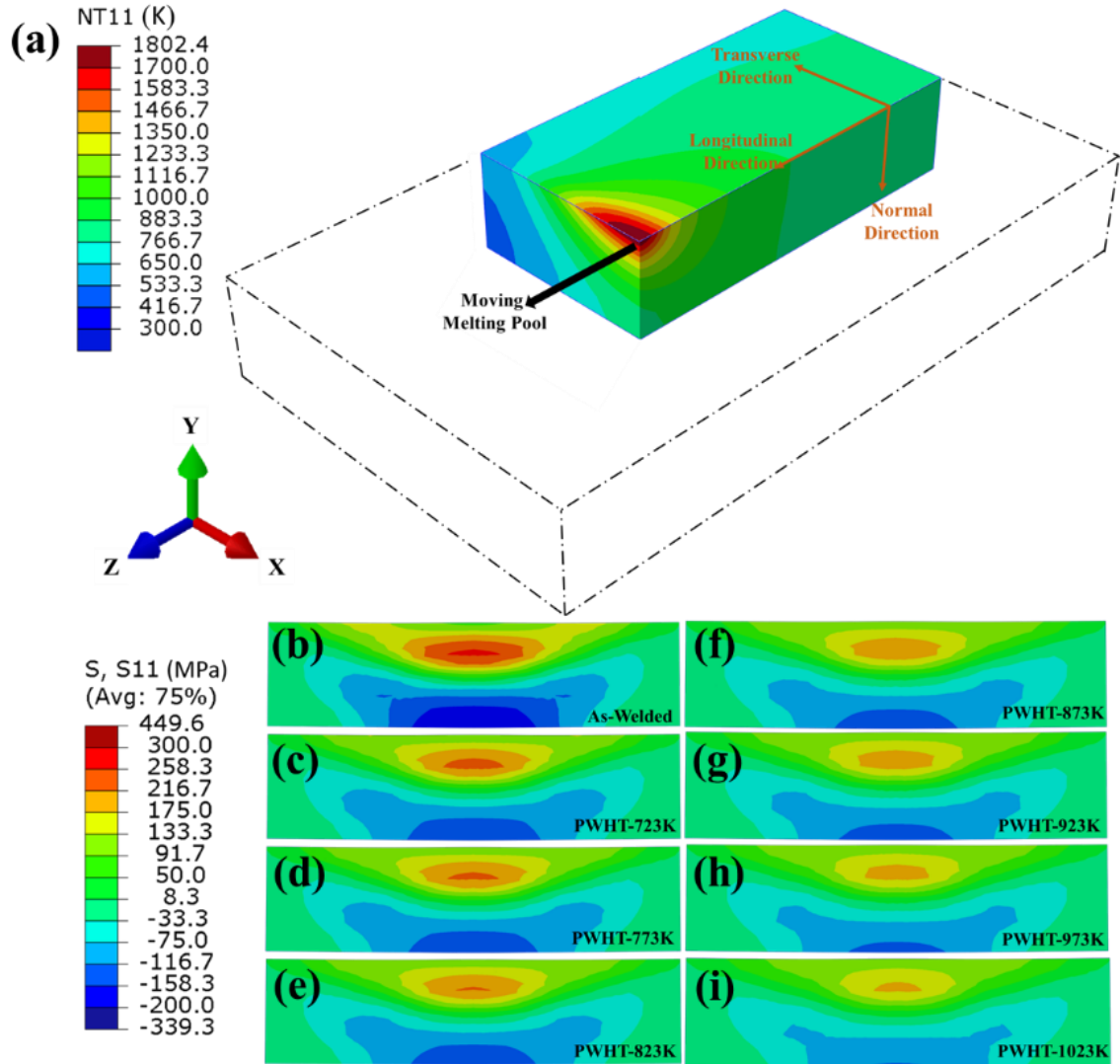


Figure 3.4. (a) Three-dimensional simulation model for mimic welding process, heat source moving direction is Z, (dark red area is melting pool); (b)-(e) Residual stress distribution of model cross-section after welding process and PWHT at 723K, 773K, 823K, 873K, 923K, 973K and 1023K for 3 hours.

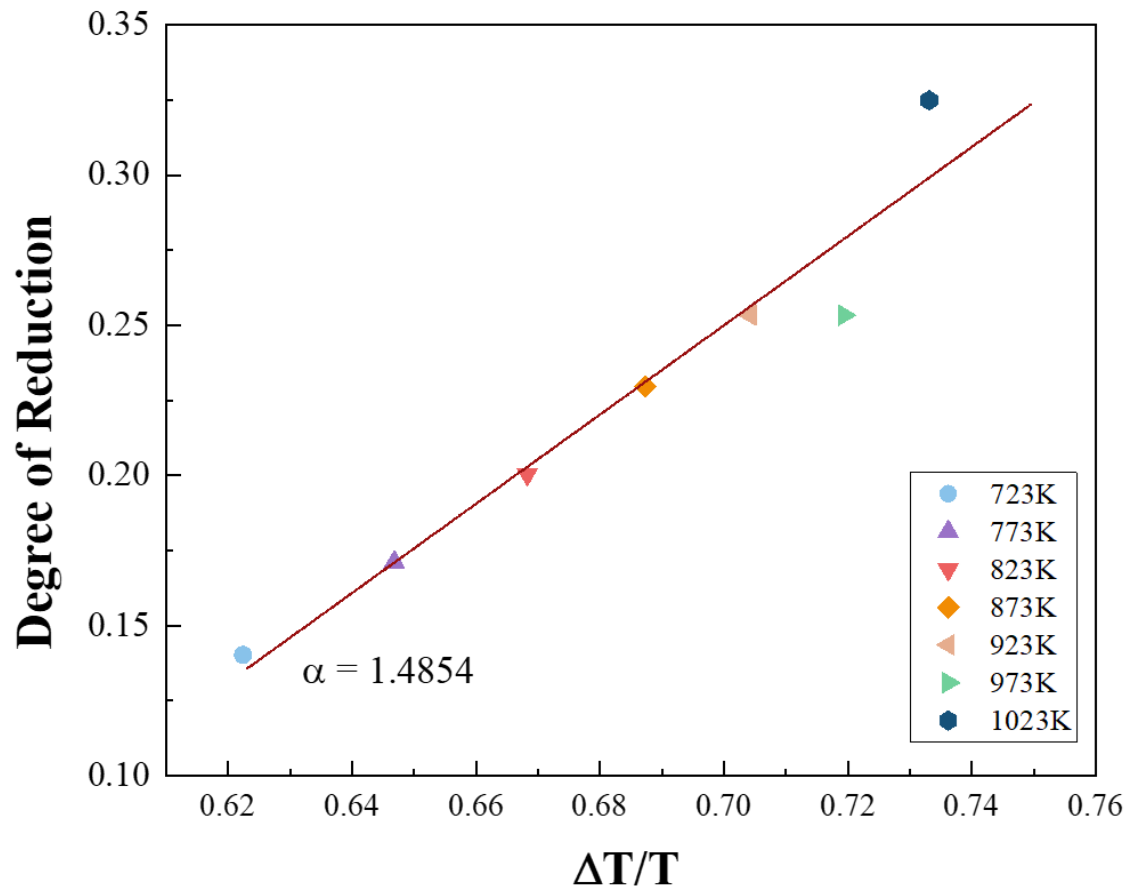


Figure 3.5. Relationship between degree of residual stress reduction and different PWHT temperature.

angle of approximately 50°~70° is usually applied to flatten the weldments. To simulate the real residual stress field distribution, a user-subroutine-defined initial stress field (SIGINI) is introduced using the following equation:

$$\sigma_r = y_0 + \frac{A}{\left(w \times \sqrt{\frac{\pi}{4 \times \ln(2)}}\right)} \times \exp(-4 \times \ln(2) \times (x - x_c)^2 / w^2) \quad (20)$$

Where y_0 , A , w and x_c are set as 50 MPa, 500MPa, 10.399 and 14.12543 respectively, they are parameters used to determine the value of residual stress. In specific computation, the basic form in equation (20) will be adjusted to relocate the highest residual stress to HAZ and regulate its magnitude.

3.4.1 Experimental data fitting

The reliability of lifetime prediction in different microstructures is based on the accuracy of base metal material parameters used in simulation model. Before deducing the material parameters for HAZ, it is necessary to compare the simulation results with creep test results on the base metal. Figure 3.6 shows the results of the creep test for base metal at four different temperature levels, which have been fit well by adjusting several main control factors and considering other factors as shown in Table 2. However, due to the limited creep data available for 347/347H stainless steels in the NIMS database, it is necessary to extrapolate the results for other temperatures using the creep and Arrhenius equations. The main influence factors, such as stress exponent n , cavity nucleation parameter F_n , grain boundary diffusivity D , grain boundary viscosity etc., were determined using the Arrhenius equation $\ln k = \ln A + \frac{-E_a}{RT}$, (k is rate constant, A is frequency factor, E_a is activation energy, R is gas constant, T is temperature in Kelvin) and the formatted creep rate relation with applied stress $\frac{\dot{\epsilon}_p}{\dot{\epsilon}_0} = \left(\frac{\sigma}{\sigma_0}\right)^n$. The comparison between the experimental discrete data and the simulation line connected data shows a good correlation between them. The experimental data also revealed two different slopes, which correspond to creep and diffusion failure mechanisms. The slope change occurs at a low stress level, as the lifetime reaches 10000 hours, especially for 973K case. The slope of experimental discrete data for $\log(\sigma) - \log(t_r)$ is approximately equal to -1, which indicates the

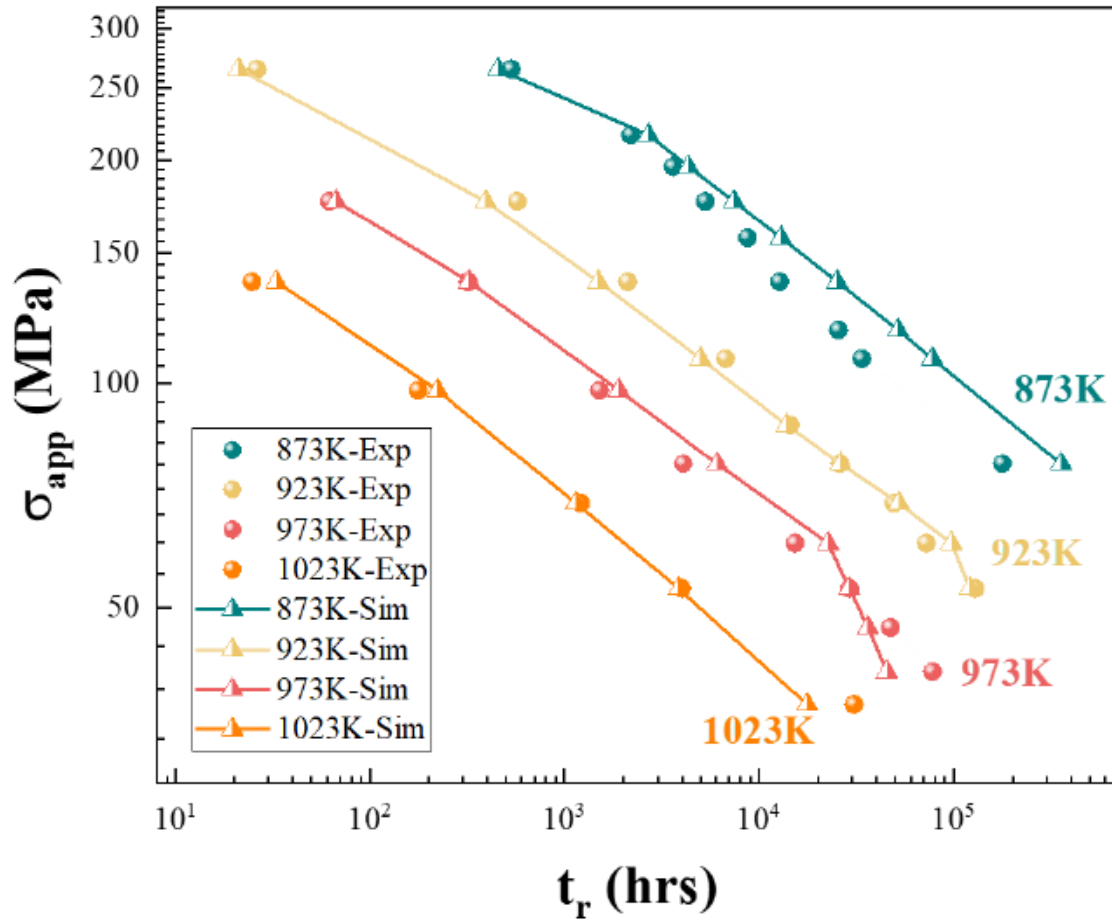


Figure 3.6. NIMS actual experimental data (rounded discrete points) of 347 SS creep test at 873K, 923K, 973K and 1023K under different applied stress fit with simulation results (triangular line connected results).

Table 2. Representative values for material parameters in base metal (BM) at different temperatures.

Parameter	873 K	923 K	973 K	1023 K
Reference stress (MPa)	142	142	142	142
Stress exponent, n	6.25	7.65	8.32	5.44
Grain size (10^{-3} mm ³)	5	5	5	5
Cavity nucleation parameter (10^{-8})	1.5	6	6	6
Heterogeneity factor	1	1	1	1
Reference stress for nucleation law (MPa)	100	100	100	100
Poisson's ratio, ν	0.3	0.3	0.3	0.3
Young's modulus, E (GPa)	146.6	142.0	137.3	132.6
Diffusion parameter, D	1.10×10^{-21}	5.33×10^{-21}	2.19×10^{-20}	5×10^{-20}
Grain boundary viscosity	5.81×10^{14}	1.20×10^{14}	2.93×10^{13}	8.2×10^{12}
Reference strain rate	1.416×10^{-9}	1.159×10^{-8}	6.099×10^{-8}	1.405×10^{-6}

lifetime is inversely proportional to the diffusional creep rate. This agrees with the conclusion of Hull and Rimmer that creep failure is purely controlled by atom diffusion type-induced cavity growth under low applied stress. As the applied stress decreases, the rupture time increases, and a no failure case will occur when the residual stress decreases to a critical value that is not available in real experiments [32-35].

3.4.2 Two-dimensional simulation results

Currently, the microstructure setting has three possible cases, each of which leads to failure due to the nature of cavity growth. Nevertheless, while a non-failure is not feasible in reality, it is achievable through simulation by governing the mechanism with pure creep deformation. As such, the lifetime of the material increases as residual stress relaxes without cavity nucleation and coalescence. Henceforth, the non-failure case also been incorporated into computation to facilitate comparison with the other three microstructure settings. Simulation results presented in Figure 3.7 depict the relationship between applied stress (residual stress) and failure time at testing temperatures of 873K and 1023K, as shown in (a) and (b), respectively. Moreover, temperature effects can also be determined by comparing the figures (a) and (b). The two simulation temperatures produce similar trends, as the lifetime under identical conditions keeps decreasing gradually with the addition of microstructure features, from Only BM without cavity, Only BM, With HAZ, to the final With HAZ & PFZ case. At the simulation temperature of 873K, the Only BM case has a failure time of 1.353×10^9 hours, which decreases to 3.332×10^8 hours in the With HAZ case, and further reduces to 1.682×10^8 hours in the With HAZ & PFZ case. The distinction between these four cases at 873K is difficult to perceive from the figure. Conversely, the testing temperature of 1023K yields a noticeable deviation in each case, with the Only BM case exhibiting a lifetime of 4.514×10^7 hours, the With HAZ case 3.541×10^6 hours, and the With HAZ & PFZ case 6.889×10^5 hours, respectively.

The influence of microstructure variation is evident, with the addition of more microstructure features accelerating the failure process and increasing the creep rate. The comparison of the four microstructure cases indicates that the microstructure gradient is

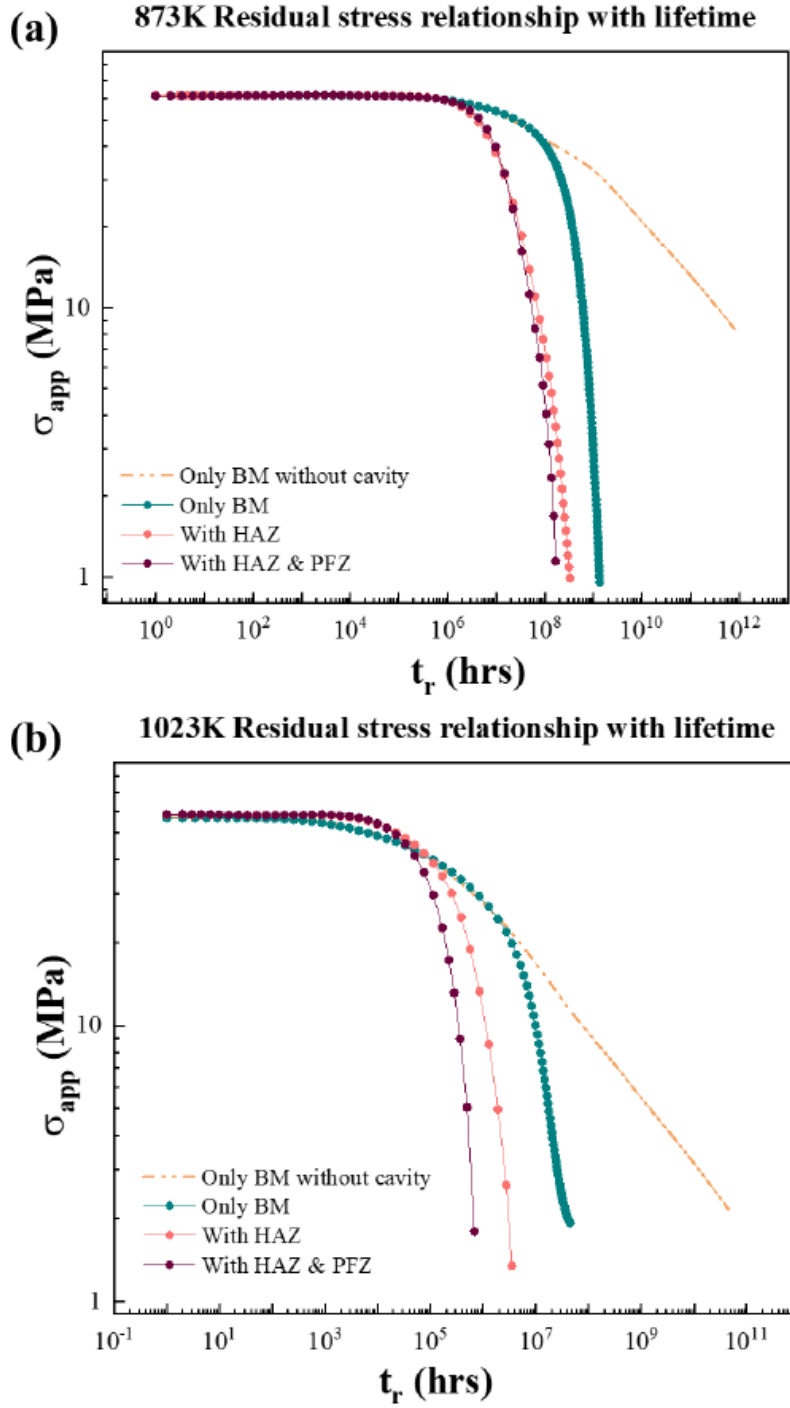


Figure 3.7. Residual stress variation in simulation models with different microstructure setting: only base metal without adding cavity into calculation; only base metal adding cavity into calculation; based on previous case, adding HAZ, final case is adding extra PFZ into calculation. Simulation calculated at four different temperatures separately, (a) is 873K and (b) is 1023K.

responsible for shortening the failure time. Despite the enhancement of mechanical properties through precipitation strengthening particle formation (NbC), the degrading impact caused by the widening of PFZ becomes more pronounced with increasing service time. The competitive relationship between these two factors is time-dependent and influenced by the service temperature. As NbC migrates into the grain interior and the volume fraction of PFZ increases, the thermal-mechanical properties difference on the grain boundary enlarges. The contribution of other types of precipitations (e.g., $M_{23}C_6$, G-phase, Z-phase etc., which preferentially formed at GB) accelerates the voids formation, coalescence, and crack propagation process. Meanwhile, the simulation prediction trend aligns with the common understanding of the ten-fold difference in the lifetime between BM and HAZ. Therefore, the material parameters for HAZ can be determined based on the fitted material parameters data set for BM in Table 2. As shown in Table 3, the material parameters for HAZ are presented at different temperatures.

In section 3.3 of the three-dimensional thermal-mechanical simulation, the residual stress relaxation rate was defined as α . To generate the C-curve map for 347H stainless steel using simulation data, the definition of α was inherited from chapter 2, with adjustments made as shown in equation (21) listed below:

$$\sigma_{res}^{magnitude}(T_{reheating}) = \sigma_{res}^{magnitude}(T_{ref}) \times \left(1 - \alpha \frac{T_{reheating} - T_{ref}}{T_{ref}}\right) \quad (21)$$

T_{ref} is the reference temperature, in this calculation, T_{ref} is room temperature. $T_{reheating}$ is gradually increasing from 873K to 1023K with a 50K interval. Here α set as the parameter reflecting the reduction rate of residual stress and it can be adjusted in a range. In this work, two values of α , 0.1 and 0.4, are used as the upper and lower limits of the reduction rate range. A C-curve map is constructed based on the material parameters combination for four different microstructure settings. Figure 3.8 shows the C-curve map for 347H stainless steel under three microstructure settings with different applied residual stress levels. As predicted, the base metal case with a higher α value promises a longer lifetime under the same working conditions. This observation is consistent with previous estimations, which suggest that increasing the residual stress and adding microstructure features lead to a significant decrease in failure time, from $\sim 10^5$ hours level to $\sim 3 * 10^4$

Table 3. Representative values for material parameters in HAZ at different temperatures.

Parameter	873 K	923 K	973 K	1023 K
Reference stress	0.142 GPa	0.142 GPa	0.142 GPa	0.142 GPa
Stress exponent, n	6.25	7.65	8.32	5.44
Grain size	5×10^{-3} mm	5×10^{-3} mm	5×10^{-3} mm	5×10^{-3} mm
Cavity nucleation parameter	5×10^8	1.7×10^9	2.6×10^9	3.2×10^9
Heterogeneity factor	1	1	1	1
Reference stress for nucleation law	0.100 GPa	0.100 GPa	0.100 GPa	0.100 GPa
Poisson's ratio, ν	0.3	0.3	0.3	0.3
Diffusion parameter, D	4×10^{-20}	1.94×10^{-19}	7.96×10^{-19}	1.82×10^{-18}
Grain boundary viscosity	5.81×10^{13}	1.20×10^{13}	2.93×10^{12}	8.2×10^{11}
Reference strain rate	1.416×10^{-8}	1.159×10^{-7}	6.099×10^{-7}	1.405×10^{-5}

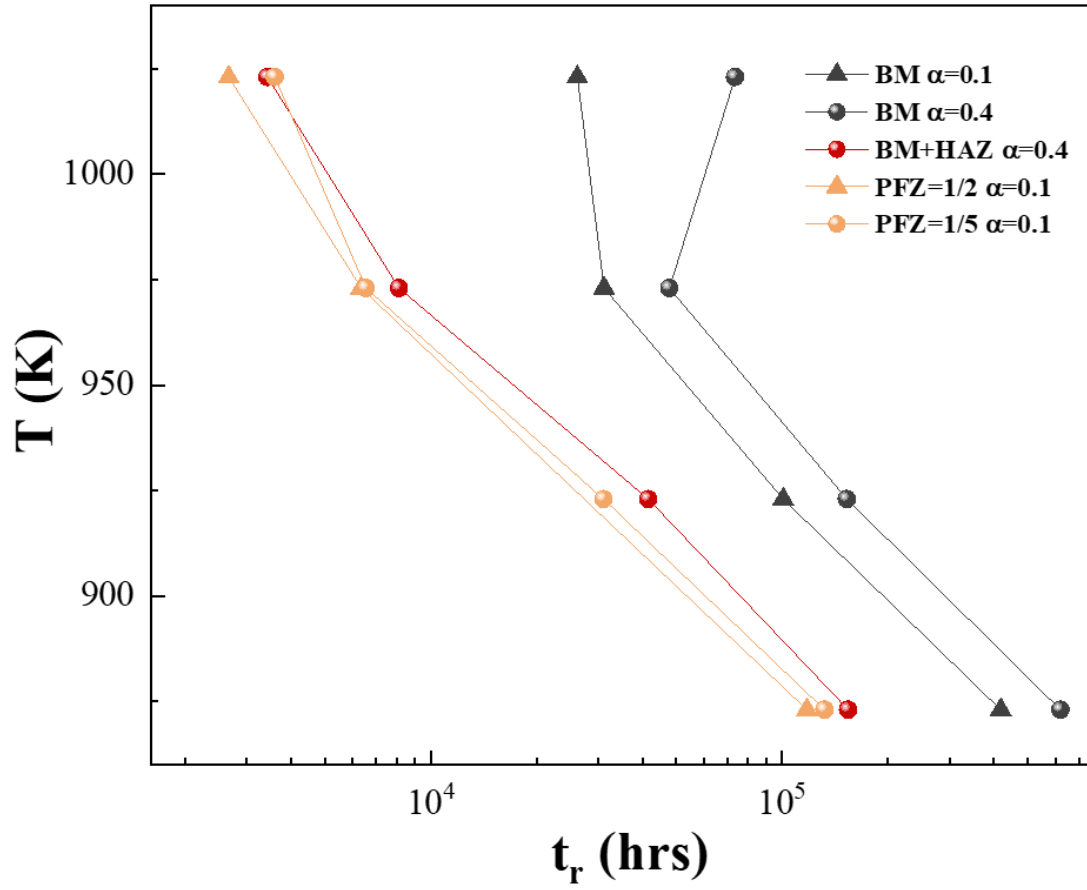


Figure 3.8. Relationship between lifetime and working temperature under different microstructure model setup (dark gray line represent the microstructure only contain base metal; orange line includes base metal and heat affected zone; red line contains base metal, heat affected zone and precipitation free zone).

hours, finally drop to the ~3000 hours with PFZ & HAZ. The testing temperature has no impact on this trend, even though the C-curve nose appears at a temperature level of ~973K. The width of the PFZ also plays a role in weakening the mechanical properties, as it increases the creep rate. The two different PFZ widths calculated in Figure 3.8 are $\frac{1}{2}$ and $\frac{1}{5}$ respectively, where the ratio of the PFZ represents the PFZ width over the radius of the grain. A larger value of this ratio indicates that the region without precipitation (NbC) is larger. The PFZ width of $\frac{1}{2}$ gives the shortest lifetime among these five conditions, which can be considered the upper limit of the PFZ width. The essence of the C-curve is its ability to provide the minimum lifetime across a wide temperature range. In Figure 8, two cases show the C-curve nose, which is consistent with experimental results. The turning trend indicates the reliability of the simulation models, as they not only choose the correct failure mechanism to predict lifetime but also include the microstructure evolution that contributes to locally thermal-mechanical properties variation.

3.4.3 Schematic prediction

The aim of this study is to generate a C-curve map that covers a wide temperature range and can be extrapolated to other alloy systems. Figure 3.9 illustrates a schematic drawing of the residual stress evolution process, starting from welding, solidifying, PWHT, and service period until final failure. The residual stress is generated after the heating process explained the starting point is not in the origin. The non-uniform residual stress accumulation, coupled with microstructure evolution, determines the residual stress field distribution during cooling. Residual stress reduction caused by PWHT and service at four different temperatures is shown in various colors. Previous predictions and estimations based on thermal-mechanical properties suggest that as the simulating temperature increases from mid to high-temperature range, the lifetime will decrease. However, considering the influence of microstructure, a similar trend like the C-curve can be developed in Figure 3.9. A comparison between the simulated temperature at 1023K and 1373K reveals that, although the lifetime is expected to be shorter at 1373K due to the higher creep rate, the failure time is longer than the 1023K case due to the precipitation

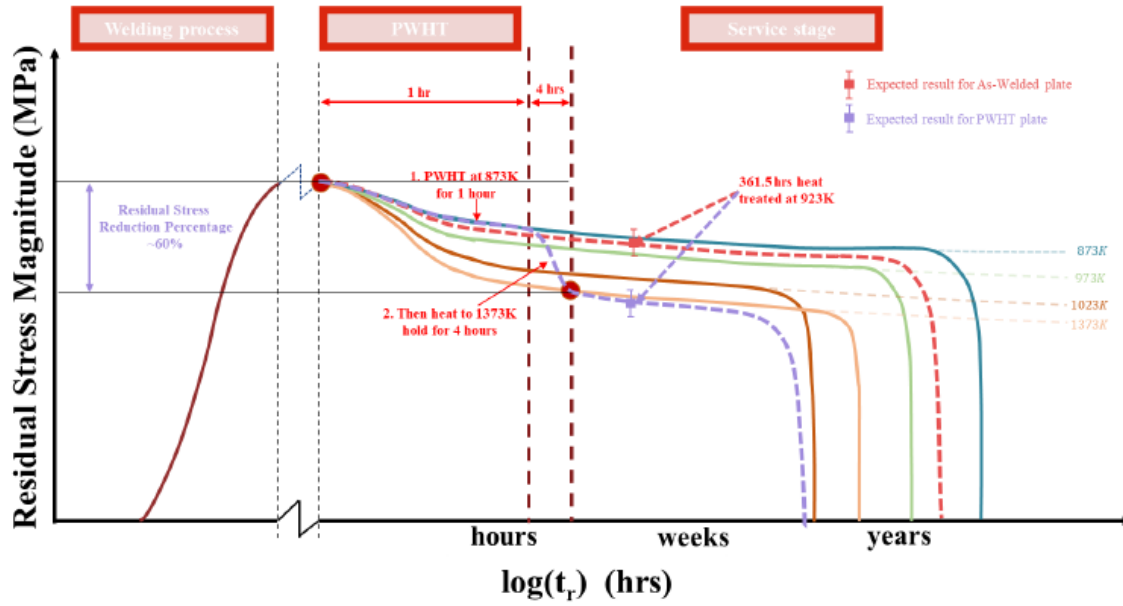


Figure 3.9. Schematic drawing of the relationship between lifetime and residual stress level. Dotted line is the residual stress variation trend for the case without cavity adding into calculation, solid line is the case adding cavity into simulation model. Both cases are embedded with creep in the simulation model.

annihilation/regeneration temperature range and precipitation strengthening effect. The schematic drawing can predict the rupture time in a specific working condition and narrow down the range when a component experience stepped thermal treatment. Neutron diffraction experiments can be used to verify this estimation, as explained in a separate paper.

The C-curve schematic drawing in Figure 3.10 highlights two important factors that have a significant impact on the lifetime of 347H austenitic stainless steel. The first factor is the residual stress reduction rate, which has been discussed in previous sections. Due to a higher α promises a lower residual stress level, which can guarantee a longer lifetime. Therefore, as when $\alpha_1 > \alpha_2 > \alpha_3$, the line with the highest reduction rate in α_1 will provide the longest lifetime. This trend can be applied to a wide temperature range and has been validated through experiments on 347H stainless steel. The C-curve nose will appear as the consequence of impact from microstructure evolution.

The second factor is $\frac{\partial \sigma_{YS}}{\partial T}$, which reflects the yield strength degradation level as temperature increases. Due to the nature of the material thermal-mechanical properties, σ_{YS} is decreasing caused by grain sliding and more effortless happened diffusion with increasing temperature. The variation rate of $\frac{\partial \sigma_{YS}}{\partial T}$ can be used to identify the resistance of deformation in material. A higher value of $\frac{\partial \sigma_{YS}}{\partial T}$ within the same temperature range indicates a faster drop in yield strength, leading to higher susceptibility to crack formation and increased creep rate. In Figure 10, the three ratios have a relationship of $\left(\frac{\partial \sigma_{YS}}{\partial T}\right)_1 < \left(\frac{\partial \sigma_{YS}}{\partial T}\right)_2 < \left(\frac{\partial \sigma_{YS}}{\partial T}\right)_3$, with the $\left(\frac{\partial \sigma_{YS}}{\partial T}\right)_1$ case having the longest lifetime. With the similar theory as the residual stress reduction rate variation, the involvement of precipitation has influence on the relationship between service temperature and lifetime. The grain line with the ratio of $\left(\frac{\partial \sigma_{YS}}{\partial T}\right)_1$ shows the C-curve nose also. The schematic drawing in Figure 3.10 elucidates the evolution of thermal-mechanical properties and microstructure involvement in 347H austenitic stainless steel. The C-curve nose as the indicator can identify the

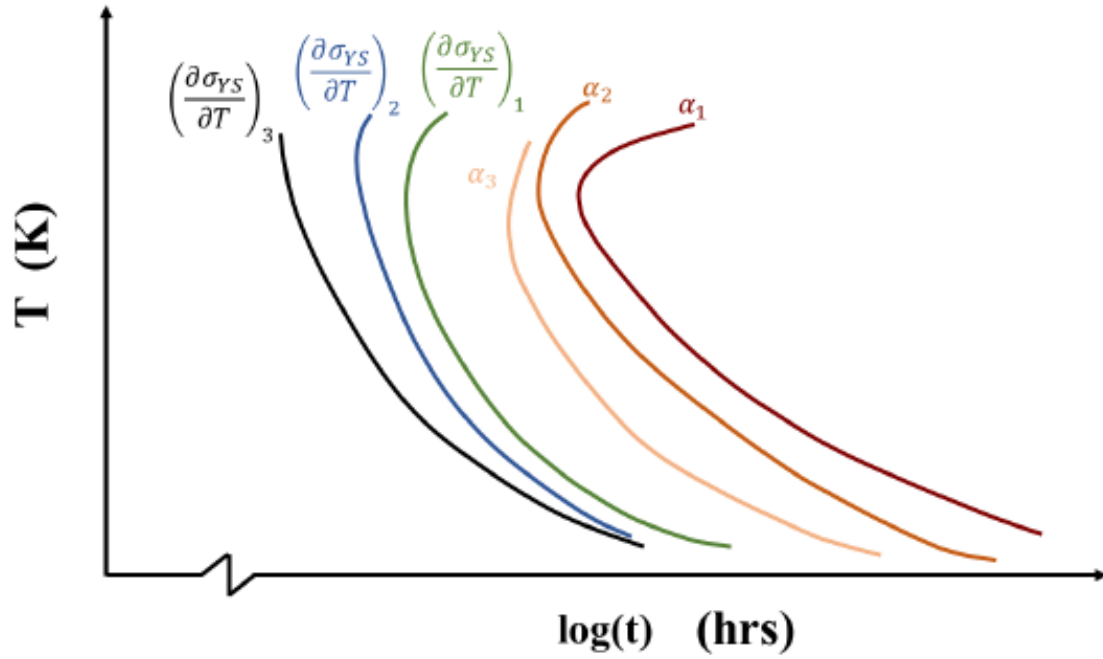


Figure 3.10. C-curve schematic drawing under different residual stress reduction rate $\alpha_1 > \alpha_2 > \alpha_3$ and different $\frac{\partial \sigma_{YS}}{\partial T}$ (σ_{YS} is the yield strength under different temperature. $\frac{\partial \sigma_{YS}}{\partial T}$ represents the yield strength variation rate at different temperatures).

governing factor transition happened in these service conditions, which can also be used as guidance to determine the minimum lifetime as shown in Figure 3.1.

3.5 Conclusion

Identifying the control failure mechanism and perform lifetime prediction consistently remains a difficulty for service weldments components due to the complex and synergistic process happened along the thermal treatment. With the unsuccessful path to track the impact level of each factor. A new comprehensive methodology using a series simulation models combined with accessible experimental results as validation tools is demonstrated in this work. Instead of considering each factor separately, this work brought a solution to predict the direction of this synergistic process. In this work, the study focuses on the 347H material parameters fitting with real creep data to generate a reliable baseline. Then using separate model to calculate the temperature and residual stress field distribution. In the meantime, the effects brought by the PWHT are also investigated. Finally, the microstructure features are gradually added from simple pure base metal to the complex case with PFZ. Hence, prediction on the failure life can be achieved through these series models. Validation on the residual stress field measurement and precipitation evolution are made through Neutron and Synchrotron diffraction experiments which will be in a separate paper.

CHAPTER FOUR

MICROSTRUCTURE CHARACTERIZATION

4.1 Atom probe tomography

4.1.1 APT mechanism and sample information

The SEM and TEM can give two-dimensional distribution of elements and precipitation. However, two-dimensional results is not sufficient to address the relationship between these particles' interaction and their relative position with the grain boundaries. Hence, here we are using APT to locate these features.

Atom probe tomography (APT) is one of the most spectacular microscopies that exist. A three-dimensional image at near atomic scale is produced with single-atom sensitivity where each atom (actually each isotope) in the image is identified. Because the fundamental data format is the 3D position and identify of individual atoms in a volume containing potentially hundreds of millions of atoms, many types of information may be gleaned. Elemental concentration may be determined in any subvolume size or shape simply by counting atoms. Concentration profiles may be calculated in any direction, even radially through a spherical feature or normal to any defined surface. Isoconcentration surface can be set to delineate and measure interfaces. Interatomic distribution functions can be determined for studying ordering, dopant interactions, cluster formation, crystal structure, diffusion, and early stages of precipitation. APT is used to study an increasingly wide variety of inorganic materials and is gradually demonstrating capabilities for analysis of organic materials as well. The unique power of APT lies in its ability to tie compositional information to structure. Other characterization techniques can determine physical structure at sub-nanometer levels, but only APT has the capacity for broad compositional identification (and quantification) at these length scales. Furthermore, APT accomplishes this with high analytical sensitivity which can reach one atomic part per million (appm). The basic theory of APT can be explained in the following equations:

$$qV = \frac{1}{2}mv^2 \quad (4.1)$$

$$\frac{m}{q} = \frac{2eVt^2}{L^2} \quad (4.2)$$

Figure 4.1 illustrates the operational mechanism of APT by demonstrating the distribution of nano-sized particles and their positional relationship. Additionally, it is crucial for this project to compare samples subjected to long-term thermal treatment with those subjected to short-term thermal treatment. Consequently, we present the outcomes of three samples: the fusion zone of the outer diameter of a thick pipe, the fusion zone of the inner diameter of a thick pipe, and the base metal region of a neutron as-welded plate [44].

4.1.2 Results of APT characterization

In Figure 4.2, the distribution of elements in the fusion zone of the outer diameter of the thick pipe is depicted. Notably, the carbon and niobium elements exhibit a dense distribution, forming NbC particles resembling needles in the sample. Simultaneously, Cr, Fe, Mn, and Ni gather abundantly in a particular region, which can be attributed to the grain boundary at present. While there is a significant overlap in the distribution of Nb and C, it is observed that P also tends to accumulate in the NbC sites. However, there is currently no reported evidence or explanation for this preference. Previous studies have reported the presence of Si in the NbC sites. However, in these APT results, Si is excluded from the atom maps due to its mobility on the APT needle surface and artificial Si enrichment in the APT atom maps, thereby avoiding confusion.

Figure 4.3 presents the percentage variation of each element as a function of distance along the needle position. The observed trends in the variation of Nb, C, and Fe indirectly support the proposition that these dense particles correspond to NbC, while nitrogen (N) also demonstrates a tendency to accumulate in the same specific location.

The sample subjected to the outer diameter experienced a relatively significant temperature change, whereas the inner diameter sample endured a relatively higher level of pressure compared to the outer diameter sample. Thus, Figure 4.4 displays atom maps illustrating the fusion zone of the inner diameter sample.

And also Figure 4.5 presents the percentage variation of each element as a function of distance along the needle position. A similar morphology is observed in the enrichment locations of Nb and C elements. However, a distinction arises in the overlap of vacant

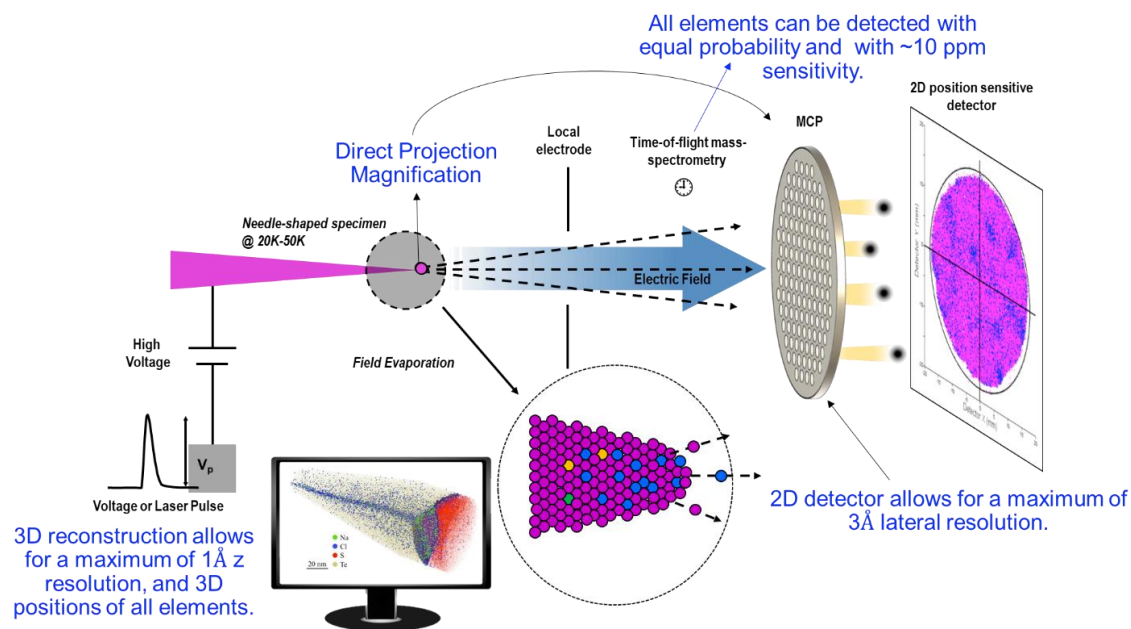


Figure 4.1. APT mechanism.

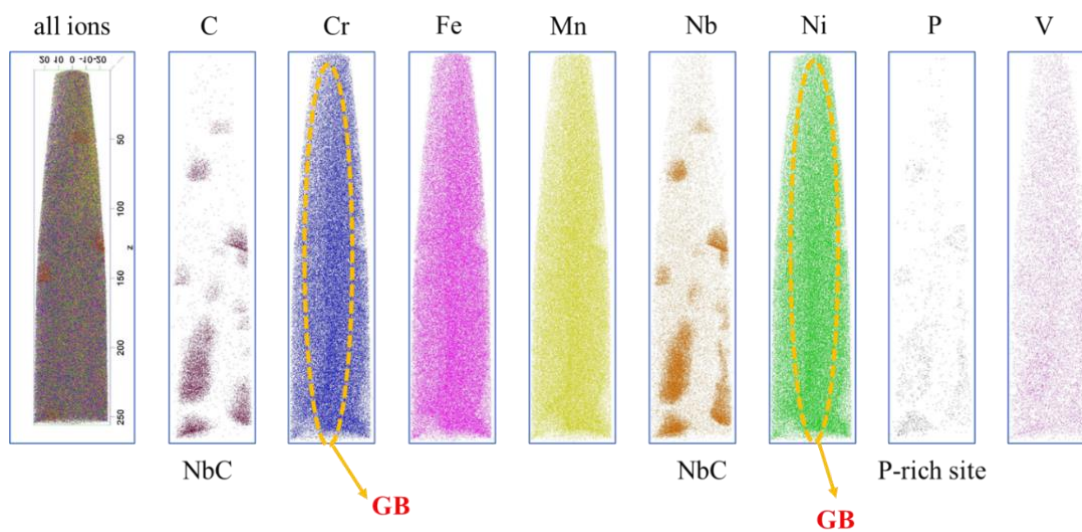


Figure 4.2. Thick pipe outer diameter FZ atoms map.

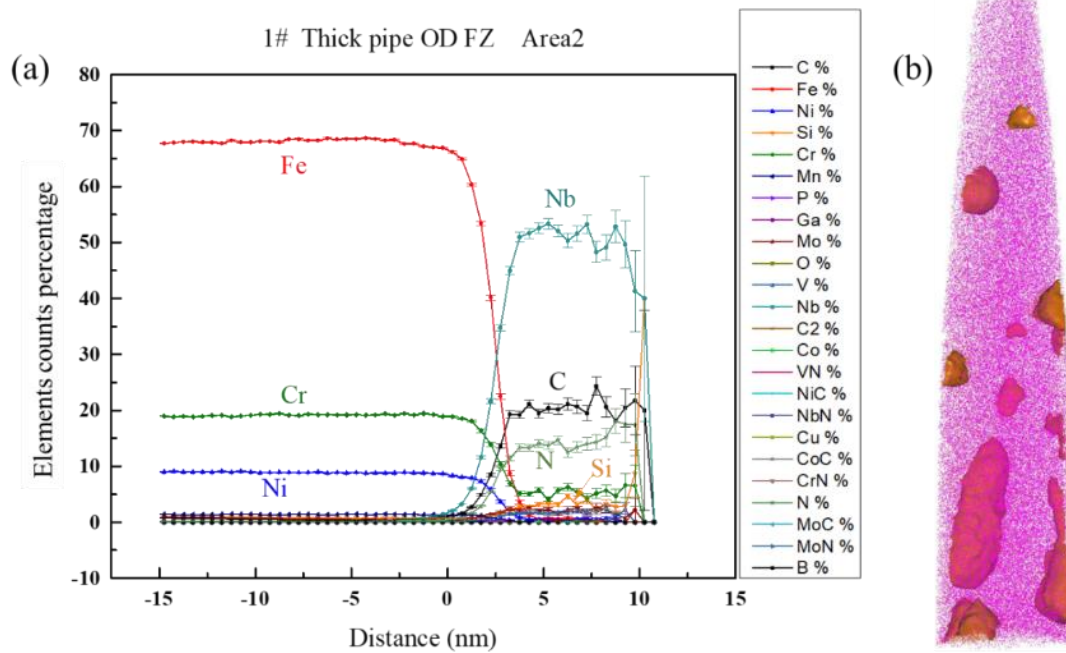


Figure 4.3. Thick pipe outer diameter FZ atoms percentage variation along the evaporation direction.

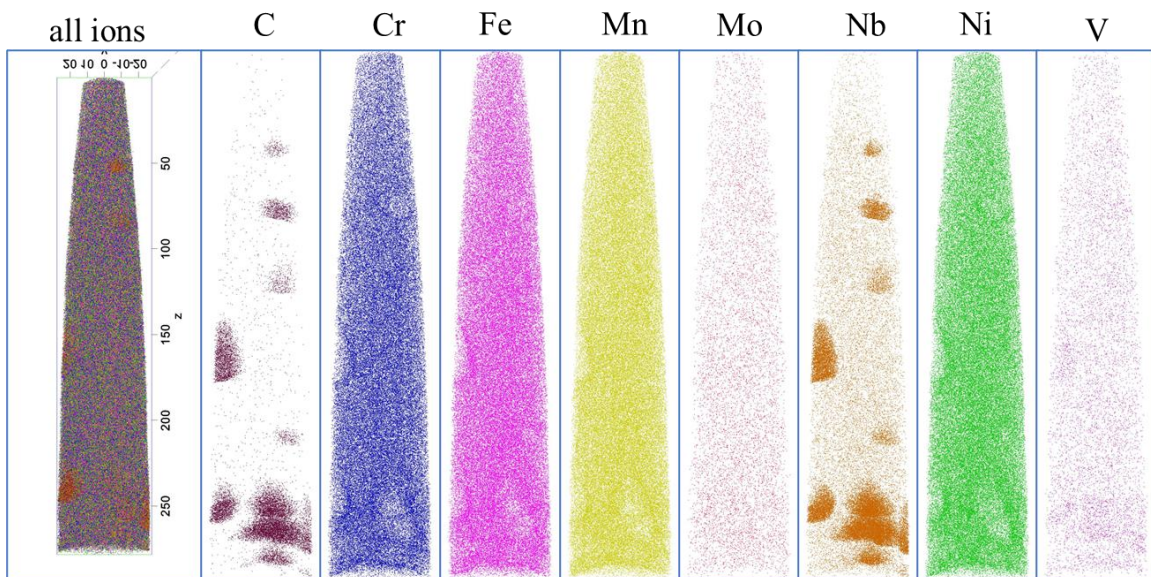


Figure 4.4. Thick pipe inner diameter FZ atoms map.

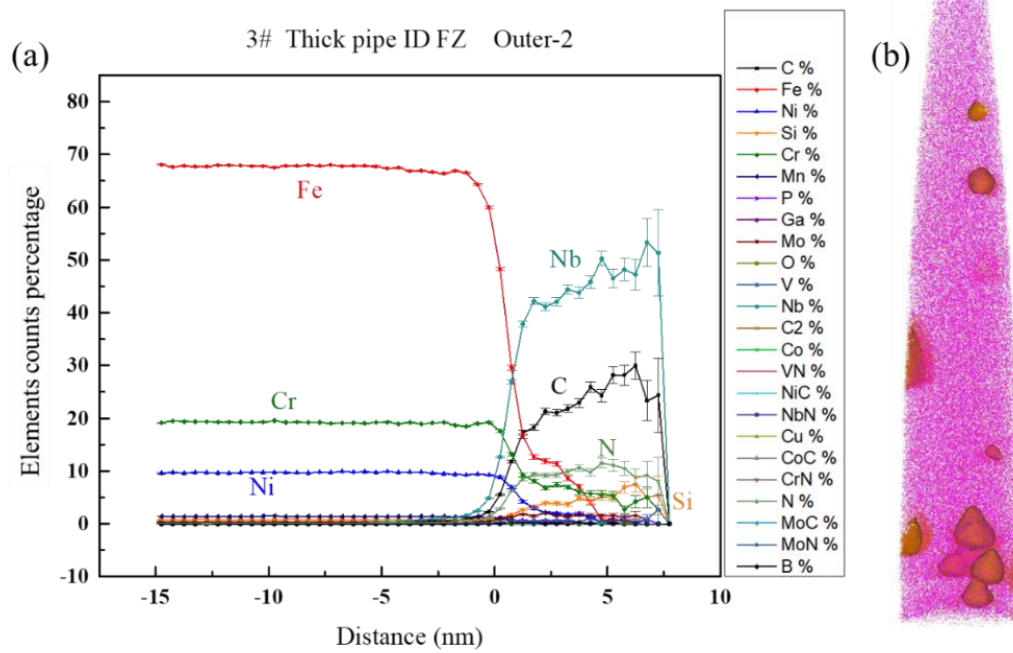


Figure 4.5. Thick pipe inner diameter FZ atoms percentage variation along the evaporation direction.

positions of Cr and Ni with the NbC sites. Furthermore, the distribution of element count percentages in the inner diameter sample resembles the findings from the outer diameter sample. In the service pipe, the detection of nano-sized NbC particles is relatively easy. This can be attributed to the fact that NbC is a precipitation that is sensitive to both time and temperature, requiring sufficient time for accumulation.

As a comparative analysis, Figure 4.6 presents atom maps of the As-Welded plate used for neutron diffraction residual stress measurement. This particular sample only underwent the welding process, and although locally dramatic temperature changes are essential for relaxation cracking of residual stress, they do not significantly contribute to the formation of nano-sized NbC particles. The uniform distribution of each element in Figure 4.6 serves as direct evidence of this.

4.2 Synchrotron diffraction

The precipitation evolution during thermal treatment is a crucial factor influencing the redistribution of residual stress fields. The changes in microstructure, characterized by the generation and annihilation of precipitates, contribute to local stress concentration, thereby affecting the redistribution of residual stress fields. While neutron diffraction is capable of accurately generating the residual stress field, it lacks the ability to track microstructure evolution. In contrast, synchrotron diffraction experiments provide opportunities for in-situ or ex-situ monitoring of precipitation evolution. However, given that failure occurs over an extended period, it is impractical to utilize synchrotron monitoring for certain types of precipitates that require long-term thermal treatment for generation. Hence, in this project, a combined approach involving both ex-situ and in-situ measurements is employed. In-situ measurements are used to monitor precipitation behavior at high temperatures (900 °C), which enhances the identification of each precipitation evolution behavior. Although the isothermal holding time is limited to 4 hours, it is sufficient to demonstrate the critical influence of temperature on the generation or annihilation of specific types of precipitates. If the temperature field does not meet the requirements, precipitates will not form or be annihilated. To elucidate the differences in microstructure evolution during isothermal holding, four distinct regions— BM, FZ, HAZ,

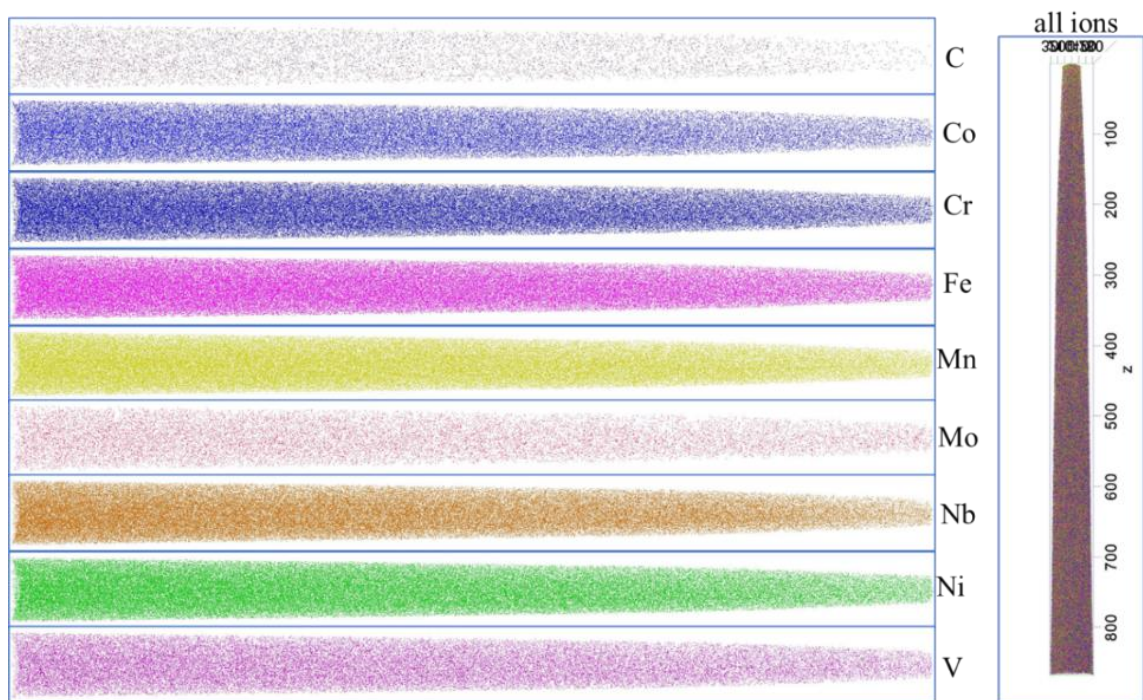


Figure 4.6. As-Welded plate BM atom map.

and $\frac{1}{2}$ HAZ $\frac{1}{2}$ FZ —were obtained by cutting the welded sheet into 2mm-thick, 6mm-diameter specimens. Furthermore, ex-situ synchrotron diffraction experiments are conducted to precisely identify different types of precipitation and calculate the reduction of residual stress resulting from thermal treatment. For the ex-situ measurements, 17 different thermal treatment conditions were applied to sheet samples. Given that the synchrotron penetration for 347H stainless steel is approximately 2.5mm, a 2mm-thick sheet is used as the ex-situ measurement sample to ensure data resolution.

4.2.1 In-situ measurement

Figure 4.7. (a) illustrates the thermal history of the in-situ measurement sample. To obtain the in-situ samples representing the four different regions, a sheet was welded to generate the FZ and HAZ. As depicted in (a), the samples were heated to the melting temperature before being cut into the desired regions. Subsequently, a heating rate of 10°C/min was applied to raise the sample temperature to 900°C, followed by an isothermal holding period of 4 hours. Finally, the sample was cooled down to room temperature at the same rate. Data acquisition commenced during the heating stage and continued until the sample returned to room temperature. A relatively high data acquisition rate (30s/point) was employed during the heating and cooling stages to capture rapid changes in temperature that could accelerate certain types of precipitates. This dense acquisition rate increases the likelihood of capturing the fast changes in precipitation. However, during the isothermal holding stage, where the holding time is 4 hours, a lower data acquisition rate (300s/point) is sufficient to track the variations, given the slower changes occurring during this period.

Figure 4.8 presents the in-situ measurement results for the base metal (BM). From the beginning of the measurement, both the matrix and NbC phases were detected. The heating and cooling stages did not reveal the generation of any other types of precipitation. Since the measurement region belongs to the base metal, where no microstructure gradient exists, this can explain why only peak shifting is observed in the scanning results, without the emergence or disappearance of peaks. The results for the HAZ and the $\frac{1}{2}$ HAZ $\frac{1}{2}$ FZ

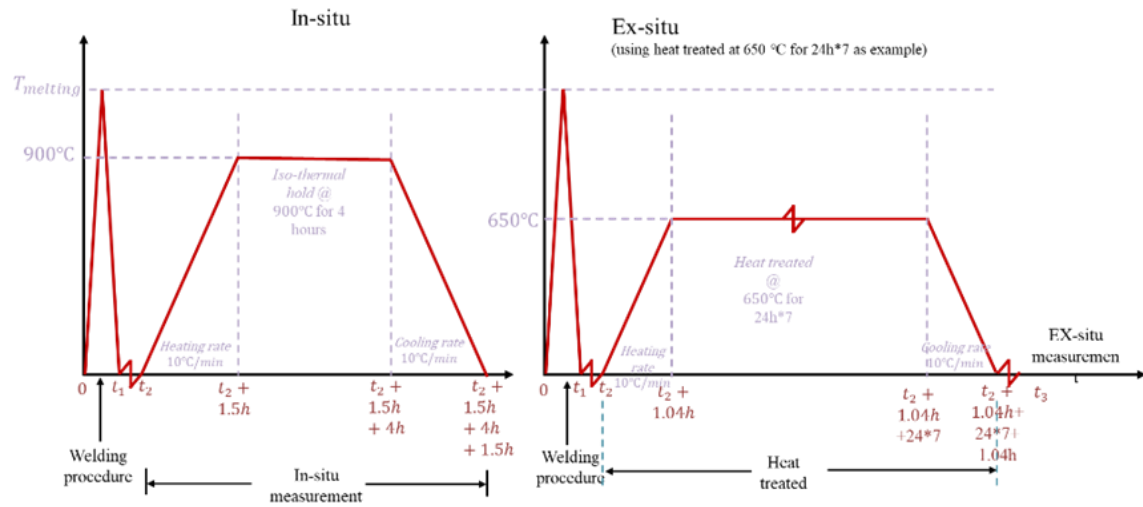


Figure 4.7. The in-situ and ex-situ sample thermal history, (a) in-situ sample thermal history; (b) ex-situ sample thermal history.

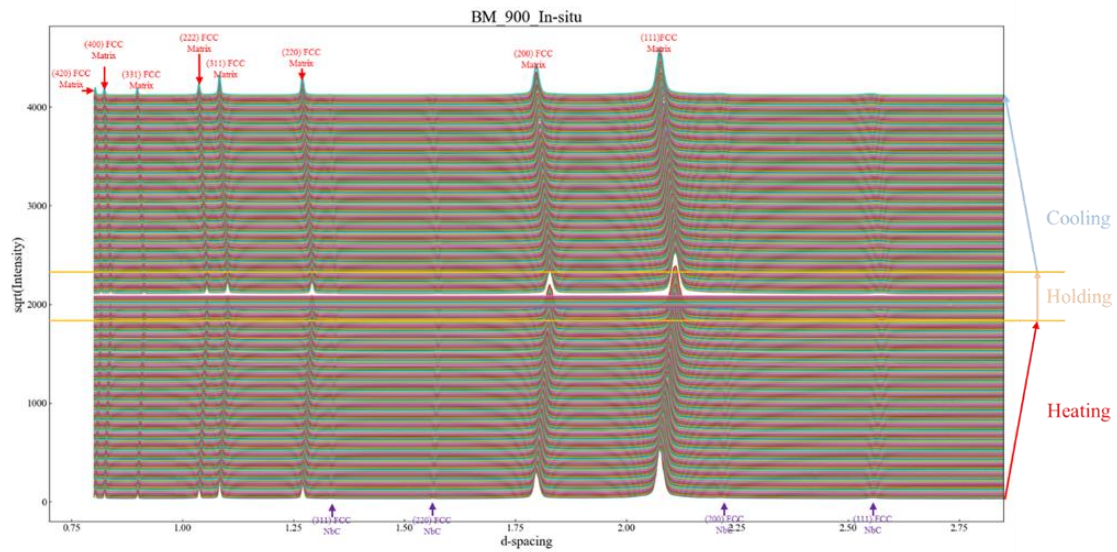


Figure 4.8. BM sample in-situ measurement result.

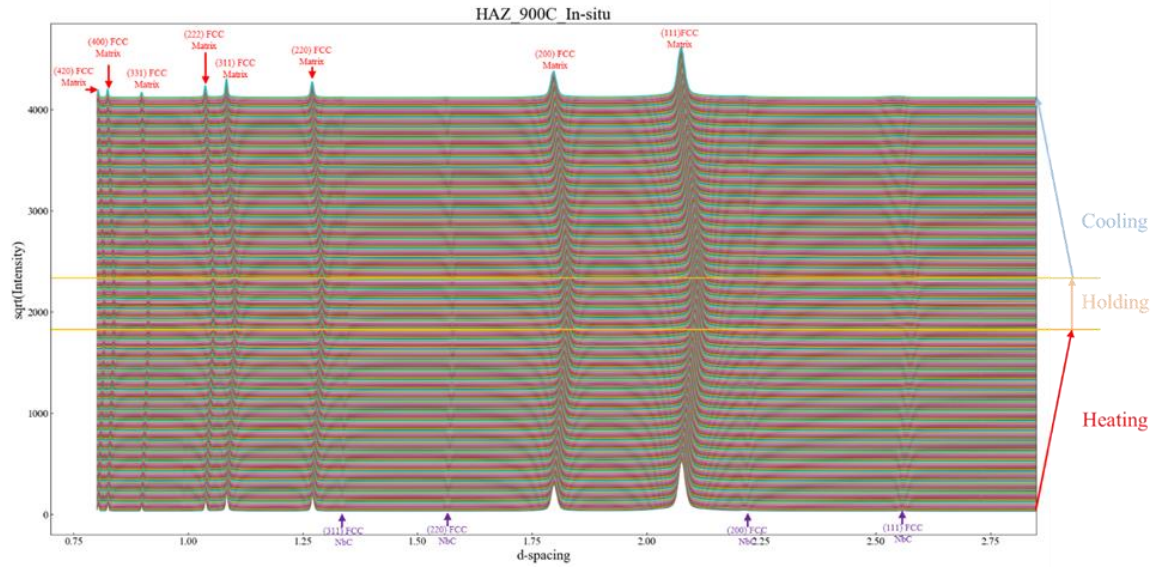


Figure 4.9. HAZ sample in-situ measurement result.

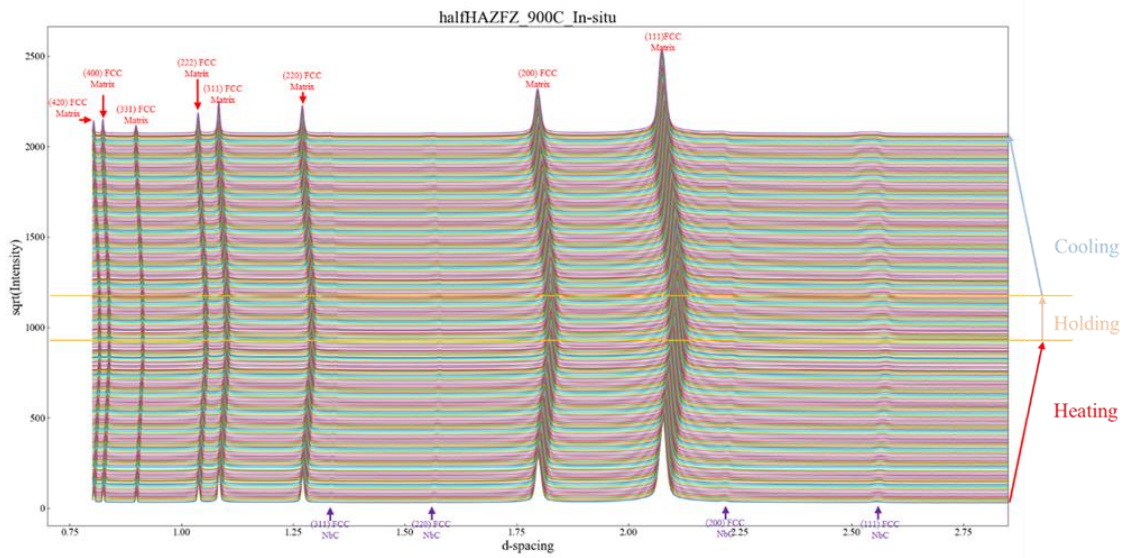


Figure 4. 10. $\frac{1}{2}$ HAZ $\frac{1}{2}$ FZ sample in-situ measurement result.

region is shown in Figure 4.9 and Figure 4.10 respectively. Although these regions exhibit microstructure variations caused by the welding process, the drastic temperature changes and the 4-hour isothermal holding at 900 °C are not sufficient to form other types of precipitates. The only noticeable changes are observed in the intensity of the NbC peaks, which decrease during heating and increase during cooling, while the matrix peaks show the opposite trend. The FZ sample undergoes melting and solidification, resulting in the most significant microstructural changes.

Figure 4.11 displays the scanning results for the FZ sample. In addition to the matrix and NbC phases, the presence of Cr₂₃C₆ and Fe₃C is also detected. As the temperature approaches 900 °C during the heating process, the peak intensities of Cr₂₃C₆ and Fe₃C gradually decrease. During the isothermal holding stage, their peak intensities remain relatively stable. Subsequently, as the furnace temperature cools down to room temperature, the peak intensities of Cr₂₃C₆ and Fe₃C gradually increase back to their original levels.

To clearly illustrate the variations between different regions, Figure 4.12 presents a comparison between the BM and FZ samples using selective scanning data, highlighting the changes induced by the temperature field. In (a), the d-spacing changes of the base metal are shown, revealing only peak shifting. However, in (b), which represents the FZ sample, two additional types of precipitates (Cr₂₃C₆ and Fe₃C) are already present before the heating step. As the temperature increases, not only peak shifting is observed, but also a decrease in peak intensity. Conversely, during the cooling stage, the opposite trend is observed. Due to limitations in beamtime and furnace capabilities, achieving long-term isothermal holding at higher temperatures is not feasible. Nevertheless, this in-situ comparison between two different regions within a weldment provides valuable insights into the different microstructural compositions that lead to distinct precipitation evolution trajectories. It indirectly indicates that the HAZ and FZ are regions with more stress concentration sites, which is also related to varying degrees of residual stress relaxation.

4.2.2 Ex-situ measurement

The in-situ measurements provide valuable insights into the relationship between

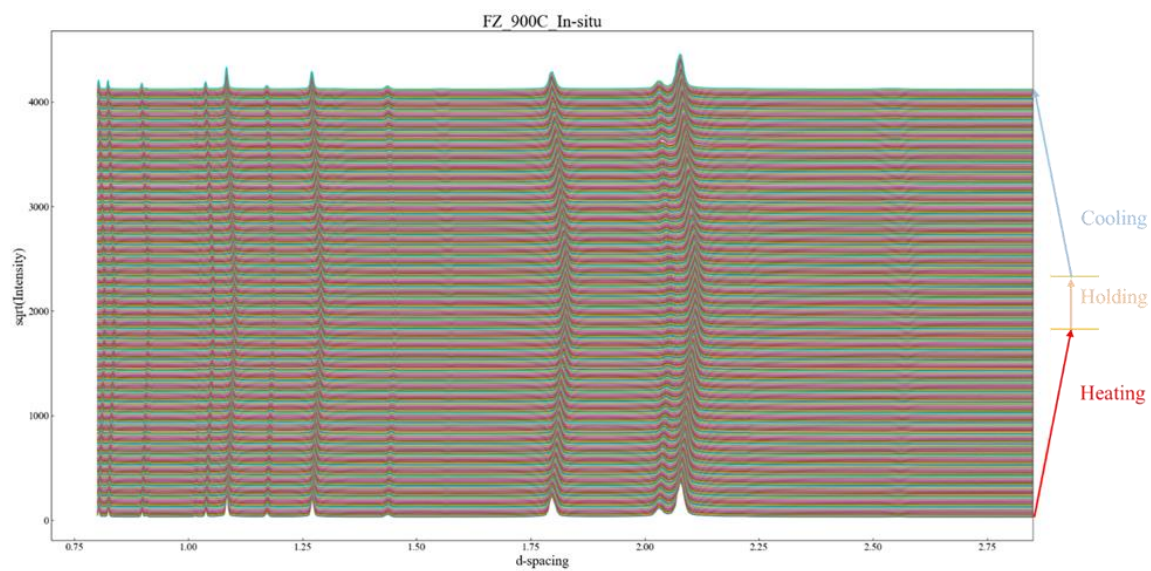


Figure 4.11. FZ sample in-situ measurement result.

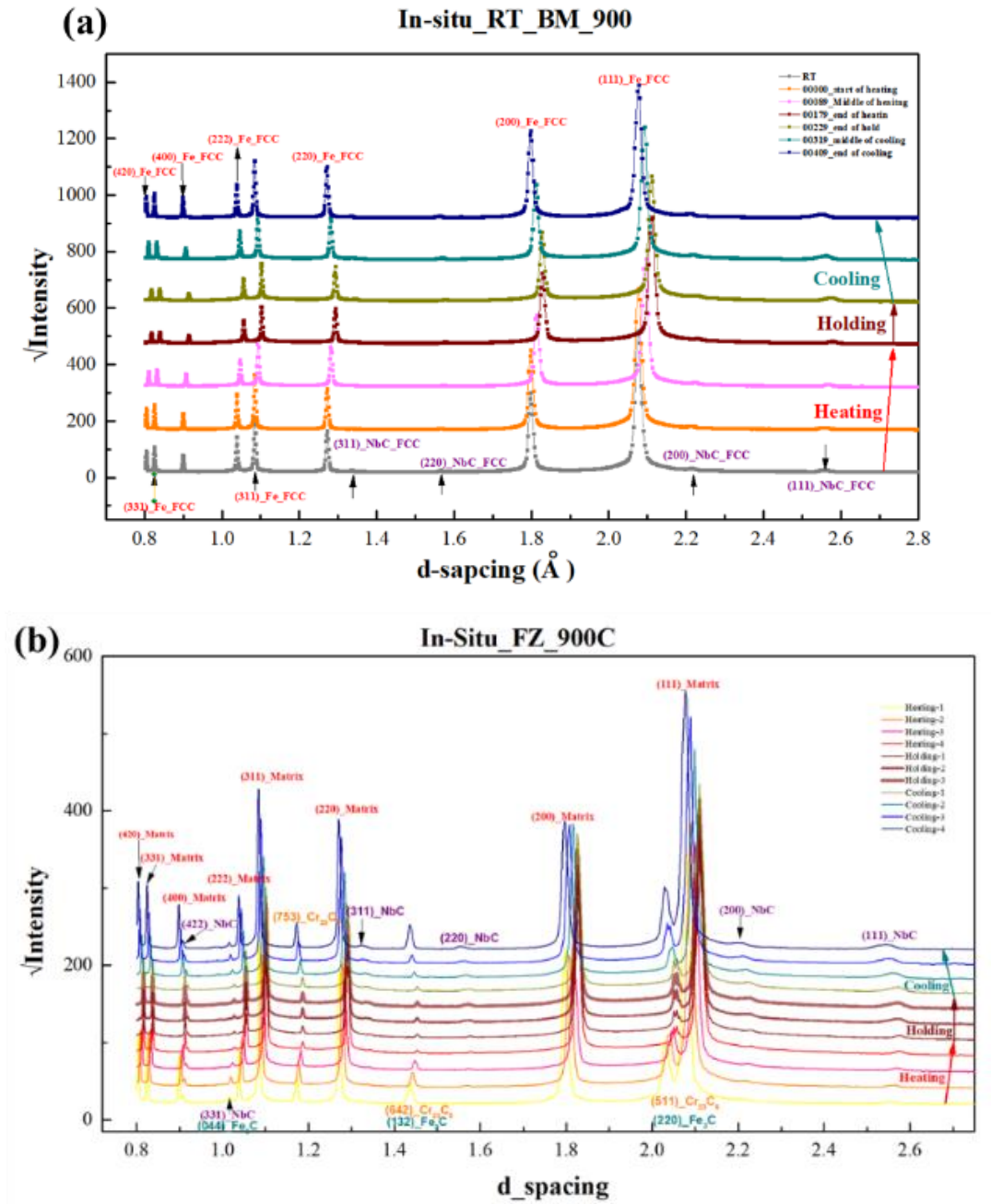


Figure 4.12. (a) in-situ BM sample selected scanning lines results; (b) in-situ FZ sample selected scanning lines results.

precipitation evolution and temperature variations. However, the long-term effects of thermal treatment cannot be directly observed through in-situ measurements. To address this limitation, we have employed ex-situ measurements to scan samples that have undergone different thermal conditions. Although ex-situ measurements cannot track the evolution of precipitation in real-time like in-situ methods, they allow us to calculate the delta lattice strain. In ex-situ measurements, the data obtained can be used to calculate the delta lattice strain by comparing each condition to a reference state, typically the "As-Welded" condition. The strain variation is then determined based on the reference state. This calculation can be performed using the following equation:

$$\text{Delta lattice strain } (\Delta\epsilon) = \frac{d_{\text{thermal treatment condition}} - d_{\text{reference condition}}}{d_{\text{reference condition}}}$$

To facilitate a comprehensive interpretation of the ex-situ data, all ex-situ samples followed a specific scanning sequence, as depicted in Figure 4.13. The scanning matrix encompasses 43 scanning points, covering the BM, HAZ, and FZ. Each region is marked with a different color in the figure, and the FZ is located in the middle, spanning a range of 6mm. Within the FZ, there are a total of 7 scanning points with a 1mm interval between them. On either side of the FZ lie the HAZ regions, each with a length of 9mm and containing 9 scanning points with the same 1mm interval as the FZ. In the ex-situ samples, only the 18mm BM region was measured, comprising 9 scanning points with a 2mm interval. The scanning points in the BM area are not as dense as those in the FZ and HAZ regions. This is because the temperature field has a lesser impact on the base metal, and it is reasonable to assume that there are no significant changes in both the residual stress field and microstructure towards the far end of the sample. It's important to note that each sample was subjected to the same scanning matrix, allowing the calculations to be performed at the corresponding positions consistently.

Before calculating the delta lattice strain for different thermal treatment cases, it is important to understand the distribution of lattice strain in the As-Received and As-Welded conditions. Due to the welding method and sample dimensions, two scanning lines were employed perpendicular to the welding line. One scanning line, referred to as the "top line," was located 20mm away from the sample edge (perpendicular to the welding line). The

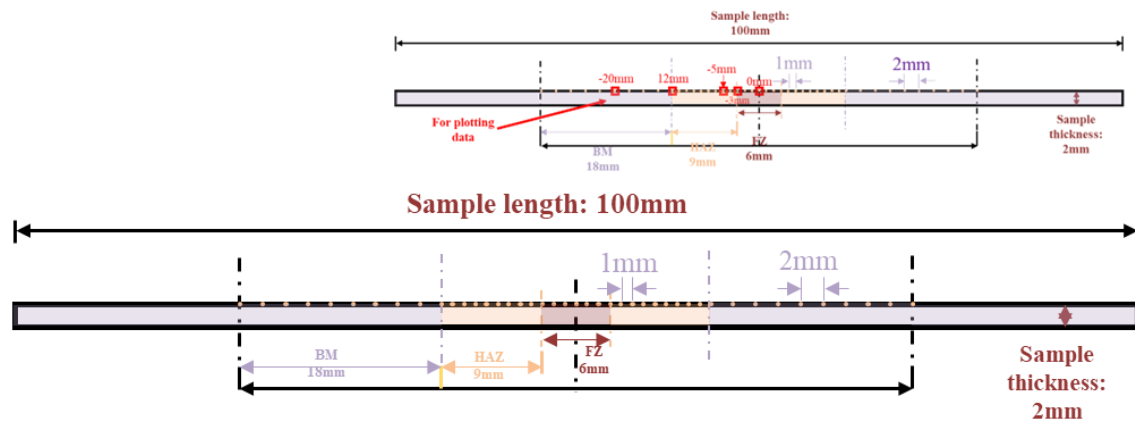


Figure 4.13. All ex-situ samples scanning map.

other scanning line, known as the "middle line," was positioned in the middle of the sample. The scanning sequence started from the left-hand side of the view and moved towards the right. As the sample was in the As-Received condition, indicating no prior thermal history, two reference d-spacing points were selected to calculate the lattice strain. One reference point was in the middle of the scanning map (referred to as 0mm), while the other was at the left-hand side (LHS) edge of the scanning map (referred to as -30mm). Theoretically, these two calculations should yield identical results since the sample is in the As-Received condition. Figure 4.14 illustrates the results of these two lattice strain calculations. Due to the difference in reference point selection, the position of the concentrated zero lattice strain varies accordingly. Using the 0mm reference point, the results show a distribution ranging from tensile lattice strain (0.0004) to compressive lattice strain (-0.0002) for both the top and middle scanning lines. Similarly, when using the -30mm reference point, a similar distribution is observed, but the range changes from compressive lattice strain (-0.0004) to tensile lattice strain (0.0002). When comparing these two reference points, the only difference observed is a shift in the data distribution. The minor variation present in the As-Received sample can be disregarded when compared to samples subjected to thermal treatment. Additionally, by examining these two reference points, it can be concluded that the base metal has no significant contribution to the variation in lattice strain.

The same analysis method was applied to the As-Welded sample, as shown in Figure 4.15. Due to the significant microstructure variation induced by the rapid temperature changes during welding, the lattice parameter across the scanning map is not a fixed value but exhibits fluctuations. By using a reference point to calculate the lattice strain, we can determine which region experiences the most pronounced or dramatic changes. The As-Welded sample exhibits similar features to the As-Received condition, with the zero lattice strain concentration occurring at 0mm when using 0mm as the reference point, and vice versa for the -30mm case. However, the key difference between the As-Welded and As-Received conditions lies in the lattice strain distribution within the FZ and HAZ. In the FZ and HAZ, a compressive lattice strain trend is observed for certain

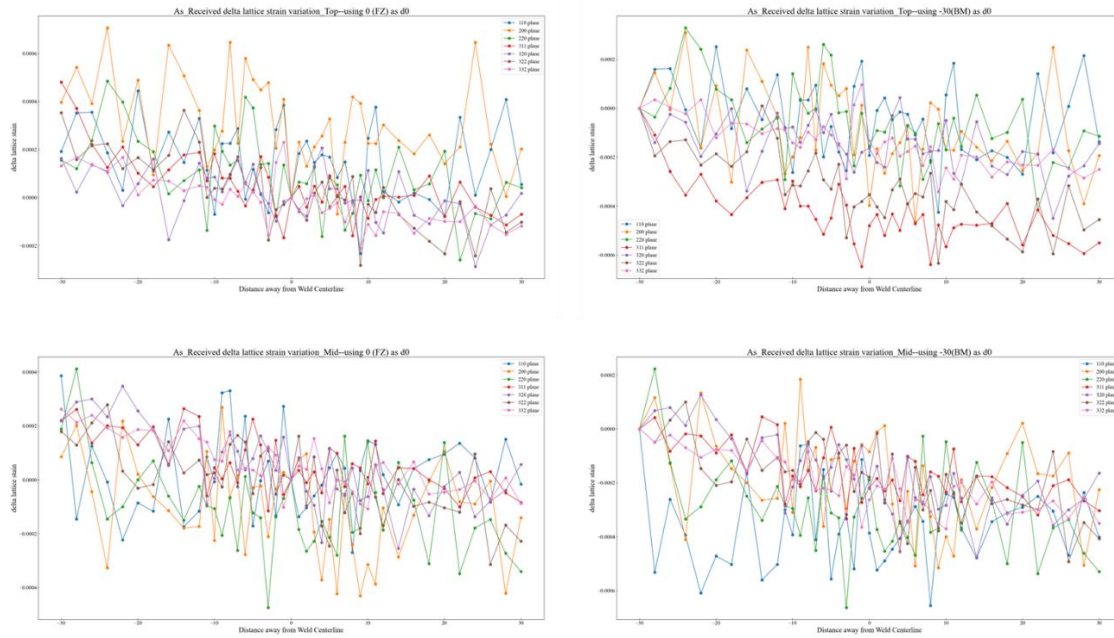


Figure 4.14. As-Received sample delta lattice strain distribution, comparison of calculation results by using two different reference points.

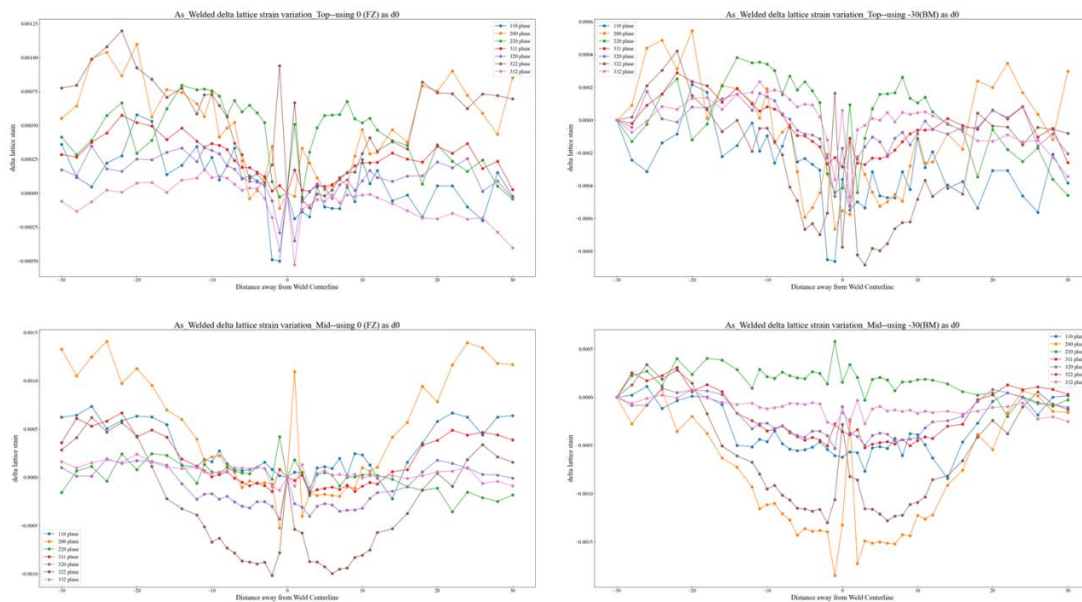


Figure 4.15. As-Welded sample delta lattice strain distribution, comparison of calculation results by using two different reference points.

lattice planes, while the base metal remains within a near-zero lattice strain range. The presence of compressive lattice strain in the FZ and HAZ indicates the accumulation of compression stress resulting from the welding process. To further analyze the effects of thermal treatment on lattice strain and residual strain, the As-Welded condition sample can be used as the reference sample. This approach helps to isolate the effects caused by the initial residual stress field and precipitates in the As-Welded condition.

In order to provide a better understanding of the minor effects brought by the As-Received condition, an additional calculation based on the equation mentioned earlier has been performed. The results of this calculation are presented in Figure 4.16.

$$lattice\ strain = \frac{d_{As-Welded}^{Position\ #} - d_{As-Received}^{Position\ #}}{d_{As-Received}^{Position\ #}}$$

The aforementioned equation has been applied to the sample in its As-Welded condition, with the As-Received sample serving as the reference sample. This approach aims to eliminate the inherent slope present in the sample. Figure 4.16. (a) & (b) present the computed results for both the middle and top scanning lines. By examining the sample morphology in Figure 4.17, it is observed that the FZ is located within a 4mm range in the middle of the scanning map, while the HAZ spans a range of 9mm on both sides of the FZ.

The remaining area corresponds to the BM. The delineation of these specific regions is indicated in Figure 4.18, which facilitates the precise identification of the regions undergoing changes. In comparison to the BM, the FZ and HAZ exhibit a decreasing trend in the top scanning line, resulting in an approximate lattice/residual strain level of 0.0002. However, in the middle scanning line, a valley is also discernible, accompanied by an increased compressive residual strain as depicted in (b). These two figures collectively demonstrate the presence of compressive residual stress in the middle region of the sample. As one moves towards the edge of the sample, the compressive residual stress gradually diminishes. Meanwhile, the BM region consistently displays a relatively uniform distribution of residual strain, fluctuating around zero residual stress. Given that the delta lattice strain under the reference condition has been determined, the delta lattice strain for other thermal treatment conditions can be computed using the equation provided below:

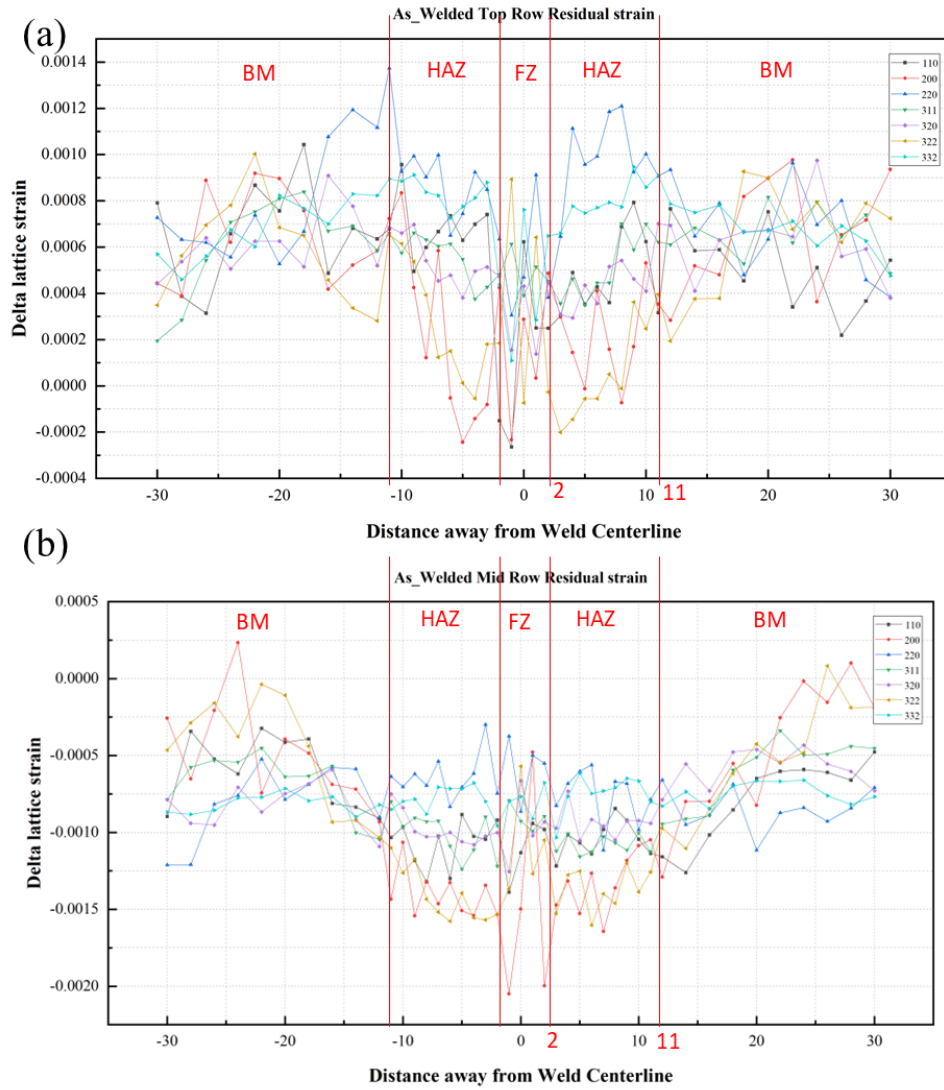


Figure 4.16. As-Welded sample two different scanning lines' delta lattice strain distribution; by using the As-Received sample as reference sample.

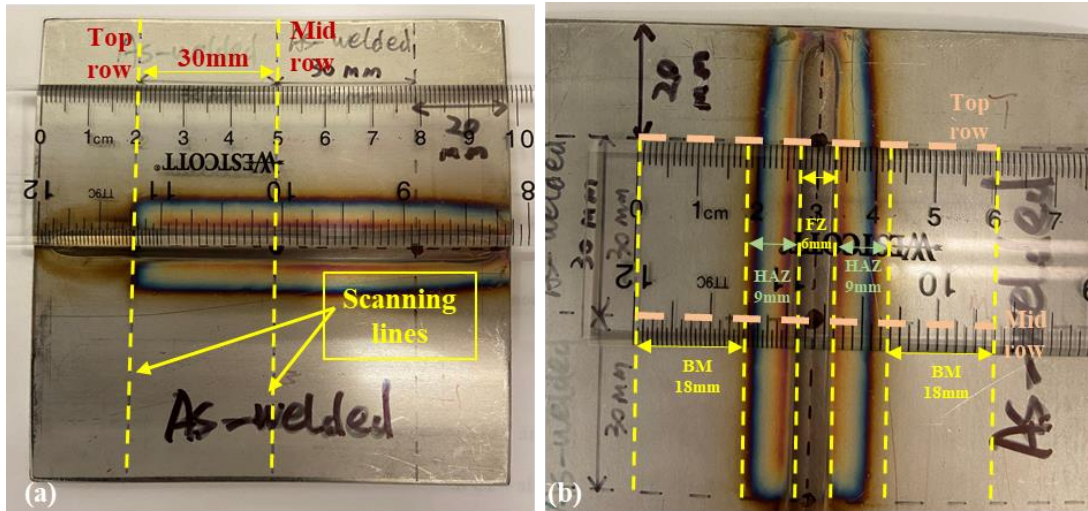
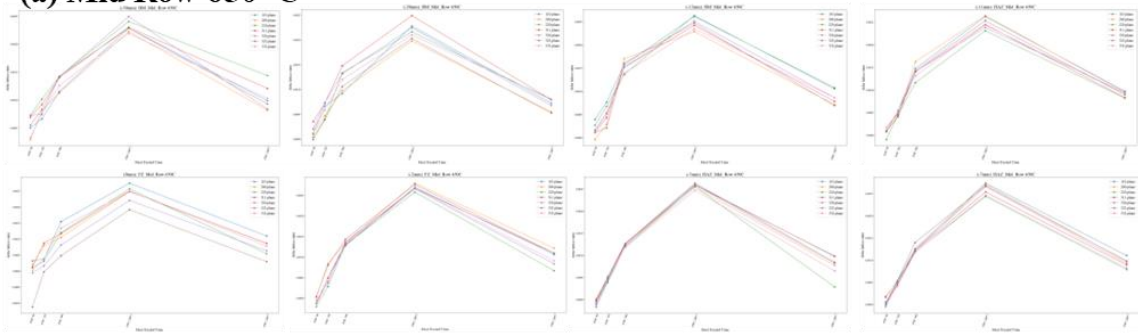
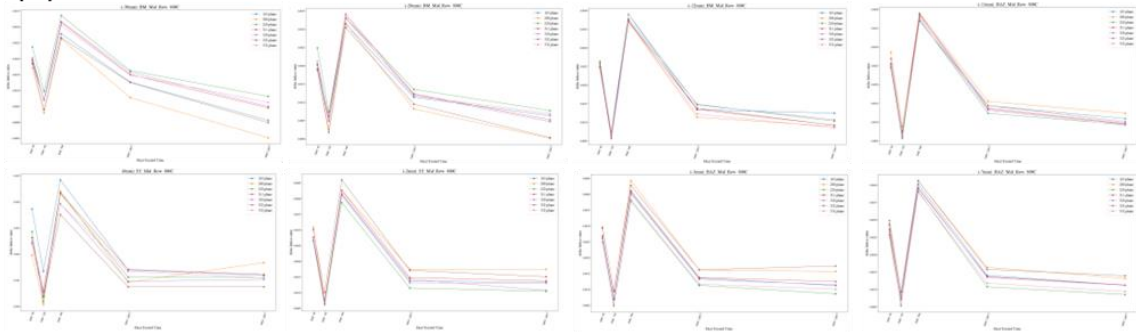


Figure 4.17. Synchrotron diffraction sample images for Ex-situ measurement.

(a) Mid Row 650 °C



(b) Mid Row 800 °C



(c) Mid Row 900 °C

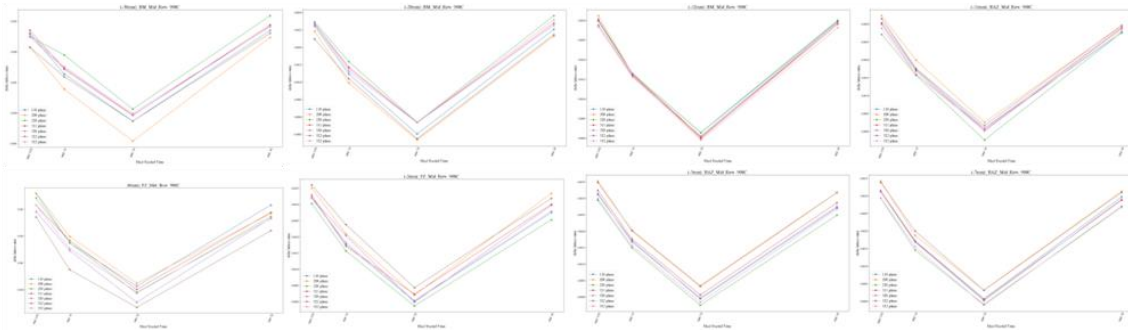


Figure 4.18. Middle Row delta lattice strain variation at three different testing temperatures.

$$\text{Delta lattice strain } (\Delta\epsilon) = \frac{d_{\text{thermal treatment condition}} - d_{\text{reference condition}}}{d_{\text{reference condition}}}$$

Apart from the As-Received and As-Welded conditions, an additional set of 15 samples underwent distinct thermal treatments, with 14 of them following a sequential time sequence to facilitate comparison between these conditions. Figure 4.18 illustrates the middle scanning lines for the 14 thermal treatment conditions. At temperatures of 650°C and 800°C, each temperature level was subjected to 5 different isothermal holding times: 4 hours, 12 hours, 24 hours, 72 hours, and 168 hours. At 900°C, four isothermal holding times were employed, corresponding to four individual samples: 0.5 hours, 1 hour, 2 hours, and 4 hours. Regarding the 650 °C samples, an increase in the isothermal holding temperature corresponds to an increase in the delta lattice strain until the isothermal holding time reaches 72 hours.

Subsequently, a decreasing trend is observed in the delta lattice strain. It should be noted that the Y-axis represents the delta lattice strain, indicating the level of residual strain variation compared to the As-Welded condition. Thus, an increasing trend signifies a continuing relaxation of residual strain compared to the As-Welded condition, while a decreasing trend suggests a slower relaxation process. Among the cases involving isothermal holding at 650°C, the 72-hour holding time exhibits the highest percentage of lattice strain relaxation. A similar approach can be applied to the 800°C isothermal holding cases. Due to the higher isothermal holding temperature, the variation trend differs from the 650°C cases. From the isothermal holding time of 4 hours to 12 hours, a decreasing trend is observed, indicating a decrease in the relaxation rate. However, after the isothermal holding time reaches 24 hours, the delta lattice strain increases to its highest value among all five cases at 800°C. Finally, as the isothermal holding time continues to increase to 72 hours and 168 hours, there is a significant drop compared to the 24-hour case, although the 168-hour case shows a slight increase compared to the 72-hour case. When the isothermal holding temperature is raised to 900°C, a turning point in the delta lattice strain variation occurs at 2 hours of holding time. From 0.5 hours to 2 hours, the delta lattice strain steadily decreases until it reaches nearly zero lattice strain. Subsequently, an increasing trend is

observed as the holding time is extended to 4 hours. For the generation of these series of figures, we selected eight distinct scanning points, each corresponding to different microstructure regions. Specifically, the points at 0mm and -2mm belong to the FZ, while -3mm, -7mm, and -11mm correspond to the HAZ. Furthermore, -12mm, -20mm, and -30mm pertain to the BM. Despite the varying distances from the weld centerline, these scanning points exhibit similar variation trends. Additionally, the middle scanning line and top scanning line, as depicted in Figure 4.19, demonstrate comparable trends, with the only distinction lying in the specific lattice plane.

4.3 Micro-scale microstructure characterization

347H austenitic stainless steel is known for its precipitation strengthening capability, which enhances its performance during the mid to late stages of surface evolution through the formation of precipitates. Therefore, it is crucial to investigate the type, distribution, size, relationship with grain boundaries, and composition of the precipitates to support the explanation of the SRC mechanism. Various microscopy techniques are commonly employed to reveal the microstructure morphology. Scanning Electron Microscope (SEM), Energy Dispersive Spectroscopy (EDS), and Transmission Electron Microscope (TEM) are typically used for this purpose. SEM provides a comprehensive view of the microstructure, while EDS enables the mapping of element distribution. TEM, on the other hand, allows for the visualization of precipitates at the nanoscale. However, SEM has limited resolution, and certain trace elements may not be clearly discernible in EDS maps. Furthermore, all these methods have a common drawback of not being able to demonstrate the three-dimensional distribution of precipitates. To address this limitation, in addition to the aforementioned techniques, Atom Probe Tomography (APT) is employed to provide a three-dimensional perspective of the precipitates. APT offers high-resolution imaging and can accurately determine the elemental composition of individual atoms within the precipitates. By utilizing APT, researchers can gain valuable insights into the spatial distribution and composition of the precipitates in three dimensions, complementing the information obtained from other microscopy techniques.

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4.3.1 Results of SEM

For the SEM characterization, a specific sample preparation method is employed. The samples are treated with a solution containing HNO_3 : Glycerol: HCl in a ratio of 1:3:7. The procedure involves warming the samples in hot water, followed by immersing them in the solution for 15 seconds. Subsequently, the samples are rinsed with water. The SEM results encompass various samples, including the as-received condition plate (which was also used to cut Gleeble samples), the fractured sample from the Gleeble test, a thick-pipe sample used for neutron diffraction residual stress measurement (which includes BM, HAZ, and FZ), and a thin sheet used for synchrotron diffraction experiments. Although these samples were obtained from different shapes and conditions, they exhibit only slight differences in their nominal composition as they are all made of 347H stainless steel. However, it should be noted that the thick pipe sample has undergone 5 years of service, which may introduce additional variations.

Figure 4.20 displays the microstructure morphology of the as-received condition sample, revealing a grain size ranging from approximately 10 to 30 μm . The composition of the sample is provided in the table. The austenitic grains are uniformly distributed. The scanning results under secondary electron (a) and backscatter electron (b) imaging highlight the distribution of precipitates. Since these precipitates likely consist of nano-sized NbC, their presence may not be easily detected using EDS, necessitating further assistance from APT. The secondary electron results at a farther distance (c) are also shown. Line scan results (d) and its insert figure (e) demonstrate a flat trend along the scanning line.

In In Figure 4.21, the element distribution of the pre-strained sample is shown. The secondary electron scanning results (a) indicate that these particles are Nb-rich sites, suggesting a high probability of NbC formation. However, due to the resolution limitations of EDS, detecting carbon (C) is challenging.

To support the assumption that precipitation fraction gradually increases after deformation, EBSD results in Figure 4.22 are compared. The undeformed EBSD image shows an evenly distributed distribution of non-deformed grains.

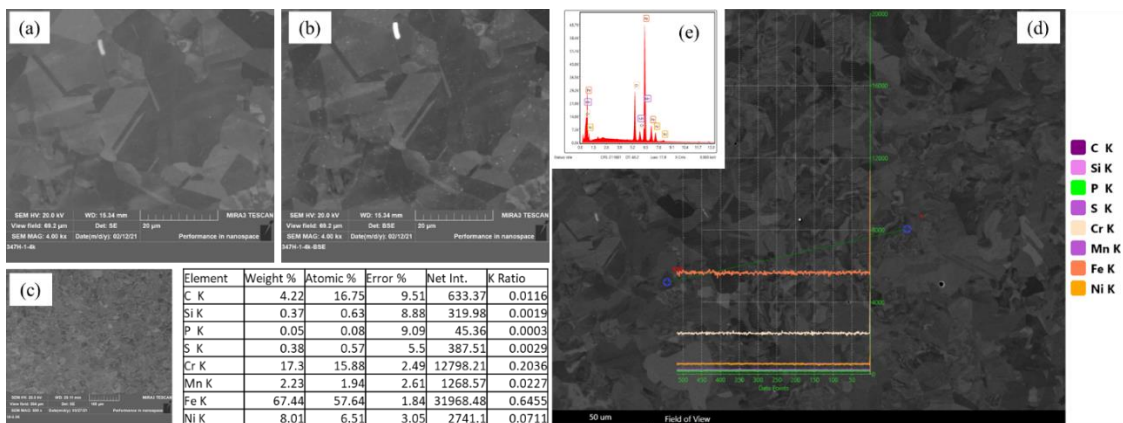


Figure 4.20. As-Received sample SE images (a-c); (d) BSE images; insert table is the elements percentage.

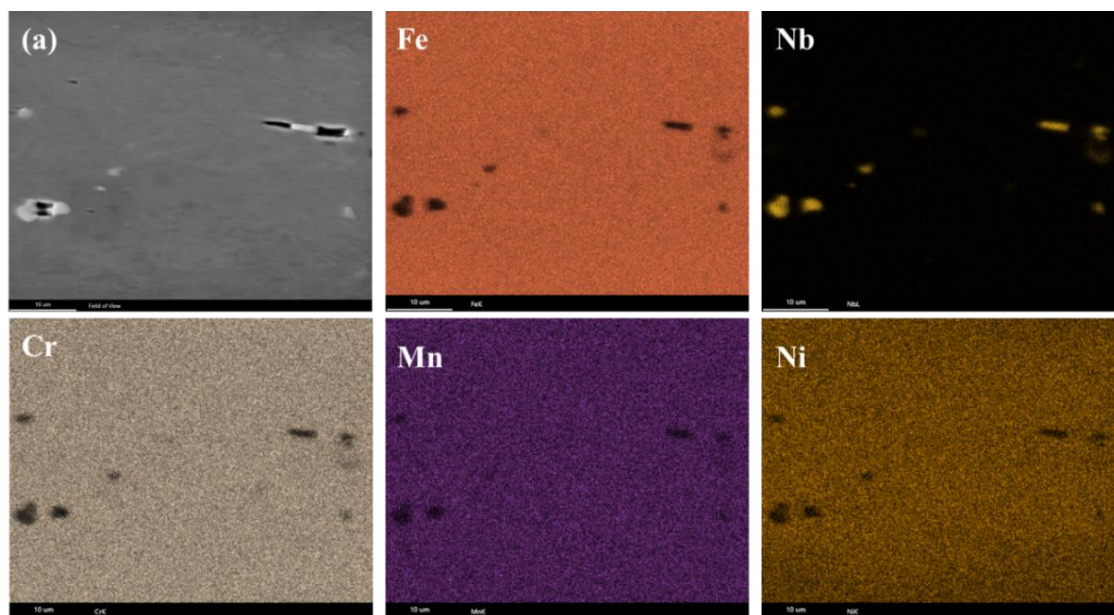


Figure 4.21. EDS map of As-Received sample.

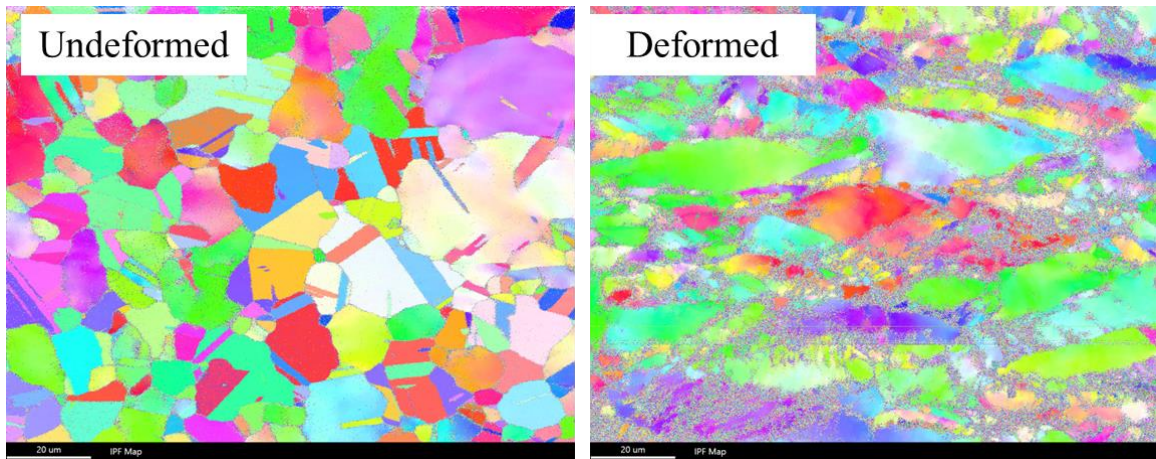


Figure 4.22. Comparison of the undeformed and deformed EBSD images.

However, in the deformed image, the grains are heavily distorted, indicating significant deformation. This distortion is also evident in the secondary electron images shown in Figure 4.23.

In addition to deformation-induced changes, thermal cycles can also induce microstructural variations. Figure 4.24 illustrates the morphology of the sample after undergoing multiple thermal cycles at different temperatures and isothermal holding times. Microcracks are visible throughout the sample, and precipitation is predominantly located at grain boundaries. This serves as direct evidence to verify the effect of thermal treatment on the SRC.

The characterization of microstructure in actual service conditions is crucial for understanding the SRC mechanism, especially for materials like 347H that are commonly used in pipelines. To address this, the thick pipe sample is utilized, which has a wall thickness of 32.07mm and is primarily employed for neutron diffraction residual stress measurement. In order to perform microstructure characterization, a slot is cut from the thick pipe sample, and this cut-off section is used for analysis. Figure 4.24 displays images of the weld region in the thick pipe. Figure 4.25 (a) depicts the inner and outer diameter of the pipe, covering both the FZ and the HAZ. Figure 4.25 (b) provides a magnified view of the middle of the FZ, revealing a significant number of precipitates. In comparison, Figure 4.25 (c) shows the edge of the FZ, which includes parts of the HAZ as well. In contrast to the dense distribution of precipitates observed in (b), (c) exhibits a relatively smaller number of precipitates, with most of them located in the FZ. Line scans are employed to gain insights into the type and composition of these particles.

As demonstrated in Figure 4.26. (b) and (c), the line scans suggest that these particles are NbC. Furthermore, the EDS mapping depicted in Figure 4.27 confirms that the particles are composed of Nb and C.

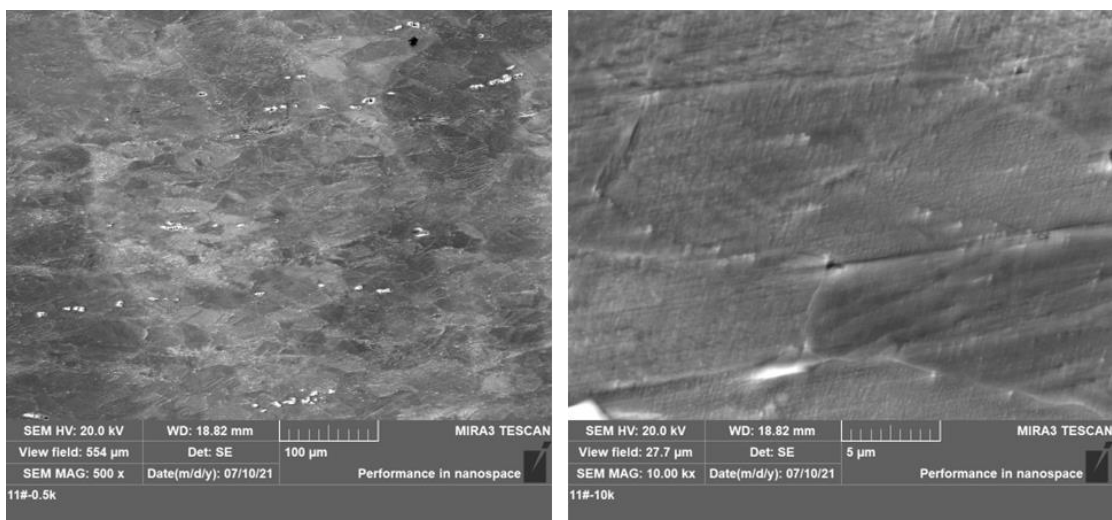


Figure 4.23. Fractured sample SE images.

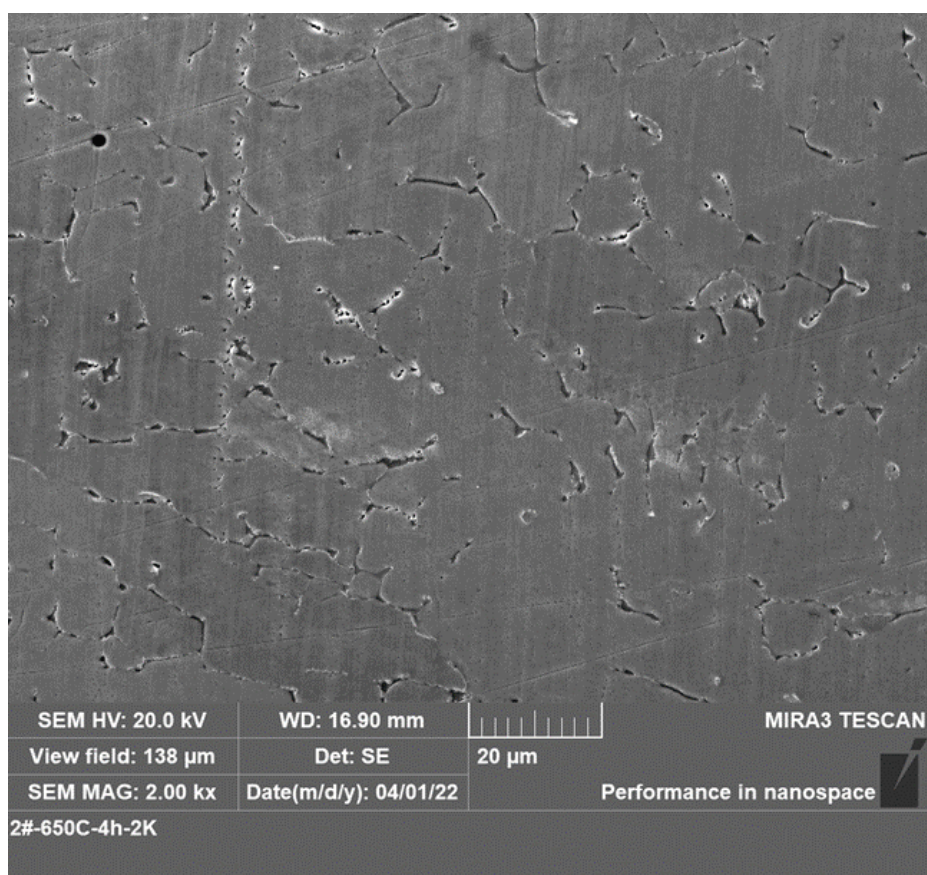


Figure 4.24. Multiple thermal cycles sample SE image.

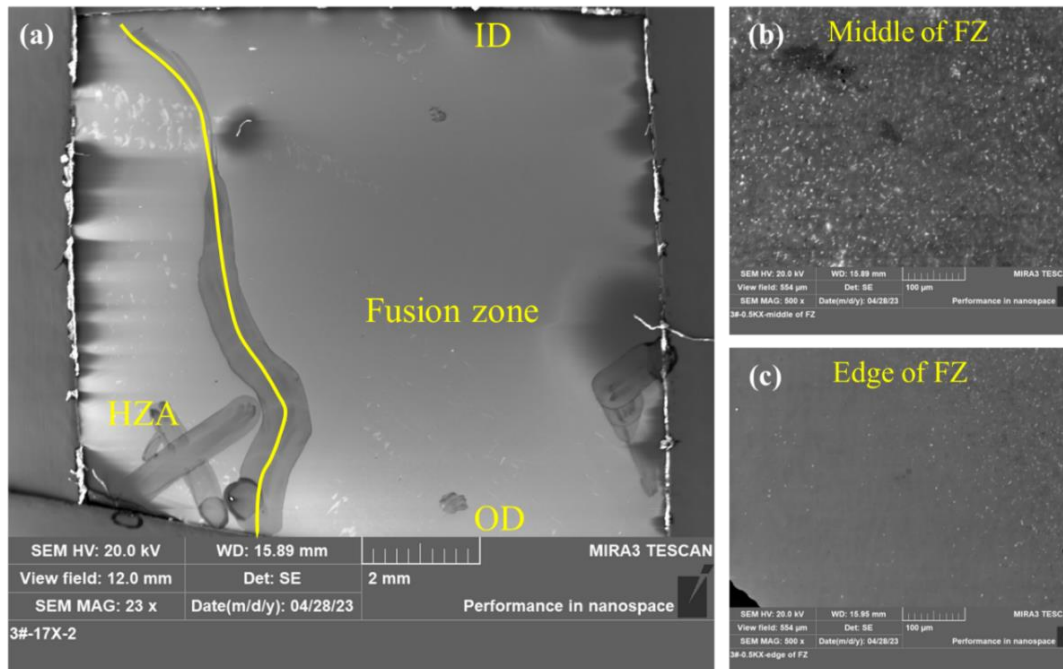


Figure 4.25. Thick pipe SE images.

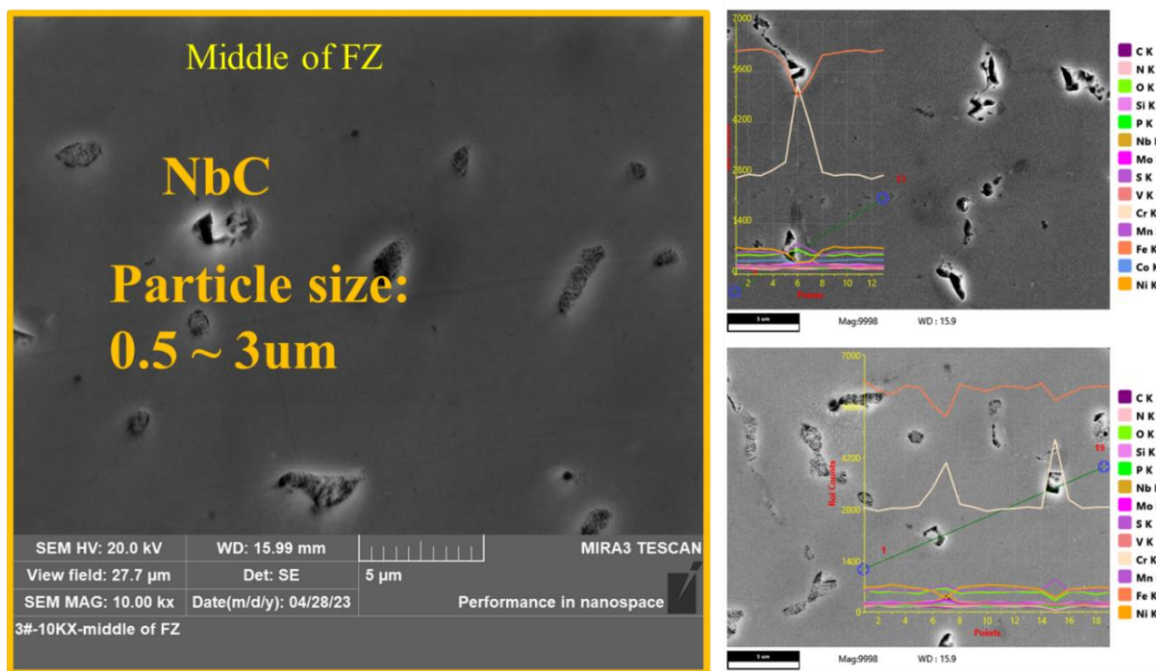


Figure 4.26. Thick pipe line scan results.

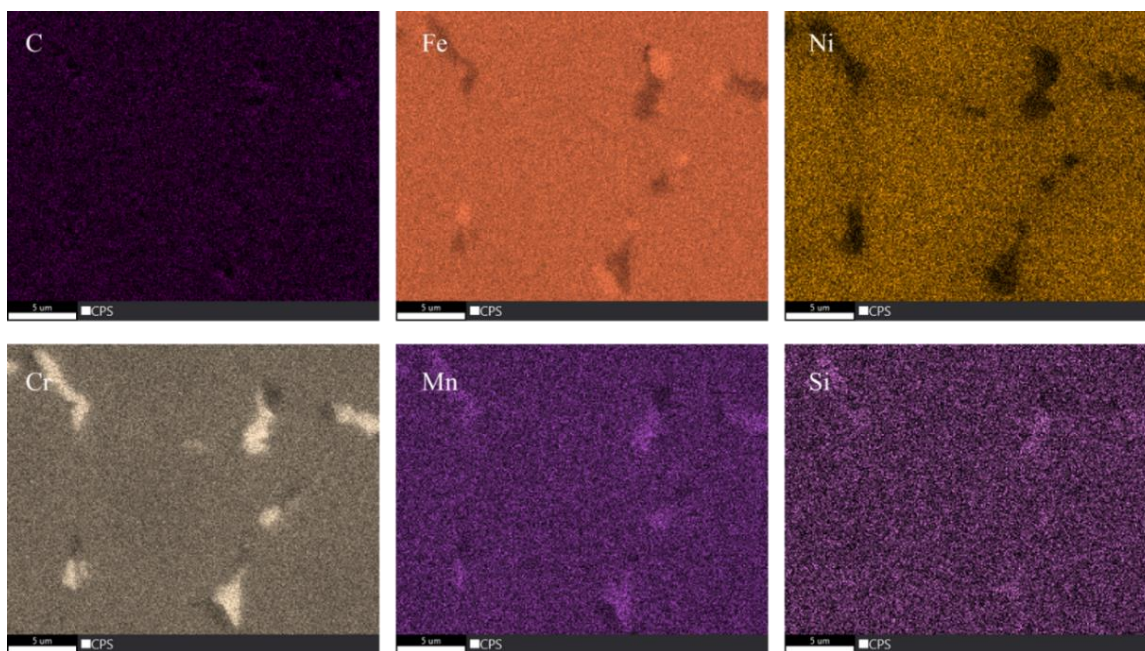


Figure 4.27. Thick sample FZ EDS mapping.

CHAPTER FIVE

THERMOMECHANICAL PROPERTIES MEASUREMENT

The variation of material properties is inherent to changes in the temperature field. Failing to accurately identify such variations can lead to inaccuracies in simulation models. To address this issue, neutron and synchrotron diffraction have been employed as supportive and verification tools for predicting the lifespan of materials. Additionally, the Gleeble thermomechanical testing system has been utilized to establish the relationship between temperature and yield strength. The evaluation of the variation in yield strength is motivated by the strain hardening effect.

Metallurgically, it has been well established that plastic deformation generates dislocations. The increase in dislocation density results in an increase in flow stress (expansion of the yield surface). At the same time, at elevated temperatures, annihilation of dislocation also takes place, which results in a decrease of dislocation density and corresponding reduction of flow stress. As the annihilation of dislocations is a thermally activated process (whereas generation of dislocations by plastic deformation is not), the rate of annihilation is higher as temperature increases. The primary metallurgical process behind the annihilation of dislocations includes recovery and recrystallization. Hence, the strain hardening behavior at a given temperature (and deformation rate) is governed by the two competing dynamic processes – the rate of dislocation generation and the rate of dislocation annihilation. We call such strain hardening behavior dynamic strain hardening.

To investigate the dynamic strain hardening effect in 347H stainless steel, a comprehensive set of experiments was conducted to measure the reduction in strain hardening. In order to induce a drop in the yield surface caused by localized thermal treatment, the samples were pre-strained at room temperature. Subsequently, the samples were subjected to the Gleeble testing system, heating the middle section to a specific temperature. Finally, the pre-strained and thermally treated samples were subjected to tension testing to obtain the final strain-stress curve. However, because of thermal treatment, when reloading the sample to the same level of strain as in the pre-strain step, the resulting curve does not perfectly match the original curve. The reduction in yield stress

at a specific pre-strain level can be defined as the reduction in strain hardening, denoted as "R." The experimental setup and methodology for calculating the R value will be detailed below.

5.1 Sample preparation

A representative sample of 347H austenitic stainless steel was obtained by cutting it from an As-Received condition plate, ensuring the material's homogeneity. The dimensions of the sample are depicted in the Figure 5.1, featuring a 7mm diameter in the 70mm gauge section, providing an adequate length for temperature observation.

5.2 Pre-strain test

In this series of tests, a pre-strain level of 10% was applied, comprising both elastic and plastic strains. The tensile machine employed a strain control model, with a set breakpoint of 100,000 microstrain for the hold position. Engineering strain and engineering stress were calculated using the following equations:

$$\text{Engineering strain} = \frac{\text{stroke column} - 1\text{st stroke number}}{\text{gauge length}}$$

$$\text{Engineering stress} = \frac{\text{Load}}{\text{cross_section area}}$$

Then the true stress-strain are generate using the equations below:

$$\text{True strain} = \ln(1 + \text{engineering strain})$$

$$\text{True stress} = \text{engineering stress} \times (1 + \text{engineering strain})$$

To begin, the pre-strained samples underwent local thermal treatment using the Gleeble testing system. The heating temperature was varied at three different levels: 650 °C , 700 °C , and 750 °C . Additionally, three different isothermal holding times were employed: 5s, 80s, and 320s. The specific Gleeble testing setup utilized in this experiment is depicted in Figure 5.2.

The objective of employing different thermal treatment temperatures and holding times was to generate distinct microstructures, thereby leading to different flow surfaces. Consequently, an R-value variation trend could be observed. To characterize the local

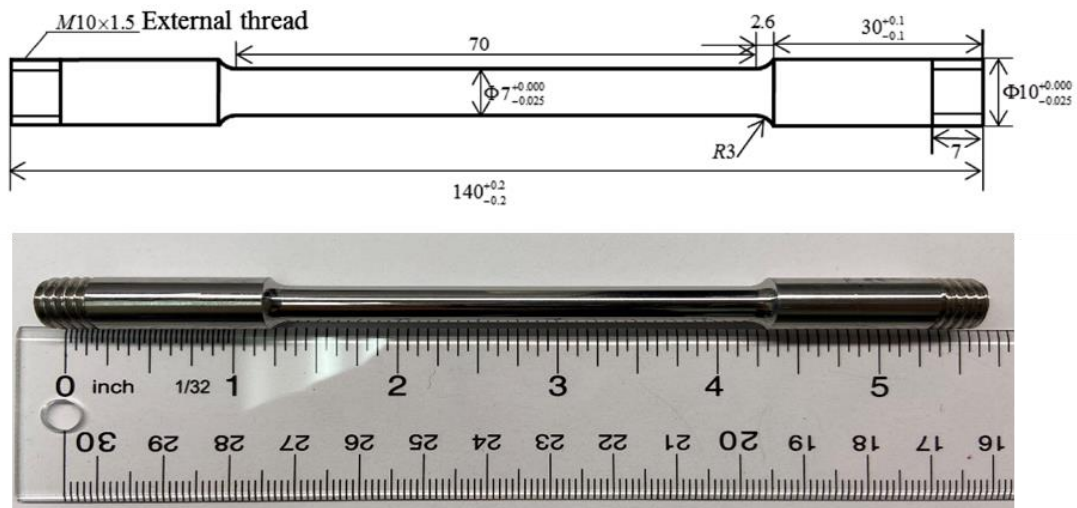


Figure 5.1. Gleeble sample geometry.

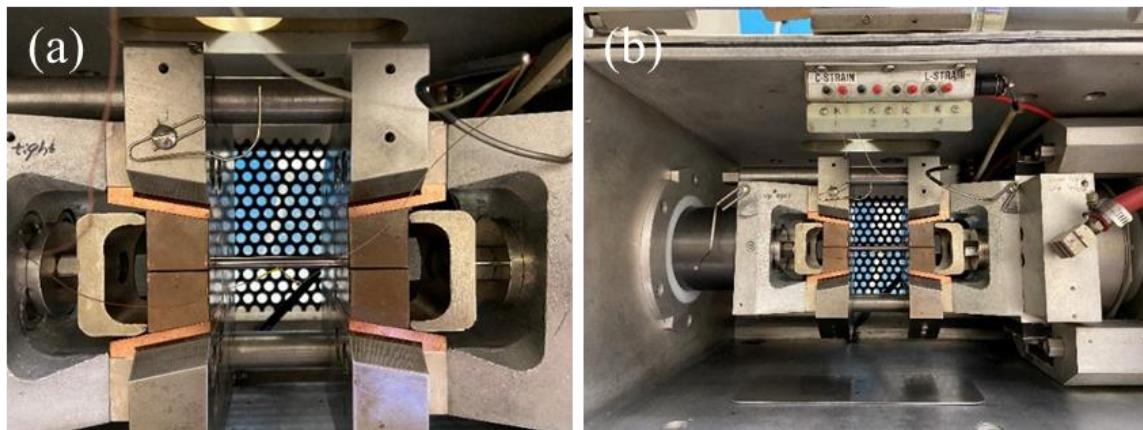


Figure 5.2. Gleeble test setup.

mechanical properties across the length of the specimen, the digital image correlation (DIC) technique was employed. Prior to installing the samples into the DIC testing system, they were coated with a thin white background and random black specks, as illustrated in Figure 5.3.

The DIC system captured images of the unloaded and deformed samples at a frame rate of 10 Hz. Subsequently, the strain field was calculated from a series of sequential images. The actual setup for the DIC system is presented in Figure 5.4. The schematic drawing in Figure 5.5 illustrates the principle of DIC.

The schematic drawing in Figure 5.5 illustrates the principle of DIC.

Following the application of various thermal treatment combinations, when the sample was reloaded to the same strain level as in the pre-strain test under the DIC monitoring system, the flow stress exhibited a different value, denoted as σ'_y . The reduction in strain hardening, represented by the R value, is defined by the following equation:

$$R = \frac{\sigma_y - \sigma'_y}{\sigma_y - \sigma_0}$$

The observed increase in yield stress from σ_0 to σ_y indicates significant strain hardening in the stainless steel. When the material is unloaded and then reloaded, elastic deformation occurs until the stress reaches σ_y . However, if the material undergoes a brief elevated temperature excursion after plastic deformation, such as being heated to 900°C for 6 seconds, the new yield point, denoted as σ'_y , is lower than σ_y . This phenomenon is attributed to the recovery and recrystallization processes that take place under certain conditions. The extent of reduction in strain hardening resulting from the high-temperature excursion can be quantified by the ratio of the reduction in the yield point, as indicated in the R-value equation.

Figure 5.6 illustrates the morphology of a fractured sample.

While Figure 5.7 displays a sequence of DIC images that track the failure process. By selecting different specks in various regions, the corresponding strain values can be obtained.



Figure 5.3. Sample prepare before testing under DIC.

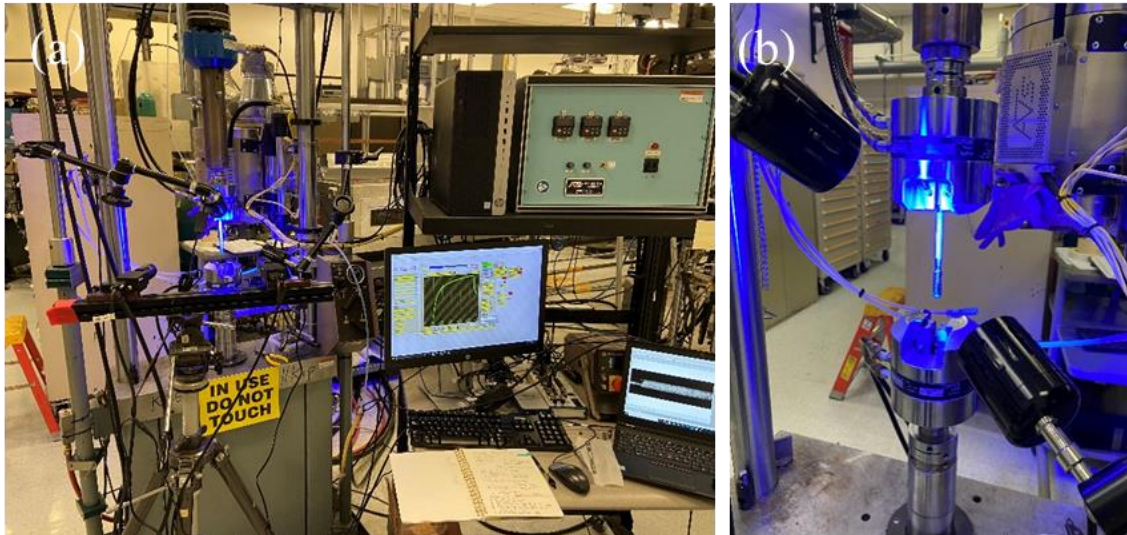


Figure 5.4. Actual setup of DIC test.

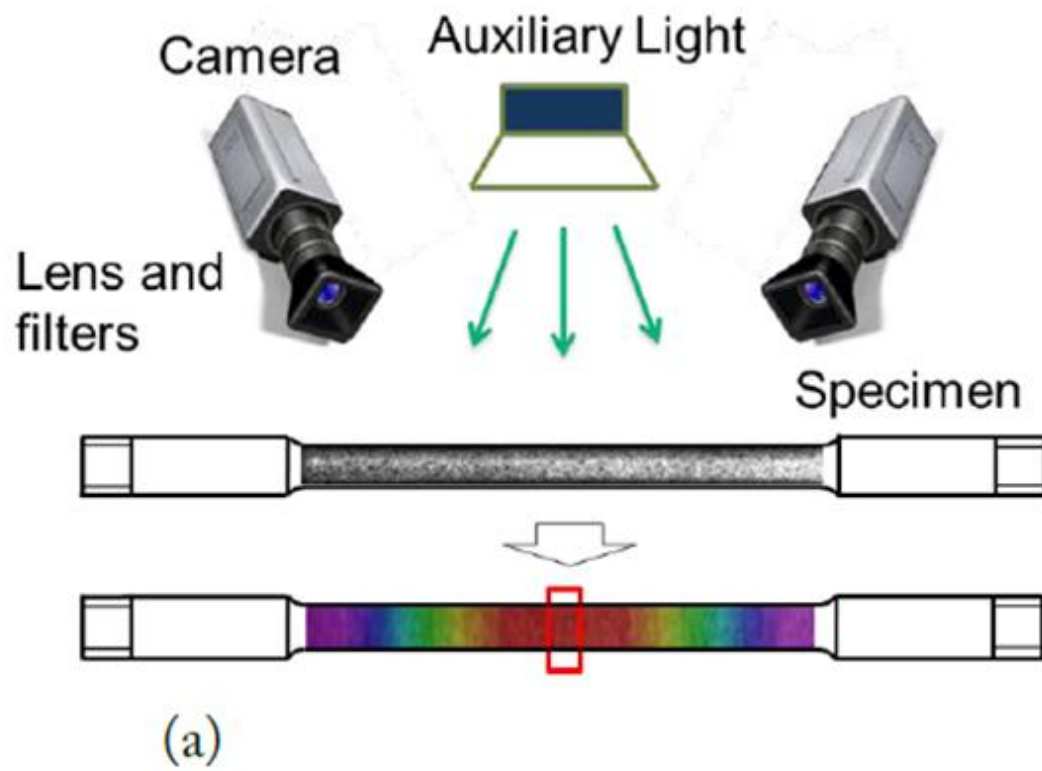


Figure 5.5. DIC mechanism schematic drawing.



Figure 5.6. Fractured DIC sample.

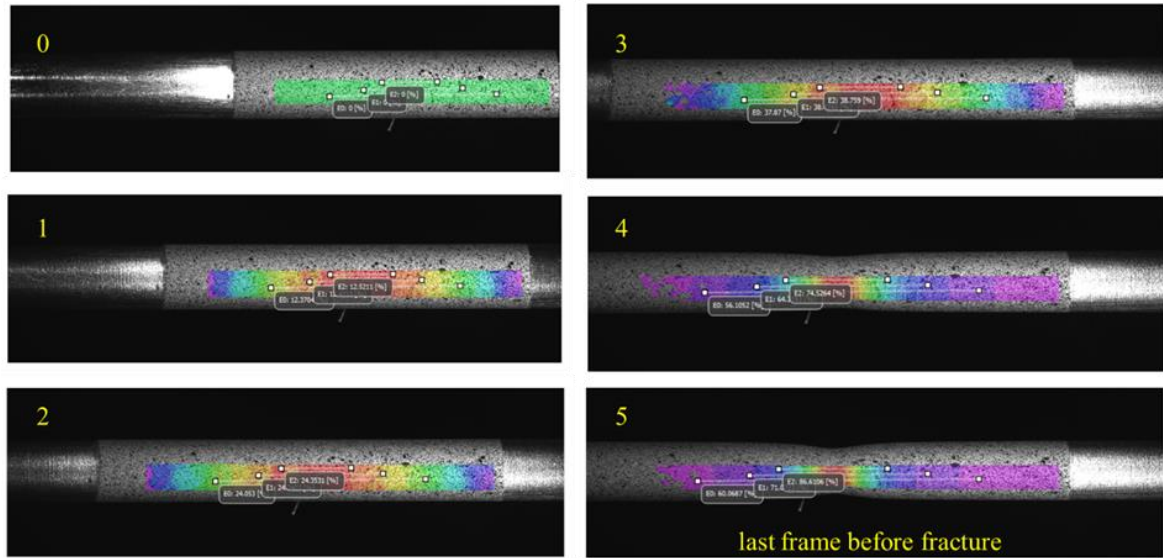


Figure 5.7. Featured images during the DIC test, to demonstrate the fracture process.

Figure 5.8 depicts the variation in the stress-strain curve caused by thermal conditions, with the dark red line representing the stress-strain curve obtained from the RT pre-strain. The remaining curves represent the results of DIC monitoring, with variations arising from the selection of different regions for analysis. Due to the material properties of 347H stainless steel, its response to short-term thermal treatments tends to be unpredictable, leading to a relatively random distribution of R values. Consequently, further investigation is required to refine the Gleeble and DIC testing methods for measuring the R-value.

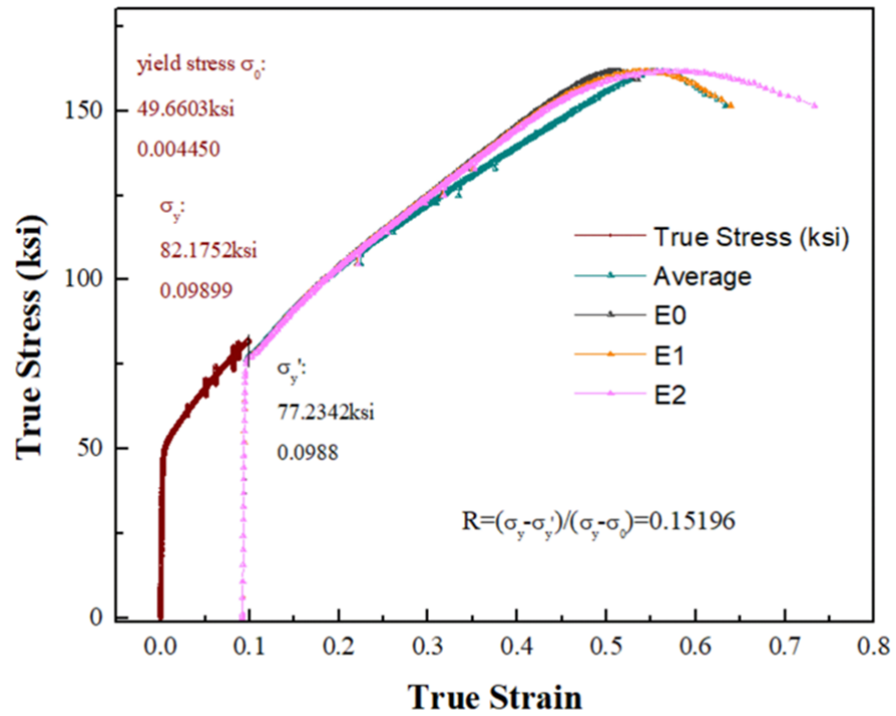


Figure 5.8. R-value calculation detail explanation .

CHAPTER SIX

CONCLUSIONS AND RECOMMENDATIONS

Identifying the control failure mechanism and perform lifetime prediction consistently remain a difficulty for service weldments components due to the complex and synergistic process happened along the thermal treatment. With the unsuccessful path to track the impact level of each factor. A new comprehensive methodology using a series simulation models combined with accessible experimental results as validation tools is demonstrated in this work. Instead of considering each factor separately, this work brought a solution to predict the direction of this synergistic process. In this work, the study focuses on the 347H material parameters fitting with real creep data to generate a reliable baseline. Then using separate model to calculate the temperature and residual stress field distribution. In the meantime, the effect bring by the PWHT are also investigated. Finally, the microstructure features are gradually added from simple pure base metal to the complex case with PFZ. Hence, prediction on the failure life can be achieved through these series models. Validation on the residual stress field measurement and precipitation evolution are made through Neutron and Synchrotron diffraction experiments, which can not only verify the simulation model's accuracy, but also provide guidance for measuring the acutal service components.

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APPENDIX

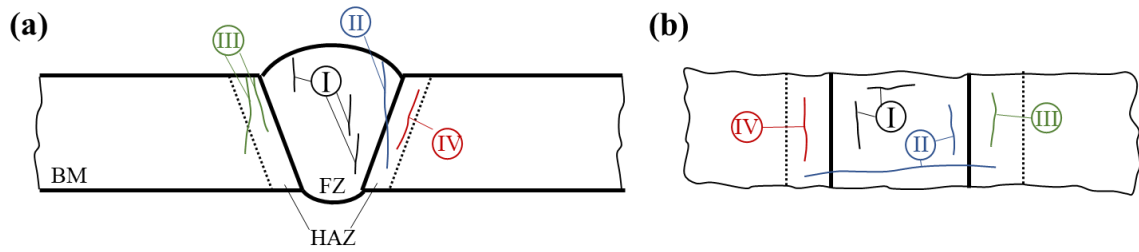


Figure A.1. Four different types of crack demonstration.

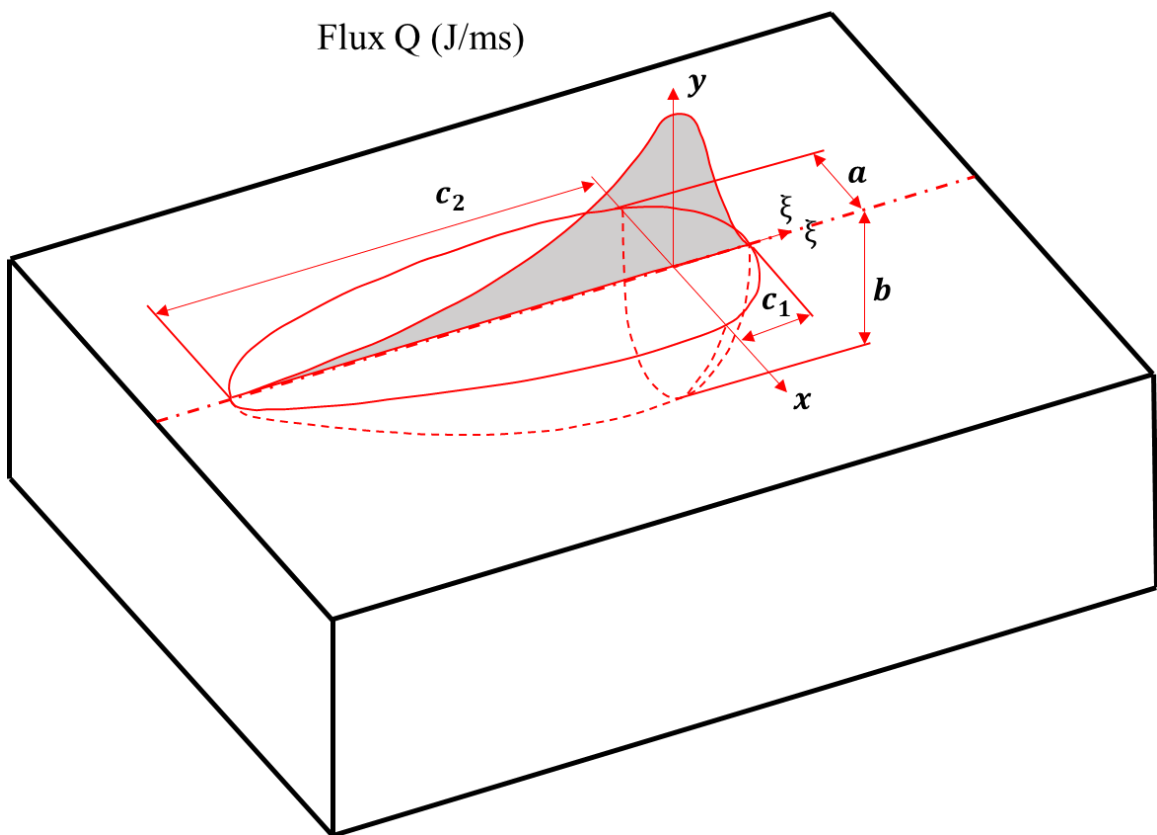


Figure A.2. Heat source model demonstration.

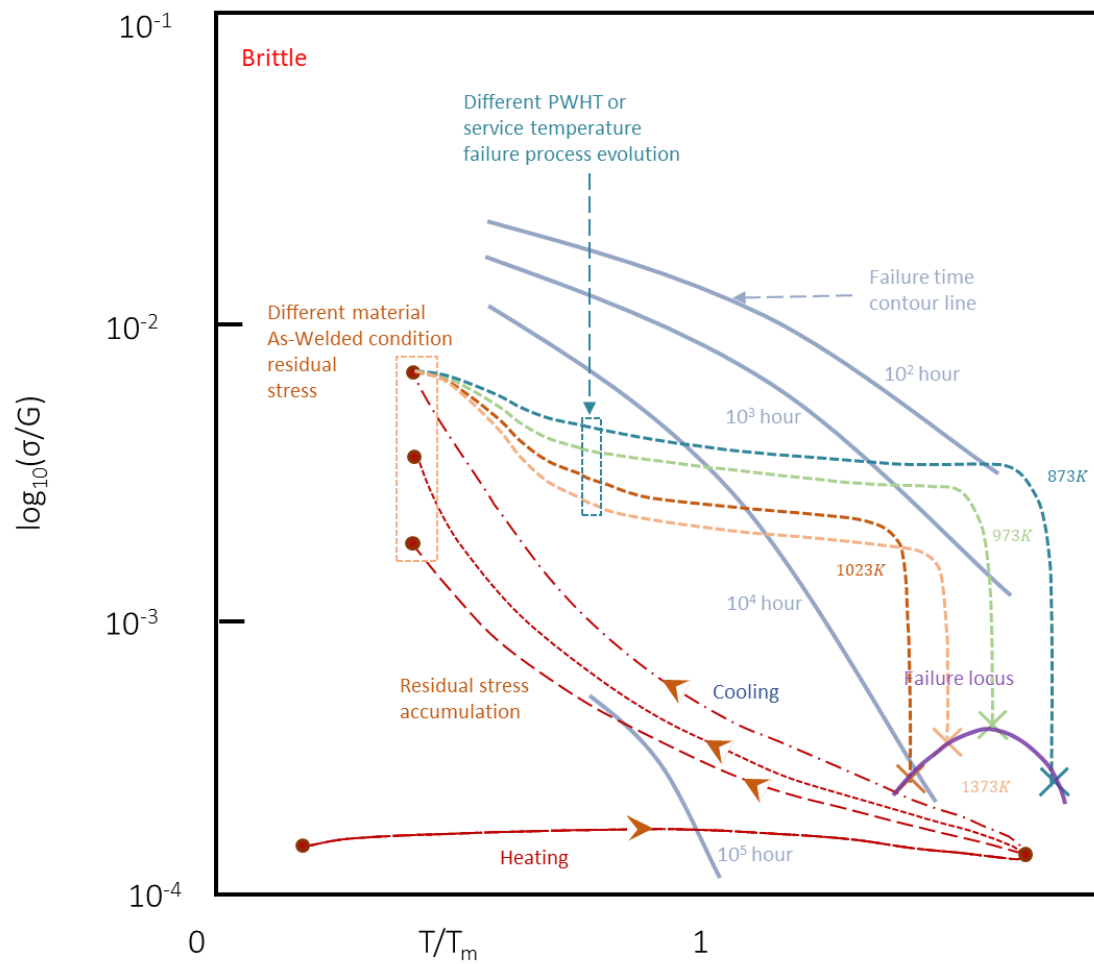


Figure A.3. Master figure of entire failure process evolution.

VITA

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