

**Development and Influence of Fusion Boundary Microstructure  
on Resistance Spot Welds in 3<sup>rd</sup> Generation  
Advanced High Strength Steels**

by

**Dileep Chandran Ramachandran**

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## **Examining Committee Membership**

The following served on the Examining Committee for this thesis. The decision of the Examining Committee is by majority vote.

External Examiner

Professor Kip O. Findley  
Dept. of Metallurgical and Materials Engineering,  
Colorado School of Mines, USA

Supervisor

Professor Elliot Biro  
Dept. of Mechanical and Mechatronics Engineering,  
University of Waterloo, Canada

Internal Member

Professor Charles Kwan  
Dept. of Mechanical and Mechatronics Engineering,  
University of Waterloo, Canada

Internal Member

Professor Kaan Inal  
Dept. of Mechanical and Mechatronics Engineering,  
University of Waterloo, Canada

Internal-External Member

Professor Michael Pope  
Dept. of Chemical Engineering,  
University of Waterloo, Canada

## **Author's Declaration**

This thesis consists of material all of which I authored or co-authored: see Statement of Contributions included in the thesis. This is a true copy of the thesis, including any required final revisions, as accepted by my examiners.

I understand that my thesis may be made electronically available to the public.

## **Statement of Contributions**

This thesis includes 8 chapters, including the introduction (Chapter 1) and literature review (Chapter 2). The series of data chapters 3 to 6 including published or submitted manuscripts the candidate authored as the primary author. The manuscripts have been slightly modified to fit the style of the thesis. It is important to highlight that for each of the works, the candidate wrote and did the primary editing as well as undertook the majority of the laboratory work, including welding, sample preparations, mechanical testing, and microstructural characterization, with exceptions as indicated below. The following co-authors have made specific contributions to the present work, as delineated below:

Professor Elliot Biro supervised the research progress, advised the research direction, assisted with data analysis, reviewed, and edited the manuscripts.

Dr. Andrew Macwan from ArcelorMittal Dofasco, Hamilton, Canada, acted as industrial supervisor, provided material, financial support, feedback on the research, and assisted with editing the manuscripts.

### **Chapter 3**

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The candidate's contribution was as described above. B. Figueredo, and O. Sherepenko, had technical discussion with the candidate. W. Jin, operated the SEM to obtain the EPMA data under the candidate's instructions and Prof. Y. Do Park provided editorial support. Prof. Biro's role was as described above.

### **Chapter 4**

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The candidate's contribution was as described above. A. Salandari-Rabori involved in technical discussions and reviewed the manuscript. Dr. Macwan and Prof. Biro's role were as described above.

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The candidate's contribution was as described above. O. Timothy Betiku, operated the MTS frame to acquire the mechanical test data used in the paper. M. Shojaee and A.R. Midawi did the tensile test of gleeble samples. J.-U. Kim supported the candidate to polish the TEM samples but all the electron microscopic analysis has been conducted by the candidate. R. Bakhtiari involved in the technical discussions during the early part of the work. A. Salandari-Rabori involved in discussions and reviewed the manuscript. Dr. Macwan and Prof. Biro's role were as described above.

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The candidate's contribution was as described above. A.R. Midawi operated the instrumented indentation test machine under the guidance of the candidate. A. Salandari-Rabori involved in technical discussions, reviewed and commented on the manuscript. Dr. Macwan and Prof. Biro's role were as described above.

## **Abstract**

The use of advanced high-strength steels (AHSS) is one of the design solutions for making new-generation auto bodies to build economical and environmentally friendly structures without compromising vehicle safety. Among various types of third generation AHSSs, quenched and partitioned (Q&P) steels, which are one of the material solutions due to their exceptional combination of strength and ductility compared to the conventional dual-phase (DP) steels. To produce body-in-white structures, resistance spot welding (RSW) is the predominant joining process used in the automotive industry. Hence, it is essential to investigate the resistance spot weldability of Q&P steels.

Recent investigations show that during welding, partitioning of alloying elements occurs at the weld fusion boundary (FB), especially in the steels with higher carbon equivalents. A narrow region around the nugget was transformed, which has a softer microstructure than the fusion zone (FZ) and heat-affected zone (HAZ), referred to as the “halo ring”, leading to premature failures in this weakened zone. When welded (AWS recommended single pulse schedule) and tested in tensile shear and cross tension geometries, it was seen that the fracture path in Q&P RSWs occurred through the halo ring. Moreover, the fracture surfaces of such welds show an intergranular fracture, which is contrary to the previous observations.

Based on these preliminary observations, the focus of the present research is to characterize the halo microstructure, find a phenomenological link between thermal history and the halo ring, and solve the discrepancies in failure behaviors by providing robust methods to eliminate halo. Modified welding schedules and post-weld heat treatments were implemented to eliminate the halo ring. The modified double-pulse schedule exhibited improved cross tensile strength (CTS) values by 33%, with an associated 110% increase in absorbed energy than the single-pulse weld. Similarly, the paint baking (PB) process was also implemented in single pulse weld, and it shows 34%, and 102% improvements in CTS and absorbed energy. Both methods improved the mechanical properties by shifting the crack path to the upper-critical heat affect

zone (UCHAZ) from the halo ring. The former method modifies the grain structure at the fusion boundary, whereas the latter one redistributes the elements segregated at the grain boundaries to the grain interiors.

The elemental diffusion in the halo ring has been discovered by making spot welds sandwiching low carbon (LC steel weld) and high carbon (HC steel weld) steels, respectively with Q&P steels on both sides, which is to tailor the chemical composition of the FZ. The halo formation is more prominent in welds made with LC steel rather than HC steel. It was found that the transient softened zone can be affected by differences in chemical composition between the FZ and UCHAZ. Furthermore, the mechanism of halo formation has been studied by characterizing the halo microstructure with transmission electron microscopic (TEM) analysis. TEM investigation disclosed that the microstructure within the halo ring is characterized as bainitic ferrite accompanied by needle-like cementite, specifically identified as upper bainite. Carbon diffusion towards the FZ in LC steel weld is attributed to higher activity resulting from differences in the chemical potential of carbon. This also accompanied by varying substitutional solute content towards the FZ from the UCHAZ is the reason for the halo formation. These advances in knowledge facilitated the development of strategies to mitigate halo formation through alloying adjustments (alloying with greater and lesser compositions of steels), paint baking, and welding schedule modifications.

Keywords: Advanced High Strength Steels; Resistance Spot Welding; Halo ring; Microstructure; Fracture; Paint baking; Double pulsing.

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## Dedication

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*I take immense pride in dedicating this work to each one of you.*

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## **List of abbreviations**

|       |                                   |
|-------|-----------------------------------|
| AHSS  | Advanced-high Strength Steel      |
| BIW   | Body-in-white                     |
| BM    | Base Material                     |
| CI    | Confidence Interval               |
| CTT   | Cross Tensile Test                |
| DP    | Dual Phase                        |
| EPMA  | Electron Probe Micro-Analyzer     |
| F     | Ferrite                           |
| FB    | Fusion Boundary                   |
| FGHAZ | Fine-Grained Heat Affected Zone   |
| FZ    | Fusion Zone                       |
| GI    | Galvanized                        |
| HAZ   | Heat-affected Zone                |
| HC    | High Carbon steel                 |
| HSLA  | High Strength Low Alloy           |
| HSS   | High Strength Steel               |
| IF    | Interfacial Fracture              |
| IPF   | Inverse Pole Figure               |
| IQ    | Image Quality                     |
| LC    | Low Carbon steel                  |
| LME   | Liquid Metal Embrittlement        |
| M     | Martensite                        |
| PAGB  | Prior Austenite Grain Boundary    |
| PB    | Paint Baking                      |
| PF    | Pullout Failure                   |
| PHS   | Press-hardened Steel              |
| PIF   | Partial Interfacial Fracture      |
| PMZ   | Partially Melted Zone             |
| PTF   | Partial Thickness Failure         |
| PWHT  | Post Welded Heat Treatment        |
| RA    | Retained austenite                |
| SCHAZ | Sub Critical Heat Affected Zone   |
| SF    | Stacking Faults                   |
| TM    | Tempered martensite               |
| TRIP  | Transformation Induced Plasticity |
| TST   | Tensile shear Test                |
| TWIP  | Twinning Induced Plasticity       |
| UCHAZ | Upper Critical Heat Affected Zone |
| UHSS  | Ultra-high Strength Steel         |
| UTS   | Ultimate Tensile Strength         |

## List of symbols

|               |                               |
|---------------|-------------------------------|
| $\mu$         | Chemical potential            |
| $a$           | Radius of the indenter        |
| $d_n$         | Nugget diameter               |
| $F_c$         | Force                         |
| $F_{\max}$    | Maximum load                  |
| $G$           | Gibbs free energy             |
| $I$           | Current                       |
| $I_{\max}$    | Expulsion current             |
| $L_{\max}$    | Displacement                  |
| $M_f$         | Martensite finish temperature |
| $M_s$         | Martensite start temperature  |
| $P_{\max}$    | Peak load                     |
| $R$           | Total resistance              |
| $R_b$         | Universal gas constant        |
| $h$           | Sheet thickness               |
| $S_{\max}$    | Normalized strength           |
| $T$           | Temperature                   |
| $t$           | Time                          |
| $T_m$         | Melting temperature           |
| $W_{\max}$    | Absorbed energy               |
| $X$           | Mole fraction                 |
| $\alpha'_p$   | Primary martensite            |
| $\alpha'_s$   | Secondary martensite          |
| $\varepsilon$ | Strain                        |
| $\sigma$      | Stress                        |
| $\sigma_y$    | Yield strength                |

# Chapter 1. Introduction

## 1.1. Background

Since the early 2000s, continuous demand for increased fuel efficiency (reducing CO<sub>2</sub> emission) for automobiles has made vehicle weight reduction one of the primary concerns for automotive manufacturers [1–4]. Light weight structures can be achieved by reducing the weight of the car body without compromising the strength and formability requirements. However, passenger safety is one of the main considerations for automotive manufacturers when designing a car body [5,6]. The use of high strength steel (HSS), in particular, advanced high strength steels (AHSS) in car bodies allow the automotive manufacturers to reduce sheet thickness, without sacrificing the crashworthiness, thereby also tackling the green-house gas emission [7–11].

There are a wide variety of thermo-mechanical processing techniques resulting in various generations of AHSS, such as first (1<sup>st</sup>), second (2<sup>nd</sup>), and third (3<sup>rd</sup>) generations. Figure 1.1 shows the generations of AHSS used in the automotive industry with its lightweight potential and energy absorption capability. The detailed classification of steels is as follows [6,12]:

1. Mild steel: IF and mild steel.
2. High strength steels: C-Mn steels and high strength low alloy (HSLA) steels.
3. 1<sup>st</sup> generation (Gen 1) AHSS: Dual phase (DP), transformation induced plasticity (TRIP), and martensitic steels.
4. 2<sup>nd</sup> generation (Gen 2) AHSS: Twinning induced plasticity (TWIP), and austenitic stainless (AS) steels.
5. 3<sup>rd</sup> generation (Gen 3) AHSS: Quenched and partitioned (Q&P) steel and TRIP assisted bainitic ferrite steels.

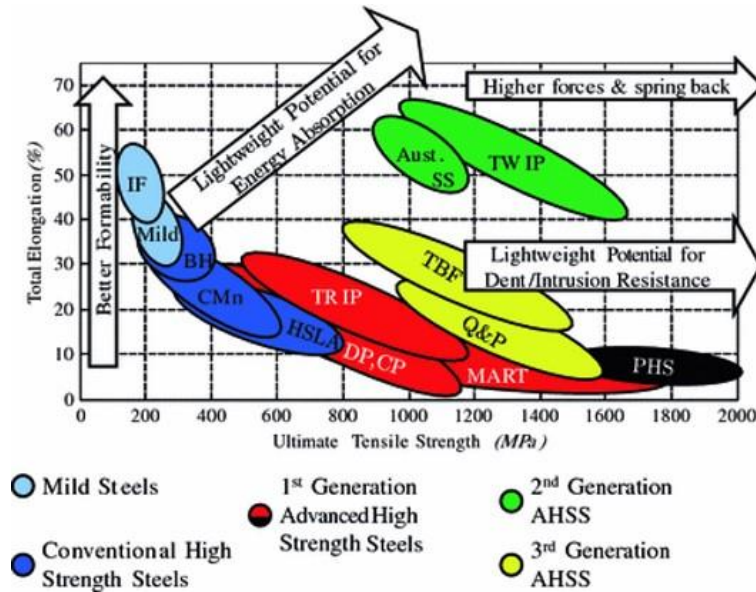


Figure 1.1: Total elongation vs. ultimate tensile strength diagram showing the different generations of AHSS [13]

A typical body-in-white structure of an automobile is composed of a wide variety of AHSSs. To assemble the components of the car body, joining is essential. Resistance spot welding (RSW) is the predominant joining technique employed in the automotive industry. As such, the safety of a vehicle is dependent by the quality of these welds. One drawback associated with the use of AHSSs over conventional mild steels is their susceptibility to unfavorable failure modes during loading [14]. Recent investigations indicate that welds made in steels with a higher carbon equivalent often exhibit a failure through the fusion boundary, commonly referred to as a failure through "halo ring" [15–17]. Despite this form of failure being observed in steels over the past decade, the reasons for its occurrence and proposal for effective mitigation remain unclear. Consequently, this study will focus primarily on investigating and detailing this aspect.

## 1.2. Objectives

This study aims to gain a fundamental understanding on the resistance spot weldability of Gen 3 AHSS, with the main focus on gaining a deep understanding of localized softening of FB (halo ring), which is a potential site for premature failure initiation in Gen 3 AHSS. The available literature does not offer a

complete understanding of the characteristics of the halo, the mechanism of its formation, failure behaviors or fracture mechanics, and how could it be eliminated. While interpreting recent literature, it can be hypothesized that the detrimental effect of the halo ring is significantly stronger in steels that have higher alloying content, especially solutes such as Mn, Si, and C. Hence, a newly developed Zn coated (GI) Gen 3 steel such as quenched and partitioned (Q&P) steel with a 1.2 mm thickness will be used to investigate the in-depth understanding of halo ring, its characteristics, formation mechanism, and solutions to prevent its detrimental effect. However, the results can apply to the other Gen 1 and Gen 3 AHSS with high carbon equivalent such as TRIP, DP, Medium Mn steels, etc. The main objectives are:

1. Develop a spot-welding schedule according to AWS standards and quantify the effect of the halo ring on the mechanical performance of Q&P 980 steel resistance spot welds (RSW).
2. Characterize the microstructure and formation mechanism of the halo ring (softened area near the HAZ and the hardened area adjacent the FZ).
3. Characterize the local fracture mechanisms (ductile or brittle, fracture mechanics is the study of crack propagation, i.e., mechanics of crack propagation) and mechanical properties of the halo ring.
4. Determine the phenomenological link between thermal history during welding and mechanical performances of the welds due to chemical inhomogeneity and phase transformations happening in the halo ring, with the help of mechanical and physical metallurgy.
5. Develop novel welding schedules to eliminate the halo ring from the spot welds by invoking metallurgical changes by modifying the thermal profiles of the halo ring.

### **1.3. Thesis Outline**

This thesis comprises of eight chapters. A comprehensive review of literature available on the Gen 3 steel spot welds and the fusion boundary softening are in chapter two. Additionally, chapter two reviews the proposed mechanism or hypotheses of halo formation in resistance spot welds. The initial main research chapter, chapter three, encompasses the optimization of spot-welding parameters, analysis of weld mechanical properties, and the application of the double pulse welding technique to eliminate the halo. It also provides a clear knowledge on the effect of local microstructural constituents on the mechanical properties. The influence of base material's chemical compositions on halo formation is detailed in chapter four. Chapter five focusses into a comprehensive understanding of the halo microstructure, proposing its formation mechanism with the assistance of TEM and EBSD. Chapter six provides an in-depth discussion on mitigating the halo ring's effects through a paint baking process, also exploring the micro-mechanical aspects of spot weld fracture with and without the halo ring. Lastly, chapter seven summarizes the major findings and offers recommendations for further advancing this research.

## Chapter 2. Literature review

### 2.1. Advanced High Strength Steels

Advanced high strength steels are typically defined as steels with ultimate tensile strengths higher than 550 MPa, and are primarily in sheet form for automotive structures [6]. They offer uniquely high strength to weight ratio and optimized formability, which allows automakers to greatly reduce the overall weight of the car body. The microstructure of these steels consisted of a combination of martensite (M), ferrite (F), and retained austenite (RA) to achieve an exceptional combination of strength and ductility.

With the continuous demands for higher strength and ductility, steelmakers have continued to develop new AHSS grades by developing new thermo-mechanical processing routes to achieve improved combinations of strength and ductility [18,19]. It is worth noting that the superior mechanical properties of these AHSSs are mainly attributed to the presence of higher alloying content, leading to the combinations of complex microstructural features [20–22]. As mentioned earlier, the Gen 1 AHSS has a higher strength to ductility ratio than the conventional high strength steels and can be produced with only slight increases in production cost compared to conventional HSS. However, it should be noted that not all Gen 1 steels are as cost-effective (cost per kg) as conventional HSS. Further requirements in the automotive industry led to the development of Gen 2 AHSSs such as TWIP and AS steels with superior strength and ductility [23,24]. The higher cost of production and complex manufacturing process made this Gen 2 steel less economically feasible in the automotive production line. Furthermore, to fill the gap between the Gen 1 and 2 steels, the Gen 3 AHSS was developed [6].

Several Gen 3 AHSS have been developed in the past more than a few years. However, only three types are currently in production. They are Q&P steels [25,26], TRIP-assisted bainitic ferrite (TBF) steel [27], and Medium Mn TRIP steel [28–30]. Figure 2.1 shows the strength comparison of typical Gen 3 AHSS with Gen 1 AHSS [31]. Note that the Gen 3 steels have much higher ductility than the similar strength Gen

1 steels. Moreover, it should also be noted that the nano steel 1200 mentioned in Figure 2.1 is only under laboratory development.

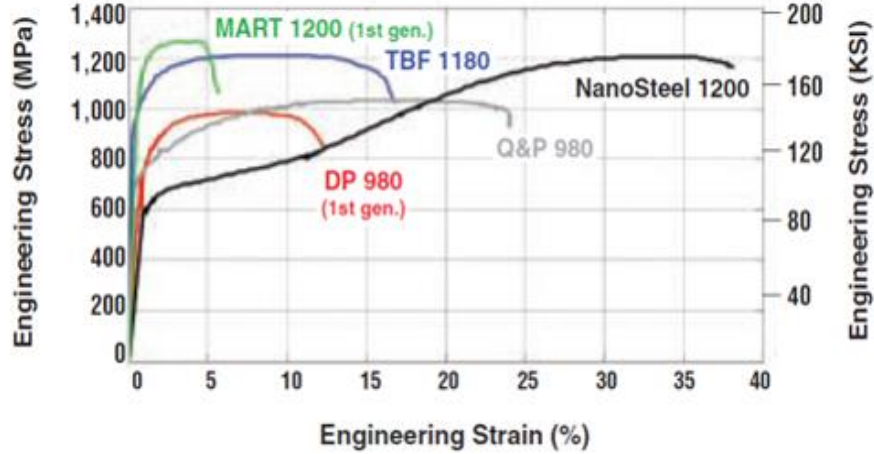


Figure 2.1: Engineering stress-strain diagram of AHSS compared with 1st gen AHSS [31]

The interest in steels processed by quenching and partitioning (Q&P) is due to its ability to achieve a good combination of strength and ductility with fewer alloying additions compared to the Gen 2 steel. The quenching and partitioning heat treatment was originally proposed by Speer et al. [32] as a new approach to producing sheet steels consisting of a martensite matrix with a considerable amount of retained austenite (RA). The heat treatment consists of three stages: an initial quenching stage, a carbon partitioning stage, and a final quenching stage as shown in Figure 2.2. In the first stage, the austenitized steel is quenched to a temperature between the martensite start ( $M_s$ ) and martensite finish ( $M_f$ ) temperature. This quenching process partially transforms the austenite microstructure into primary martensite ( $\alpha'_p$ ) and the remaining austenite will stay untransformed. The carbon content in  $\alpha'_p$  and the untransformed austenite is the same as that of austenite after this stage. However, the untransformed austenite present in the steel is not stable. During the partitioning stage, the steel is heated slightly above the quenching temperature for a short period of time, so carbon atoms diffuse from the supersaturated  $\alpha'_p$  into the untransformed austenite. This movement of C from the  $\alpha'_p$ , increases the C content of the remaining retained austenite, lowering the  $M_s$  temperature of the now carbon enriched austenite, thereby stabilizing it when the material is cooled to room

temperature. Thus, the stability of the untransformed austenite is a function of carbon content. Furthermore, if the amount of carbon in the steel is insufficient to fully stabilize the untransformed austenite, some of the untransformed austenite will transform to secondary martensite ( $\alpha'_s$ ) rather than remaining as retained austenite, during the final stage of quenching [33].

The final microstructure depends mainly on the initial quenching temperature [34]. When a lower quenching temperature (closer to the  $M_f$ ) is used, the final microstructure will consist of carbon enriched austenite, and  $\alpha'_p$  with a very low solute carbon content. When the quenching temperature is higher, the microstructure will consist of austenite with less enriched carbon,  $\alpha'_p$  with very low carbon content, and  $\alpha'_s$  with the same carbon content as the austenite phase. Furthermore, the higher the initial quenching temperature, the higher the amount of RA in the final microstructure [35]. However, there is an optimal quenching temperature required to maximize the amount of RA in the microstructure and the quenching temperature depends on the composition the steel being used [36].

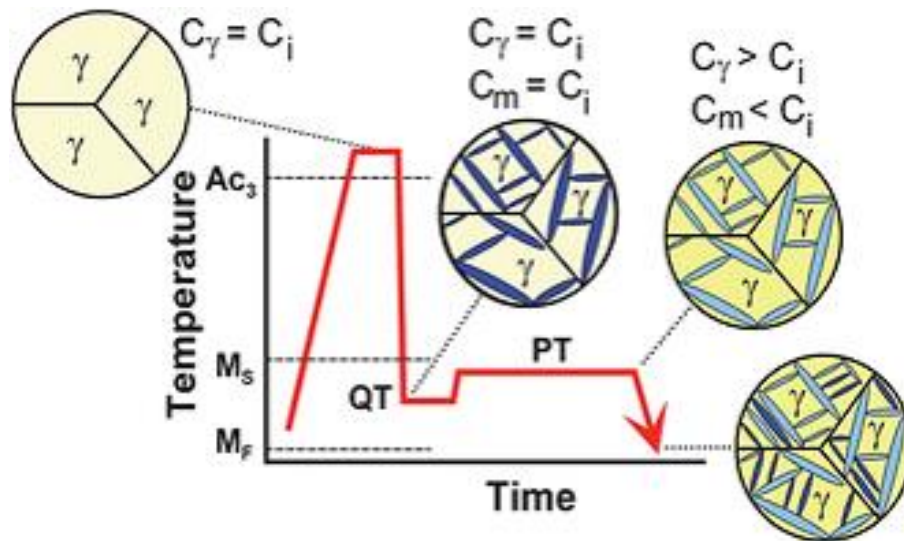


Figure 2.2: Schematic diagram showing the thermal profiles and phase transformation happening in Q&P steels [33]

## 2.2. Resistance Spot Welding

To produce body-in-white structures, resistance spot welding (RSW) is predominantly utilized in the automotive industry. A typical auto body consists of 3000 to 6000 spot welds, which also include a wide variety of steel sheet combinations. This large number of spot welds in a particular auto body makes it the most widely used joining technique in auto body assembly [37].

The four stages of the RSW process when making a typical weld are shown in Figure 2.3 [38]. First, the two or more sheets of materials are placed between water-cooled copper electrodes mounted in a pneumatic/hydraulic jaw. In the second stage, the sheets are squeezed by the electrodes, and the electrodes maintain a predefined load through the clamping system on the samples followed by a high current passing through the electrodes for a short period of time. Fundamentally, the electrodes transfer a high current through the work piece while applying a particular force to the sample and act as a heat sink to promote the nugget formation (the nugget is a pool of molten metal that quickly cools and solidifies into an elliptical joint). During the third stage (cooling and post-clamping), the current is shut off and the nugget formation occurred by cooling through the electrodes (heat dissipation). Once the weld cycle ends, the electrodes are retracted from the sample leaving behind a sound weld nugget in the sheet-sheet interface, in the last stage.

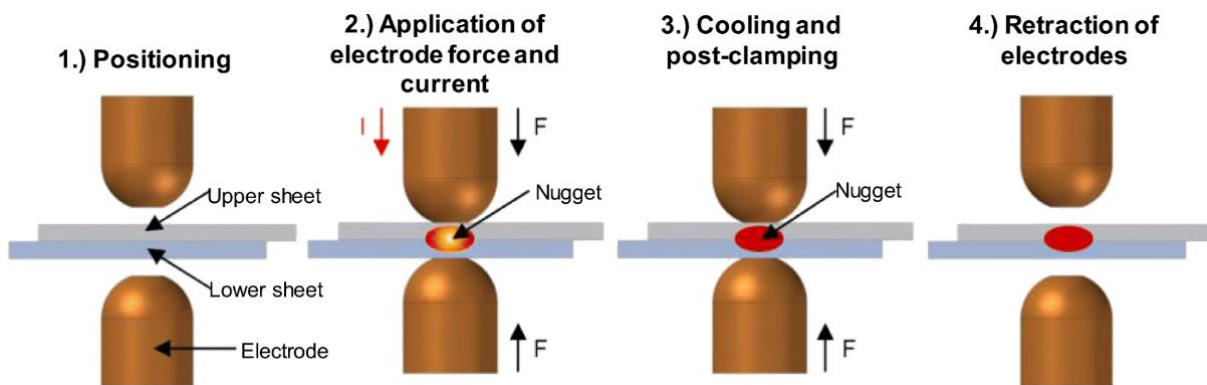


Figure 2.3: Stages of RSW process showing the nugget formation [38]

RSW process uses joule heating to produce the necessary heat to melt the material in the weld [39]. As a high current passes through a conductor, heat is generated due to the collided movement of electrons within the workpieces being joined, which converts the high electrical energy into thermal energy. The amount of heat generated ( $Q$ ) can be modeled by Equation 2.1.

$$Q = \int_0^t I^2 R(T) dt \quad 2.1$$

Where  $I$  represent the welding current,  $R$  is the total resistance,  $T$  is the temperature, and  $t$  is time. The amount of heat generated is a function of current, bulk resistance, and the extent of time the current passes through the conductor. As seen from the Equation 2.1, current plays a major role in heat generation. In a typical RSW process, the total resistance is a combination of seven individual resistance as mentioned in Figure 2.4. It consisted of two separate bulk resistance of the electrode ( $R_1$  and  $R_7$ ), two electrode-to-sheet contact resistance ( $R_2$  and  $R_6$ ), two material bulk resistance ( $R_3$  and  $R_5$ ), and sheet-to-sheet contact resistance ( $R_4$ ) [40]. In general sheet to sheet, the contact resistance was considered to be the highest resistance of all, and it leads to the highest heat generation in that area. Hence, the first melting happens and will lead to the nugget formation on the sheet-to-sheet interface.

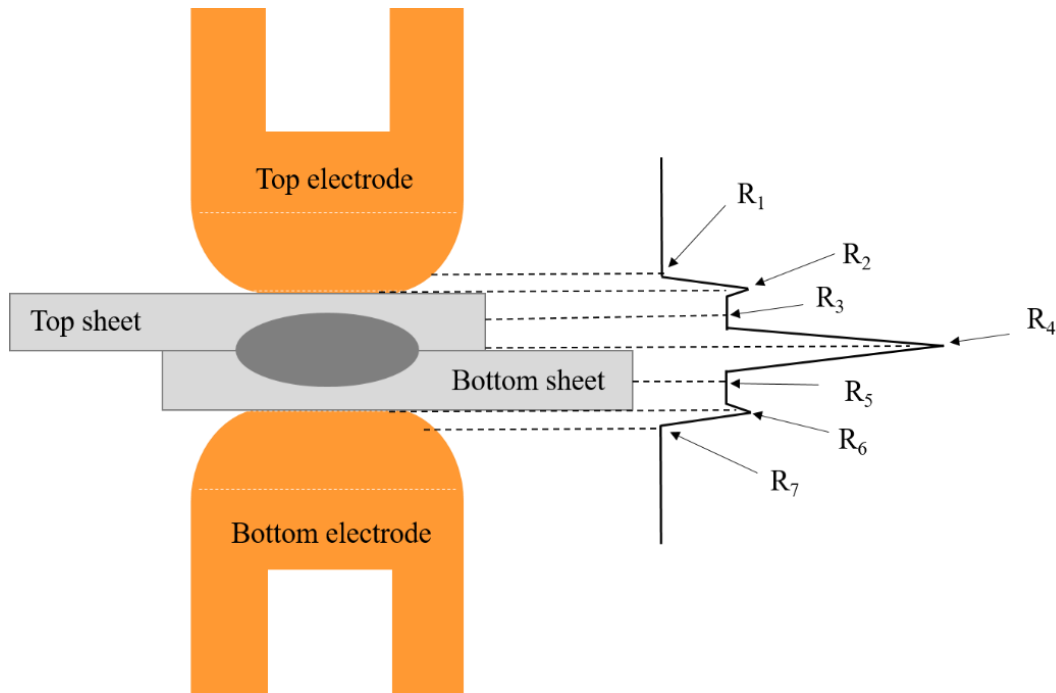


*Figure 2.4: Schematic representation of RSW and the relative resistance across a spot weld.*

Electrical resistance is a function of temperature (see Equation 2.1). It is known that electrical resistivity increases with an increase in temperature [41]. Longer welding time during spot welding generates more heat, which leads to a comparatively bigger nugget. Furthermore, the contact resistances ( $R_2$ ,  $R_6$ , and  $R_4$ ) depend on the surface conditions of the material being welded, and the electrode force (the pressure applied through the electrode). High electrode forces break down the surface asperities on the sheet surfaces, thereby lowering the contact resistance, that reduces the heat generation.

The time duration between the current cut-off and the retraction of the electrodes is referred to as the cooling time. Higher cooling times increase heat dissipation through the electrodes. The cooling time promotes nugget solidification. In the case of steels with high hardenability, higher cooling rate promote high carbon martensitic microstructures, whereas the low cooling rate could lead to the auto tempering of the martensite, which in-turn leads to the formation of softer microstructures in the nugget. However, it should be noted that the cooling rate during the spot welding is always higher than the critical cooling rate needed to form martensite in fusion zones [42].

In spot welding, weld nuggets are considered acceptable, if they are of sufficient diameter (at least  $4\sqrt{h}$ , where  $h$  is the thickness of the single sheet used), and are free of detrimental defects such as shrinkage, porosity, and cracks [43]. Acceptable weld nuggets are formed, when the welding current and welding time varied such that the range at which the combination of these parameters results in a sound weld nugget is defined as the weld lobe. A typical weld lobe for a Gen 3 980 steel is shown in Figure 2.5 [44]. It should be noted that the weld lobe curve is plotted with a constant electrode force. The lower limit is defined as the combination of current and time resulting in a nugget diameter of  $4\sqrt{h}$ , according to the AWS D8.9 standard [45]. While keeping the constant welding current, a higher welding time causes a higher nugget size. The upper limit of the weld lobe is defined by a phenomenon called expulsion, where molten material is ejected from the joint due to extremely high heat input. Therefore, expulsion (reduces the nugget diameter) should be avoided, since it deteriorates the mechanical performance of the welded joints [46,47].

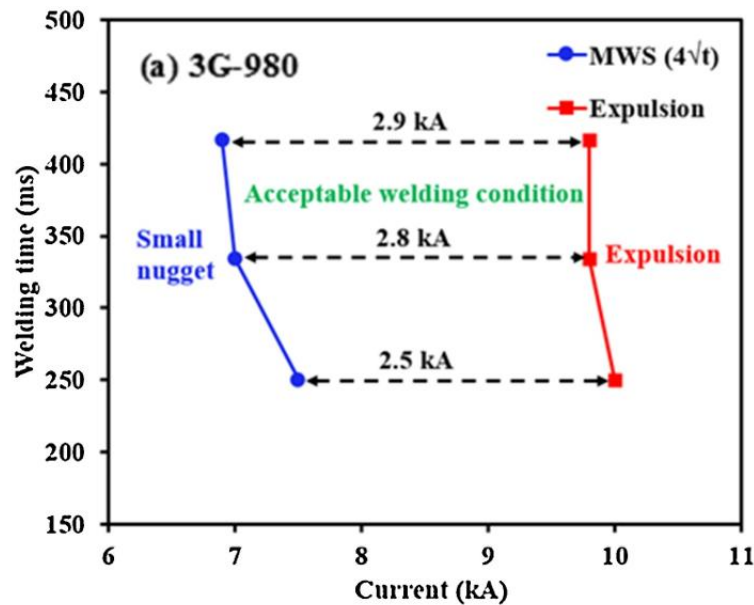


Figure 2.5: A typical weld lobe curve of Gen 3 980 steel [44]

Aside from factors from Equation 2.1, electrode geometry, electrode force, and cooling water flow rate are the other factors that influence the nugget formation. The weld lobe can be adjusted by changing the electrode force. When the electrode force is applied during welding, it lowers the contact resistance due to

the breakdown of surface asperities. In other words, higher electrode force will lead to less heating due to the reduced contact resistance [40]. In summary, electrode force is highly related to the contact resistance, especially in extremely low electrode force cases, since small changes in electrode force can significantly vary the contact resistance. According to Wohner et al. [48], a lower electrode force promotes early expulsion due to a decrease in retention pressure of the molten metal. Therefore, electrode force should be chosen accounting for steel grade and thickness in such a way that the chosen electrode force will not favor either early expulsion or delayed heat generation [49]. Suggested forces for a wide variety of materials were given in the AWS D8.9 standard [45]. Furthermore, electrode geometry is related to the current density. In general dome, and radius typed electrodes were commonly used in the automotive industry. Electrodes with radius contact faces have more size to surface area ratio compared to the dome shaped electrodes. Dome-type electrodes can provide a higher current density than the electrodes with radius contact faces. The electrode geometry also changes the nugget growth kinetics, because electrode geometry affects the cooling rate by changing the contact area between the electrode and the material being welded [50].

In addition to the process parameters, material thickness, properties, and coating also have a strong effect on weld development. As mentioned earlier, the minimum nugget size required for an acceptable spot weld is a function of material thickness. Higher thickness material will also have higher bulk resistance due to longer current path. Oikawa et al. [13] showed that high-strength materials are resistant to indentation thereby decreasing the weldability and may induce hot cracking. On the other hand, softer materials can experience deformation under electrode force, which can significantly reduce the nugget size. The type of coating used in the material and its thickness (or coating weight) also affects the nugget growth thereby the mechanical performance of the welded parts. Murugan et al. [51] reported that Zn coating in AHSS leads to liquid metal embrittlement (LME) cracking which may deteriorate the mechanical performance of the weld [37]. However, the influences of material parameters are out of the scope of the current study.

### 2.3. Microstructural Transformations During the RSW Process

The temperature experienced during the RSW process affects the surrounding microstructure of the nugget through different solid state phase transformations. The transformations that happen in each region are a function of the temperature and the time at which that temperature is applied [52]. A schematic diagram of a typical Q&P 980 steel spot weld showing different regions of the heat-affected zone (HAZ) is shown in Figure 2.6a. The fusion zone (FZ) is the solidified molten metal that reaches above the melting temperature ( $T_m$ ) of the material during RSW (see Figure 2.6b). The material surrounding the FZ that undergoes solid state transformations of microstructure due to the temperature exerted in the FZ is called the HAZ [53]. The peak temperature in the HAZ is below the  $T_m$  at the FB and the temperature gradually decreases towards the BM. Due to these various temperatures, different combinations of microstructural constituents can be expected in the HAZ as shown in Figure 2.6c through f [54].

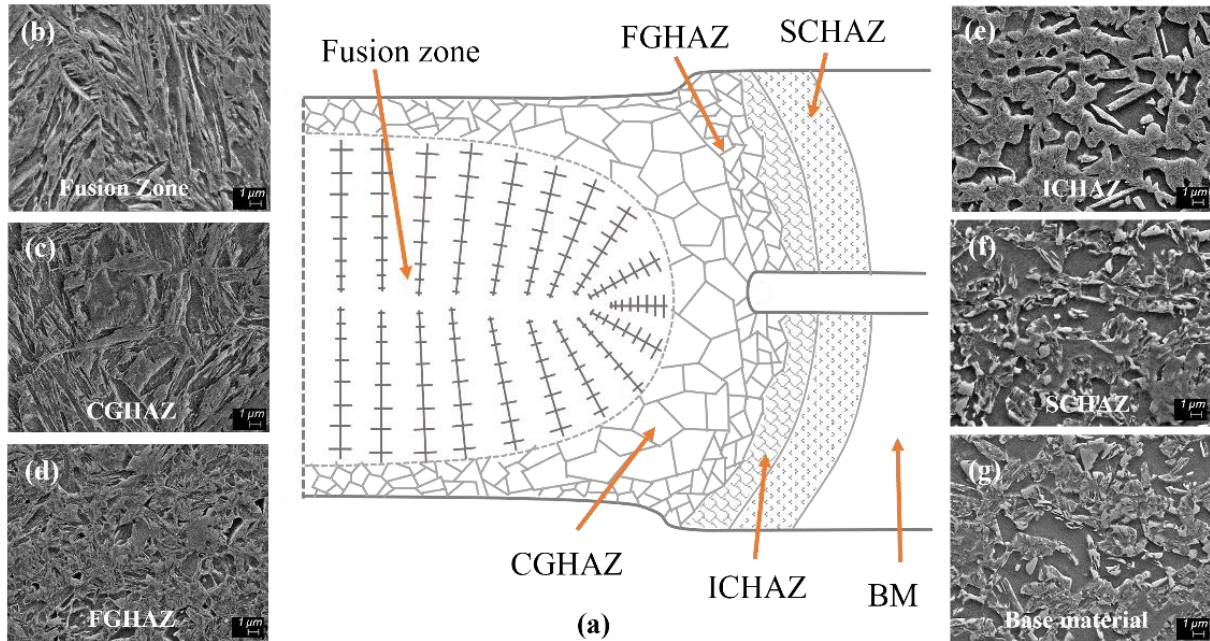


Figure 2.6: Schematic diagram showing (a) microstructural transformation that happened at a typical Q&P 980 steel weld [54], and corresponding microstructures at (b) Fusion zone, (c) CGHAZ, (d) FGHAZ, (e) ICHAZ, (f) SCHAZ and (g) Base material

The HAZ can be characterized into four sub-regions. The peak temperature reached in the region adjacent to the FZ is just below the  $T_m$ , this is called coarse-grained HAZ (CGHAZ). It is widely accepted that the temperature in the CGHAZ region surpasses 1100 °C in steel and is enough to promote grain growth. Complete austenization followed by the martensite transformation is the key microstructural transformation happening in this region. Large martensite packets with higher prior austenitic grain boundaries (PAGB) compared to the other parts of the weld are the characteristics of CGHAZ and are shown in Figure 2.6c. In the area where the peak temperature is below 1100 °C but still above  $A_{c3}$  (the temperature at which a fully austenitic microstructure develops during heating) is the region where recrystallization dominates and is characterized as fine-grained HAZ (FGHAZ). Figure 2.6d shows an example of an FGHAZ microstructure of a Q&P 980 steel spot weld. Martensite transformation from fully austenite microstructure is the dominant phase transformation that occurs in the FGHAZ. Therefore, fine martensite grains forming from relatively fine PAGBs are the characteristics of FGHAZ. The area where the peak temperature reaches between  $A_{c1}$  (the temperature at which austenite starts forming during heating) and  $A_{c3}$  is characterized as intercritical HAZ (ICHAZ). The final microstructure of the ICHAZ generally depends on the BM microstructure [55]. For instance, Q&P steel with a mixture of ferrite, martensite, and RA in the BM microstructure, can partially transform into austenite during welding. This austenite can be transformed back to a complex martensite austenite constituent, with the untransformed microstructures from the BM during cooling, as shown in Figure 2.6e. The region outside of the ICHAZ, with a peak temperature below  $A_{c1}$  is called subcritical HAZ (SCHAZ). SCHAZ is the region where either the softening or secondary hardening happens [56]. Tempering of martensite present in the BM leads to the formation of soft microstructures such as tempered martensite (TM) (see Figure 2.6f), and is referred to as softening in SCHAZ, whereas the bainite present in the BM can provide carbon by dissociating the carbides present in the bainite, which can promote the formation of secondary carbides such as  $M_2C$  or  $M_{23}C_6$  and is referred to as secondary hardening [56,57]. A typical microstructure of Q&P 980 steel with ferrite, martensite, and retained austenite is also shown in Figure 2.6g. These differences in microstructures in a particular spot weld make it complex while

mechanical loading since each microstructural constituent has its specific mechanical behaviors. For instance, the martensitic microstructure is hard and brittle, compared to the ferritic microstructure, which is soft and ductile. More detailed explanations of these behaviors will be discussed in further sections.

## 2.4. Mechanical Performance of the Weld

In applications, spot welds are subjected to complex loading conditions, which specifically include shear (TS) loading (relative displacement of adjacent sheets), and tensile (CT) loading (sheets are pulled apart along their normal directions). Otherwise, the tensile shear test (TST) and cross-tensile test (CTT) are the most common tests used to evaluate the mechanical performances of the spot welds [58,59]. Standard coupon sizes recommended by AWS D8.9M are used to perform these mechanical tests [60]. A typical example of the TST and CTT test coupons were shown in Figure 2.7.

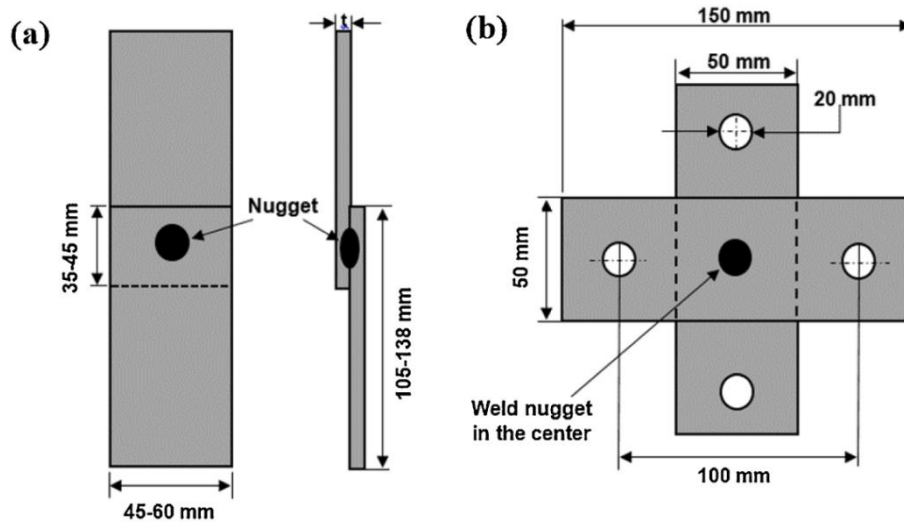


Figure 2.7: Schematic representation of (a) TST and (b) CTT coupons [44]

The measured data after TST and CTT test comprises a load-displacement curve, as shown in Figure 2.8. Apart from the uniaxial tensile test of the materials, the strength of the spot welds is usually represented in peak load (kN), which is represented as  $P_{\max}$ . Absorbed energy (see  $W_{\max}$ ) and displacement (see  $L_{\max}$ ) before failure initiation is also important parameters used to characterize the spot welds. The area under the

load-displacement curve up to peak load is considered the absorbed energy, which is an indication of the toughness of the spot welds [61]. Several studies reported the area under the whole load-displacement curve as the energy absorbed [62–64]. Although both have been used in different studies, the accurate choice of the curve in different situations still has not been resolved. According to the candidate's knowledge, the area under the curve up to peak load should be considered when analyzing the mechanical properties of a single spot weld, since the test coupon experiences significant rotation after the peak load. Shojaee et al. [65] conducted a comprehensive investigation into how these changes in loading orientations affect the mechanical properties of spot welds due to coupon rotation during loading. They evaluated the mechanical properties using KS-II tests (two U-shaped formed channels welded together at the center of the mating surfaces and pulled in different orientations) and determined that there was minimal rotation and slippage of the coupon within the KS-II testing fixture. To establish a force-based failure criterion, they proposed a novel triangulation method in conjunction with a Digital Image Correlation (DIC) based imaging technique. Additionally, the proposed method enables the calculation of energy dissipation by nugget deformation independently in both the shear and tensile directions.

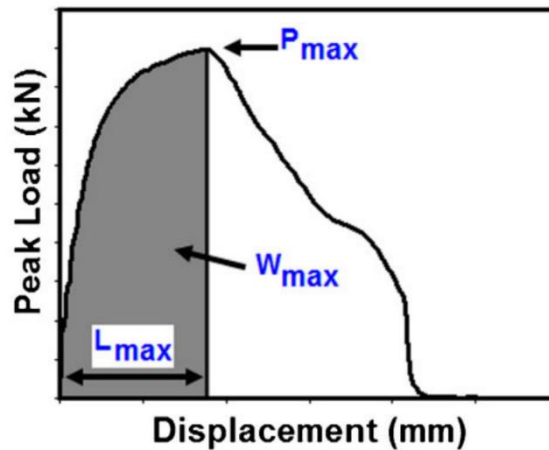


Figure 2.8: Typical load-displacement curve during the mechanical test [61]

It is widely believed that the nugget is subjected to greater shear stress in TS loading conditions. Therefore, the larger the size of the nugget, the greater its ability to resist shear stress [58,66,67]. The peak load and

energy absorption are proportional to the connection area between the sheets. In the case of normal loading conditions, the tensile stress on the nugget occupies a dominant position rather than the shear stress. Hence, the toughness of the nugget plays a dominant role during CTT. On the other hand, the peak load of CTT is directly proportional to the fracture toughness of FZ and inversely proportional to the hardness of FZ [68]. As mentioned above, maximizing the contact area between the sheets by maximizing the nugget diameter is favorable for improving the TS performance of the weldment, whereas, reducing the hardness of the FZ and making tougher microstructures is beneficial to improving the CT performance. However, the nugget size obtained by the conventional welding process is limited, which greatly limits the TS properties of steel welded joints. Moreover, the high C content and alloying elements lead to the formation of brittle martensite with high hardness in the nugget, which further deteriorates the CT performance of the joints [69,70]. Hence, it is important to develop new welding schedules and processing techniques to overcome the above-mentioned limitations for using the Q&P steels in the auto bodies.

#### 2.4.1. Failure Characterization

Post failure analysis of spot welds provides insights into the local strength in the spot welds. This analysis includes an understanding of failure locations, crack propagation path, type of fracture, the overall shape of the weld after separation, etc. One commonly noted metric is failure mode, a qualitative metric, that indicates the mechanical performance of that particular joint [53]. Figure 2.9a shows typical failure modes that most likely occurs in spot welds during mechanical loading [61]. Interfacial fracture (IF) is the failure that occurs through the weld nugget, which is not desirable for a spot weld joint. It is a brittle mode of failure with poor energy absorption capability. Numerous studies pointed out IF during CTT of welds even with the best welding parameters due to the presence of hard and brittle martensite in the FZ [62,64,71–73]. IF failures also result from small nuggets and defects such as shrinkage and porosity [74]. That means IF failure occurs if the force required to initiate fracture across the faying surface is lower than the force required for through-thickness pullout [75]. According to the AWS D8.1 standard IF failure is characterized

as a mode 7 fracture. Partial interfacial fracture (PIF) (mode 5) starts as IF mode and then transforms into the through-thickness direction (see Figure 2.9b). Some critical microstructural features present in the periphery of the FZ, the presence of defects, geometrical factors such as sharp notch geometry, and complex loading conditions could be the reasons behind this failure mode. Figure 2.9c shows pullout fracture (PF) through upper critical HAZ (UCHAZ). The UCHAZ is consist of CGHAZ and FGHAZ. This failure can also happen through SCHAZ. PF type of failure mode (mode 1) is considered the most common and desirable type of failure in spot welds due to the high energy absorption capacity [53,61,76,77]. Apart from these failure modes, there is mode 2, and 4, which is corresponding to partial thickness fracture with button pull, and interfacial fracture with button pull and partial thickness fracture, respectively. These failure modes 2, and 4 are considered as the transitional failure modes from mode 1 to 3 and mode 3 to 5, respectively. Furthermore, Mode 6 is an interfacial fracture with partial thickness fracture. Although there are 7 failure modes according to the AWS D8.1 standard [78], modes 4, and 6 are not commonly seen in 3<sup>rd</sup> generation AHSS.

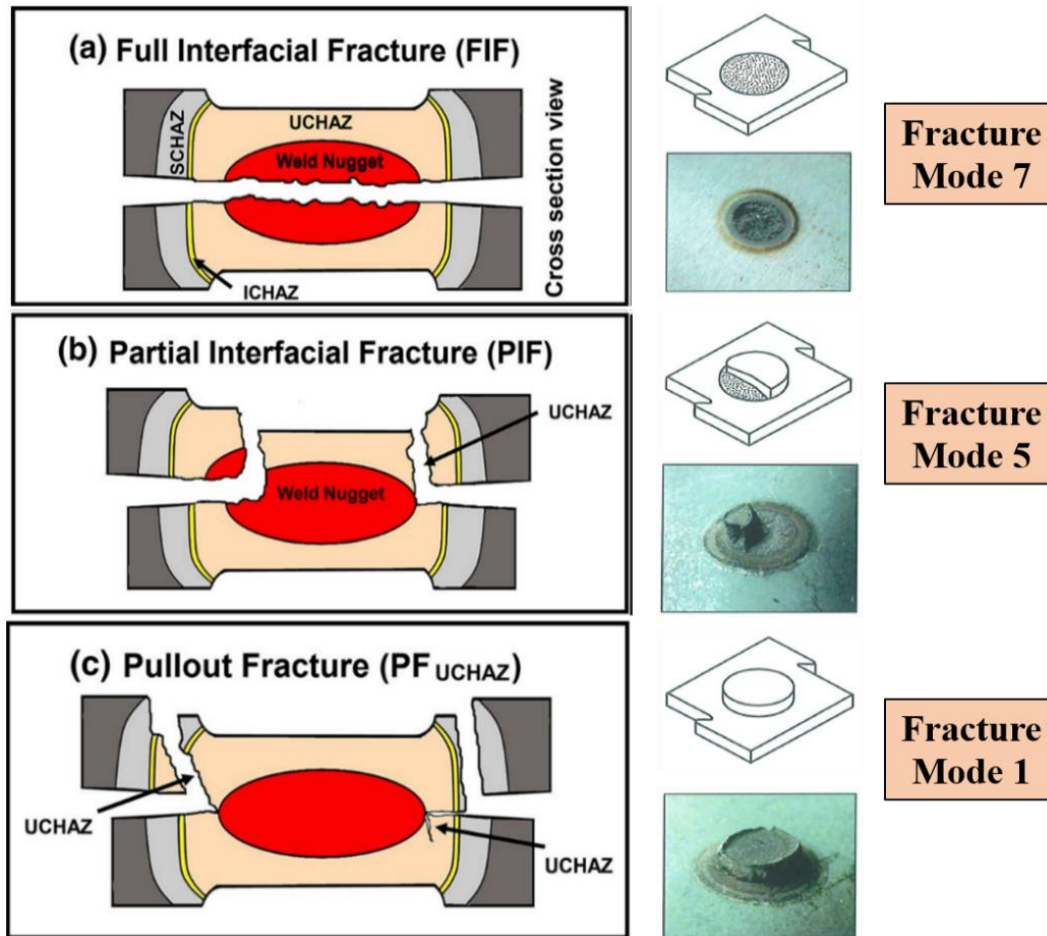


Figure 2.9: Typical failure modes after mechanical tests of RSW and its corresponding fracture mode according to the AWS D8.1 standard [53,78]

Recent investigations show a new failure location, which progresses along the fusion boundary (FB) of the spot welds, as shown in Figure 2.10 [79]. The failure is occurring exactly through the FB of the nugget and results in comparatively poor energy absorption capacity than the PF. This type of partial-thickness failure (PTF) can be characterized as mode 3 fracture. It has been reported that due to the geometrical factors, failure starts at the sheet-sheet interface and is followed through the FB due to the comparatively lower hardness of the FB [72,73,80–82].

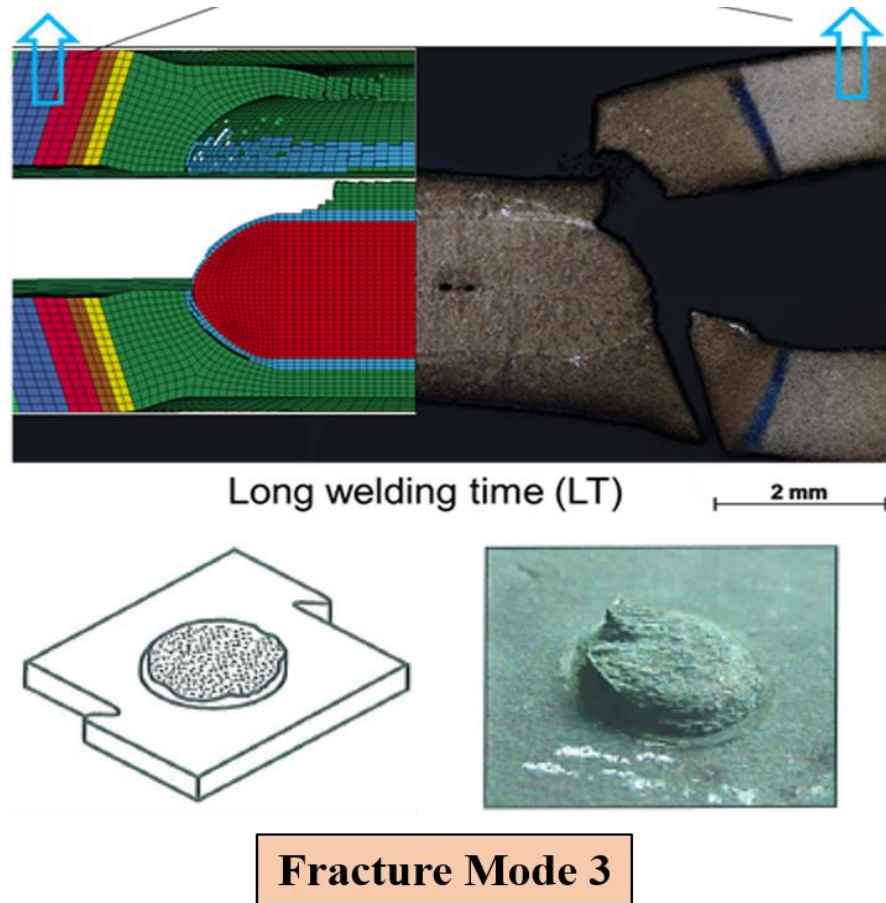


*Figure 2.10: Partial thickness type failure mode (mode 3) along the FB of the spot welds [79]*

As mentioned above, other than the geometrical factors and loading characteristics, properties of the regions in and around the weld play a crucial role in the FB failures in the spot welds. Characteristics of the FZ, different regions of HAZ, and BM changes the loading response of a weld [83]. The local properties of these regions can be generally evaluated by microhardness analysis. Typically, FZ and UCHAZ are the regions with the highest hardness in Gen 3 AHSS since martensite is the primary component in BM microstructure. The martensitic transformation is related to the cooling rate. Generally, the critical cooling rate required to form martensite should be greater than  $\sim 27^{\circ}\text{C/s}$  and the cooling rate is a function of material composition and PAGS [84]. It should be noted that the cooling rate associated with the spot welding is in the order of  $2000^{\circ}\text{C/s}$  [1,85].

Understanding fracture initiation and propagation in new Gen 3 AHSS are complex because of the complex microstructure. As mentioned above, cracks can be initiated and propagated through either the UCHAZ, SCHAZ, or base material, with mode 1 failures. However, failure through the FB could be either mode 2, 3, 4, 5, or 6 failures. It should be noted that these failure modes are variants of the PIF failure mode. Different modes of failure are aided by separate mechanisms. For example, failure along the SCHAZ (mode 1) is due to the stress concentration and necking phenomenon, whereas the failure through the halo (mode 3), or UCHAZ (mode 1) was due to the crack initiation and propagation mechanism. To elaborate on these ideas, necking occurs in comparatively low-stress levels and high strain during failure through the soft SCHAZ. In Gen 3 steels, the microstructure within the SCHAZ is more ductile and strain hardening can happen. Figueredo et al. [17,86] recently reported a secondary hardening phenomenon in SCHAZ of Gen 3 steel, which agrees with the previous studies showing that the dislocation movement can be pinned by the fine carbides responsible for the secondary hardening. On the other hand, the presence of microstructure with lower toughness due to high hardenability shifts location of the stress concentration area to the geometrical notch (sheet-sheet interface) in low strain values which can cause premature failure of the spot welds [61].

## **2.5. Post Pulsing of Resistance Spot Welds**

One effective method for enhancing the mechanical properties of resistance spot welds, by altering their microstructure, involves reheating the weld through the application of a secondary current pulse, commonly referred to as post pulsing [10,51,87–89]. Various welding schedules were implemented in the literature to improve the mechanical performance of spot welds. Chabok et al. [89] employed a post welded heat treatment (PWHT) on DP steels and observed a modification in the microstructure from columnar to equiaxed structures in the outer part of the FZ. They established that the microstructure of this zone was responsible for preventing the crack propagation. EftekhariMilani et al. [14] also studied the effect of post pulsing on the 3G AHSS and determined that application of PWHT led to the homogenization of alloying elements at the edge of the FZ. This reduced segregation of the elements led to the further enhancement of

the mechanical properties. However, the main disadvantages with this welding schedules is the implementation of long cooling time between the welding pulses, making it unfavorable for industrial applications.

Betiku et al. [87] on the other hand proposed a comprehensive understanding on how in-situ post weld heat treatment parameters modifies the grain morphology, improving the mechanical performance of the press hardened steel (PHS) spot welds (see Figure 2.11). They applied a very short cooling time between the pre- and post-weld pulses and proved that employing a post-weld pulse during the austenitic phase (short cooling time), which proved more effective for refining grain compared to a longer cooling time that leads to martensite formation prior to the post-weld pulse application. It was found that a cool time of 100 ms, prior to post-pulsing, resulted in the formation of equiaxed grains at the edge of the FZ as shown in Figure 2.11a with a lower aspect ratio as shown in Figure 2.11b. For a cool time of 300 ms, it was observed that the weld first transformed to martensite and on re-heating austenite reversion occurred. After the cooling schedule, the austenite transformed back to martensite retaining the initial epitaxial solidification structure. Thus, a shorter cool time where austenite is formed during the intermediate cooling proved effective for in-situ grain refinement than the longer cool time where austenite reversion occurred on reheating of the weld.

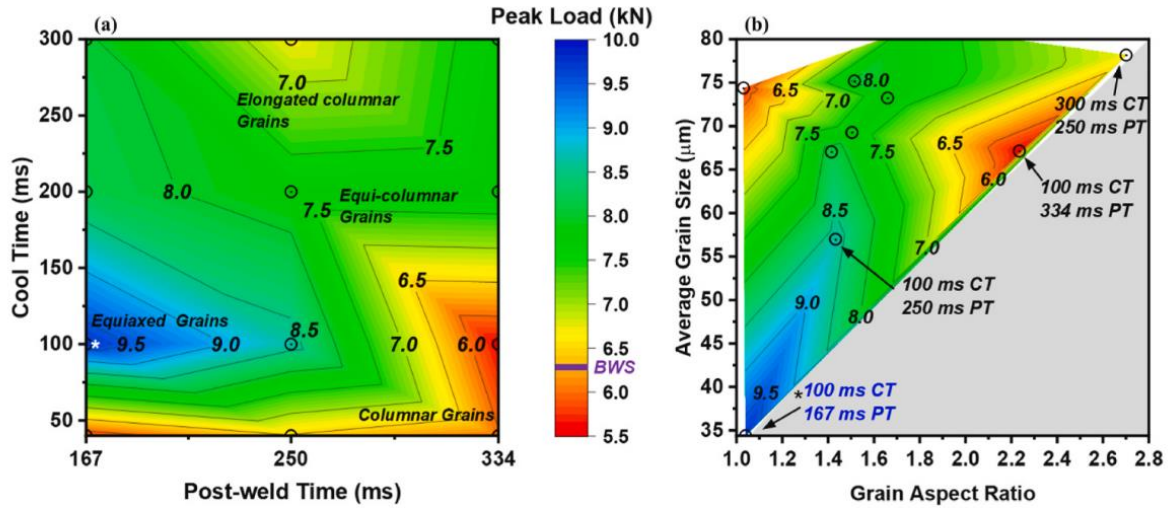


Figure 2.11: (a) Peak load of selected PWHT schedules, and (b) corresponding changes in grain size and morphology with peak load [87]

These experimental observations suggests that post pulsing can serve as a viable solution for altering the microstructures of resistance spot-welded AHSSs. The application of post pulsing has the potential to enhance the microstructure specifically at the weld edge. However, it is important to note that while post pulsing proved effective, it also prolongs the welding cycle and, consequently, the overall welding time. This extension of welding time could be considered a drawback, particularly in an industrial setting.

### 2.5.1. Paint Baking (PB) Effect

Followed by the completion of the fabrication processes and assembly of different components, the automotive body-in-white undergoes a painting procedure to apply a protective and visually appealing coating to the vehicle structure. Subsequently, the applied paint undergoes a curing process, necessitating the placement of the entire body-in-white in an oven at around 180 °C for a duration of 20-30 minutes. The purpose of this cycle is to bake the paint layers on the structure while simultaneously hardening and curing the coatings and adhesives. After this curing cycle, resistance spot welds in steels shows an enhancement in strength and energy absorption capability [42,54,90–97]. Additionally, the thermal cycle may temper the martensite. Depending on the alloying content of the base material, the PB cycle could induce different

stages of martensite tempering [54]. It has been reported that the segregation of carbon and the precipitation of  $\epsilon$  carbide occurs in the initial stages of the tempering, typically within the temperature range of 100-200°C [98].

One of the typical examples [54] highlighting the impact of PB on spot weld performance is presented in Figure 2.12. In Figure 2.12a, the as-welded crash exhibits unfavorable performance, characterized by weld failure and sheet delamination. Conversely, the post-PB scenario in Figure 2.12b demonstrates substantial improvements, underscoring the importance of comprehending the influence of PB on the mechanical properties of welds.

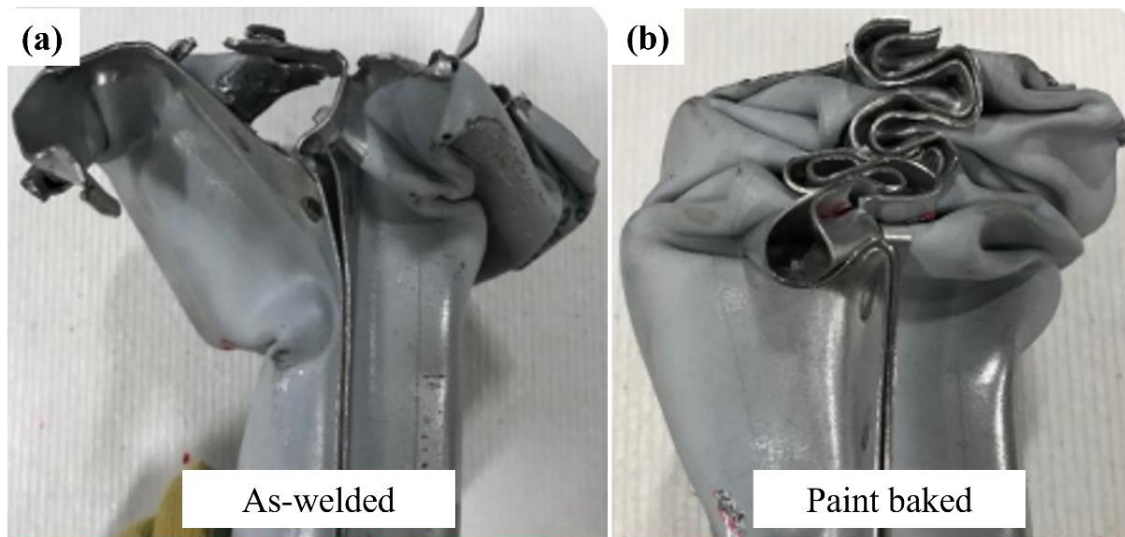


*Figure 2.12: A typical Gen 3 steel crash can (a) as welded, and (b) paint baked. After the crash test shows the higher energy absorption capacity of the welds after paint baking [54]*

Tumuluru et al. [42] documented the impact of a baking cycle on the structure and properties of resistance spot welds in 780 MPa DP and TRIP steels. The study reported that post-weld baking led to a roughly 6% increase in the load-bearing capacity of the weld in shear tension compared to the as-welded condition. This enhancement was attributed to the strain aging of ferrite and the tempering of martensite after baking in dual-phase and TRIP steels, respectively. Shamsujjoha et al. [94] compared the PB on mechanical property sensitivity of 780 MPa hot-formed (HF) and TRIP steels as shown in Figure 2.13. The microhardness map

showed that in the DP steel, there was a negligible difference in microhardness between the as welded and PB conditions, as shown in Figure 2.13a. Conversely, in HF steel, a slight reduction in hardness was observed after the PB process. Following the PB process, the energy absorption of the HF steel spot welds rose from  $21.4 \pm 0.1$  to  $46.4 \pm 6.1$  J. In contrast, the energy absorption of the DP980 welds remained constant. Similarly, the CTS improved from  $3.5 \pm 0.1$  to  $6.4 \pm 0.8$  kN for the HF steel, as illustrated in Figure 2.13b. It was found that precipitation of fine carbides which will pins the movement of dislocations, from the decomposition of fresh martensite during PB, is the reason for improving mechanical properties of the HF steel.

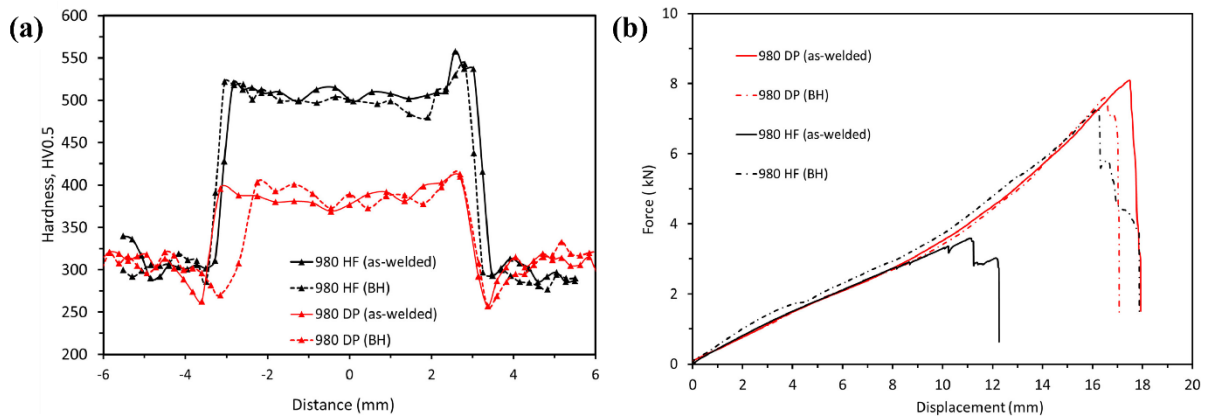


Figure 2.13: (a) Microhardness profile and (b) cross tension load-displacement curve of both 980 HF and 980 DP before and after paint baking, respectively [94]

Recently, Manladan et al. [16] investigated the effect of PB on 30MnB5 hot-stamped steel resistance spot welds. They observed that the as-welded condition failed through the FB and the PB condition the failure occurred in the FZ, as shown in the Figure 2.14. Although they observed that PB improved the energy absorption capacity of the spot welds, the reason for the crack propagation mechanism before and after the PB process was unknown.

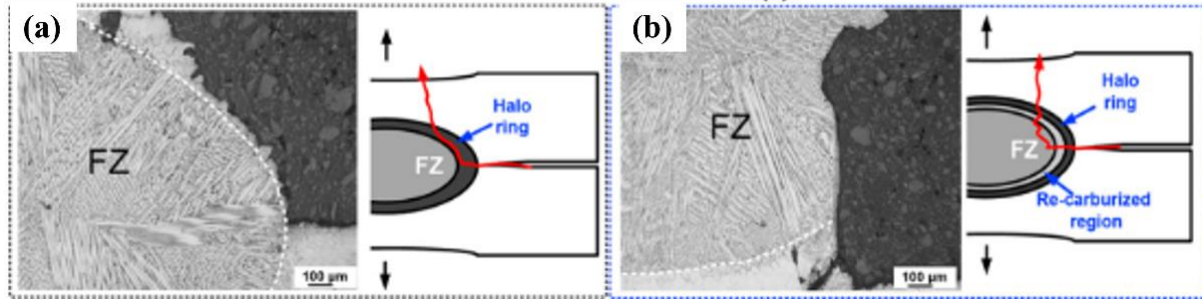


Figure 2.14: Fracture location of the 30MnB5 spot welds (a) as welded, and (b) PB [16]

In summary, the conventional PB cycle, characterized by a low-temperature PWHT, has the potential to influence the microstructure and mechanical performance of spot welds. The metallurgical transformations induced by this thermal cycle have been identified as a means to enhance the mechanical properties of the welds. While it is established that the PB cycle can improve the mechanical properties of the base material, such as yield strength, a comprehensive study on the specific impact of the PB on the on the macro and micro-fracture behavior of the AHSS spot welds is currently lacking. Further research is needed to delve into the detailed effects of the PB on AHSS resistance spot welds.

## 2.6. Localized Softening in Resistance Spot Welds

### 2.6.1. SCHAZ Softening

Martensite is one of the major microstructural constituents in AHSS with a strength higher than 1 GPa. Within the SCHAZ of the weld, martensite present in the BM can decompose to TM (a mix of ferrite and cementite), causing a decrease in local hardness [53,99–106]. Tamizi et al. [53] investigated the resistance spot weldability of a martensitic steel (MS1400) and observed a notable decrease in hardness within the SCHAZ, as illustrated in Figure 2.15a and b. They identified the presence of TM microstructure in the SCHAZ and attributed to the reduction in hardness. The TM formed (see Figure 2.15d) from the martensite present at the BM (see Figure 2.15c), leading to strain localization in the SCHAZ under CT loading. Saha et al. [52,57] and Biro et al. [107,108] reported that this martensitic tempering is prominent in DP 780

steels, which provides an easy path for crack propagation, deteriorating the strength of the DP steel spot welds. Although the crack propagates through the SCHAZ, failure surface consistently exhibits ductile behavior, attributed to the superior toughness of tempered martensite compared to the martensite microstructure. The extent of tempering depends on the local temperature and time of high-temperature exposure to that particular area. Numerous studies have been conducted by different researchers to address this phenomenon in a wide variety of AHSS. It is worth noting that there was also reported a secondary hardening (increase in hardness) at SCHAZ due to the formation of fine secondary carbides in steels with bainitic microstructure [56,109,110]. Nevertheless, no studies have been conducted so far to understand tempering in Gen 3 steels. Hence, there is also a research gap to address the mechanism behind the softening or secondary hardening phenomenon happening in the Gen 3 AHSS.

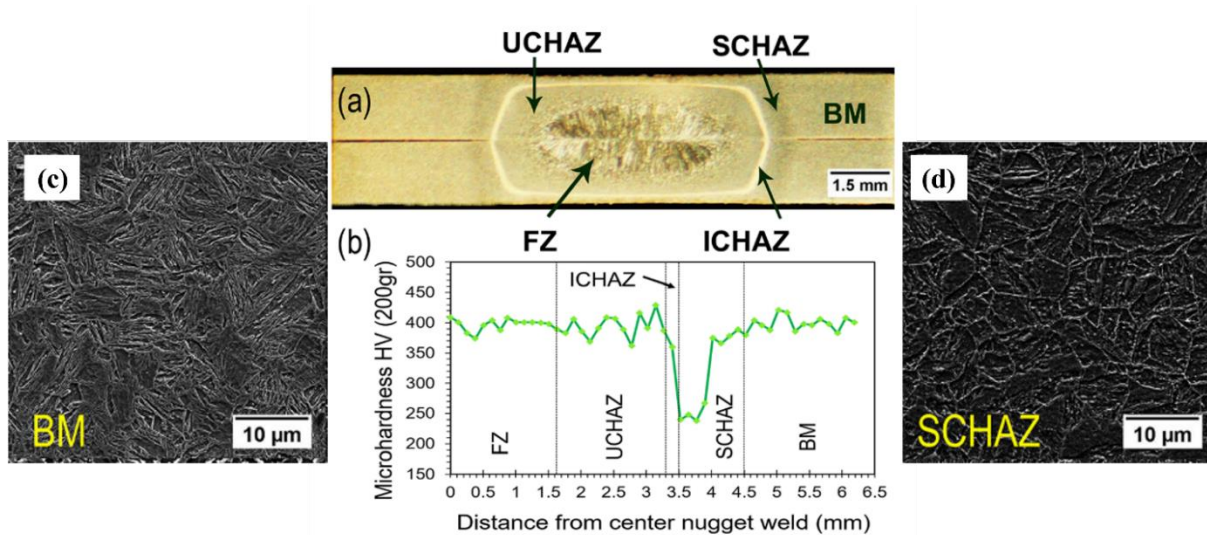


Figure 2.15: (a) Microstructure and corresponding (b) hardness profile of a MS1400 martensitic steel resistance spot weld showing softening in the SCHAZ. Electron micrographs of (c) BM showing martensite, and (d) SCHAZ showing tempered martensite [53]

## 2.6.2. Fusion Boundary Softening (Halo Ring)

Post-welded properties of the spot welds are determined by the microstructure present in them. Recent investigations show the presence of a narrow region adjacent to the FB with a local decrease in hardness

compared to surrounded FZ and HAZ. Sherepenko et al. [80,81], Mohamadizadeh et al. [72,73], Figueredo et al. [82], and Zhang et al. [111] reported that this softening phenomenon at the FB deteriorates the post-weld properties. This transient softened zone at the FB is referred to as the “halo ring”. A typical example of the halo ring in 22MnB5 steel is shown in Figure 2.16 as the blue-coloured region surrounding the nugget. The blue shade in this specific microstructure was due to the special etching followed by illuminating the surface with DIC imaging under an optical microscope. Although interpreting studies (changes in microstructural contrast) conducted by Raath et al. [112,113], Lee et al. [114], Spena et al. [47], Liu et al. [68], and Li et al. [115], show that the presence of halo ring, however, they did not report it as localized softening along the FB. In other words, they failed to interpret the halo phenomenon in their spot welds. Hence, it can be summarized that although halo is reported as a new phenomenon in spot welds, even though evidence of it may also be seen in studies published from as old as the early 2000s. A detailed summary of the literature shows the effect of halo on mechanical properties were summarized in Table 2.1. From Table 2.1 it can be also interpreted that the halo is prominent in the steels with high hardenability, such as 22MnB5, Usibor, Q&P steels, etc.

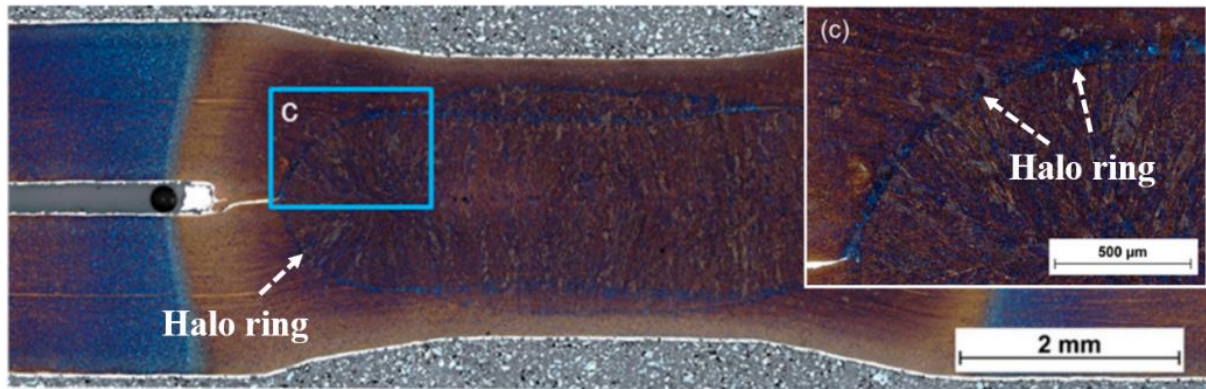


*Figure 2.16: A typical example of halo ring (blue) in 22MnB5 steel [80]*

Table 2.1: Summary of available literature on halo formation

| Reference                    | Materials and methods                        | Observations                                                                                                                                                                                                                                                  | Hypothesis on halo formation                                                                                                                                                                                                          | Remarks                                                                                                                                                |
|------------------------------|----------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------|
| Li et al. [115]              | ➤ Magnetically assisted dual-phase steel RSW | ➤ Halo region might consist of austenite and bainite or ferrite and martensite.<br>➤ Magnetically assisted spot welds show a wider halo                                                                                                                       | ➤ Carbon depletion from the grain boundary could be the reason behind the halo formation                                                                                                                                              | ➤ The authors did not provide any evidence for the probable microstructure and elemental partitioning.<br>➤ No evidence of fracture                    |
| Sherepenko et al. [80,81]    | ➤ RSW of Al-Si coated 22MnB5                 | ➤ $\delta$ -ferrite is the microstructure along with the halo<br>➤ Material close to the FB is heated above the $\gamma \rightarrow \delta$ transition temperature (1494°C).<br>➤ 400 ms welding time is the maximum time observed for the movement of the FB | ➤ Macro segregation of carbon and alloying elements may cause halo formation.<br>➤ $\delta$ -ferrite formation due to the isothermal soaking of the fusion zone in the peritectic region for a long time may cause the halo formation | ➤ No evidence for the $\delta$ -ferrite formation                                                                                                      |
| Chabok et al. [89,116,117]   | ➤ RSW of dual-phase 1000 steel               | ➤ The Halo region is called as recrystallized zone.<br>➤ Low fraction of high angle grain boundaries in the halo region<br>➤ Coarser structure of Bain groups in the halo region                                                                              | ➤ Low welding current produced with double pulse welds attributed to the formation of halo.<br>➤ This leads to lower resistance to crack initiation and propagation                                                                   | ➤ Even though they implemented pulsing in welding schedules the fracture path does not change.                                                         |
| Mohamadizadeh et al. [72,73] | ➤ RSW of Usibor 1500                         | ➤ Using tint etching, the weld periphery was identified clearly.<br>➤ Welds failed at FZ/HAZ interface during STT and CTT<br>➤ A 104 $\mu\text{m}$ wide halo ring was observed with a minimum hardness of 470HV compared to 508HV of FZ                       | ➤ Solute elements such as carbon and other alloying elements diffuse towards the molten weld pool from solid HAZ could be the reason for the halo formation.                                                                          | ➤ Shear band formation through the halo ring during loading was observed during STT                                                                    |
| Lee et al. [114]             | ➤ RSW on 15B22 hot-stamped boron steel       | ➤ A sudden hardness drop was observed near the FB and the authors think it is in PMZ.<br>➤ It causes premature failure during STT.                                                                                                                            | ➤ Due to the formation of a large amount of soft ferrite in the rapid solidification process.                                                                                                                                         | ➤ Evidence of the softer ferrite on the FB was not promising.<br>➤ The microstructure seems to be martensitic from the morphological characterization. |
| Zhang et al. [111]           | ➤ RSW on Q&P 980 steel                       | ➤ Presence of Mn free zone at the interface of FZ and HAZ with a banded bainite microstructure                                                                                                                                                                | ➤ Depletion of Mn in the interface of FZ and HAZ                                                                                                                                                                                      | ➤ Even though the crack followed the FB, the authors thought that it was due to the softened microstructure formed due to the tempering pulse          |
| Raath et al. [112,113]       | ➤ RSW of boron steel and DP 600              | ➤ A sudden drop in hardness at the nugget periphery was observed.<br>➤ The decrease in hardness is more significant in boron steel rather than the DP 600                                                                                                     | ➤ No mention about the mechanism                                                                                                                                                                                                      | ➤ Even though the presence of a halo was observed in this study the authors think the effect of halo is less significant.                              |
| Figueredo et al. [82]        | ➤ RSW of Zn coated Q&P 980 steel             | ➤ Evidence of transient softened region along the FB                                                                                                                                                                                                          | ➤ No mention about the mechanism                                                                                                                                                                                                      | ➤ Failure exactly concentrated in the FB                                                                                                               |
| Manladan et al. [16]         | ➤ RSW of 30MnB5                              | ➤ Failure along the FB and characterized as halo                                                                                                                                                                                                              | ➤ No mention about the mechanism                                                                                                                                                                                                      | ➤ PB improved the strength and energy absorption capacity                                                                                              |

From Table 2.1, the effects of the halo ring on mechanical properties have been investigated adequately in some steels. However, limited studies are focusing on the exact formation mechanism, and how it affects the fracture mechanics. Moreover, there is a knowledge gap between the effects of the halo ring in different locations of the spot weld (either near sheet-to-sheet interface or at the boundary of the nugget near sheet electrode interface) on the mechanical performance. Sherepenko et al. [80,81], and Mohamadizadeh et al. [72,73] showed that the width of the halo ring will be larger near the sheet-electrode interface than the sheet-sheet interface.

The available literature on Q&P steel spot welds show a range of CTS values. To compare the literature, a normalized CTS value [118] is calculated with the following Equation 2.2 and was plotted to the nugget diameter (ND), as shown in Figure 2.17.

$$S_{max} = F_{max} / d_n \cdot h \quad 2.2$$

Where  $S_{max}$  is the normalized strength,  $F_{max}$  is the maximum load,  $d_n$  is the nugget diameter, and  $h$  is the sheet thickness. Spot welds with halo rings has comparatively lower CTS values compared to the weld exhibiting no halo conditions.

As shown in Figure 2.10 the failure path of the spot welds with halo ring are through the fusion boundaries. From the normalized CTS plotted against nugget diameter graph shown in Figure 2.17, it can be interpreted that, the failure along the halo ring significantly influences the CT strength of the spot welds, since the halo can act as a channel for crack propagation. It should be also noted that even though spot welds have a halo ring in most of the recent investigations by different researchers, very few of them gave proper attention to this phenomenon.

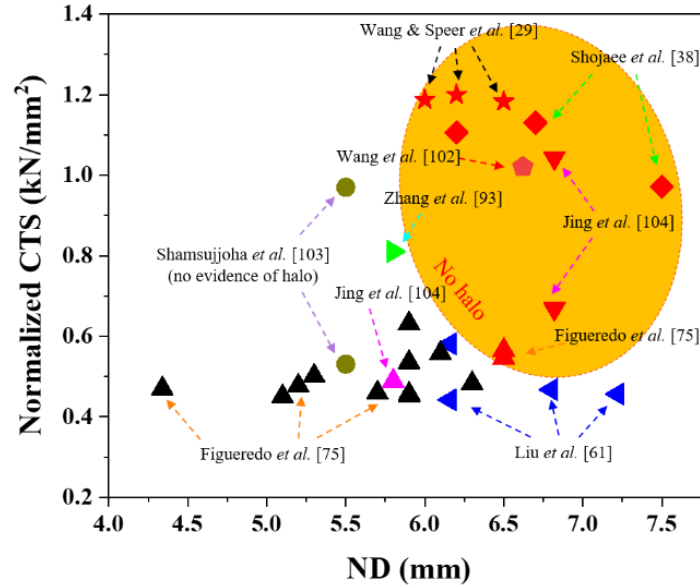


Figure 2.17: Mechanical property comparison between Gen 3 980 samples with halo and without halo [33,44,68,82,111,119–121]

Figueredo et al. [82] recently investigated the mechanical performance of Q&P 980 steel spot welds. It was observed that the failure path followed the fusion boundary (halo) in all the conditions, as shown in Figure 2.18. Although the failure of the welds in the research by Figueredo et al. [82] was attributed to the halo, many other welds reported in the literature that failed through the FB were attributed to other mechanisms. For example, Liu et al. [68], and Jing et al. [122] claimed that during CT loading conditions the crack in Q&P 980 steel spot welds propagates through liquation cracks, which are in the partially melted zone (PMZ) of the welds. However, the experimental evidence also shows softening along FB which is a potential failure path. Wang et al. [119] pointed out that a clear softening of the SCHAZ, and the cracks are propagating through the SCHAZ. Furthermore, Shojaee et al. [44] investigated the spot weldability of Gen 3 steels and reported that failure occurred through the CGHAZ during CTT. Therefore, although FB failure is commonly seen in Q&P steels, other failure paths also occur such as through SCHAZ, and CGHAZ. There is no understanding in the literature about the conditions necessary to promote failure preferentially through anyone of the above three failure paths.

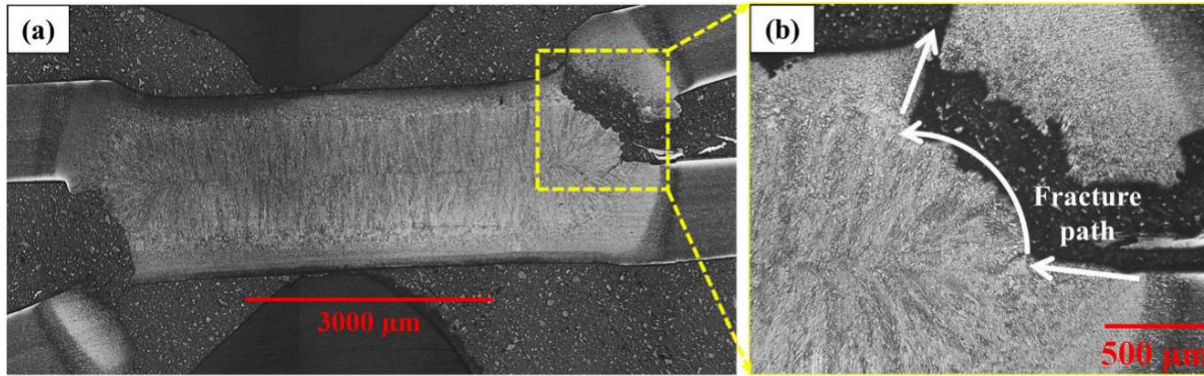


Figure 2.18: A typical example of halo failure during CTT in Q&P 980 steels [82]

The mechanism of halo formation has been studied by different researchers, as summarised in Figure 2.19. The first study was done by Li et al. [115] in 2016 on DP 590 steel resistance spot welds. They believed that C depletion from the GB leads to “fluid-flow-controlled negative macro segregation of C during solidification” [115] resulted in halo formation. However, they were unable to provide evidence for carbon depletion. In 2018 Sherepenko et al. [15,80,81] studied the resistance spot weldability in 22MnB5 steel and showed that  $\delta$  ferrite formed in the FB due to the isothermal soaking of the fusion zone in the peritectic region for a long time could be the reason for the halo formation. They also provide evidence for carbon depletion in the FB by carbon mapping from the phase field simulations. However, they believed that this could not be the sole reason for the halo formation, as all welds will be in  $\delta$  ferrite due to the temperature in the FB.  $\delta$  ferrite on the halo in this particular 22MnB5 steel could be due to its thermodynamic stability at the FB temperature. Mohamadizadeh et al. [72,73] proposed that various solutes such as carbon and other alloying elements diffuse towards the molten weld pool from solid HAZ might be the key factor to form the halo. Due to this solute diffusion, they also speculated that a softer lower C martensite will be formed adjacent to the FB, forming the halo ring. In 2020, Lee et al. [114] studied the RSW of 15B22 hot-stamped steel and found that the halo consisted of soft ferrite, which supported the observation from Sherepenko et al. [15,80,81] study. Zhang et al. [111] investigated the spot weldability of Q&P 980 steel and observed a strong Mn depletion in the FB, which they thought caused the decrease in hardness adjacent to the fusion zone. Recently, Manladan et al. [16] studied the effect of paint baking on the mechanical behaviour of the

30MnB5 hot-stamped steel and proposed that segregation of carbon and other alloying elements at the FZ during extended welding time causes the depletion of carbon at the FB.

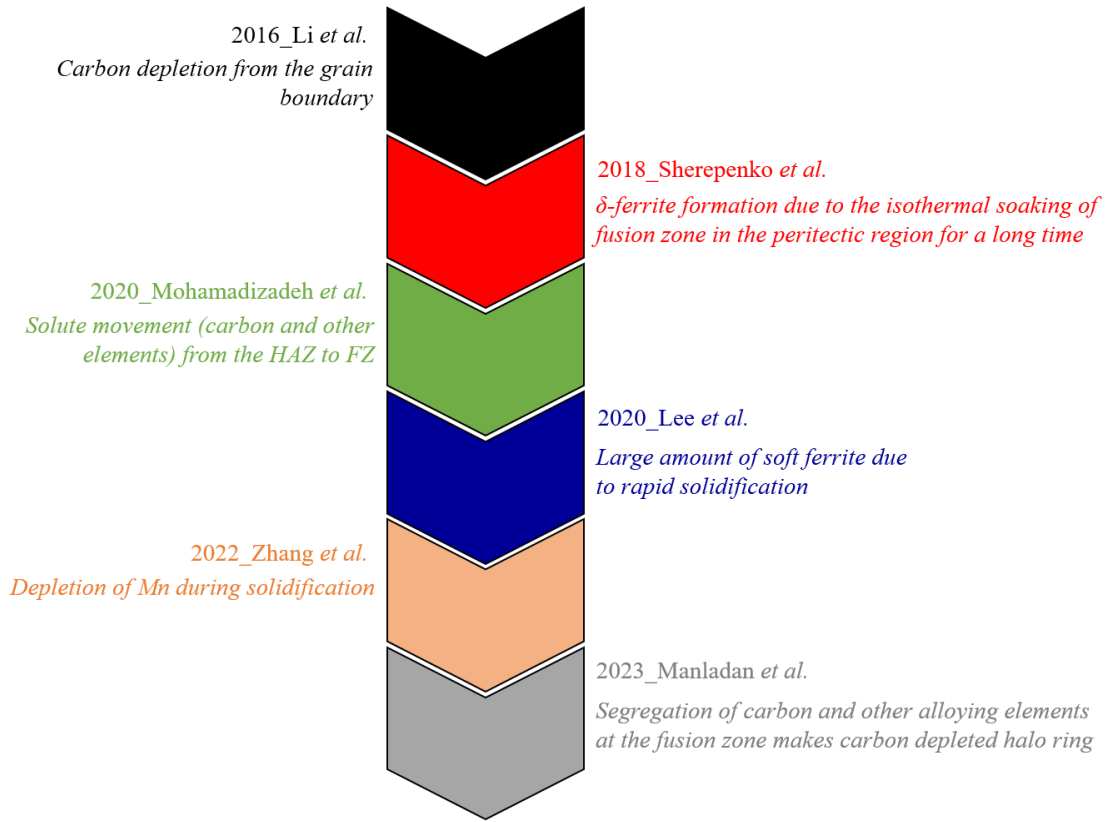


Figure 2.19: Timeline showing the mechanism of FB softening and proposed hypothesis  
[15,16,72,73,80,81,111,115–117,123]

Interpretation of the available literature does not provide a clear knowledge on the mechanism of halo formation and its macro-mechanical behaviours. Therefore, there exists a clear knowledge gap in the fundamental understanding of the halo ring and its effect on the mechanical properties of the weld. Hence, these discrepancies in the formation mechanism of the halo ring and how to mitigate the effect of halo will be fundamentally addressed in my research.

Although efforts have been made to understand the impact of the halo ring on mechanical properties, there are still many questions to address. The fundamental cause of mode 3 failures characterized by brittle

fracture surfaces in spot welds has yet to be determined, and there is a lack of literature addressing the microstructure of the halo ring and its correlation with thermal history. A comprehensive understanding of the halo ring is essential to propose effective strategies for its mitigation. Additionally, the influence of material chemical composition on the halo ring remains unclear. These insights are crucial for understanding the formation mechanism of the halo ring, enabling automakers to adjust welding parameters to produce spot welds with out halo ring. This thesis aims to bridge the above-mentioned research gaps.

## **Chapter 3. Effect of Fusion Boundary Softening on Mechanical**

### **Properties <sup>1</sup>**

#### **3.1. Overview**

Despite frequent reports of transient softened zone in resistance spot welds affecting mechanical properties, no work has delved into investigate how local microstructural constituents influences the mechanical properties of the spot welds. This chapter explores the effect of halo ring on mechanical properties of the Q&P 980 steel spot welds under normal loading conditions. Additionally, this chapter establishes a new welding schedule to improve the mechanical properties by the eliminating the halo ring with modification of the grain structure. Detailed microstructural characterization, and fractographic and elemental analysis were conducted to obtain a comprehensive insight. Results indicate a direct relationship between the mechanical properties and local microstructure at the fusion boundary. Fractographic analysis using scanning electron microscope (SEM), and electron probe micro-analyzer (EPMA) were used to confirm the brittle fracture and the elemental segregation, respectively. These findings will have high impact on the knowledge to understand how local microstructures impact the mechanical properties of AHSS.

#### **3.2. Background**

Knowledge of the resistance spot weldability of Q&P steels is limited in the available literature [68,119,120,124]. Pouranvari et al. [53] reported that, the mechanical performance of the resistance spot welds was predominantly related to the failure location in the spot welds. The load-bearing capacity of the spot welds improves when the failure location shifts from sheet-sheet interface (interfacial failure) to through-thickness failure with a failure path going through either the base metal or the HAZ, known as

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<sup>1</sup> This chapter is composed of a published research paper as outlined in the statement of contributions. D.C. Ramachandran, B. Figueredo, O. Sherepenko, W. Jin, Y. Do Park, E. Biro, A study on improving the mechanical performance by controlling the halo ring in the Q&P 980 steel resistance spot welds, J. Manuf. Process. 75 (2022) 320–330.

button pullout [53]. Recently, Liu et al. [68] examined the effect of double-pulse welding schedules on the mechanical properties of Q&P980 steel spot welds. Weld failures were concentrated on the fusion boundary, and they associated the deterioration of mechanical properties of the investigated spot welds to liquation cracking. However, recent studies [72,80,81] showed that failure around the fusion zone was related to the localized softening of the fusion boundary called as ‘halo ring’. These studies hypothesized that the formation of this halo ring at the fusion boundary was due to partitioning of solute atoms, such as carbon, from the solid fusion boundary to the molten weld nugget during extended welding times.

In addition to the localized softening observed at the fusion boundary, there has also been recognition of softening within the SCHAZ of the spot welds. This softening is attributed to the tempering of martensite and has been widely documented in previous generations of AHSS such as DP steels [53,103,125,126]. Even though the SCHAZ softening is also prominent in most of the third-generation AHSS [68,124], recent investigations show the presence of a halo ring [72,73,80,81,113,115], which affects the mechanical performances of the spot welds. Raath et al. [113] investigated the effect of welding schedules on the residual stress distribution of boron steel spot welds. They reported an increased hardness drop around the fusion zone with increasing welding time. Meanwhile, Li et al. [115] studied the magnetically assisted spot weldability of dual-phase steels. A clear drop in hardness around the fusion zone was detected. They suspected the microstructure of this region consisted of a mixture of bainite and austenite or ferrite and martensite. Whereas, Sherepenko et al. [80,81] investigated the effect of transient softening at the spot weld fusion boundary on mechanical properties by analyzing the weldability of 22MnB5 martensitic steel. The transient softening of the fusion boundary deteriorates the mechanical performance of the spot welds, and the failure follows exactly through the fusion boundary. Mohamadizadeh et al. [73] examined the failure behavior of spot-welded ultra-high-strength 22MnB5 steel. Other than the reported softening in the SCHAZ, a transient softening at the fusion boundary was observed. The failure under tensile, and shear loading initiated and propagated through the softened fusion boundary, which deteriorated the mechanical properties of spot welds due to the formation of shear bands. Furthermore, Eftekharimilani et al. [10]

pointed out that implication of short secondary pulse current on the third-generation 1 GPa AHSS anneals the primary nugget at the weld edge and second pulse current homogenizes the alloying elements such as phosphorous and as a result, it significantly improves the cross-tension strength (CTS). Recently, Figueredo et al. [82] also observed the failure around the fusion boundary on the Q&P980 spot welds under cross-tension tests, which deteriorates the strength compared to the same strength DP steels.

Based on the literature, it can be summarized that localized softening around the fusion zone could be detrimental to the mechanical performance of spot welds. To date, only a handful of studies have been reported that looked at the effects of double-pulse welding on the mechanical performance of third generation AHSS. Hence, this chapter focuses on improving the mechanical performance of Q&P980 steel spot welds via welding schedule optimization (chemical homogenization) and possibly eliminating the influence of localized softening on the mechanical performance.

### 3.3. Materials and Methods

#### 3.3.1. Material

The present study examines an industrially produced zinc-coated 1.2 mm thick Q&P980 steel with a composition of 0.23 wt.% C, 2.0 wt.% Mn, 1 wt.% Si, and other alloying additions, as described in Table 3.1. The carbon equivalent of the materials was calculated according to the method described by Yurioka et al. [127], where it was found that the carbon equivalent for Q&P980, is 0.65.

*Table 3.1: Chemical composition of the 1.2 mm Q&P980 steel*

|               | C   | Mn  | Si  | Cr+Mo+Ti+Nb | CE   |
|---------------|-----|-----|-----|-------------|------|
| Q&P 980 steel | 0.2 | 2.0 | 1.1 | 0.22        | 0.65 |

The microstructure of the base material consists of a combination of about 38% ferrite, 47% tempered martensite, 9% fresh martensite, and 6% retained austenite, as shown in Figure 3.1a, and b. The microstructural constituents were identified according to their morphology as reported in previous

literatures [57,61,125,128]. The RA was characterized using the EBSD technique; the phase map is shown in Figure 3.1c.

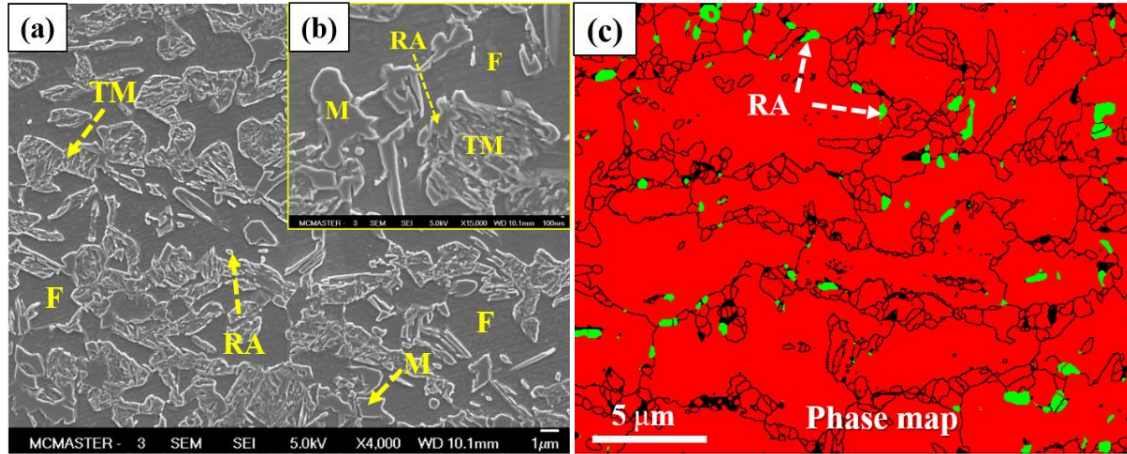


Figure 3.1: Base metal micrographs of the Q&P 980 steel (a) electron micrograph (b) EBSD phase map to quantify retained austenite (RA).

The Q&P 980 steels' material properties, including ultimate tensile strength (UTS), yield strength (YS), elongation, and hardness, are presented in Table 3.2.

Table 3.2: Mechanical properties of the Q&P 980 steel

| Material | YS (MPa) | UTS (MPa) | Elongation (%) | Hardness (HV) |
|----------|----------|-----------|----------------|---------------|
| Q&P 980  | 640      | 1013      | 20             | 323           |

### 3.3.2. Resistance Spot Welding

RSW was carried out using a robotic spot welder with a C-type transformer spot welding gun and a Rexroth Bosch weld controller. A 6 mm dome-shaped Cu-Cr electrode with an electrode force of 4 kN was used for the welding. AWS D8.9 recommended a single-pulse schedule [45] and a novel double-pulse welding schedule was experimented as well [51]. Both welding schedules are shown schematically in Figure 3.2. For the single-pulse schedule, the welding current was selected as 8.0 kA, which is 0.2 kA less than the expulsion current ( $I_{max}$ ), to maximize the nugget diameter (see Figure 3.2 a). For double-pulse schedules, a pause in the welding current (7.8 kA) between 15 to 60 ms was allowed before initiation of the second

current pulse, then a constant current pulse of 8.2 kA was applied. Since the primary current was used to maximize the nugget diameter in this study, 7.8 kA was the maximum current that can be used without the expulsion, combined with a constant 8.2 kA secondary current, as shown in Figure 3.2b. These conditions provide a chemical homogenization effect in double-pulse welds.

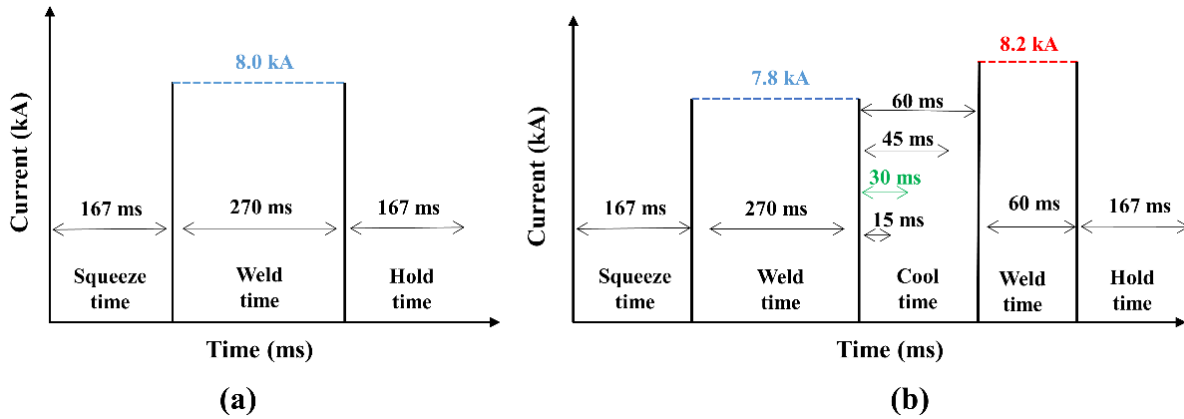


Figure 3.2: Schematic diagram showing the (a) single, and (b) double-pulse welding schedule parameters.

### 3.3.3. Microstructural Characterization

Metallography was characterized using optical microscopy (Olympus BX51M) and scanning electron microscopy (SEM) after etching the samples with a 3% nital solution. The samples were prepared with conventional metallographic methods and etched with 3% nital to reveal the microstructure. A JEOL JSM 7000F field emission SEM was used for imaging and electron backscattered diffraction (EBSD) techniques. The EDAX orientation image microscopy (OIM) software was used for the EBSD data acquisition and post-processing. For EBSD analysis a step size of 0.2  $\mu\text{m}$  was used for quantitative characterization of retained austenite. Electron probe micro analysis (FE-EPMA, model: JEOL JXA-8530F, Japan) was performed to characterize the elemental distribution across the fusion boundary.

### 3.3.4. Microhardness Analysis

Hardness measurements were carried out using an automated Clemex microhardness machine interfaced with a Vickers indenter. Hardness line scans were made using a 0.2 kg force and a spacing of 200  $\mu\text{m}$

between the consecutive indents, whereas a 0.05 kgf force was used for hardness mapping with a spacing of 50  $\mu\text{m}$  between the consecutive indentations. The hardness values are reported with a 95% confidence interval.

### 3.3.5. Mechanical Properties

The CTS was measured according to the AWS D8.9 standard [60]. Mechanical tests were performed at a constant crosshead speed of 10 mm min<sup>-1</sup> using a 100 kN MTS frame universal tensile testing machine. The peak load and failure energy were extracted from the load-displacement curves. The absorbed energy was calculated by integrating the area under the curve up to the peak load. All reported results are an average of three repetitions and the error bars show a 95% confidence interval (CI). To identify the crack initiation and propagation mechanism during the CTS test, interrupted CTS tests were carried out. The tests were stopped after reaching 90% of the weld's peak load, after which, the weld was cross-sectioned and analyzed by optical microscopy.

## 3.4. Results

### 3.4.1. Microstructural Characterization

The weld made using a single-pulse condition was cross-sectioned. It had a weld nugget with a nugget diameter (ND) of  $6.02 \pm 0.11$  mm, as shown in Figure 3.3a, which is the maximum achievable nugget diameter without an expulsion. On the other hand, Figure 3.3b shows the macrostructure of the double-pulse schedule (7.8 kA-30 ms-8.2 kA) with an ND of  $5.64 \pm 0.14$  mm. The double-pulse welds exhibited two distinct nuggets; a primary weld nugget produced by 7.8 kA of welding current followed by a secondary weld nugget produced by 8.2 kA of secondary welding current, as shown in Figure 3.3c. The secondary nugget grows by remelting the primary nugget with the secondary current pulses, as the cooling time between the pulses is comparatively short (30 ms). Furthermore, an equiaxed grain structure was seen in the interface between the primary and secondary nugget (see Figure 3.3d) which confirms the secondary

current reheats the primary nugget FZ nearer to the fusion line. This secondary nugget also contributes to achieving a chemically homogeneous final nugget.

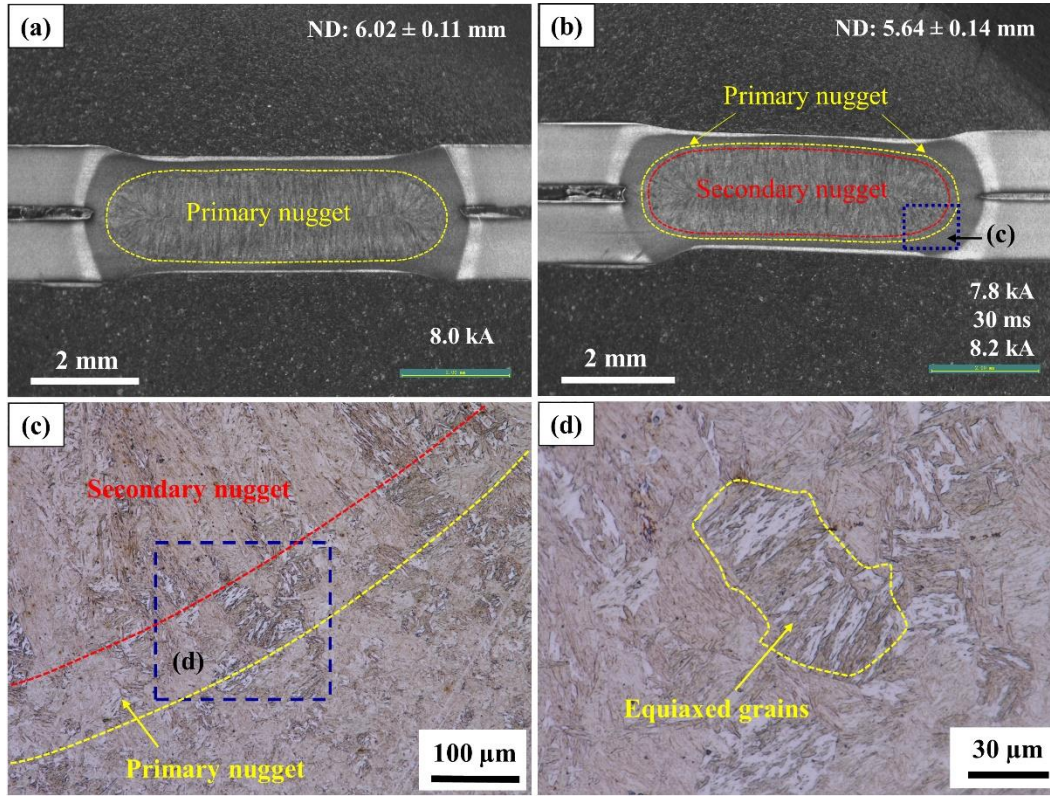


*Figure 3.3: Cross-sectional micrographs of (a) single-pulse, and (b) double-pulse with 7.8 kA primary current. (c) Higher magnification image showing the primary secondary nugget interface, and (e) equiaxed grain present in the interface*

The optical micrograph of the single-pulse weld in Figure 3.4a shows different areas of heat-affected zones (HAZ). The microstructures were characterized by analyzing their morphology and identified using similar micrographs found in the literature [57,129]. The FZ consisted of a fully martensitic structure, with columnar grains oriented towards the weld centerline as shown in Figure 3.4b. It has been reported that the cooling rate after spot welding is on the order of 2000 °C/s [53]. Hence the microstructures of the FZ and HAZ, resulting from both welding schedules, were predominately martensitic. Similarly, the CGHAZ and FGHAZ are predominantly martensite (see Figure 3.4c and d).

ICHAZ (where the peak temperature during welding ranged between the Ac1 and Ac3 temperatures of the material) consisted of a combination of complex microstructures such as TM, F, and RA, as shown in Figure 3.4e. The RA was identified by the EBSD analysis. The intercritical temperature, rich chemistry, and high cooling rate stabilize the RA even at room temperature. Figure 3.4f shows that in SCHAZ there is a combination of TM and F is the predominant microstructures. This tempered martensite is a mixture of TM from the base material, and TM formed during the welding cycle, as it has been widely reported that temperatures between 300 to 650°C are likely susceptible to the tempering of martensite [57,125]. The double-pulse condition showed a similar microstructure as the weld made using the single-pulse condition within the FZ and HAZ.

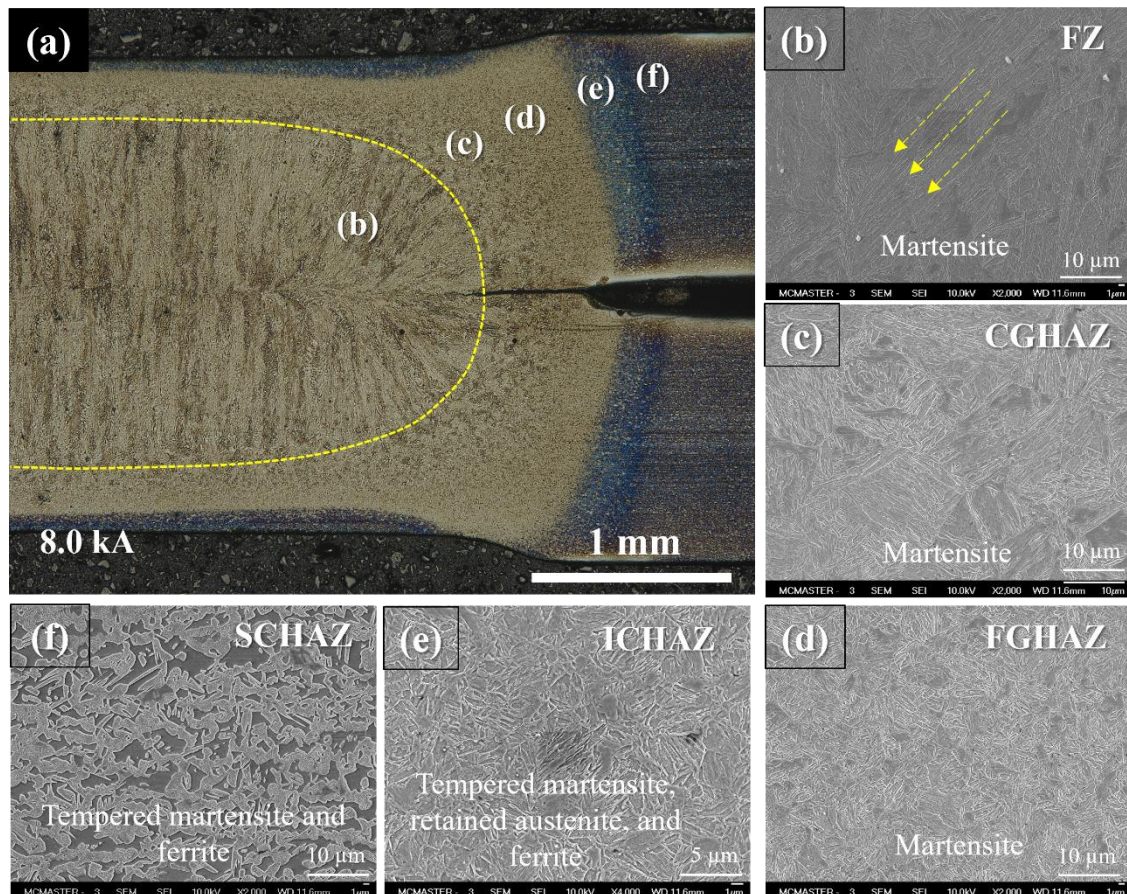


Figure 3.4: Microstructure analysis of the Q&P 980 spot weld. (a) Macrograph showing the overall view of the FZ, HAZ, and, base material, (b) FZ, (c) CGHAZ, (d) FGHAZ, (e) ICHAZ, and (f) SCHAZ

### 3.4.2. Cross Tensile Strength of Welds

Figure 3.5a shows the load-displacement curve during the cross-tension test of both single and double-pulse welds. Pulsing significantly improved the mechanical performance of the welds. Moreover, as the cooling time between the pulses increases, the CTS values increase to a maximum and subsequently decrease after 30ms cooling time. Here the area under the load-displacement curve up to failure is considered as the absorbed energy on the spot welds during loading until fracture [78]. The higher absorbed energy represents the improved weld performance. Hence, the absorbed energy is also in-line with the higher CTS values, as shown in Figure 3.5b. It has been reported that CTS values of the spot welds are predominantly related to the nugget diameter of the welds, as the nugget is the primary load-bearing area in the cross-tension test [43]. Despite a decrease in nugget diameter from  $6.02 \pm 0.11$  mm to  $5.64 \pm 0.14$  mm (see Figure 3.3), as interpulse cooling time increased, and the smaller nugget diameter of the double-pulse weld made with 30 ms cooling time compared to the single-pulse condition, CTS and absorbed energy values for the double-pulse condition using the 30 ms cooling time are approximately 33% and 110% higher, respectively, than those of the single-pulse condition. In other words, the mechanical performance of the welds was improved to 33% by implementing double-pulse welding schedules, specifically in the case of 30 ms cooling time even though the nugget diameter for the double-pulsed condition was smaller. It has been noted that the authors tried to match the single and double-pulse welds nugget diameters. However, early expulsion occurring in double-pulse welds due to the high current near to the maximum possible current ( $I_{\max}$ ) made this impossible.

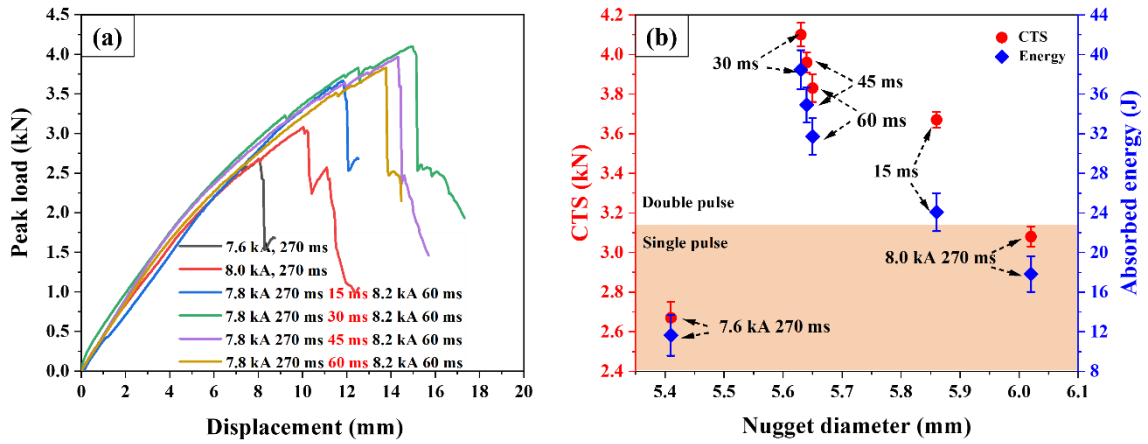


Figure 3.5: The cross-tension test results of the spot welds. (a) load-displacement curves, and (b) relationship between nugget diameter, CTS, and absorbed energy

An interrupted cross-tension test was also conducted to understand the failure behavior of the weld joints. The single-pulse and double-pulse conditions with a 30 ms inter-pulse cooling time were chosen for the comparison as these conditions exhibit the lowest and highest CTS, respectively. Although in past studies RSW made with high-strength steels have failed in the SCHAZ [5,53,103], this was not seen in the present work. Examination of the welds in the current study that were loaded to 90% of the failure load showed that failure initiated at the fusion boundary for both the single and double-pulse welding conditions, as seen from Figure 3.6a-f. Recent studies show that in high-strength steels with rich chemistry, stress concentration always occurs at weld notch due to the geometrical factors, especially in steels that do not exhibit SCHAZ softening [72]. Hence it is likely that failure can also initiate at the sheet-sheet interface. The cross-sectional micrographs after the interrupted cross-tension test are shown in Figure 3.6e and f confirm that the failure occurred along the fusion boundary or through the CGHAZ despite the welding conditions. In other words, failure occurred along the fusion boundary in single-pulse weld, whereas the failure likely occurred through the CGHAZ in double-pulse weld. How pulsing and this failure initiation location resulted in the difference in peak load and absorbed energy between the single and double-pulse welds will be discussed in the following sections.

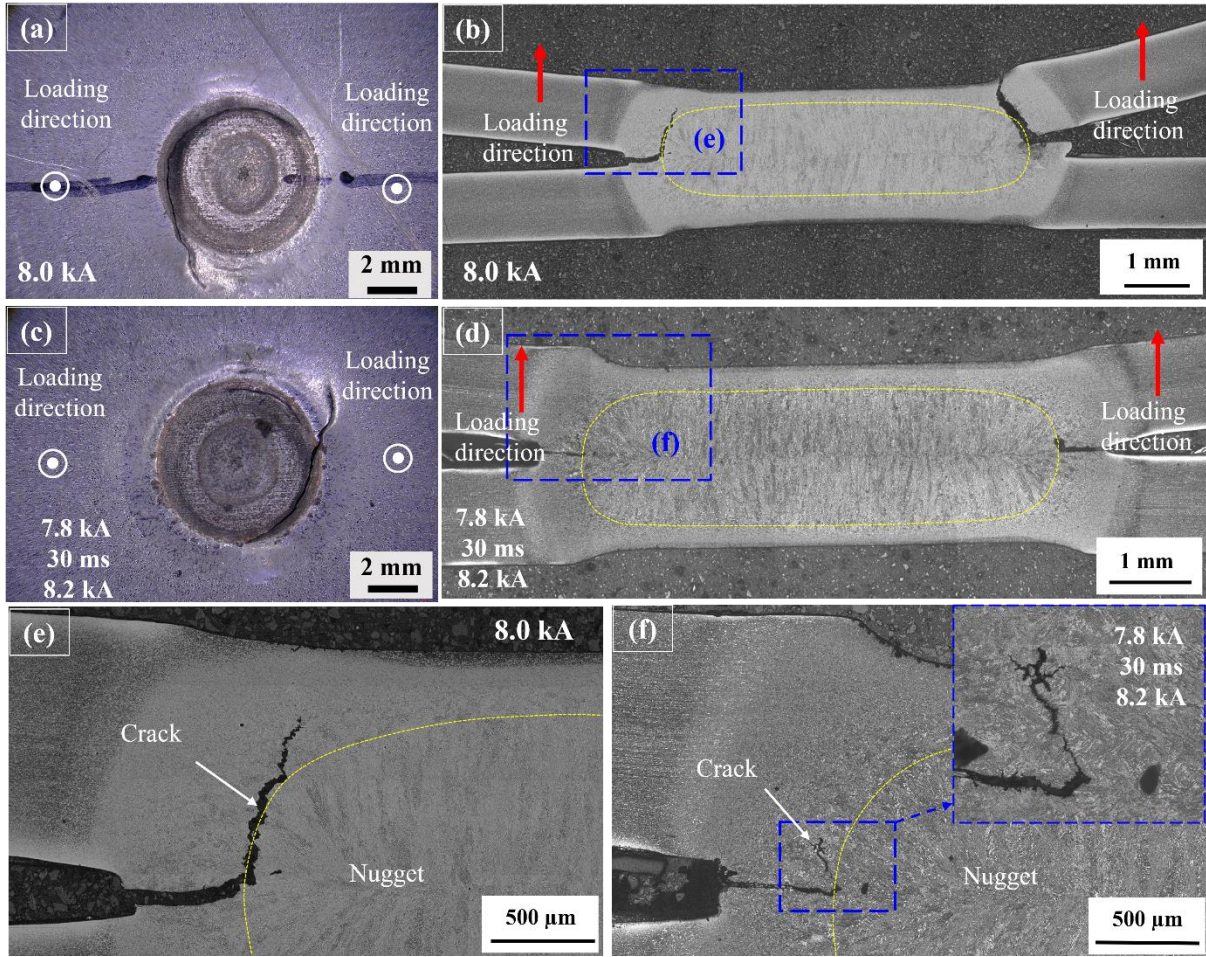


Figure 3.6: Macroscopic images and weld cross-section after the failure and interrupted cross-tension test respectively. (a) and (b) single-pulse weld, (c) and (d) double-pulse weld. Optical micrograph showing the (e and f) failure path in single and double-pulse welds after the interrupted test.

### 3.5. Discussion

#### 3.5.1. Classification of Halo Ring

The high-density microhardness map and corresponding microstructure in Figure 3.7 show that the soft zone exists at the nugget periphery in the single-pulsed weld (Figure 3.7a and c). The hardness of this soft zone was still relatively higher than the base material hardness. Interestingly, the double-pulse welding schedule using a 30 ms cooling time, greatly reduces the severity of the softened zone along the fusion boundary, having a hardness of 519 HV compared to 436 HV in the softened zone of the fusion boundary

of the single-pulsed weld, as seen from Figure 3.7b and d. These features are similar to those observed by Sherepenko et al. [80,81] who reported that, in ultra-high-strength steels, localized softening may occur at the fusion zone boundary. It was claimed that this occurs by diffusion of carbon from the solid heat-affected zone to the molten weld pool causing the transient softened zone around the nugget [72,81], which has been called the ‘halo ring’.

The average base metal and CGHAZ hardness values for weld made in both the single and double-pulsed conditions are  $314 \pm 3$  HV, and  $540 \pm 5$  HV, respectively, as seen in Figure 3.7e. However, the average fusion zone hardness is slightly different in both welds, where the average fusion zone hardness of the single-pulse and double-pulse welds are  $550 \pm 5$  HV, and  $536 \pm 5$  HV, respectively. The width of the halo ring is approximately  $50 \mu\text{m}$  and the hardness of the halo ring region is around  $428 \pm 37$  HV, which is  $\sim 20\%$  lower than the fusion zone hardness in the single-pulsed condition. Furthermore, as mentioned above, only a minor hardness decrease was detected at the fusion boundary of the weld made using the double-pulsed condition. The high-density hardness map along the fusion boundary is also shown in Figure 3.7e. It is worth noting that, the difference in fusion zone hardness between the two conditions could be attributed to the cooling rate associated with the two different fusion zones.

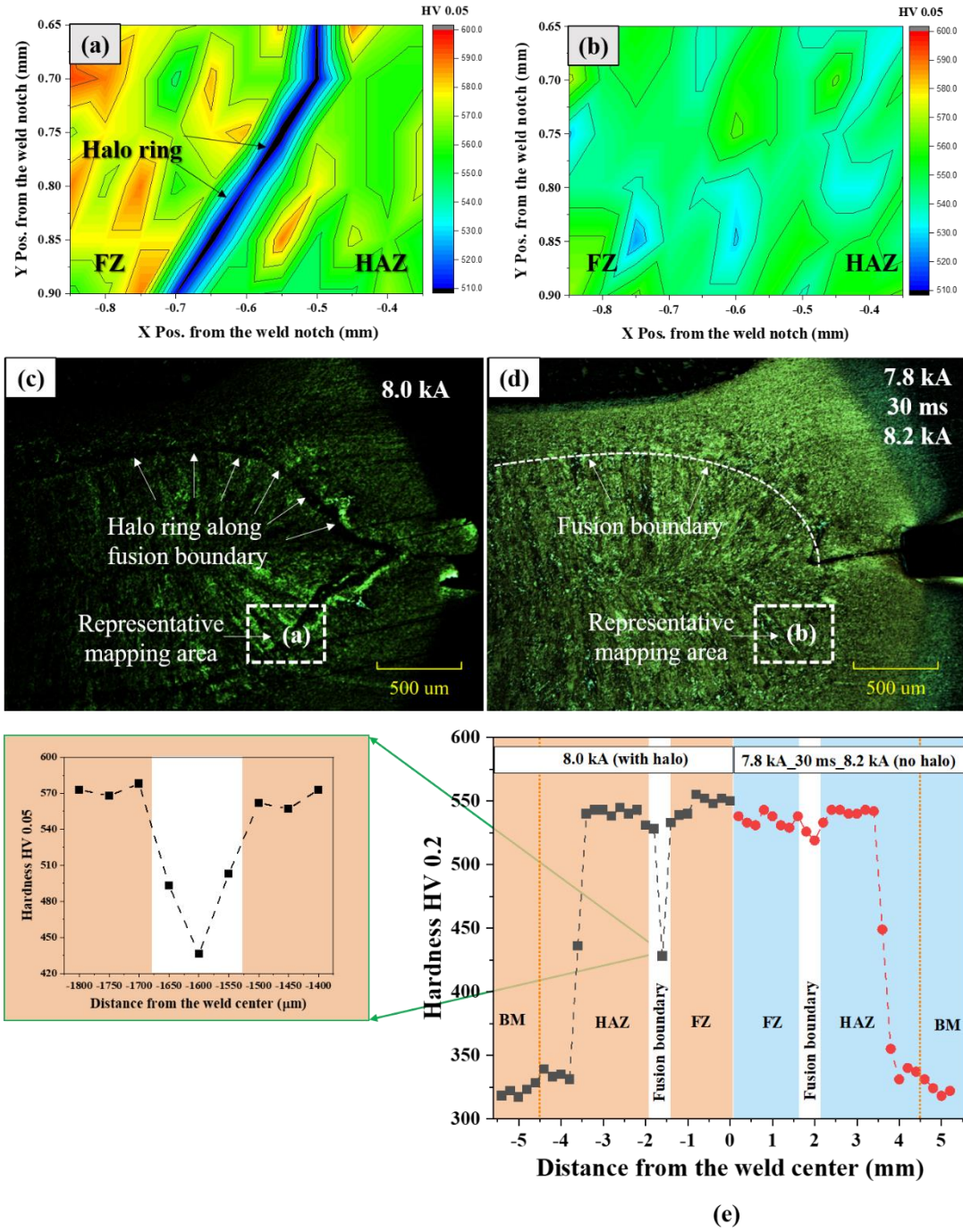


Figure 3.7: High-density microhardness map and corresponding macrostructure of (a) and (c) single-pulse weld, (b) and (d) double-pulse weld, and (e) quantitative hardness map from center of the weld to base metal for both single and double-pulse condition.

### 3.5.2. Fracture Characteristics of Halo Ring

Recall, cross-sections of the interrupted cross-tension tests (see Figure 3.6) showed both welds failed near the fusion boundary. However, a clear difference in fracture morphology can be seen when the fracture surfaces were viewed at higher magnification in Figure 3.8b and c. Figure 3.8a and d show the electron micrographs of the fracture surface of single and double-pulse conditions, respectively. The crack initiation area has been identified by the interrupted CTS and is shown in Figure 3.6e and f. It is worth noting that failure surface resulting from the area where the crack starts at the sheet-sheet interface and propagates towards the fusion boundary through CGHAZ shows a small area of ductile dimple fracture in both the conditions (see Figure 3.6a and b). In the single-pulse welds, once the crack reaches the halo ring, it propagates through the fusion boundary in a brittle manner as shown in Figure 3.8e and f, due to the halo ring's mechanical contrast with the adjacent microstructure. Even though the crack mainly propagates through the halo ring, some portion of crack (~21% of total crack length) also went through the CGHAZ, in the single-pulse weld (see Figure 3.9a). The described small changes in fracture path towards the FZ or CGHAZ during normal loading are also reported in the literature [68,73,81]. The areas where the crack propagated through the CGHAZ failed in an intergranular fashion, as shown in Figure 3.9c. The failure mostly occurred (~ 54% of total crack length) through the halo ring, where it propagated in a brittle (cleavage) fashion (see Figure 3.8e and Figure 3.9d). Once the sample exhibits significant bending at the end of the test failure proceeds by shearing through the remainder of the area (~25% of total crack length). The double-pulse weld, failed through a similar path, however, the fracture mode differed. When the crack propagated through the CGHAZ, failure progressed in a mixed failure mode (brittle and ductile), as shown in Figure 3.8b, d, g, and h. Despite the failure location, all the failure surfaces show a mixed mode of fracture (mixed with cleavage and dimple fractures). The failure path is chaotic and passes through fusion boundary, CGHAZ, and FGHAZ with a failure percentage of ~7%, ~59%, and ~34% of total crack length, respectively (see Figure 3.9b and e). These percentages were determined from the cross-sectional analysis

of the fractured sample. Failure through the FGHAZ is complete shearing as shown in Figure 3.8b. Moreover, the load-bearing capacity (Figure 3.5) of the welds shows in line with the current observations.

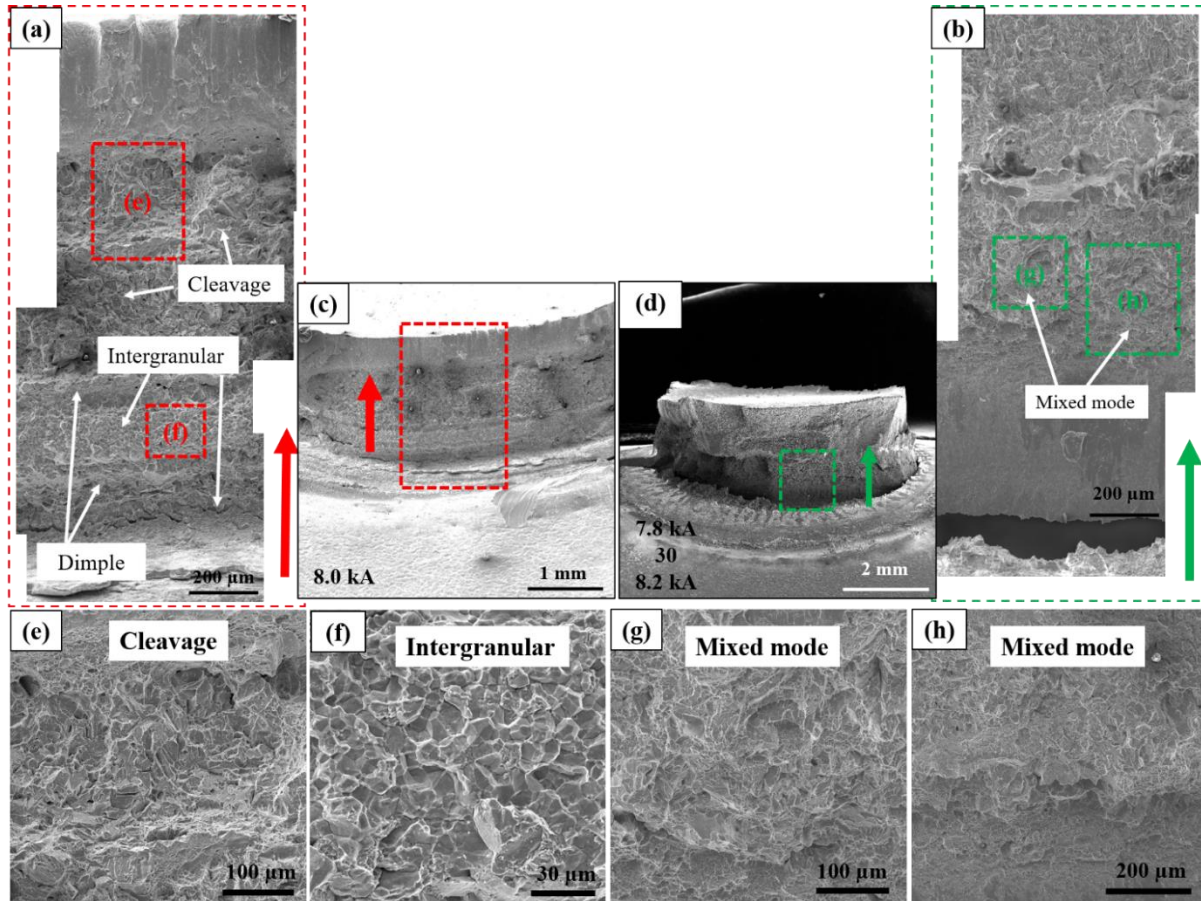


Figure 3.8: Fractographic analysis of (c) and (d) single-pulse welds and double-pulse weld, respectively. (a) and (b) Panoramic images of the highlighted area in Figure 3.8C, and Figure 3.8d, respectively. High magnification images of the highlighted area (e) and (f) in Figure 3.8a show cleavage and intergranular fracture, and (g) and (h) in Figure 3.8b shows a mixed mode of fracture.

The absorbed energy (see Figure 3.5b) of the joints agrees well with the above observations on fracture morphology. In other words, the fracture surface is brittle in the welds made with a single-pulse, whereas mixed mode fractures with significant ductile components in the welds were made in the double-pulsed condition, as shown in Figure 3.9. Moreover, secondary cracks through the grain boundary also can be seen on the single-pulse weld, as seen from Figure 3.9c. It should be noted though, Mohamadizadeh et al. [73]

observed shear banding along the halo ring in 22MnB5 spot welds during normal loading, which was contrary to the brittle fracture observed through the fusion boundary in the present study. It can be hypothesized that the observed brittle intergranular fracture in the present study occurred through the near side of the CGHAZ or hard interface adjacent to the halo ring. EftekhariMilani et al. [10], reported that the Mn, Si, and P segregation at dendritic boundaries of third-generation 1GPa AHSS during single-pulse welds, embrittled the grain boundaries, thereby deteriorating the CTS values. This phenomenon could be the reason responsible for the weakening of CGHAZ in the current study. Due to the microstructural complexity and small width of the halo ring, further investigation of the fracture surface is necessary to find out the exact crack initiation and propagation regions and will be presented in future works.

According to the previous research, Q&P steel spot welds fail along the SCHAZ due to martensite tempering or along the partially melted zone (PMZ) due to liquation cracking [68,119]. Interestingly, in the current work, it has been observed that failure likely occurred along the fusion boundary and CGHAZ. The presence of a halo ring along the fusion boundary is the primary cause for the decreased CTS of single-pulse welds. However, a 33 % increase in peak load and a 110% increase in absorbed energy were observed in double-pulse (30 ms cooling time) welds, as shown in Figure 3.5b. It has been hypothesized that prolonged heating of the weld nugget by providing a long welding time could be the main reason to form the halo ring around the nugget. It can be either controlled by remelting the primary nugget boundary by secondary nugget for a very short time or the chemical homogenization (reheating) of primary nugget by secondary nugget. In this research, the authors approached the latter idea.

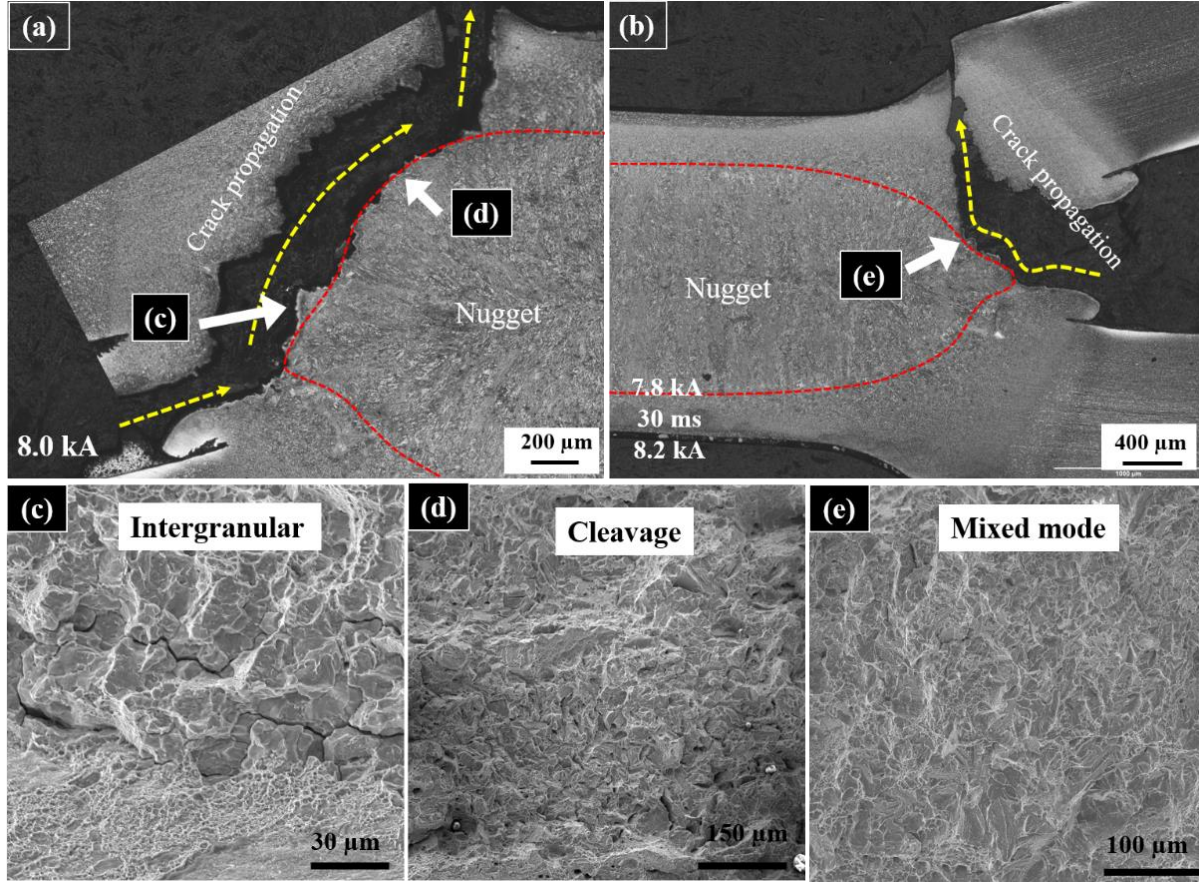


Figure 3.9: Cross-sectional images showing the samples after CTS (a) single-pulse weld, (b) double-pulse weld, (c) and (d) brittle intergranular and cleavage fracture observed in Figure 3.9a, and (e) mixed-mode fracture observed in Figure 3.9b.

### 3.5.3. Elemental Distribution Across the Halo Ring

Several hypotheses have been asserted to explain the formation of the halo ring recently. Li et al. [115] suggested that the carbon depletion from the fusion boundary during welding could be responsible for this localized softening. Whereas, Sherepenko et al. [80,81] proposed that formation of  $\delta$ -ferrite due to the long isothermal soaking of the fusion zone in the peritectic region caused this softening. Nevertheless, it can be hypothesized that fundamentally the halo formed due to diffusion of carbon and other alloying elements from the solid fusion boundary into the molten nugget. To validate this hypothesis, the elemental analysis across the fusion boundary was investigated using the electron probe micro analysis (EPMA). The EPMA measurements showed strong segregation of Mn, Si, and C at the fusion boundary (Figure 3.10). The

locations of this EPMA line analysis on single and double-pulse welds were shown in Figure 3.10a and b. In the single-pulse weld, the segregation of Mn, and Si were seen in the fusion boundary, which is approximately 10  $\mu\text{m}$  in width towards the center of the nugget (see Figure 3.10c). Moreover, the C depleted region was seen inside the halo ring, and a strong C gradient was seen near the fusion boundary (see Figure 3.10e). The average C content in the FZ is 0.25 wt.%, whereas in the halo it is 0.21 wt.% (~16% reduction). This C gradient forms due to a relatively long time that the fusion boundary remains stationary when welding for long weld times, such as 270 ms (see Figure 3.2a). During the welding cycle, nugget growth ends before the termination of the welding current due to the cooling from the electrode. The growth of the fusion boundary stops when heat generation slows to the point where it equals the rate of heat extraction at the electrodes. At this point elements in the solid HAZ will diffuse to the liquid nugget, where their solubility is higher, as proposed by Sherepenko et al. [80]. The strong partitioning of Mn, Si, and C in the vicinity of fusion boundary creates a hardness gradient which can potentially act as local weak point during deformation. Taniguchi et al. [130] similarly observed an area enriched with manganese near the fusion boundary, noting that the presence of this high-hardness region led to cleavage fracture. Thus, it can be inferred that the brittle fracture occurs within the high hardness region of the halo ring.

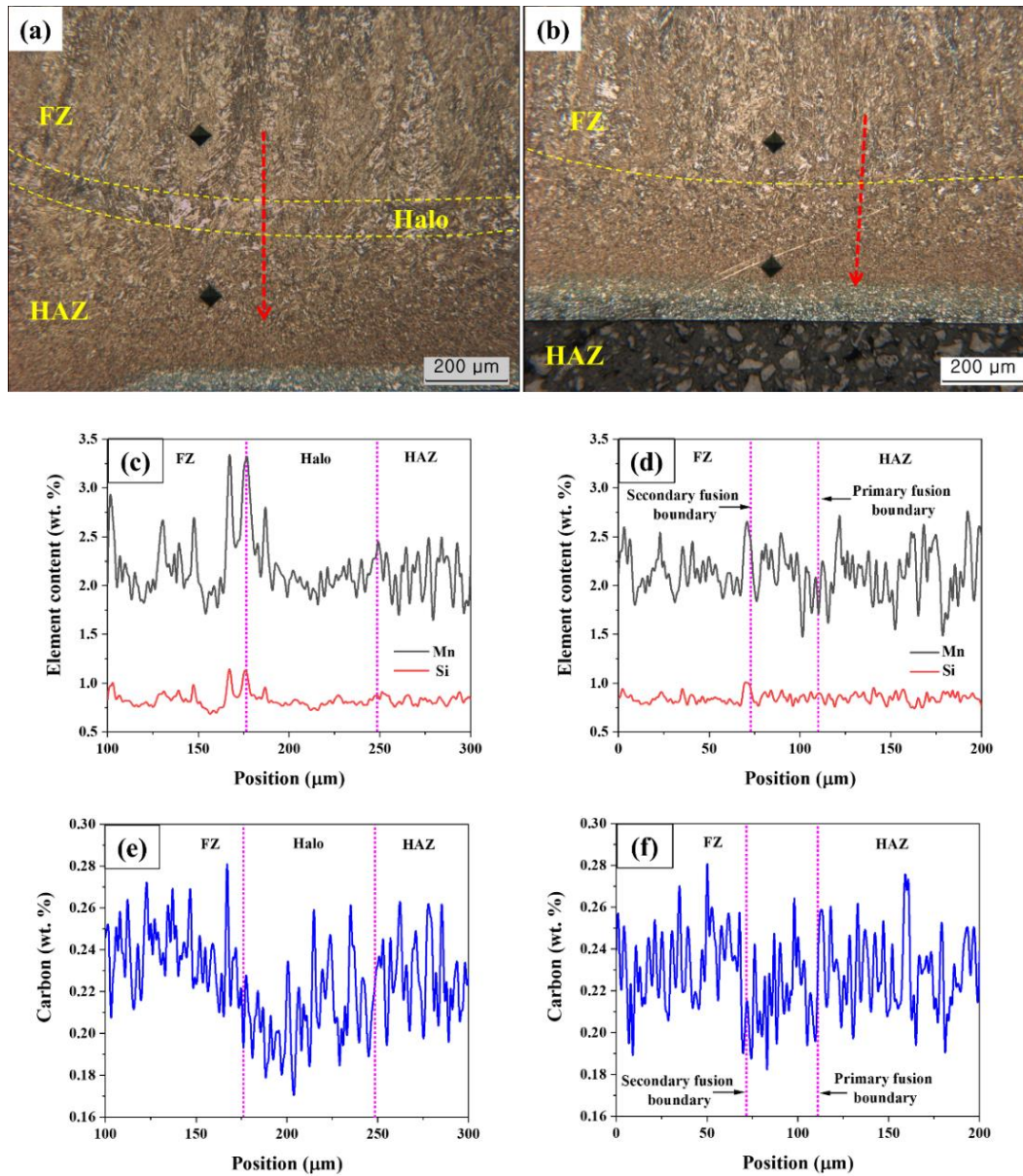


Figure 3.10: Optical micrographs and corresponding electron probe micro analysis of Mn, Si and C across the fusion boundary: (a, c, and e) single-pulse weld with halo ring, and (b, d, and f) double-pulse weld without halo ring

Elemental partitioning has been considerably reduced in the fusion boundary of double-pulse welds (see Figure 3.10b, d, and f). However, there was still a small area ( $<5\ \mu\text{m}$ ) which shows the segregation of Mn, and Si near the secondary fusion boundary, as seen from Figure 3.10d. The region between the primary and secondary fusion boundaries were shown in Figure 3.10d and f. Furthermore, in the double-pulse weld, the

average C content was 0.23 and 0.22 wt.% in the secondary nugget, and between the primary and secondary nuggets, respectively. This lower C content in the FZ also justifies the lower hardness value of the double-pulse weld compared to the single-pulse weld (see Figure 3.7e). Hence, C partitioning was not significant in the fusion boundary of double-pulse welds, as compared to single-pulse welds, as the second pulse homogenizes the alloying elements in the primary nugget boundaries, redistributing the alloying elements at the primary and secondary nugget interfaces. The homogenization process improves the mechanical performance of the double pulse welds. Chen et al. [131,132] also reported that the implementation of stepped current pulses (more than one pulse) in welding schedules can improve the mechanical performances of the spot welds. It should also be noted that the double-pulse welding schedule implemented in this study needs further improvement to completely eliminate this elemental partitioning.

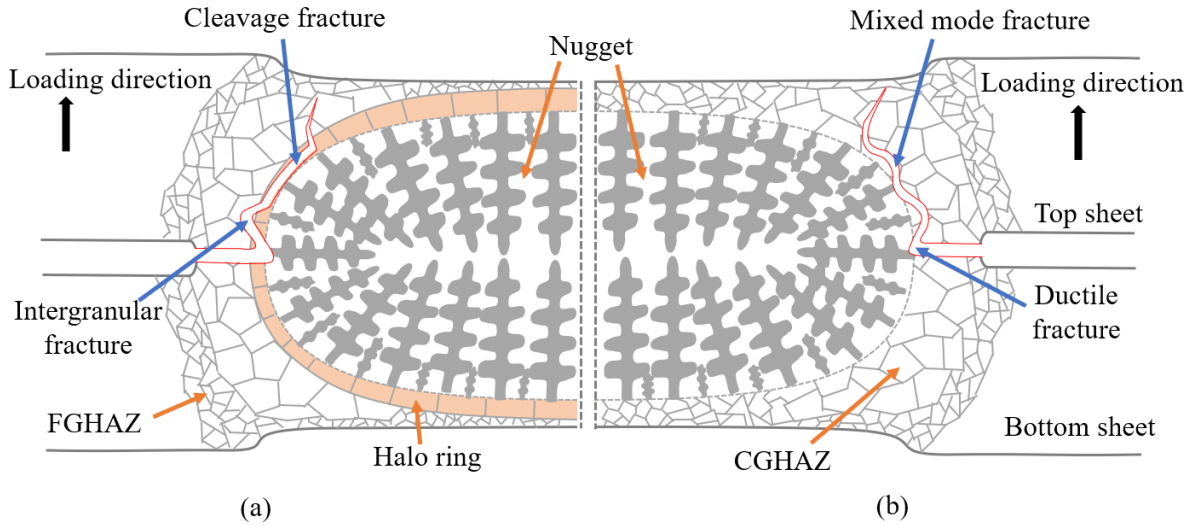


*Figure 3.11: Schematic representation of the fracture path in (a) single-pulse weld showing fracture along the halo ring, and (b) double-pulse weld showing fracture along the CGHAZ.*

The failure path of single and double-pulse spot welds is schematically shown in Figure 3.11. The failure path resulted in a full pullout failure mode in both conditions. However, the fracture path is significantly different in single and double-pulse conditions as shown in Figure 3.11a and b, respectively. In single-pulse condition, the failure predominantly progresses through the fusion boundary (halo), whereas in double-

pulse it remained in the CGHAZ. It is worth noting that, in both conditions, the fracture initiated at the sheet-sheet interface propagated towards the fusion boundary before following an independent path based on the microstructure resulting from the welding parameters used.

### **3.6. Summary**

This chapter conducted a systematic study on improving the mechanical performance by implementing a double-pulse welding schedule to minimize softened zone around the fusion boundary of Q&P 980 steel spot welds, and the following conclusions were drawn from the study. Detailed microstructural characterization and mechanical property correlation were also done in this study.

- 1) Double-pulse welding schedules were successfully implemented in Q&P 980 steels. Applying a higher second current with a short cooling time (30 ms) between the current pulses improves CTS and absorbed energy by 33 % and 110%, respectively, compared to the mechanical properties of a single-pulse weld.
- 2) Spot welds made with a single current pulse had poor CTS in the order of  $3.1 \pm 0.05$  kN, whereas in double-pulse welds CTS improved to  $4.1 \pm 0.06$  kN.
- 3) The halo ring was seen in the single-pulse welds, and it was eliminated through the pulsing technique (high secondary current for a short period of time).
- 4) The halo ring was chemically characterized by EPMA analysis. Carbon partitioning (carbon depleted halo ring), accompanied by Mn and Si segregation, was observed at the fusion boundary, which was attributed as the responsible mechanism for halo formation.
- 5) The strength of the Q&P980 spot welds is predominantly related to the fracture type and its location. Both the spot welds show plug failure mode, however, the spot weld made with a single-pulse condition shows brittle fracture along the fusion boundary (halo), whereas the spot welds made with double-pulse condition failed along the CGHAZ in a mixed mode manner.

# **Chapter 4. Effect of Material Chemical Composition on Halo Formation <sup>2</sup>**

## **4.1. Overview**

Thus far, the research on halo studies has been limited to similar sheet combinations. However, automotive assembly lines utilize various dissimilar steel combinations to construct auto body parts. Therefore, it is imperative to examine how the chemical compositions of materials being welded affect halo formation. This chapter investigates the impact of fusion zone chemical compositions on the formation of the transient softened zone in Q&P 980 steel. To achieve this, spot welds were made in both similar Q&P steel joints as well as joints sandwiching low carbon (LC) or high carbon (HC) steels with Q&P steels, which tailored the chemical composition of the fusion zone. EBSD and EPMA characterizations was conducted on spot welded samples to elucidate the influence of material chemical composition on halo formation. The findings of this study shed light on automakers to choose the right material and modify the welding parameters as necessary.

## **4.2. Introduction**

An automotive body typically contains five thousand spot-welds in its body-in-white structures [71]. These welds joints contain similar or dissimilar combinations of sheet steels, which plays a crucial role in the crashworthiness of the structure [133]. Welding dissimilar materials demands greater precautions compared to the conventional welding of similar steel combinations, since the welding thermal cycle destroys the engineered multiphase microstructures [134]. This is due to potential differences in thickness, thermal conductivity, melting points, and even coating types between the steels being joined [135–137]. The

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<sup>2</sup> This chapter is composed of a submitted manuscript in Metallurgical and Materials Transactions A as outlined in the statement of contributions.

D.C. Ramachandran, A. Salandari-Rabori, A. Macwan, E. Biro, Effect of material chemical composition on the formation of the transient softened zone in a Gen 3 Q&P steel, Metall. Mater. Trans. A (under review).

primary challenges of a RSW process arise from the intricate interplay of chemical and physical factors, such as fusion zone (FZ) composition and heat input, respectively, that come into play during the joining of the sheets [138,139].

The weldability of Q&P steels is one of the most important key factors in controlling the possible usage of this steel grade in the automotive industry. Pouranvari et al. [61] reported that the mechanical performance of the RSW predominantly depends on the failure location in the welds during the application of strain. Previous studies [140,141] showed that failure in welds made without care to optimizing process parameters often failed along a local soft area in the FB, known as the halo ring. The formation of the halo ring in RSW is a result of thermal and metallurgical processes that occurs during the welding [140]. The halo ring region is considered as depleted C, Mn, and Si zone in the resistance spot welds [140,141]. It is believed that these elements diffused from halo ring to the FZ. Amirthalingam et al. [142] showed that the solidification of the weld pool progresses with a strong partitioning of C and P to the unsolidified liquid. Which was cited as the primary reason for the lower level of C at the FB. Furthermore, Zhang et al. [143] reported that Si and Mn that were present in the AHSS, generally segregated on the grain boundaries during welding. This segregation led to the stress concentration at grain boundaries during loading resulted in the reduction of weld strength due to the lower toughness. The authors' previous study showed that implementing a double-pulse welding schedule [140] and post-weld heat treatment such as paint baking [141] homogenizes the elements in the FB by virtue of the heat-treatment temperature and time, leading to the fully or partially elimination of the halo region in two Q&P steel sheet combination. However, in automotive body manufacturing, welds typically involve various sheet combinations, and these diverse steel combinations may influence the formation of the halo ring. To clarify, diffusion of C, Mn and Si towards the halo ring depends on the chemical composition of the steel sheets being used. Only a limited number of studies has been conducted on the weldability of Q&P steels in similar and dissimilar configurations. The spot welding of Q&P steels with diverse steels combination requires a rigorous control of the joining parameters in order

to achieve a suitable mechanical strength, and energy absorption capability for the automotive industry [118].

Despite considerable efforts to elucidate the mechanical behavior and failure mechanisms in resistance spot welds of Q&P 980 steel, there remains a notable gap in understanding the influence of material composition on the microstructural evolution of the weld nugget, and surrounding HAZ. Additionally, due to the constraints regarding the size of the halo ring, prolonged welding times with a welding current nearly at the far end of the weld lobe were employed to enhance halo ring formation [80,81]. This research focuses on providing insights into the microstructural characteristics of 1.2 mm thick Q&P 980 steel sandwiched between low carbon mild steel and high carbon steel with thicknesses of 1.5 mm and 1.4 mm, respectively. To bridge the existing knowledge deficit, the investigation explores into the impact of steel chemistry on the microstructural changes in the nugget, HAZ, and halo ring, employing advanced characterization techniques such as electron probe micro-analysis and electron back-scattered diffraction (EBSD). This study is crucial for advancing our understanding of the intricate relationships between material composition and microstructural evolution in Q&P steel resistance spot welds, providing valuable insights for optimizing welding processes in the automotive industry.

### 4.3. Materials and Methods

#### 4.3.1. Material

A 1.5 mm low C (denoted as LC) steel and a 1.4 mm high C (denoted as HC) steel other than the Q&P 980 steel mentioned in the Table 3.1 were used in this study and their nominal chemical composition also is given in the Table 4.1.

*Table 4.1: Detailed chemical composition (in wt.%) of the steel used in this study.*

|          | C     | Mn   | Si    | Cr+Mo+Ti+Nb | CE   |
|----------|-------|------|-------|-------------|------|
| LC steel | 0.002 | 0.08 | 0.004 | 0.08        | 0.01 |
| HC steel | 0.4   | 0.6  | 0.6   | 0.57        | 0.69 |

#### 4.3.2. Resistance Spot Welding

The welding parameters used in this study are shown in Table 4.2 and Figure 4.1. This study employed extended welding schedules, in which the welding time is longer than the AWS-recommended schedule for similar two sheet combination of 1.2 mm Q&P 980 steels. An electrode force of 4 kN was used in all the experimented conditions. The samples were welded 0.2 kA below the expulsion current. The average nugget diameter of all the conditions is shown in Table 4.2.

*Table 4.2: Welding parameters used in this study and the corresponding nugget diameters.*

|               | Welding time (ms) | Welding current (kA) | ND (mm) |
|---------------|-------------------|----------------------|---------|
| Q&P 980 steel | 470               | 9.5                  | 7.29    |
| LC steel weld | 470               | 9.8                  | 6.96    |
| HC steel weld | 470               | 9.6                  | 7.11    |

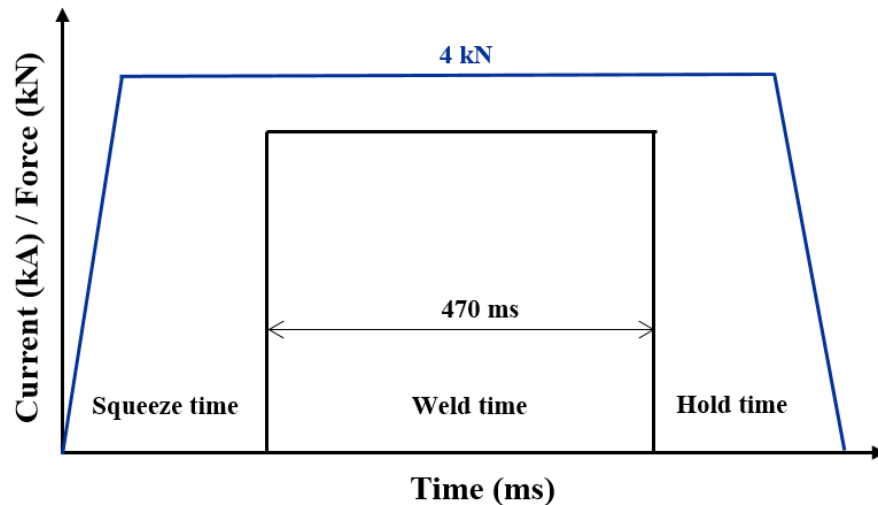


*Figure 4.1: Schematic diagram showing the welding schedule used in this study.*

#### 4.3.3. Microstructural Characterization

For metallographic analysis, the samples were prepared by conventional metallographic techniques, as documented in the previous chapter. The elements and its distribution along the FB were determined using an electron probe microanalysis (FE-EPMA; JEOL JXA-8530F). The EPMA maps were performed with a

1 $\mu$ m step size and a 100 ms dwell time, whereas the quantified line scans were done with a 1  $\mu$ m step size and a 10 s dwell time.

#### 4.4. Results and Discussion

##### 4.4.1. Microstructural Characterization

The cross-sectional micrographs illustrating the microstructure of the 3-sheet Q&P combination, sandwich weld made with LC steel (LC steel weld), and sandwich weld made with HC steel (HC steel weld) combination are shown in Figure 4.2a-c. As can be seen in Figure 4.2, the nugget shapes are rectangular in nature. The weld nugget formation is a balance between heat generation and heat dissipation, which is a function of welding schedule, steel sheet combination, sheet thickness, and heat conductivity [133]. Gould et al. [144] reported that cooling rate in RSW varies, ranging from  $1 \times 10^6$  °C/s for sheet thickness less than 0.5 mm to approximately  $2 \times 10^3$  °C/s for a sheet thickness of 2.0 mm. It is noteworthy that, despite minor differences in welding current depending on the steel combination, the welding parameters are kept constant in all three conditions. The microstructure of the nugget in all three scenarios is characterized by columnar dendrites, indicative of a rapid cooling rate.

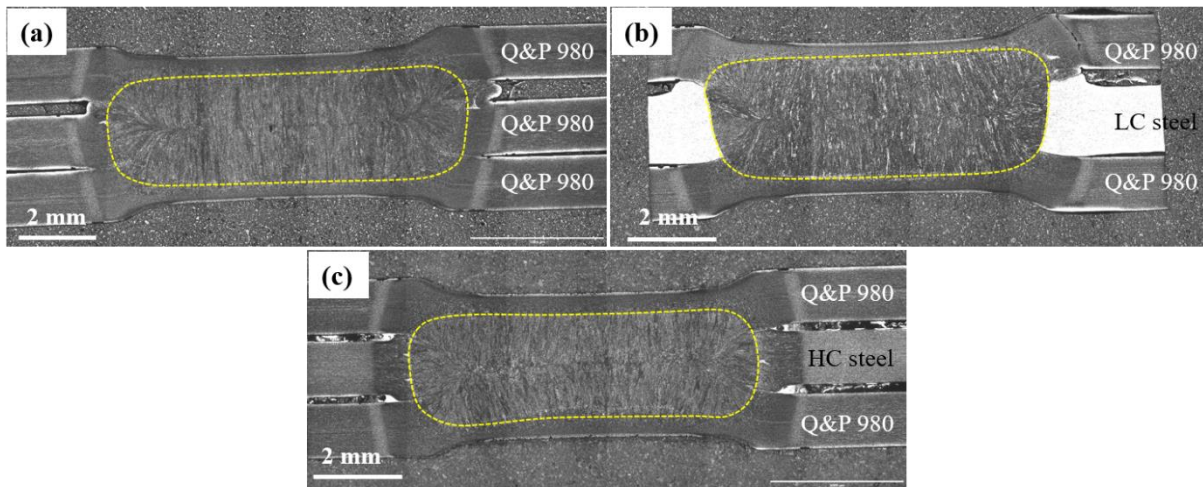


Figure 4.2: Optical microstructure of (a) 3-sheet Q&P 980 (b) LC steel sandwich weld, and (c) HC steel sandwich weld.

Detailed microstructural analysis of both the FZ and HAZ is crucial for gaining insights into the quality of spot welds. The microstructures observed in the FZ and upper critical heat affected zone (UCHAZ) of the 3-sheet Q&P steel configuration closely resemble those found in the two-sheet Q&P steel combinations, as reported in our prior study [140]. In both the FZ and the UCHAZ, the predominant constituents are lath martensite, and block martensite, respectively, depending on its respective morphologies [140,141]. On the contrary, the LC steel sandwich exhibits a combination of martensite, Widmanstätten ferrite (ferrite side plate), and grain boundary ferrite (allotriomorphic ferrite) microstructures, as shown in Figure 4.3. From the image quality (IQ) map of the LC steel sandwich (Figure 4.3a), both ferrite (body-centered cubic-BCC structure) and martensite (body-centered tetragonal-BCT structure) phases are visible. The darker appearance of the martensite phases is attributed to their higher dislocation density, signifying higher lattice distortion compared to the ferrite. Figure 4.3b illustrates the inverse pole figure (IPF) map of the weld, indicating the directional solidification structure aligned with the direction of the highest cooling rate. Within the side of the FZ closer to HAZ (see Figure 4.3a), distinct micrographs depict the presence of ferrite side-plates and grain boundary ferrite, as seen in Figure 4.3c and d, respectively. The characterization of these microstructures was conducted based on their morphologies [145]. It is noteworthy that the FZ microstructure reveals a relatively significant volume of ferrite morphologies, particularly ferrite side-plates, whereas the UCHAZ, the Q&P steel side exhibits coarse packets of martensite, while the LC steel side is predominantly ferritic.

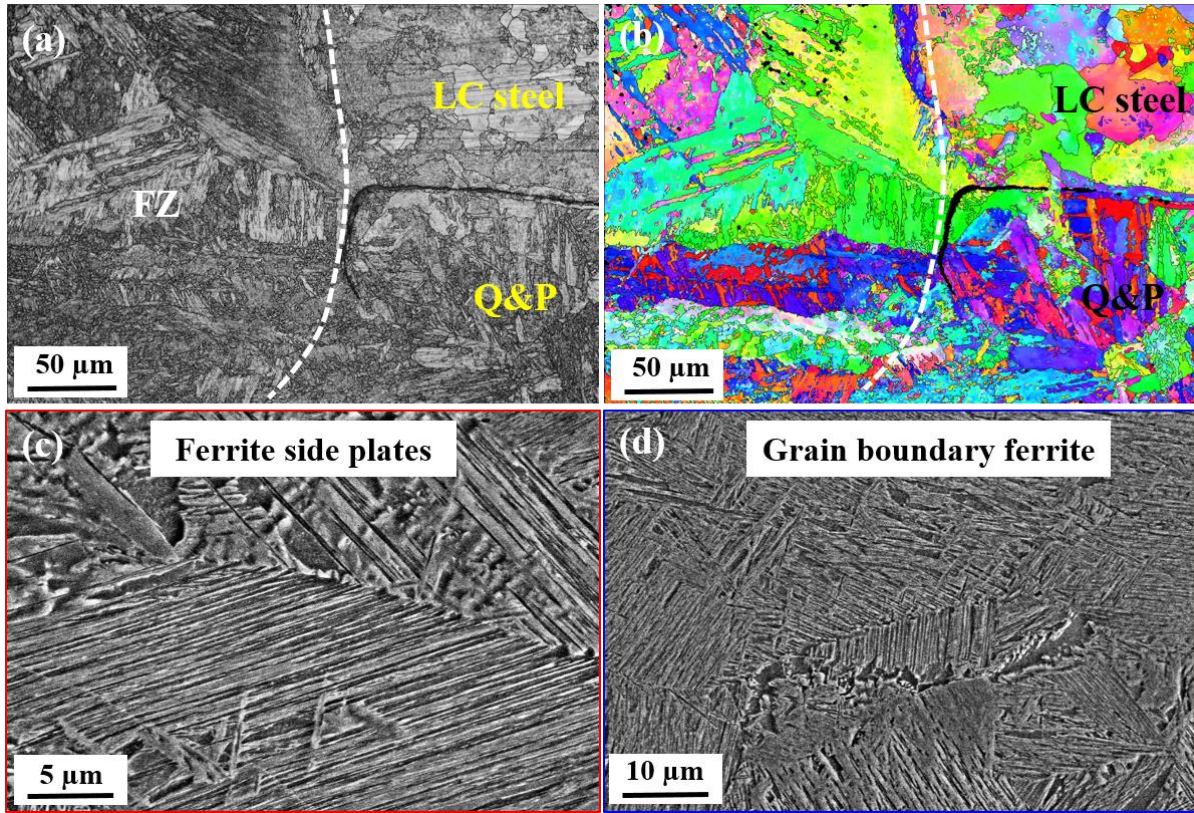


Figure 4.3: A typical EBSD microstructure of FZ and HAZ of the LC steel sandwich weld: (a) IQ map, (b) IPF map, (c and d) FZ microstructure showing different ferrite morphologies; side-plate and grain boundary ferrite, respectively.

A typical EBSD micrograph of the HC steel sandwich weld is shown in Figure 4.4a. The IQ map reveals the Q&P steel on the top and the HC steel in the middle of the 3-sheet stack ups. The IPF map shown in Figure 4.4b indicates the FZ microstructure shares a similar texture with UCHAZ. This also implies that both microstructures are similar in nature, mainly martensitic, and the only difference lying in the martensite morphology. FZ consist of lath martensite whereas the HAZ consist of block martensite. The higher carbon content in the FZ compared to the LC steel case facilitates the formation of martensite in the HC steel weld. The FZ microstructure shows a lath martensitic structure (see Figure 4.4c). The IQ map depicts that there is no ferrite (relatively low dislocation density) microstructure in the characterized area. It also worth noting that the UCHAZ of the HC steel consists of a comparatively smaller grain size than the Q&P steel side (see Figure 4.4b).

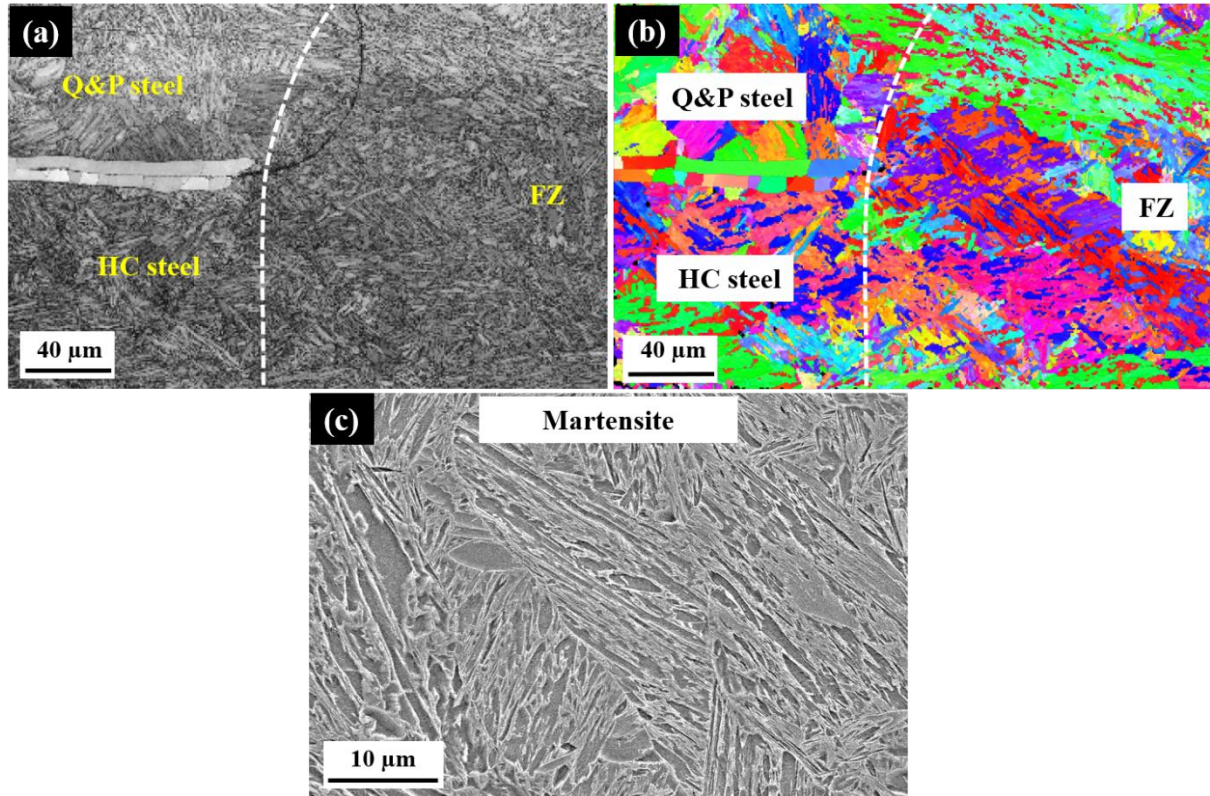


Figure 4.4: A typical EBSD micrograph of HC steel weld showing FZ and HAZ. (a) IQ map, (b) IPF map, and (c) FZ microstructure showing a lath martensite morphology.

#### 4.4.2. Hardness Analysis

The measured cross-weld hardness profile from base metal to FZ in different Q&P steel sheet stack-ups is shown in Figure 4.5. For consistency, hardness analysis was only conducted on the Q&P steel side of the joint (see Figure 4.5a). As shown in Figure 4.5b, the FZ hardness increased to 570 HV in the case of the HC steel sandwiched between the two Q&P steels. Similarly, the FZ hardness significantly decreased to 372 HV when alloyed with a LC steel. This observation is in a good agreement with the observed FZ microstructure, where ferrite side-plate and grain boundary ferrite structures were observed in the FZ of LC steel sandwich weld. It is also imperative to note that spot welding with the HC steel increases the presence of high-carbon martensite which in turn increases the FZ hardness. It is also worth noting that within each dissimilar weld, the hardness was quite constant along the Q&P steel HAZ implying the similar microstructure along the HAZ in all stack-up combinations. Furthermore, in the HC steel case, there is a

slight increase in the hardness near the base material (at HAZ/BM interface) which will be aptly elaborated in the following section.

The high-density hardness analysis along the FB region of Q&P steel side on all the three combinations is shown in Figure 4.5c. Softening in the FB is visible in all the three combinations, namely, the halo region. The extent of softening can be measured by introducing “degree of softening” parameter, the ratio of the difference in hardness between the halo ring and the hardness of the surrounding microstructure (HAZ). The degree of softening of all three combinations are calculated and shown in Figure 4.5d. The degree of softening gives a clear understanding on the severity of the halo ring. For instance, 3 sheet Q&P steel combination has 19% reduction in halo ring hardness compared to a no halo condition (spot welds with no halo region). Similarly, LC steel and HC steel weld conditions have 46% and 16% reduction in hardness compared to the no halo condition, respectively. It can be seen from the Figure 4.5d that the LC steel weld combination has the highest and the HC steel combination has the lowest severity of halo, respectively. It can be seen from the Figure 4.5c that the hardness of the HAZ does not reach a consistent value across different weld combinations. Moreover, the reduction in hardness at the halo ring is directly related to the distribution of alloying elements on it. Further explanation regarding the differences in alloying elements within the halo ring will be provided subsequently.

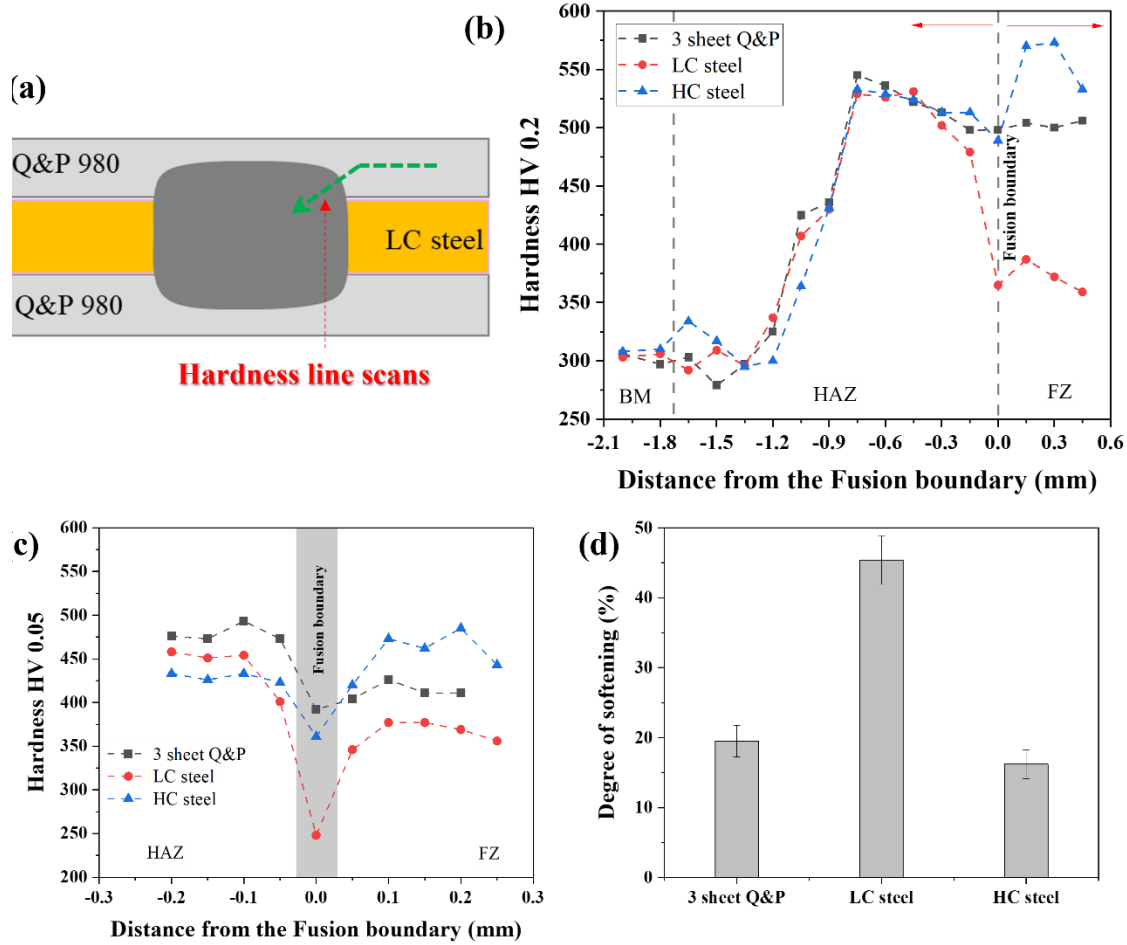


Figure 4.5: (a) Schematic diagram showing the position of hardness analysis, (b) hardness profile of the three combinations, (c) high density hardness along the FB, (d) degree of softening.

#### 4.4.3. Elemental Distribution

The elemental distribution at the FB is one of the most important factors in defining the halo ring in all three combinations of resistance spot welds. For this purpose, a comprehensive EPMA study was conducted on the samples focussing on the FB. EPMA results of similar combinations of the two-sheet Q&P steel spot welds were reported in our previous study [140] where a carbon depleted region was found near the FB, closer to the fusion zone side. Moreover, a segregation of Mn and Si near the FB, closer to the HAZ side was also observed. Figure 4.6 shows the elemental distribution map of the 3-sheet Q&P steel weld. Elemental map of C, Si, and Mn are shown in Figure 4.6b-d, respectively. It can be seen there is no

significant difference in C, Si and Mn concentration between the FZ and the HAZ in Q&P 3-sheet stack-up. This could be due to comparatively wider scanning area compared to the width of the halo ring. However, close observation shows that there is a slight reduction in C content at the FB of the top Q&P steel in the stack up (see Figure 4.6b). The segregation of Si and Mn on the inter-dendritic region can be seen from the Figure 4.6c and d. Eftekharmilani et al. [10] reported that during solidification the elements such as Mn, Si, and P tend to segregate at the inter-dendritic region due to their preferential partitioning behaviour. Although the solidification process can potentially draw more solute Mn and Si towards the molten weld pool from the solid HAZ in 3-sheet Q&P weld, the composition of the FZ and the welding schedule employed in this case do not facilitate this phenomenon. Therefore, there is no significant depletion of Si and Mn observed in the halo of the 3-sheet Q&P stack-up at this welding conditions.

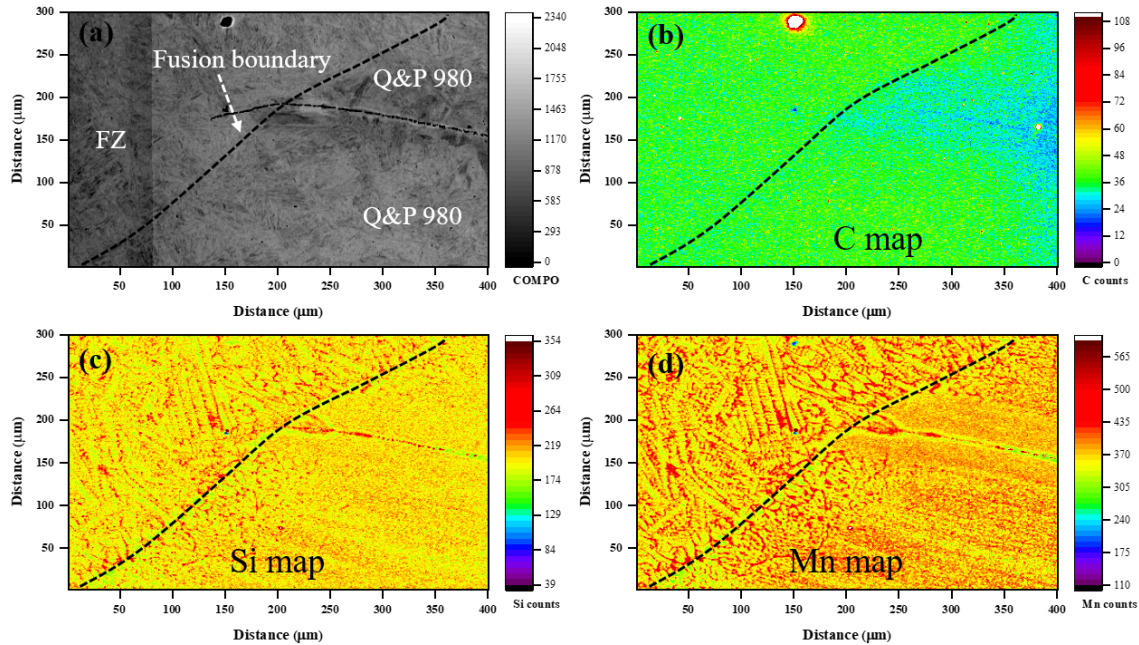


Figure 4.6: 3-sheet Q&P steel stack-up, (a) SEM backscatter (BSE) image; Elemental distribution map: (b) Carbon, (c) Silicon, and (d) Manganese.

The elemental distribution map of the LC steel stack-up weld is shown in Figure 4.7. BSE image of the interface between Q&P and LC steel is shown in Figure 4.7a. In Figure 4.7b, the EPMA map highlights a

strong depletion of C near the FB. Notably, the C depleted region extends approximately 75  $\mu\text{m}$  from the FB (see white arrows in Figure 4.7b) and is within the HAZ side of the FB. Additionally, the carbon content of both the FZ and the halo region is comparable to each other and lower than that of the Q&P steel base metal. It should be noted that the overall carbon content decreased in the FZ due to the mixing of lower alloyed IF steel with a Q&P steel, which has a comparatively higher carbon content. The observed C depletion in this area aligns well with this measured local reduction in hardness in the LC steel FZ (see Figure 4.5b).

As shown in Figure 4.7c and d, Mn and Si compositions are significantly lower in the case of IF steel compared to Q&P steel base metal. Moreover, there are no changes in the chemical composition visible on the IF steel side. A significant depletion of C, Mn, and Si, approximately 75  $\mu\text{m}$  from the FB, is observed on the Q&P steel side (see Figure 4.7b-d). Within this 75  $\mu\text{m}$  wide region adjacent to the FB, the Mn and Si-depleted region, the 25  $\mu\text{m}$  region nearer to the FB, experiences a substantial depletion of alloying elements, while the rest of the area maintains similar Mn and Si content as the FZ. As described in the authors' previous work [140,141], the region near the FB, characterized by a depletion in C, Mn, and Si, is termed the "halo ring". When alloyed with a lower alloyed steel, the halo ring exhibits more severe C, Si, and Mn depletion due to the triggered diffusion (downhill diffusion) of these alloying elements towards the FZ. Additionally, it can be also seen that there is a potential for diffusion of C from the halo to the HAZ (see Figure 4.9). This can be attributed to the higher activity (difference in chemical potential) of C caused by the variation in substitutional solute content, towards the HAZ from the halo ring. This difference in chemical potential promotes the diffusion of C from the halo ring to the HAZ, intensifying the C depletion in the halo ring. As shown in the Chapter 5 the microstructure of the halo ring was identified as upper bainite. This implies that the absence of carbide suppressing element such as Si, promotes the carbide formations (cementite) in the halo region, resulting in a reduced availability of carbon in solid solution and causing the area to transition into bainitic ferrite. To substantiate this, there is also a notable increase in C, Mn, and Si content (approximately 20  $\mu\text{m}$  inside the FZ from the FB) as illustrated in Figure 4.7b, c and d.

Additionally our previous study [141] has shown that, the grain boundaries in the UCHAZ are depleted in Mn and Si, this implies that these elements diffuse into the FZ from regions farther away from the HAZ through grain boundary diffusion. In summary, the width of C depleted zone and the level of C depends on the welding time, temperature, the carbon content in the materials, co-diffusion of Mn and Si, and the carbon diffusivities [146].

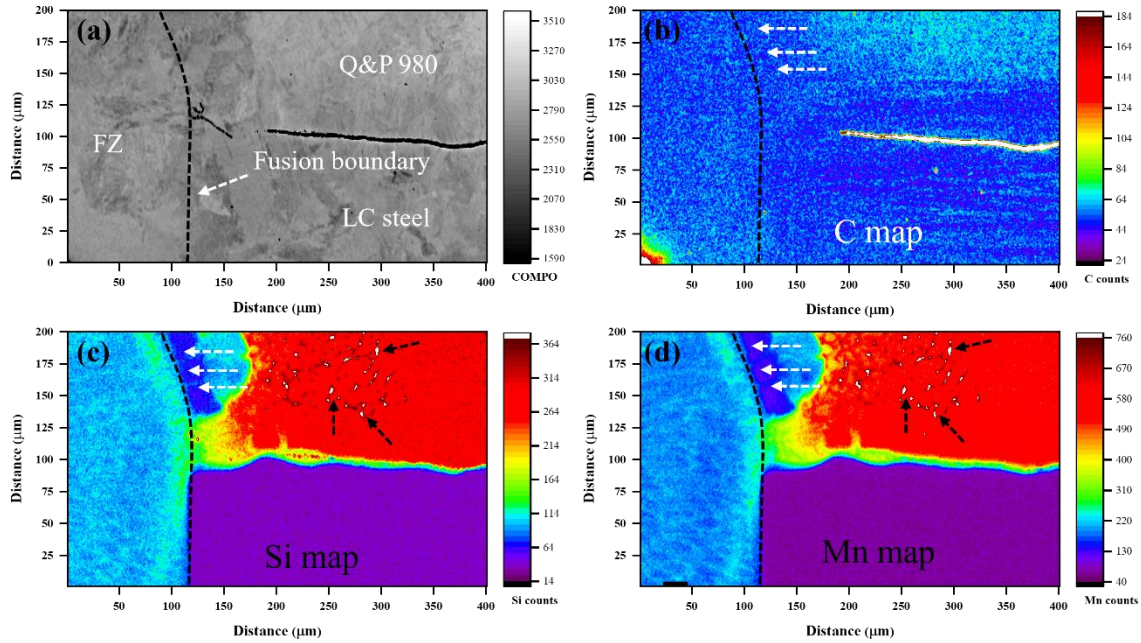


Figure 4.7: LC steel sandwich weld (a) BSE SEM image; Elemental distribution map: (b) C, (c) Si, and (d) Mn.

The segregation of Mn and Si at the grain boundaries followed by the 75 μm depleted region from FB (see black arrows, which is at least ~120 μm away from FB) can be seen from Figure 4.7c and d. Microstructural analysis of this segregated region reveals that segregation occurs at the grain boundaries of the UCHAZ. A comprehensive examination of the elemental segregation in the UCHAZ microstructure and its effect on mechanical properties is documented elsewhere [141].

The HC steel sandwich welds, on the other hand, behaved differently than the LC steel case. Although, a slight reduction in C, Mn and Si observed at the FB of Q&P steel side, major elemental diffusion happened

in the high carbon steel side of the joint, as shown in Figure 4.8. The carbon map in the Figure 4.8b shows that there is no visible carbon depleted region in the Q&P steel side. However, a significant C depleted region ( $\sim 30 \mu\text{m}$ ) was seen in the HC steel side (see white arrows). The high C region (base metal composition) in the HC steel side starts around  $110 \mu\text{m}$  away from the FB. Similarly, there is no difference in Mn and Si near to the FB in the Q&P steel side (see Figure 4.8c and d). Furthermore, likewise in the LC steel case, there was no visible segregation of C, Mn and Si inside the FZ near to the FB.

It is worth noting that C, Mn and Si depletion is shifted to the high carbon steel side. That means, alloying the Q&P steel with a higher alloyed steel causes depletion of elements in the higher alloyed steel side. On the other hand, alloying with a higher alloyed steel will shift the halo to the higher alloyed steel side due to the concentration difference between the steels (downhill diffusion). This can be also seen from the hardness scan (Figure 4.5c) that the difference in hardness between the halo ring and HAZ is minimal in Q&P steel side. However, the hardness of the FZ is significantly higher than the HAZ and halo ring. This is obvious that alloying with a higher alloyed steel will increase the overall hardness of the weld FZ.

Although there is no visible C depletion observed in the Q&P steel side of weld made with HC steel, a slight decrease in hardness is evident on the hardness map (see Figure 4.5c). It is worth noting that in Q&P steel side, C diffusion from the FB to the HAZ occurs due to the activity (uphill diffusion), resulting in a carbon depleted halo. However, this depleted C in the halo is replaced by C from the FZ, because of the concentration difference (see Figure 4.9). Nevertheless, the carbon loss from the halo to the HAZ, driven by activity, is more significant than the carbon diffusion from the FZ to the halo due to concentration.

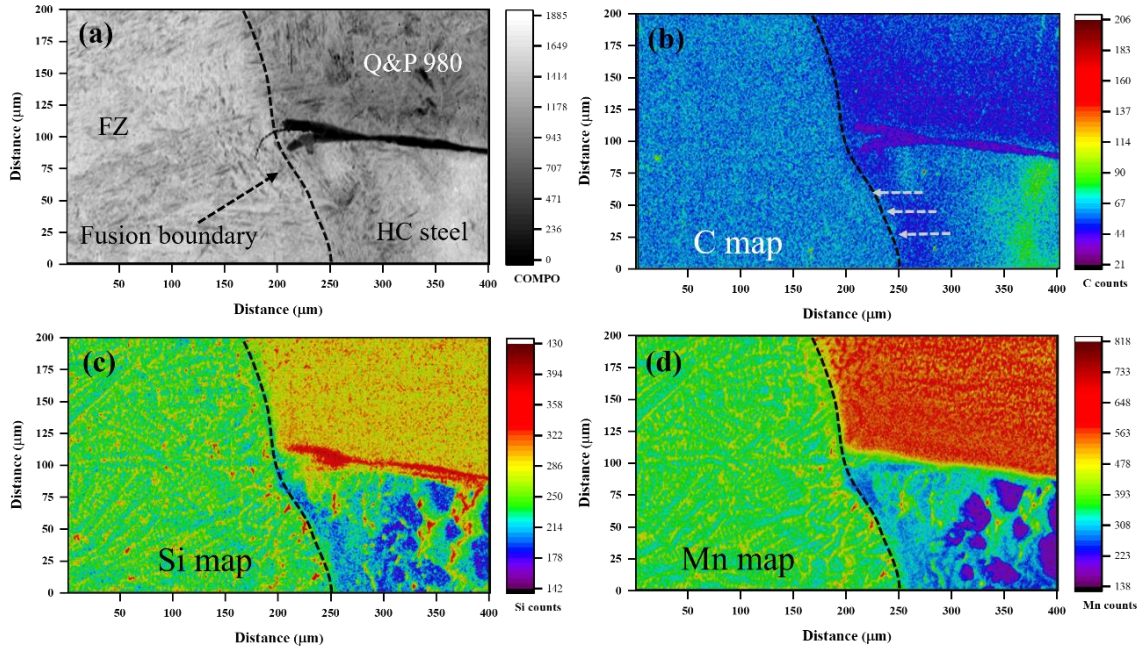


Figure 4.8: HC steel sandwich weld (a) BSE SEM image; Elemental distribution map: (b) C, (c) Si, and (d) Mn.

To quantitatively understand the behaviour of carbon throughout the weld area of the Q&P steel, EPMA line scans were conducted, and the results are summarised in Figure 4.9. In the 3-sheet Q&P case, where no mixing with other steels happened, it is evident that the C content in the HAZ and FZ is similar. Conversely, mixing with a lower alloyed steel (LC steel), reduces the overall C content in the FZ. Correspondingly, alloying with high carbon steel increased the overall C content in the FZ. These observations align well with the hardness values presented in Figure 4.5. Notably, in the LC steel case, the C content in the HAZ is slightly higher toward the base material. This observation was consistently confirmed through three repetitions of the line scans, indicating a potential uphill diffusion of C towards the HAZ due to its difference in chemical activity. It is crucial to highlight that the EPMA analysis precisely measures the amount of carbon and other alloying elements in the material. The determined C content, approximately 0.22 wt.%, demonstrates good agreement with the reported composition in Table 3.1.

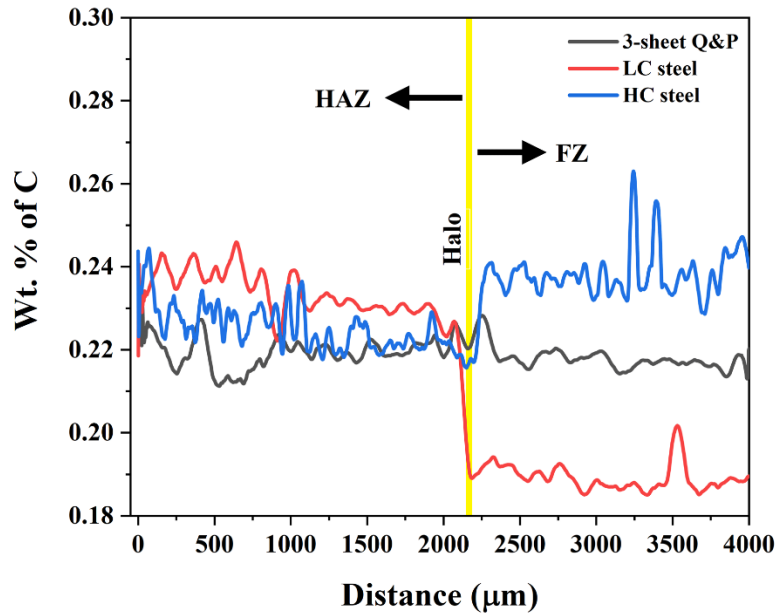


Figure 4.9: EPMA line scans taken across the FB approximately through the middle of the Q&P steel side of weld.

The EPMA maps indicate a dissimilarity between the C, Si, and Mn content measured in the FZ and the calculated alloying elements based on each sheet's contribution to the total volume. The corresponding measured and calculated C, Mn, and Si contents are shown in Figure 4.10. To illustrate, the calculated C content for an LC steel weld was determined by computing the volume percentage of the nugget, by considering volume contributions from each steel sheet being joined. To ensure accuracy, five samples were selected for each condition to validate the results, with the nugget's volume estimated from the cross-sectional area. The measured and calculated C contents with the FZ of the experimented welds are shown in Figure 4.10a. In 3-sheet Q&P weld the measured and calculated C content ( $\sim 0.21$  wt.%) exhibit a close agreement with each other. However, within the FZ of the LC steel weld, the measured carbon content is notably 53% higher than the calculated value. Conversely, in the HC steel weld, the measured C content is 40% lower than the experimental value. This discrepancy provides valuable insights into the carbon diffusion towards the FZ, elucidating the influence of concentration difference. Additionally, the reduction in carbon observed in the FB of the Q&P steel is attributed to uphill diffusion of carbon.

The diffusion of carbon from the halo ring is related with either its chemical potential ( $\mu$ ) or activity ( $a$ ), indicating the measure of a tendency of an atom to leave a solution. When the chemical potential is low, the atoms exhibit reluctance to leave the solution. The chemical potential of a C atom, assuming an ideal solution, can be expressed by Equation 4.1[147]. It's important to note that this equation offers a simplified representation of carbon's chemical potential in steels.

$$\mu_C = G_C + R_b T \ln X_C \quad 4.1$$

where  $\mu_C$  is the chemical potential of carbon,  $G_C$  denotes the Gibbs free energy of the carbon,  $R_b$  stands for the universal gas constant,  $T$  is the temperature, and  $X_C$  represents the mole fraction of carbon. Thus, it is evident that the chemical potential is directly related to the temperature of the system. Additionally, the change in free energy also decreases with increase in solute concentrations [147]. Consequently, in LC steel weld, the chemical potential of the halo is higher than that of the HAZ. Therefore, the C diffusion happens towards the HAZ from higher activity halo to the lower activity HAZ (see Figure 4.11b). Conversely, in 3-sheet Q&P weld, the diffusion of carbon is minimal. Nevertheless, the diffusion of carbon occurs due to the difference in chemical potential aided by the temperature difference. It's worth noting that in future studies, the diffusion of C will be further assessed by taking into account the equilibrium composition of C at the solid, liquid, and solid-liquid interface as a function of temperature, along with the associated chemical potential difference compared to the actual composition.

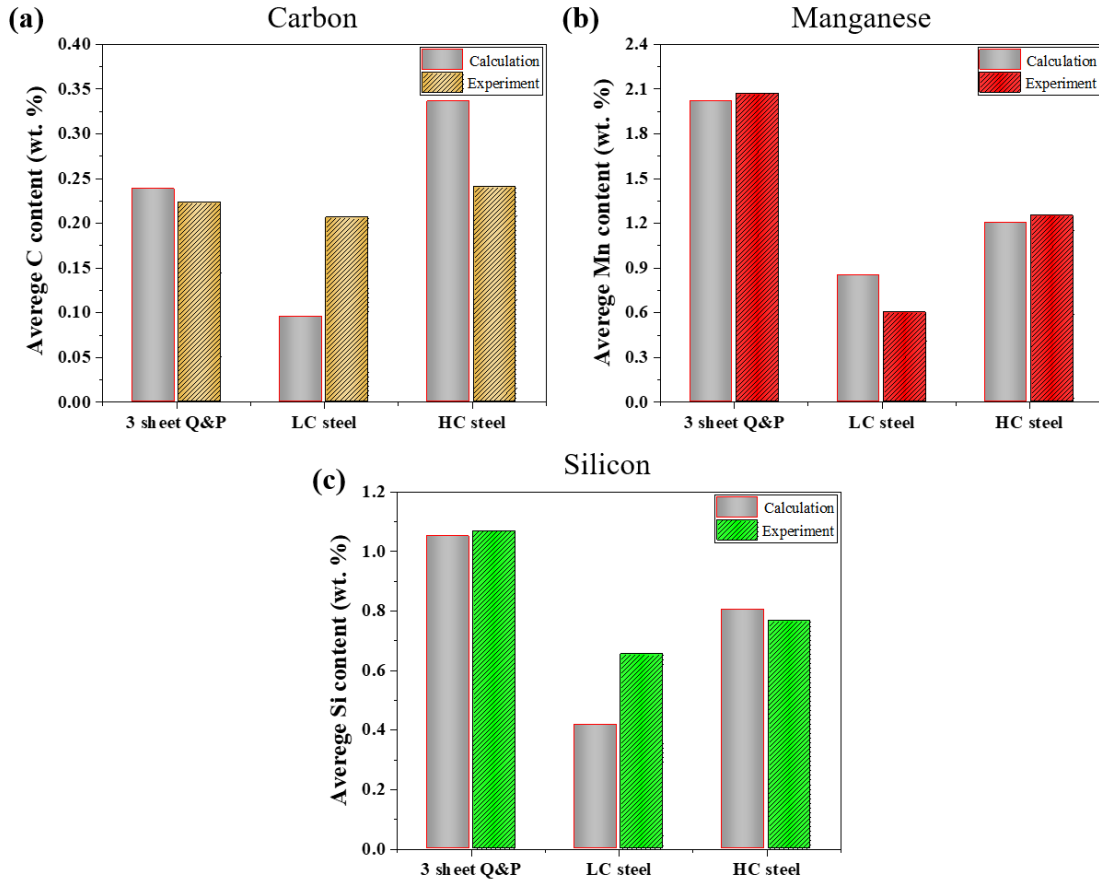


Figure 4.10: Measured and calculated elemental composition within the FZ near to the FB of Q&P steel side (a) C, (b) Mn, and (c) Si.

In contrast, the Mn content in the 3-sheet Q&P and HC steel welds demonstrates a close agreement between the measured and calculated amounts (see Figure 4.10b). The difference in Mn content are 2.4% and 3.5%, respectively, for the 3-sheet Q&P and HC steel welds. Conversely, a significant 41% decrease in Mn content is observed in the FZ of the LC steel weld. As illustrated from Figure 4.7d, the Mn segregate near the FB, which is inside the FZ, and then Mn diffuses into the inter-dendritic regions in the UCHAZ. Huang et al. [146] reported that the diffusion of carbon in welds is linked to the precipitation of metal carbides like  $M_7C_3$ , and  $M_{23}C_6$ , depending on the available carbon content in solid solution. Therefore, it can be assumed that the Mn may form metal carbides in combination with carbon at the grain boundaries, which need further validation.

The Si content follows a similar trend to Mn in the 3-sheet Q&P and HC steel cases. However, in the LC steel weld, there is a notable 36% increase in Si content during measurement. This increased Si content is concurrent with the observed decrease in Mn in the FZ. Examination of EPMA results presented in Figure 4.6-9, reveals intriguing Si and Mn segregation in the inter-dendritic region. A comparison between 3-sheet Q&P weld with HC, and LC steel welds highlights more pronounced inter-dendritic segregation (see Figure 4.7 and Figure 4.8) in HC and LC steel welds, respectively, could be attributed to the lower boron content in the FZ composition [112]. Essentially, it can be speculated that the reduction in boron levels during alloying with LC and HC steel in Q&P diminishes grain boundary stabilization by boron, facilitating greater diffusion of Mn and Si towards the FZ [142].

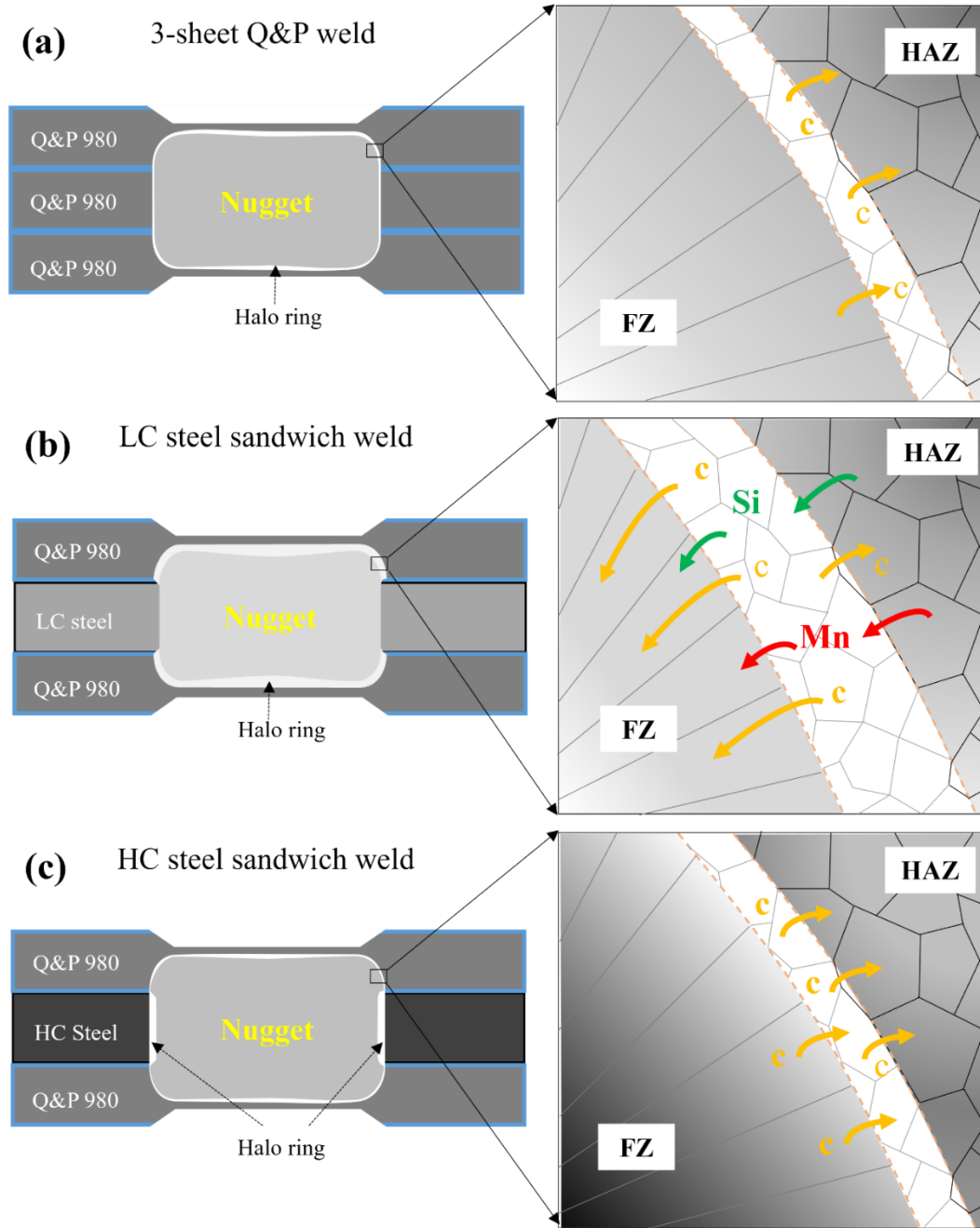


Figure 4.11: Schematic diagram showing the diffusion of C, Mn, and Si in (a) 3 sheet Q&P weld, (b) LC steel weld, and (c) HC steel weld.

In summary, the diffusion of alloying elements from the halo to the FZ and HAZ of all the experimented welds are summarised in Figure 4.11. The high degree of softening seen at the FB of LC steel is attributed to the combined effect of downhill diffusion of C towards the FZ from the halo due to the concentration

difference, and uphill diffusion of C to the HAZ due to the activity (see Figure 4.11b). Kong et al. [148] estimated the time required for C depletion to happen in martensite at 320°C and 260°C to be 0.1 to 0.5 s, respectively. However, the diffusion of C depends on the crystal structure. This estimation aligns well with the welding time employed in this study, which is 470 ms. Moreover, the temperature at the UCHAZ during welding is close to the steel's melting point, suggesting that carbon diffusion occurs even faster due to elevated temperatures. To further substantiate, the diffusion of Mn and Si also promotes this process. It is also worth noting that the factors such as welding temperature, time, pressure, and the composition of the base materials all influence the C diffusion behavior during resistance spot welding. A comprehensive investigation of this phenomenon will necessitate detailed transmission electron microscopy analysis, which is the focus of an ongoing research.

#### **4.5. Summary**

The effect of material chemical composition on the halo formation and its characteristics were systematically studied by alloying with a lower (LC) and higher alloyed (HC) steel. A comprehensive analysis, employing EBSD analysis for microstructural assessment and EPMA analysis for chemical analysis, led to the following key findings:

1. Resistance spot welds produced by sandwiching 1.5 mm LC steel and a 1.2 mm HC steel between two sheets of Q&P980 steel sheet, revealed a significant decrease in the compositions of C, Mn, and Si in the FZ of weld made with LC steel due to the admixture of low-alloyed material in the FZ, as observed through EPMA. This change corresponded to a 46 % reduction in hardness at the FB in LC steel sandwich welds.
2. The halo formation was more prominent in welds with lesser alloyed FZ compared to those with higher alloyed FZ. The degree of softening followed a decreasing order of: LC steel sandwich weld, 3-sheet Q&P weld, and the HC steel sandwich weld.

3. In LC steel sandwich welds, a region of high Si and Mn segregation inside the FZ, closer to the FB, was observed on the Q&P steel side. Conversely, no significant depletion of C, Mn, and Si was observed in the HC steel sandwich weld case on the Q&P steel side. Interestingly, the halo formation shifted to the HC steel side (middle sheet) in weld made with HC steel. The halo formation in weld made with HC steel was attributed to the uphill diffusion of C from halo to the HAZ due to the higher activity.
4. The transient softened zone can be modified by alloying with greater and lesser compositions of steel in dissimilar welded joints. Halo formation in weld made with LC steel is attributed to the combined effect of downhill diffusion of C towards the FZ from the halo due to the concentration difference, and uphill diffusion of C to the HAZ due to the activity.

## Chapter 5. Microstructure and its Formation Mechanism of the Halo Ring<sup>3</sup>

### 5.1. Overview

Although progress has been made in understanding the microstructure of the halo ring, the microstructural evolution and its formation mechanism remain poorly understood. In the previous chapter, the diffusion of carbon (resulting in a carbon depleted halo ring) along with Mn and Si was observed at the fusion boundary. It was believed that the depleted carbon region would transform most likely into a low carbon martensite structure during solidification. Even though this hypothesis can be validated through transmission electron microscopic (TEM) analysis, the small size of the region around the nugget poses challenges for conventional TEM sample preparations. To address this issue, the width of the halo ring should be increased to enable characterization of this region using EBSD and TEM analysis.

In order to increase the width of the halo ring, a dissimilar spot-welding approach involving three sheets comprising a 1.5 mm IF steel sandwiched between two 1.2 mm Q&P steel sheets, was used to maximize the heat input. A prolonged welding time of 470 ms was adopted at a welding current of  $I_{\max} - 0.2$  kA, while other welding parameters were kept constant to minimize their effects. Advanced TEM analysis techniques were employed to characterize the halo and propose its formation mechanism in this chapter.

### 5.2. Background

Various investigations indicate that the softening phenomenon at the FB adversely impacts the failure mode of the spot welds thereby it promotes low energy failures [15,16,72,80,81,111,115]. To effectively mitigate

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<sup>3</sup> This chapter is composed of a submitted manuscript in Scripta Materialia as outlined in the statement of contributions.

D.C. Ramachandran, A. Salandari-Rabori, A. Macwan, E. Biro, Microstructural evolution and formation mechanism of the transient softened zone along the fusion boundary in resistance spot welding of a 3G advanced high strength steel, Scripta Materialia, 2024 (under review)

the effect of this localized softening, a fundamental understanding of the halo microstructure and its characteristics is required. There are limited studies dealing with the microstructure and mechanism of halo formation. Li et al. [115] examined a DP 590 steel spot weld and anticipated that C depletion from the boundary to the grain interior possibly led to halo formation, though they lacked evidence for this claim. Sherepenko et al. [15,80,81] investigated resistance spot weldability of a 22MnB5 steel and proposed a mechanism for halo formation where  $\delta$  ferrite formed in the FB due to the prolonged isothermal soaking of the fusion zone (FZ) in the peritectic region. They supported their theory with evidence of carbon depletion in the FB through carbon mapping from phase-field simulations. Mohamadizadeh et al. [72] proposed that various solutes, including carbon and other alloying elements, diffuse from the solid heat-affected zone (HAZ) into the molten weld pool, potentially being a key factor in halo formation. They also proposed that solute partitioning could result in the formation of softer lower-carbon martensite adjacent to the FB, creating the halo ring. Lee et al. [114], on the other hand, examined the spot weldability of 15B22 hot-stamped steel and discovered that the halo consisted of soft ferrite, aligning with Sherepenko et al.'s [15,80,81] observations. Additionally, Zhang et al. [111] explored the spot weldability of Q&P 980 steel and observed a strong Mn depletion at the FB. They proposed that the decrease in hardness near the fusion zone is aligned with this phenomenon. However, despite their findings, Zhang et al. [111] failed to characterize the microstructure and propose a possible halo formation mechanism. Recently, Manladan et al. [16] identified a lath martensite structure in the halo ring, hypothesizing that the halo ring formed due to the macro-segregation of C and other alloying elements from the austenite to the weld pool. Despite numerous attempts to unveil the formation mechanism and microstructure of the halo ring, the studies suffer from a lack of in-depth characterizations and analysis of formation mechanisms. Accordingly, a fundamental understanding of the halo ring is essential in the scientific community to mitigate this transient softening region, improving the post-welded properties of the resistance spot welds. Therefore, in this study, we conducted a fundamental thorough analysis of the halo ring microstructure and proposed a formation

mechanism by using the state of art electron microscopy; electron backscattered diffraction (EBSD) and transmission electron microscopic (TEM) analysis.

### 5.3. Materials and Methods

In the present study, two 1.2 mm thick Q&P 980 steel samples were resistance spot welded, with a 1.5 mm interstitial-free (IF) steel sheet in the middle (three-sheet stack up). The composition of the Q&P 980 steel and IF steel were given in Table 3.1, and Table 4.1, respectively. The three-sheet stack-ups underwent resistance spot welding with a long welding time (470 ms) at its  $I_{\max}$ -0.2 kA current. RSW was carried out using a MFDC transformer with a C-type gun and a Rexroth Bosh weld controller as described in the section 2.2. Other welding parameters are kept constant and were reported section 4.3.2. The microstructure and mechanical properties of the Q&P steel were reported in section 3.3.1. The microstructure of the halo ring was analysed using SEM and EBSD in a JEOL JSM 7000F microscope. TEM analysis was conducted on the focussed ion beam (FIB) milled samples extracted from the halo region using a Talos 200 TEM at an accelerating voltage of 200 kV.

### 5.4. Results and discussion

#### 5.4.1. Charecterization of the Halo Microstructure

An SEM image of the weld illustrating the interphase between Q&P and IF steel, with a distinctive halo ring on the Q&P steel side is shown in Figure 5.1a. Details from the areas of the halo and HAZ, enclosed by the black rectangle, are shown in the IPF map in Figure 5.1b. The IPF map reveals that the halo ring is composed of coarse grains with relatively homogeneous microstructure and no evident lath or packet boundaries, which is in contrast to the lath martensitic structure observed in the HAZ. It has been reported that long welding time induces grain growth in the microstructure surrounding the weld nugget [87]. The width of the halo varies throughout the cross-section since the cooling rate significantly affects its formation. The average width of the halo ring is approximately 30  $\mu\text{m}$  near the sheet-sheet interphase (red arrows in Figure 5.1a) and it varies to  $\sim 150$   $\mu\text{m}$  near the electrode imprint region. In other words, the cooling

rate is higher, leading to an earlier arrest of nugget growth [149] in the electrode imprint area compared to the sheet-sheet interphase, which leads to the formation of a wider halo. A higher the cooling rate facilitates greater extraction of heat through the electrodes. Moreover, as seen in Figure 5.1b, it is evident that all the microstructural constituents within an individual grain of halo share similar crystallographic orientations. This implies that the microstructural constituents in that specific location have undergone transformation from a common parent grain [34].

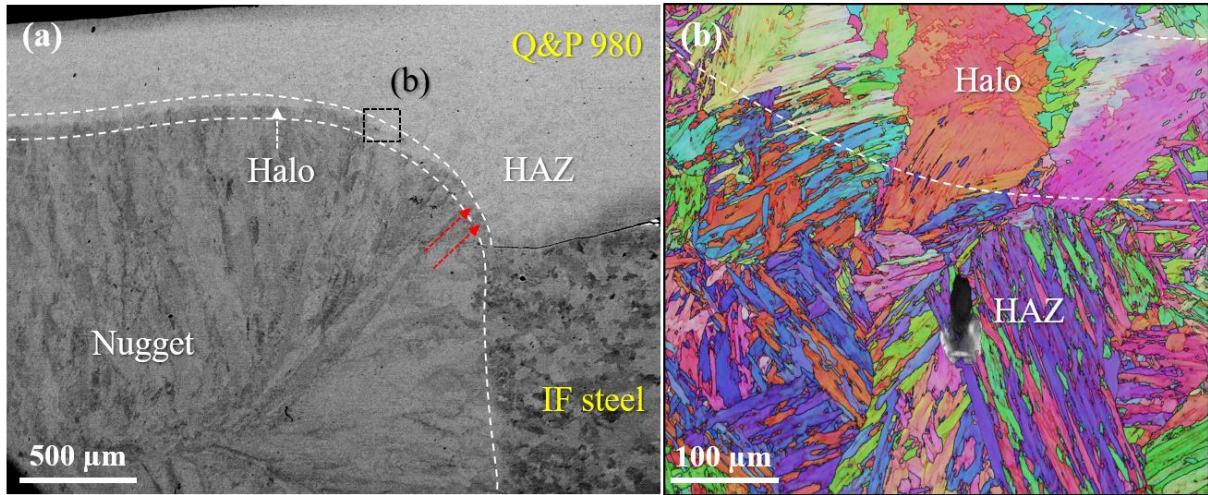


Figure 5.1: Microstructure of the spot weld three sheet stack up: (a) SEM cross-sectional micrograph of the weld and (b) EBSD IPF map of halo ring taken from the area shown in (a).

In-depth microstructural characterization of the halo ring using TEM is shown in Figure 5.2. The area from where the sample was extracted, and the extracted sample are shown in Figure 5.2a. By analyzing the morphology from the TEM bright field image (Figure 5.2b), it can be seen that the microstructure of the halo consisted of an upper bainitic structure (no carbides were found in the ferrite matrix). It was observed that dislocation pile-ups, caused by deformation from electrode force, and structures were not consistent throughout the structure, with some scatter pile-ups at initial prior-austenitic grain boundaries (PAGs) (see Figure 5.2b). A selected area diffraction (SAD) pattern was extracted from the circle shown in Figure 5.2b and is depicted in Figure 5.2c. The interpretation of the SAD pattern shows that the microstructure consists of ferrite and cementite. The ferrite has an  $[\bar{3}27]_{\alpha} \parallel [0\bar{1}1]_c$  orientation relationship with cementite, which

is not commonly documented in literatures. To gain deeper insight, dark field TEM images were taken by isolating one spot (d in Figure 5.2c) from the other reflections. The reflection from the ferrite pattern shown in Figure 5.2d, confirms the presence of bainitic ferrite microstructure in the halo ring, and the other reflection (see Figure 5.2c) confirms the presence of cementite as shown in Figure 5.2g. The HAADF-STEM images were captured by placing the bainitic ferrite microstructure in the [100] zone axis, and the interplanar (d) spacing was measured using Image J software, as shown in Figure 5.2e and f, respectively. The calculated d spacing was 2.04 Å, which shows good agreement with the d spacing of the ferrite along the (100) plane. Similarly, the HAADF-STEM image and its corresponding d spacing calculation of the cementite are shown in Figure 5.2h and 2i, respectively. The observed d spacing was 2.13 Å, which can be interpreted as cementite.

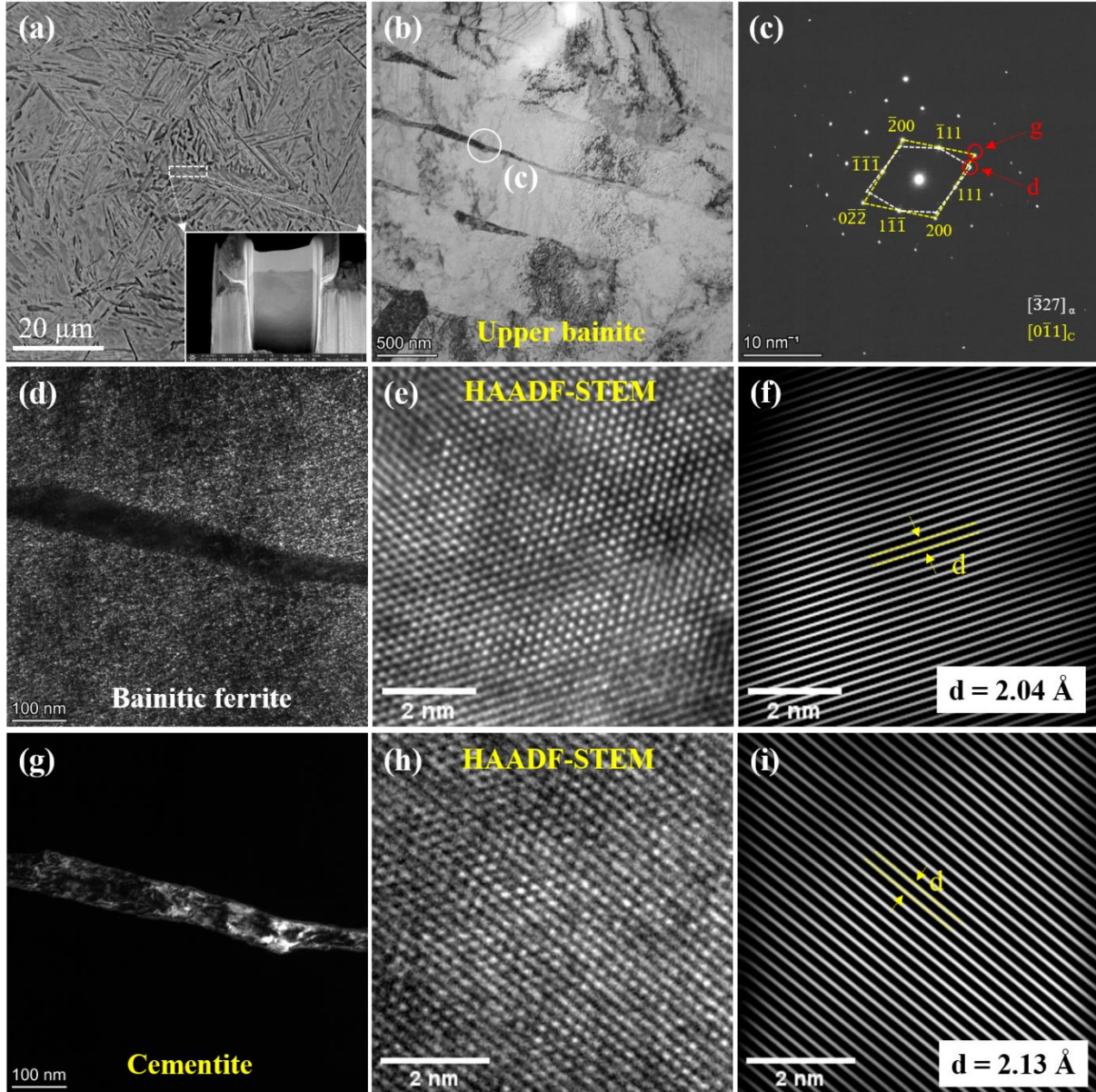


Figure 5.2: (a) FIB area and the sample (b) bright field image of the halo microstructure, (c) SAD pattern extracted from the white circle mentioned in (b), (d) dark field image of the bainitic ferrite by isolating the BCC reflection, (e) HAADF-STEM image of the ferrite in the  $[100]$  zone axis, (f) calculation of its  $d$  spacing, (g) dark field image of the cementite by isolating the corresponding reflection, (h) HAADF-STEM image of the cementite in the  $[\bar{3}27]$  zone axis, and (i) calculation of its  $d$  spacing.

Electron energy loss spectroscopy (EELS) map was employed to gain a deeper insight into the chemical nature of the halo ring microstructure. Figure 5.3a displays a representative TEM micrograph featuring the halo ring and the regions from which EELS maps were derived. EELS maps depicting the distribution of

carbon, iron, manganese, and silicon are presented in Figure 5.3b-e, respectively. These maps reveal higher carbon content in the cementite region, confirming the microstructure as upper bainite. Notably, no manganese and silicon were observed, given that the presence of silicon can impede cementite formation [150]. However, the fringe-like patterns are speculated to be stacking faults (SFs) adjacent to the cementite, denoted by white arrows in Figure 5.3f, which was confirmed by HAADF-STEM images (Figure 5.3g). SFs could form in the materials due to either deformation, the presence of crystal imperfections, or during high temperature process [151]. In this case, these SFs are believed to have formed during heating and cooling cycles, as well as due to the residual stresses and phase transformations during the welding process. In addition, it has been known [152] that decreasing carbon content reduces the stacking fault energy, making their formation more likely. On the other hand, carbon is an austenite stabilizer and the reduction in the carbon content increases the chance of austenite phase transformation upon cooling, promoting SF formation [152–154]. Surprisingly, it can be seen that SFs first are formed adjacent to the carbides at low-carbon regions and then extended into carbide particles. Carbides are deformed through the movement of SFs. It is speculated that these SFs are the result of the thermal stresses due to the ease of formation. Therefore, their existence in the microstructure at low carbon regions (areas nearer cementite) and within cementite is proof for the formation of an upper bainitic microstructure.

For an in-depth analysis of the internal structures, a Fast Fourier Transform (FFT) was extracted from the HAADF-STEM images and analyzed, as illustrated in Figure 5.3h. The FFT analysis confirms the presence of stacking faults containing a nano twin with slight atomic arrangement deviations ( $\sim 16^\circ$ ) of four layers, further supported by the inverse FFT analysis (see Figure 5.3i).

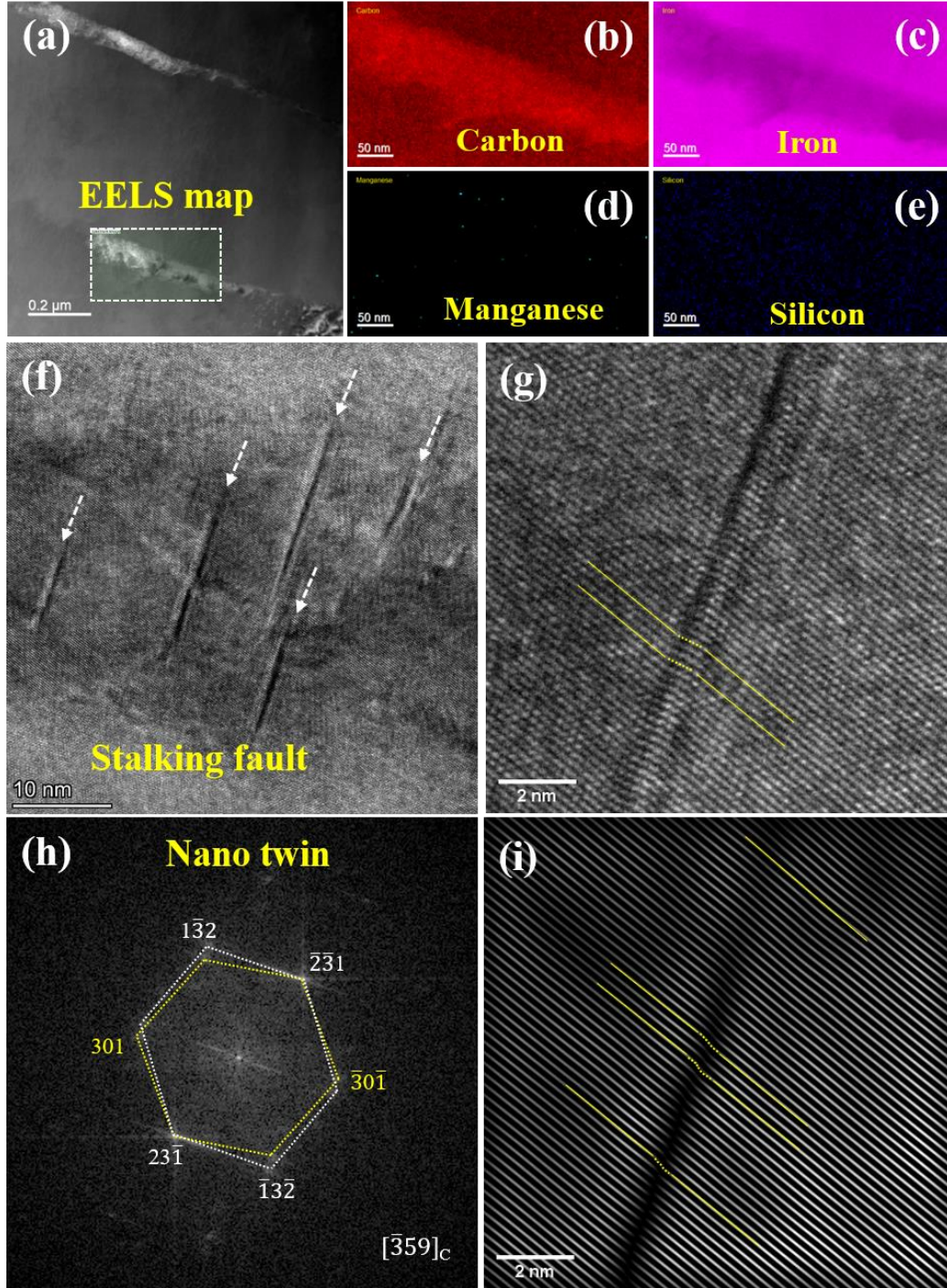


Figure 5.3: Electron energy loss spectroscopy analysis of the microstructure. (a) STEM image of the cementite, (b-e) carbon, iron, manganese, and silicon map, respectively, (f) presence of stalking faults pointed out by white arrows, (g) HAADF-STEM image of the cementite showing a stalking fault in the middle, (h) FFT extracted from the (g) corresponding to  $[\bar{3}59]_C$  zone axis, (i) inverse FFT showing the change in arrangement of planes.

#### 5.4.2. Mechanism Behind the Halo Formation

The microstructure identified in the carbon-depleted halo ring consists of the upper bainite. The schematic diagram in Figure 5.4 summarizes the halo formation mechanism and progression of bainite microstructure formation from the initial austenite. In case of short welding time conditions, upon cooling, the austenite next to the FB transforms into the lath martensite [15], as shown in Figure 5.4b. This diffusion-less transformation occurs when a medium or high-carbon steel undergoes resistance spot welding under a single-pulse welding current with a short welding time [140]. This can be associated with the rapid cooling rate during the RSW process following the welding period. Consequently, the time available for the interplay between heat generation and heat dissipation at the FB is inadequate to form the halo ring. In contrast, when RSW are made with a prolonged welding time (470 ms) at welding currents ( $\sim 0.1$  to  $0.3$  kA) less than expulsion current, nugget growth reaches its maximum (approximately around 200 ms). Subsequently, the following  $\sim 270$  ms, the FB experiences a cooling, which is dominated by heat dissipation through the electrodes. In other words, during later welding stages, after approximately 200 ms at  $I_{\max}$  current in this study [140], the FB temperature decreases below the steel's solidus temperature, during the welding time itself, contributing to the equiaxed grain structure [140]. The extent of this drop in temperature gradient towards the center of the fusion zone depends on the welding time. As a result, there is carbon diffusion from the FB to the molten metal during solidification, driven by the elevated activity at the FZ attributed to its higher temperature [146]. This, in turn, leads to the transformation of the low-carbon region in the FB into bainite, as illustrated in Figure 5.4c.

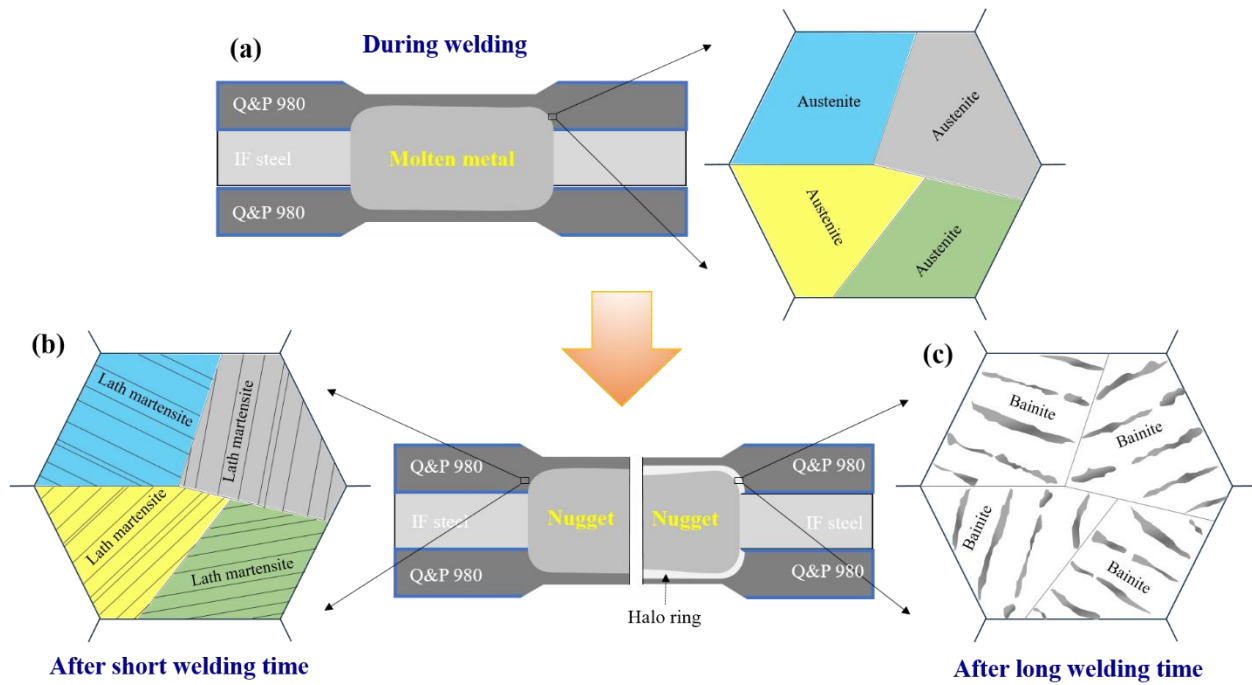


Figure 5.4: Schematic diagram showing (a) formation of austenite during the welding process, (b) transformation of austenite into martensite during cooling after a short welding time (c) transformation of austenite into bainite after a long welding time.

## 5.5. Summary

To summarize, a systematic TEM investigation disclosed that the microstructure within the halo ring is characterized by bainitic ferrite accompanied by needle-like cementite, specifically identified as upper bainite. This was further substantiated through high-resolution TEM (HRTEM) analysis, focusing on d-spacing and EELS mapping. Additionally, stacking faults and nano-twins were identified adjacent to the cementite, confirming the bainitic nature (two microstructural constituents) of the halo microstructure.

## Chapter 6. Effect of Paint Baking on Fusion Boundary

### Microstructure and Mechanical Properties <sup>4</sup>

#### 6.1. Overview

During automotive assembly, vehicles undergo low-temperature heat treatment (paint baking) to harden the paint. Although paint baking (PB) occurs at a relatively low temperature, it can remarkably affect the weld's mechanical and fracture behavior. Numerous studies have been conducted to analyse the PB response in resistance spot welds. It has been reported that the PB improves the mechanical properties of the spot welds by tempering of martensite and precipitation of  $\epsilon$  carbides. However, there were no studies focusing on the effect of PB on the transient softened zone at the fusion boundary. Although the metallurgical aspects of PB and its general hardening mechanisms are widely accepted, the micro- and macro-mechanical aspects of PB need further understanding. How PB alters the load-bearing capacity of the spot welds, work hardening behavior and the governing mechanisms have not been addressed systematically. Hence, this chapter, aims to provide a comprehensive understanding of the changes in the local mechanical properties in the UCHAZ of the Q&P 980 steel spot welds, before and after the paint baking treatment.

#### 6.2. Introduction

Post-welded heat treatments have proven to be an efficient method to modify the microstructure, and subsequently improve the mechanical properties of the spot welds. Betiku et al. [87,155] showed that the application of a post-welded tempering pulse in spot welded press-hardened steel (PHS) can increase weld

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<sup>4</sup> This chapter is composed of one published and one submitted research paper as outlined in the statement of contributions.

D.C. Ramachandran, O. Timothy Betiku, M. Shojaei, A. Salandari-Rabori, A.R. Midawi, J.-U. Kim, R. Bakhtiari, A. Macwan, E. Biro, Effect of paint baking on the fusion boundary softening and fracture behavior of Q&P 980 steel resistance spot welds, Mater. Charact. 206 (2023) 113383.

D.C. Ramachandran, A. Salandari-Rabori, A.R. Midawi, A. Macwan, E. Biro, Effect of paint baking treatment on mechanical properties of resistance spot welded Q&P 980 steel, ISIJ International (under review).

strength by 36%, compared to no post-heat treatment condition. The improvements in peak load were attributed to the transformation of martensitic lath structure into comparatively soft-tempered martensite structure with higher toughness. Eftekharimilani et al. [88] reported that the second current pulse partially anneals the solidified primary weld nugget by providing enough energy to redistribute the impurity element phosphorous, which changes the overall phosphorous distribution along the nugget boundary, improving the CTS performance. Work described in Chapter 3 confirmed that the improvement of mechanical properties of the double pulse welds (DPS) is related to the redistribution of alloying elements such as C, Mn, and Si in the fusion boundary, which could potentially eliminate the halo ring. Therefore, the manipulation of the microstructure via post-weld treatments is shown effective in enhancing the mechanical properties, while very limited works can be found dealing with such improvements in fracture behavior along with changes in the crack propagation path through the microstructure.

In automotive manufacturing, paint baking (PB), as a low-temperature heat treatment, is a mandatory process that is carried out to harden the automotive paint. This process is also considered to be similar to a low-temperature tempering process [156]. Eftekharimilani et al. [157] reported that applying the baking cycle on the bainitic AHSS significantly improved the CTS and absorbed energy. However, they found no significant effect on the failure modes of the spot welds after CTT. The improvements in the mechanical performance were attributed to the precipitation of  $\epsilon$ -carbides in the martensite, as the precipitation of  $\epsilon$ -carbide can improve the overall toughness, compared to the martensite [157]. Park et al. [54] investigated the effect of PB on spot welds made with medium Mn-TRIP and DP steels and showed that PB up to 210°C can improve the spot weld strength due to the nanoscale austenite reversion, softening the martensite. Furthermore, the segregation of Ti and Mo to prior-austenitic grain (PAG) boundaries led to a reduction in boundary cohesion, that decreased after the PB process. Shamsujjoha et al. [94] also observed an improvement in mechanical properties after the PB of 980 hot-formed (HF) steel. They theorized, evidence was not provided, that the improvement in CTS and toughness was due to the precipitation of carbides from the decomposition of martensite. Manladan et al. [158] reported that PB improved the energy absorption of

the spot welds by changing the fracture path from the halo ring to the nugget area. Although PB improved mechanical properties, they failed to investigate the fracture characteristics and crack propagation mechanisms before and after the PB treatment. However, in all these studies the PB effect is mainly discussed in the context of CTS improvement with few studies dealing with toughness changes. Accordingly, they are falling short of comprehensively investigating the effects of bake hardening on the macro and micro-fracture behavior of the AHSS spot welds.

Chapter 3 showed that the mechanical properties of these resistance spot welds can be significantly improved by applying a double pulse welding schedule by implementing a relatively high current for a short time after the primary pulse [159]. However, there were no comprehensive studies to evaluate the macro and microfracture mechanisms before and after PB treatment of spot welds. Hence, this chapter focuses on the effect of PB on the microstructure, texture, and fracture behavior of the Zn-coated 3rd generation Q&P 980 steel resistance spot-welds. Furthermore, the authors explore the crack propagation mechanism in more detail by isolating the microstructure of UCHAZ, away from the surrounding microstructure, by simulating the thermal history of this area of the HAZ using Gleeble thermo-mechanical simulation. By understanding these mechanisms, welding, and post-processing parameters may be modified in such a way as to eliminate the detrimental effects of the local microstructural features.

### **6.3. Experimental Procedure**

#### **6.3.1. Material**

This study examines an industrially produced Zn-coated 1.2 mm thick Q&P 980 steel, and the detailed description of the material is given at section 3.3.1.

#### **6.3.2. Resistance Spot Welding**

This study employed two different welding schedules: one is AWS-recommended single pulse welding (it will be named, AWS, hereafter) and the double pulse weld (DPS) as shown in Figure 6.1. Optimization of

these welding schedules and their effect on mechanical properties was already reported in our previous Chapter 3. The average nugget diameter of both conditions was kept constant at  $6.67 \pm 0.11$  mm.

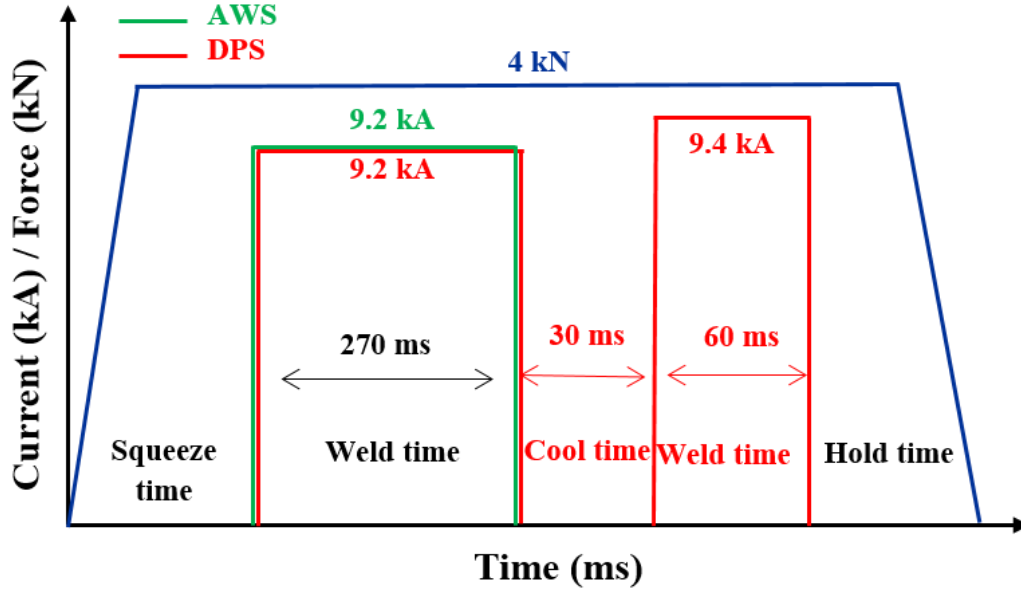


Figure 6.1: The welding schedules (AWS and DPS) used in this study.

### 6.3.3. Thermo-mechanical Simulation

Rapid tempering heat treatments were carried out to understand the mechanical properties of the UCHAZ in the as-welded and paint-baked conditions (e.g., AWS and AWS-PB samples, respectively). The UCHAZ was simulated (G-AWS: Gleeble simulation of the sample welded by AWS-recommended single pulse condition) with a Gleeble thermo-mechanical simulator. The thermal cycle used for the heat treatment and the dimensions of the test coupon is illustrated in Figure. 6.2. To validate the ability of the heat treatment to reproduce the microstructure and mechanical properties of the UCHAZ in the weld, the hardness and grain size of the Gleeble simulated structure were assessed and matched to those of the weld (AWS sample). More details about the Gleeble simulations and discrepancies in the target and sample temperature were reported elsewhere [160]. The specimens were then machined into dog-bone-shaped samples for mechanical testing; the dimensions are shown in Figure. 6.2b.

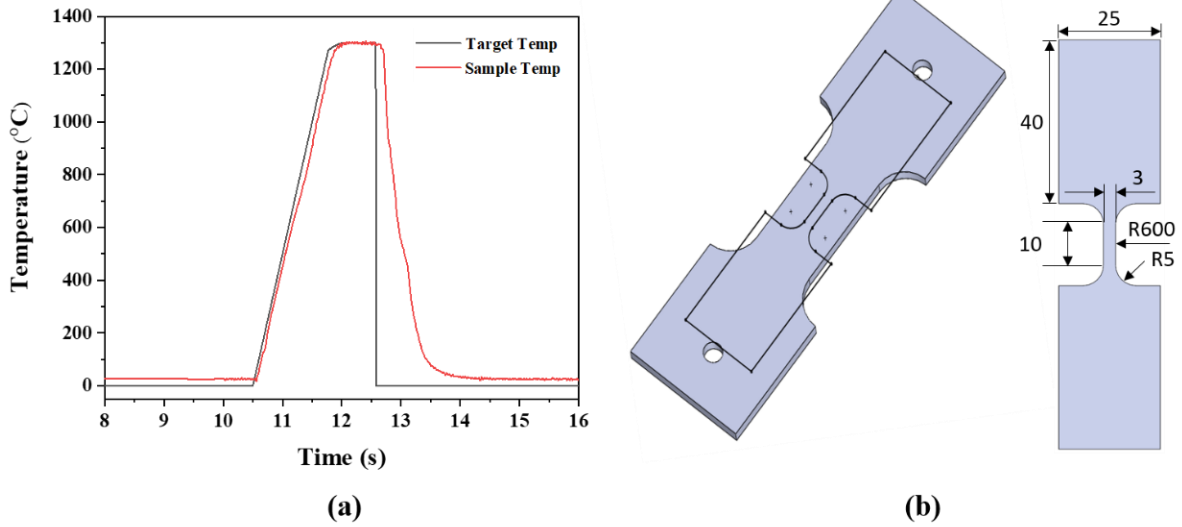


Figure. 6.2: (a) Gleeble simulation curve and (b) sample geometry before and after machining.

#### 6.3.4. Paint Baking Treatment.

During automotive production, the car body will undergo a baking treatment in an oven to process the paint. To simulate those conditions in the laboratory, the welded samples were heat-treated in a muffle furnace at a temperature of 180°C for 27 min. Five samples were heat treated per welding condition to be used for mechanical testing and microstructural analysis. PB treatments were also carried out in the Gleeble samples (G-AWS PB) with the same heat treatment condition, as mentioned above.

#### 6.3.5. Uniaxial Tensile Test

The uniaxial tensile tests (UTT) were conducted on the Gleeble samples to understand the mechanical properties of the samples after PB. The tests were conducted in a Shimadzu universal tensile frame with a strain rate of  $1 \times 10^{-3} \text{ s}^{-1}$  ( $\sim 0.54 \text{ mm.min}^{-1}$ ) and elongation was measured with coupled digital image correlation (DIC) technique using a 10 mm virtual gauge length.

#### 6.3.6. Microstructural Analysis

Standard metallographic techniques were used to prepare thin foils of TEM samples followed by electro-polishing. The 3 mm discs were thinned using a TenuPol-5 twin jet electro-polishing equipment in a

solution of 10% Perchloric acid and 90% Methanol maintained at -40°C, at a voltage of 15 V. A Talos 200 TEM at an accelerating voltage of 200 keV were used to analyze the samples. The mapping setup varies with the thickness of the sample and the spot size of the scanning-transmission electron microscopy (STEM) beam, as the intensity of the X-ray depends on the thickness and the beam current. Hence, the dwell time was maximized to obtain more accurate chemical distribution maps.

#### 6.3.7. Instrumented Indentations

Instrumented indentation testing was performed on a Nanovea-M1 indentation tester. Metallographic sample polished up to 1µm diamond suspension were used for the test. To estimate the yield strength, a nearly flat tip indenter with a diameter of 50 µm was used by following the procedure suggested by Midawi et al.[161] The yield strength of the weld before and after paint baking was calculated by Equation 6.1 as suggested by Midawi et al.[161].

$$\sigma_y = \frac{F_c}{2.2\pi a^2} \quad 6.1$$

where  $\sigma_y$  is the yield strength,  $F_c$  is the force, and  $a$  is the radius of the indenter.

### 6.4. Results

The Q&P steel coupons were initially welded according to the AWS D8.9 recommendations [45], which had a single pulse current. In previous chapter 3, a double-pulse welding schedule was designed to improve the mechanical properties. Furthermore, the welds were tested using the CTT, and the resulting failure was observed in the cross-sectional view. It was shown that the AWS sample failed along the fusion boundary, resulting in a low-strength failure. The fusion boundary failure path, the halo ring, had a lower hardness. In what follows, the effect of paint baking on the mechanical properties, fracture behavior, and microstructural evolution will be presented.

#### 6.4.1. Mechanical Properties

The CTT data and its representative load-displacement curves for AWS, DPS, AWS-PB, and DPS-PB samples are shown in Figure 6.3a. The absorbed energy (area under the curve up to peak load) is representative of the crashworthiness of the weld, and is shown in Figure 6.3b.

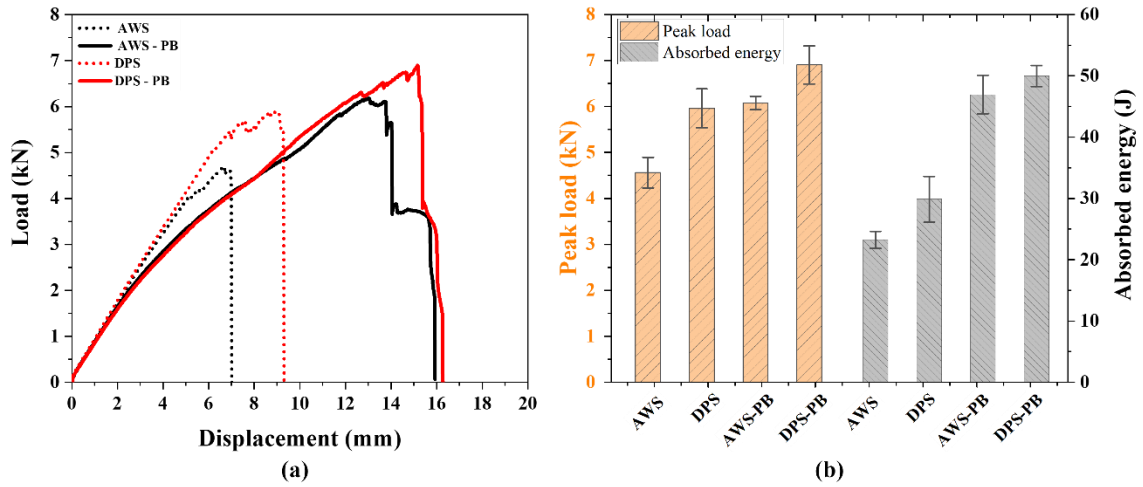


Figure 6.3: (a) Load-displacement curves of CTT test and (b) absorbed energy up to peak load of all experimented conditions.

As shown, a notable trend can be seen where a tangible increase in peak load and absorbed energy can be realized by applying PB on both AWS and DPS samples. The average mechanical properties (CTS data) and its absorbed energy up to the peak load are summarized in Table 6.1. The highest CTS performance and absorbed energy are achieved by the DPS-PB sample with an average value of  $6.9 \pm 0.4$  kN and  $50.0 \pm 1.7$  J, respectively. So, it can be clearly understood that the AWS-PB sample shows more significant improvements in properties than the AWS sample. The increase in strength and absorbed energy after PB is 34% and 102%, respectively. Moreover, there is a significant difference in the average strength (6.1 to 6.9 kN) between AWS PB and DPS PB samples (see Table 6.1).

*Table 6.1: Cross-tension test data and failure mode of the spot-welds.*

| Samples | Peak load (kN) | Absorbed energy (J) | Failure mode (AWS D8.1) [78] |
|---------|----------------|---------------------|------------------------------|
| AWS     | $4.6 \pm 0.3$  | $23.2 \pm 1.4$      | Mode 3                       |
| DPS     | $5.9 \pm 0.4$  | $29.9 \pm 3.7$      | Mode 1                       |
| AWS-PB  | $6.1 \pm 0.2$  | $46.9 \pm 3.1$      | Mode 1                       |
| DPS-PB  | $6.9 \pm 0.4$  | $50.0 \pm 1.7$      | Mode 1                       |

Representative failure areas and their corresponding cross-sectional micrographs of different welds are shown in Figure 6.4. A distinct change in failure modes between the as-welded (AWS and DPS samples) and as-welded and then paint-baked (AWS-PB and DPS-PB) samples can be observed after the CTT. AWS sample (welded according to AWS D8.1 [78]) shows partial thickness failure mode (known as Mode 3); in which the failure happens along the fusion boundary (see Figure 6.4a). However, DPS, AWS-PB, and DPS-PB samples all show pull-out fractures through the UCHAZ (see Figure 6.4b-d). Recently, Manladan et al. [158] reported a change in the failure mode from Mode 3 to Mode 2 after the PB treatment of 30MnB5 spot weld. However, no studies are reporting the changes in the failure mode from Mode 3 to Mode 1 due to the PB treatment. According to the literature, the pull-out failure mode exhibits the most satisfactory failure mode in the spot welds compared to the interfacial failure mode since it is associated with higher plastic deformation and absorbed energy before the fracture [94]. Thus, the application of PB after welding significantly eliminated the strength and toughness difference between the AWS and DPS conditions since both conditions (AWS-PB and DPS-PB samples) failed in a button pull-out mode, where the fracture path progressed through the UCHAZ. On the other hand, the PB treatment does not change the failure mode of the DPS sample after CTT. Furthermore, the DPS schedule eliminates the microstructural inhomogeneity of the fusion boundary by reheating the fusion boundary with the short second current pulse. This second current pulse homogenizes the microstructure near the fusion boundary, which mitigates the effect of the halo ring. A detailed study on the effect of DPS on microstructure and mechanical properties is reported elsewhere [162].

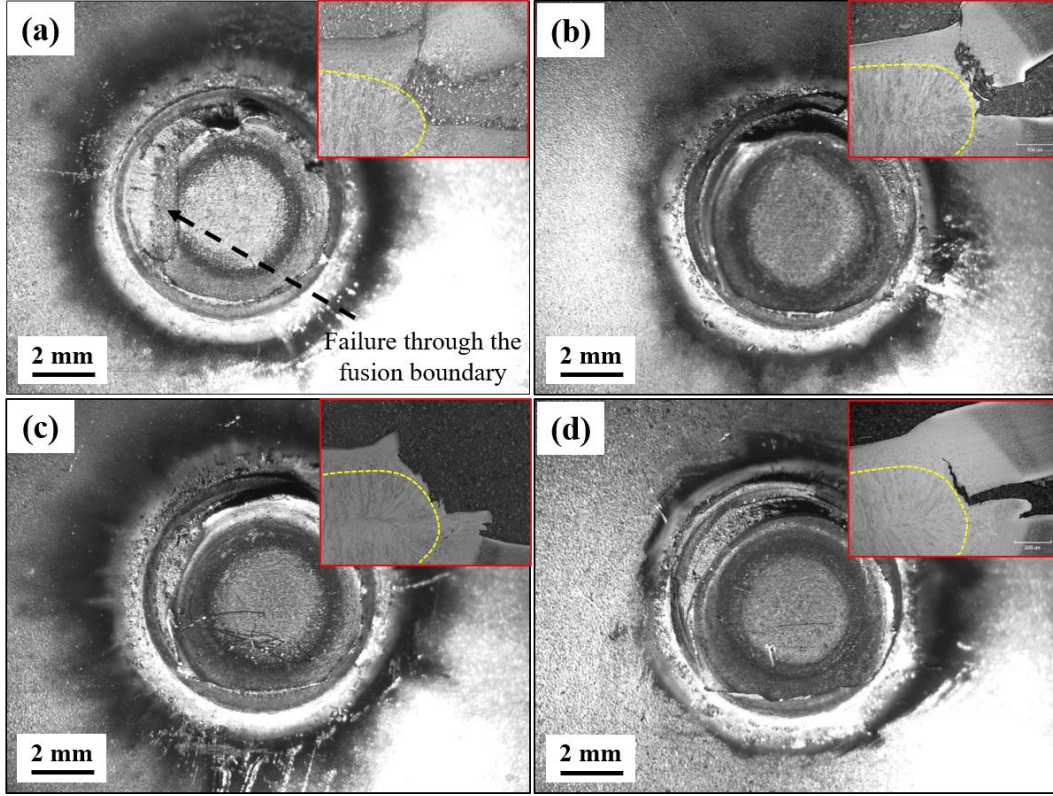


Figure 6.4: Representative failure modes and their corresponding cross-sectional micrographs of samples (a) AWS (Mode 3), (b) AWS-PB (Mode 1), (c) DPS (Mode 1), and (d) DPS-PB (Mode 1).

Often in a simplified form, changes in mechanical properties (e.g., yield strength) can be measured as changes in hardness. From the microhardness profile of the studied weld samples (according to the SEP 1220 [163]), the average nugget microhardness of the AWS and AWS-PB samples are  $541 \pm 5.5$  and  $506 \pm 4.0$   $HV_{0.1}$ , respectively. The average nugget hardness of the DPS and DPS-PB is reduced compared to the AWS and AWS-PB conditions and are  $518 \pm 5.0$ , and  $495 \pm 5.5$   $HV_{0.1}$ , respectively (see Figure 6.5). The average microhardness of the heat-affected zone for AWS and AWS-PB samples are  $532 \pm 11.0$  and  $530 \pm 7.5$   $HV_{0.1}$ , respectively, and are  $544 \pm 8.5$  and  $516 \pm 9.5$   $HV_{0.1}$  for DPS and DPS-PB samples, respectively. The error bars of the microhardness measurements represent the 95% confidence interval. From the hardness profile, it may be seen that the average hardness decreases after PB. A decrease in hardness is typically associated with a decrease in material strength. Therefore, it is believed that the change in hardness resulting from PB

is due to the microstructural changes which can thereby lead to a different joint response during tensile testing, as well. Detailed discussions on the material's tensile strength will be addressed in the later sections.

Here, it should be noted that the PB also dramatically mitigates the halo ring, formed in the AWS sample. After PB, the average hardness of the region representing the halo ring increased from 417 to 447 HV<sub>0.1</sub>. Moreover, the difference in hardness between the halo ring and surrounding microstructure (nugget and HAZ) decreased significantly, which reduces the possibility of stress concentration in the halo ring. For the DPS sample, however, the halo ring is eradicated and further exposure of samples to the the PB treatment (DPS-PB) reduces the hardness differences at the fusion boundary to a higher extent (Figure 6.5b).

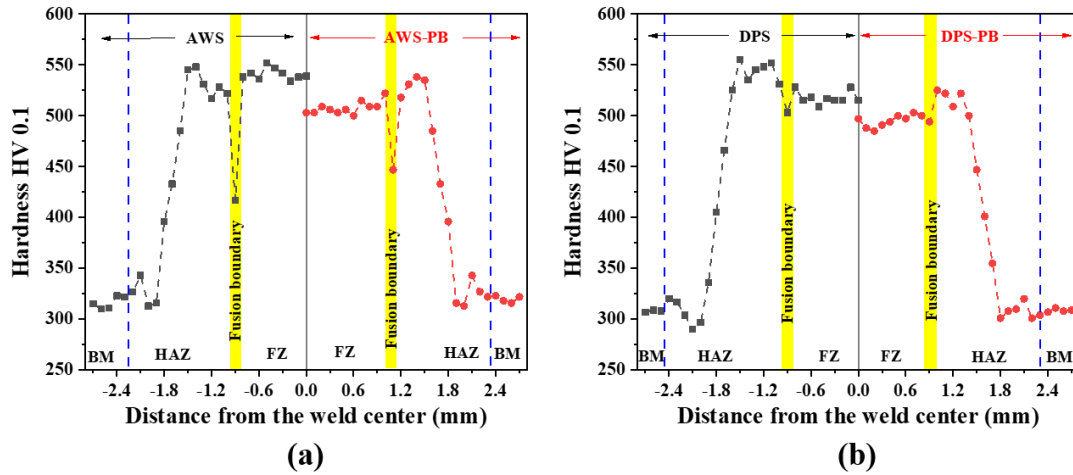


Figure 6.5: Hardness profile of the samples (a) AWS and AWS-PB samples, (b) DPS and DPS-PB samples (where halo rings are not formed).

To further substantiate the results of CTT and microhardness tests, the UCHAZ, where the failure happens in the spot-welded samples, was simulated using Gleeble. Then, uniaxial tensile tests were performed on the Gleeble samples, namely, G-AWS and G-AWS PB (performing PB on the simulated UCHAZ Gleeble sample). Engineering stress-strain curves are shown in Figure 6.6. As can be seen, after PB the ultimate tensile strength (UTS) and the elongation to fracture are noticeably improved by 7.6 % and 833%, respectively. Furthermore, the area under the stress-strain curve, up to the fracture, represents the toughness

which is dramatically larger in the case of the PB. The G-AWS sample failed near 1.1% strain, whereas the G-AWS PB sample can accommodate the strain up to 11%. The absorbed energy of G-AWS and G-AWS PB samples is 9.4, and 151.0 J/m<sup>3</sup>, respectively. It is also clear from the DIC images that the G-AWS sample experienced a brittle behavior as compared to the G-AWS PB sample, which shows a necking behavior implying a higher amount of energy absorbed during the tensile loading. Here, it is worth mentioning that the yield strength (YS) is slightly lower for the G-AWS PB sample, compared to the G-AWS sample (1161 vs. 1278 MPa) which agrees with the hardness measurements, as well. Tensile mechanical properties are shown in Table 6.2. To understand the reason behind the fracture, fracture surface analysis on the actual spot welds and the Gleeble samples are thoroughly investigated, and the results are summarised in the following section.

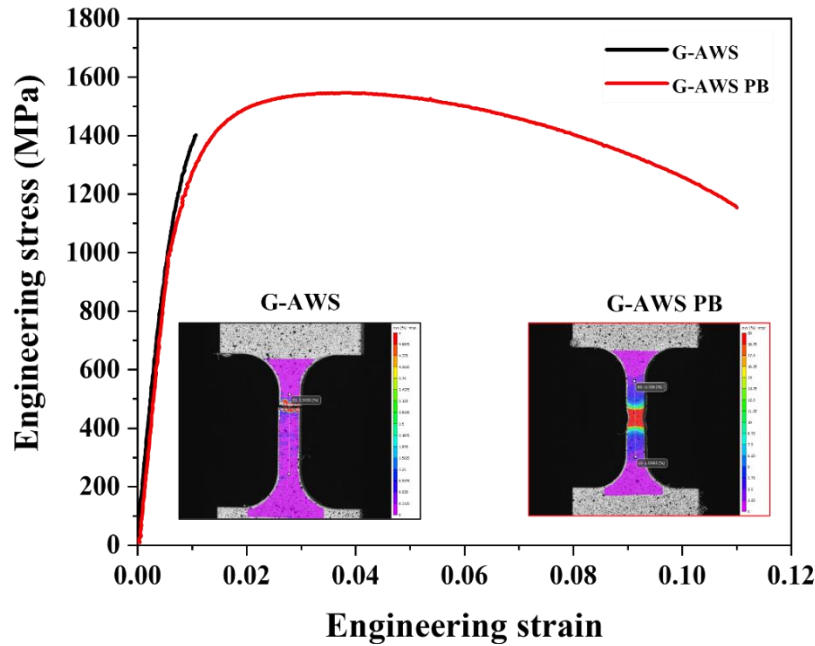


Figure 6.6: Tensile engineering stress-strain curves of G-AWS and G-AWS PB samples.

Table 6.2: Mechanical properties of the G-AWS and G-AWS PB samples after uniaxial tensile test.

| Samples  | YS (MPa)    | UTS (MPa)   | Absorbed energy (J.m <sup>-3</sup> ) |
|----------|-------------|-------------|--------------------------------------|
| G-AWS    | 1278 ± 2.8  | 1414 ± 81.4 | 9.4                                  |
| G-AWS PB | 1161 ± 19.6 | 1521 ± 20.0 | 151.0                                |

#### 6.4.2. Fracture Behavior

Interpretation of the failure locations in the spot welds is important to understand the underlying fracture mechanisms. Optical micrographs of the weld cross-sections (showing the hardness indents) for the AWS and AWS-PB samples are shown in Figure 6.7a and b, respectively.

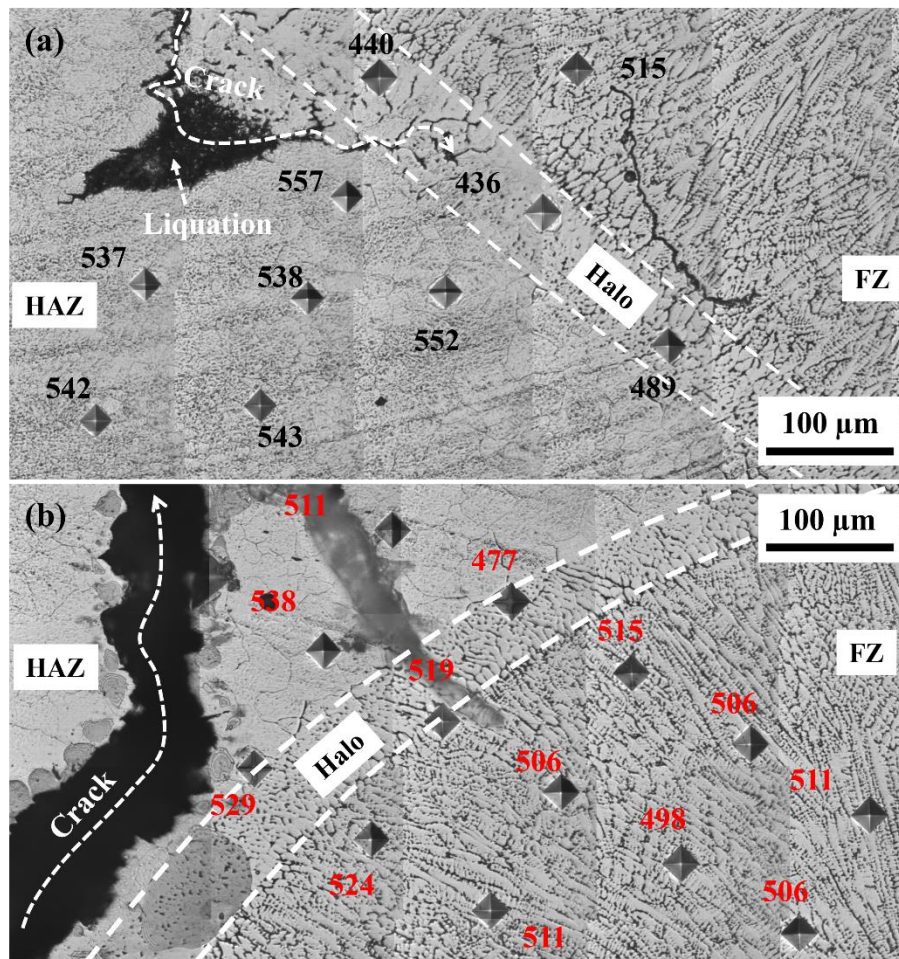


Figure 6.7: OM cross-section of the fracture surface and detailed hardness values of (a) AWS and (b) AWS-PB samples (all the hardness measurements are in HV<sub>0.2</sub>).

It can be seen that the fracture occurs through the fusion boundary (halo) and propagates through the solidified liquation (mentioned in the Figure 6.7a) region in the AWS sample (see Figure 6.7a). Nonetheless, the crack path deviates to UCHAZ in the sample subjected to the PB treatment, and solidified liquation product is not seen in that cross-section (see Figure 6.7b). Furthermore, the decrease in hardness

along the fusion boundary, compared to the FZ and HAZ is clear in Figure 6.7a. It is also worth noting that existence of a high hardness region around the halo (higher hardness of FZ and UCHAZ/HAZ than the halo), makes a sharp hardness gradient across the fusion boundary of the spot weld, which promotes a low energy failure path or stress concentration region during the mechanical testing. However, after PB, this gradient in the hardness from FZ to the HAZ is mitigated as shown in Figure 6.7b. In other words, due to a sharper reduction in the hardness of the HAZ side of the fusion boundary, an increase in the hardness at the fusion boundary and a slight reduction in the hardness of the FZ (compared to the AWS sample), the sharp hardness gradient significantly reduced (Figure 6.7b).

The fracture surfaces of the AWS and AWS-PB samples are shown in Figure 6.8. As shown in Figure 6.8a, the AWS sample fractured in a brittle manner, and showed evidence of intergranular fracture (Figure 6.8c) near the notch, and cleavage fracture (Figure 6.8b) throughout the other areas. However, the fracture surface of the AWS-PB sample shows an overall ductile mode of fracture (Figure 6.8d). Although the fracture initiated near the fusion boundary (close to the sheet-to-sheet interface) was like the AWS sample, the crack grew predominantly in a ductile manner (Figure 6.8e). Here, a clear change in the fracture macro-mechanism can be seen from an intergranular to the trans-granular propagation of the crack. This led to a change from a brittle to a ductile fracture, as shown by dimples on the fracture surface of the AWS-PB sample (Figure 6.8e). Moreover, it can be noted that this observation is in good agreement with the reported mechanical properties since the change from cleavage to dimple fracture provides significant improvements in strength and energy absorption capacity, as presented in section 3.1.

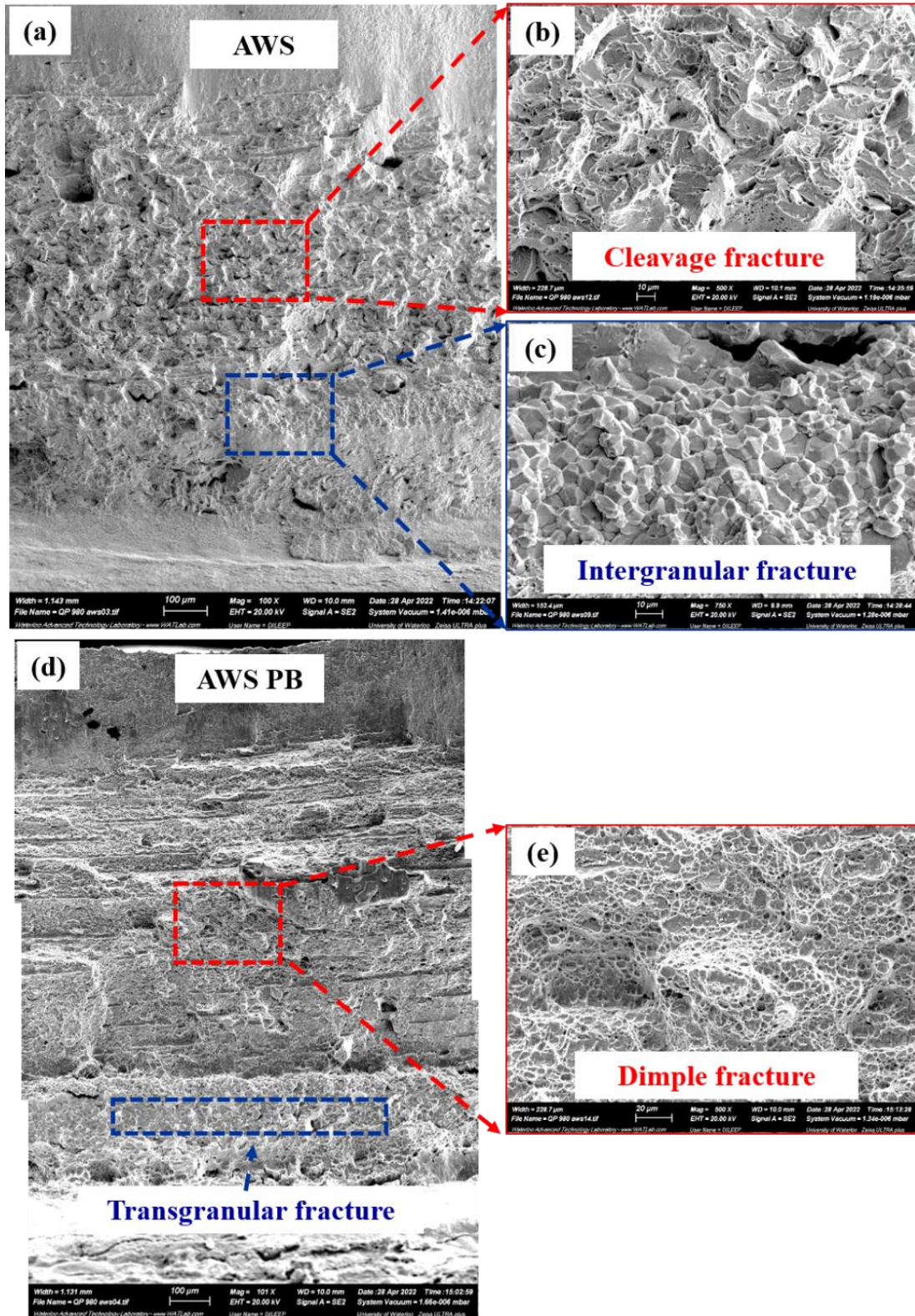


Figure 6.8: SEM micrographs showing fracture surface of (a) AWS sample showing (b) cleavage and (c) intergranular fractures and (d) AWS-PB sample with (e) ductile fracture.

The scanning electron micrographs of the fractured sample (cross-sectional view) for the AWS-PB sample after the CTT are shown in Figure 6.9. The plastic deformation occurs in the microstructure near the crack propagation area (Figure 6.9a). Close observation depicts that the microstructure present in that region is the partially disintegrated martensite (Figure 6.9a). This partially disintegrated martensite can absorb comparatively more energy during deformation; thereby increasing the energy required to propagate the crack. A typical example of partially disintegrated martensite is also shown in Figure 6.9b.

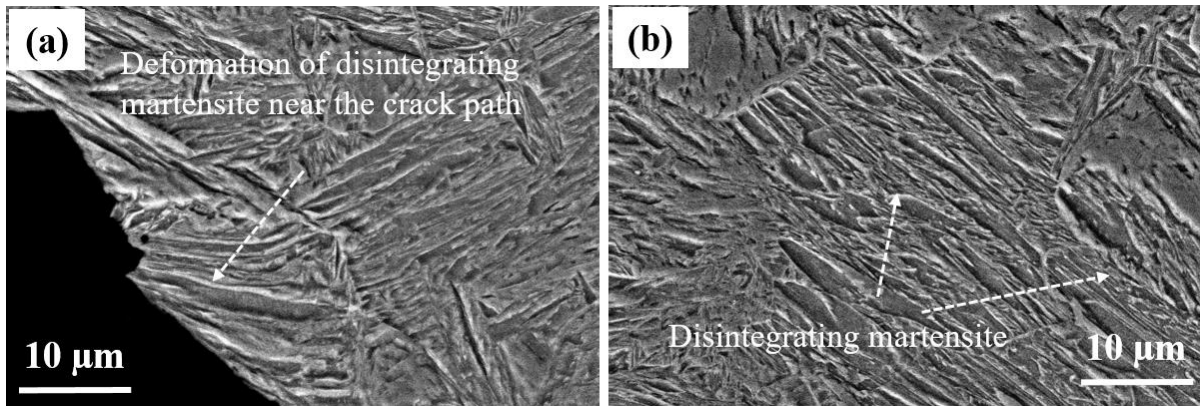


*Figure 6.9: SEM micrograph showing the (a) deformation of disintegrated martensite near the crack path, and (b) typical disintegrating martensite, in the AWS-PB sample after CTT.*

To understand the difference in fracture behavior of the UCHAZ region in AWS and AWS-PB samples (where the failure happens) simulated via Gleeble (G-AWS and G-AWS PB samples), fractographic analyses were conducted after the tensile tests. The fracture surfaces of both G-AWS and G-AWS PB are compared in Figure 6.10. As seen in Figure 6.10a, in G-AWS samples also the crack propagation proceeds along the PAGBs with an intergranular type of fracture (in agreement with the AWS sample, see Figure 6.8c). Moreover, limited trans-granular cleavage fracture characteristics can be also seen in Figure 6.10a. It could be due to the presence of different types of grain boundaries with different energies. On the other hand, the specimens after the PB (G-AWS PB) show a relatively ductile fracture behavior with dimples (in line with observations in Figure 6.8e). More importantly, Figure 6.10b shows the presence of solidified segregations along the grain boundaries, pointed by arrows.

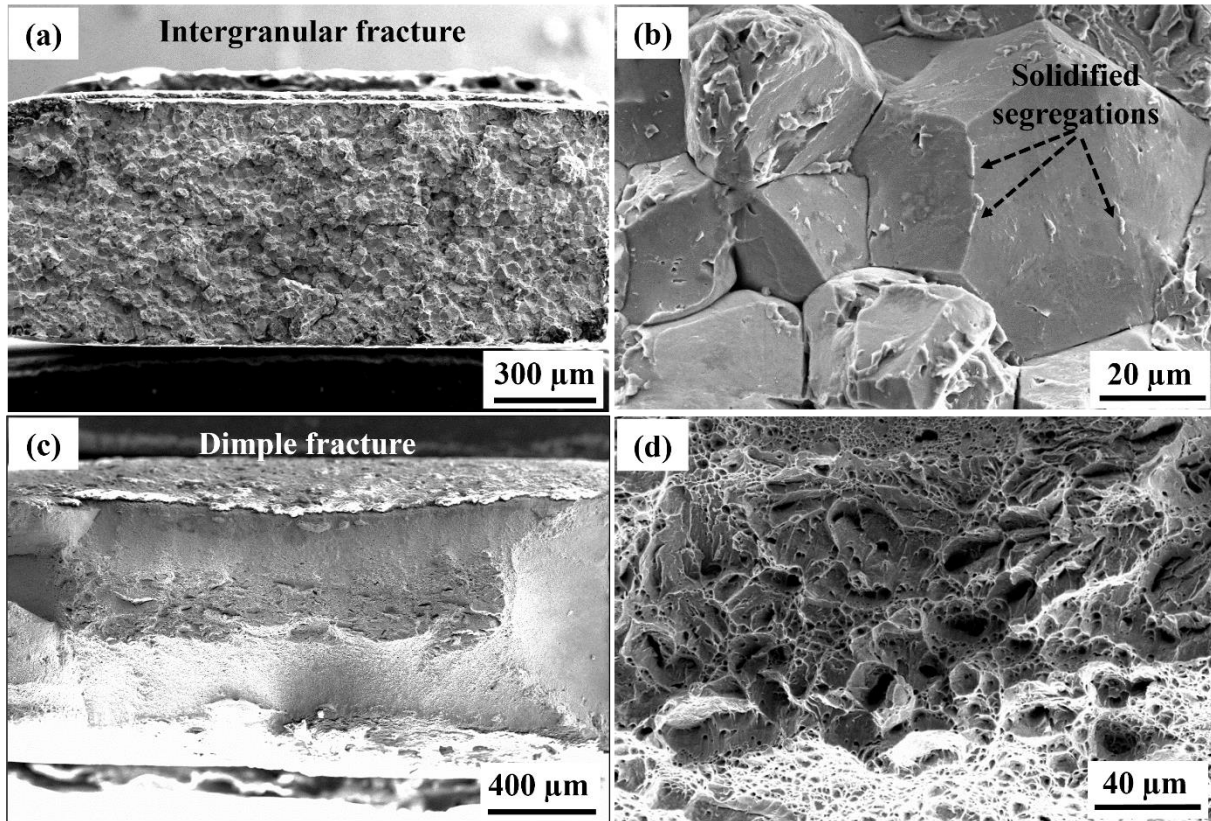


*Figure 6.10: SEM fractography micrographs of the tensile samples (a and b) G-AWS showing Intergranular-brittle fracture, (c and d) G-AWS PB showing dimple fracture.*

Figure 6.11 shows the difference in fracture behaviour from the crack beneath the surface (secondary crack analysis). Figure 6.11a shows more evidence of intergranular fracture in the G-AWS sample. The PAGBs are marked in yellow color. On the contrary, the G-AWS PB sample shows a trans-granular failure with deformed regions on the microstructures (see Figure 6.11b). These observations agree with Figure 6.10. EBSD IQ and IPF maps (see Figure 6.11c and d) on the G-AWS sample show a typical secondary crack propagating through the PAGB (an intergranular mechanism). It can be summarized that the resistance spot weld and its corresponding Gleeble samples are in acceptable agreement in terms of mechanical properties and fracture characteristics. Overall, the G-AWS sample shows brittle fracture behaviour where the crack is propagating through the grain boundaries, whereas a transition to ductile behavior is seen that is governing the fracture surface of the G-AWS PB sample with a trans-granular crack propagation mechanism.

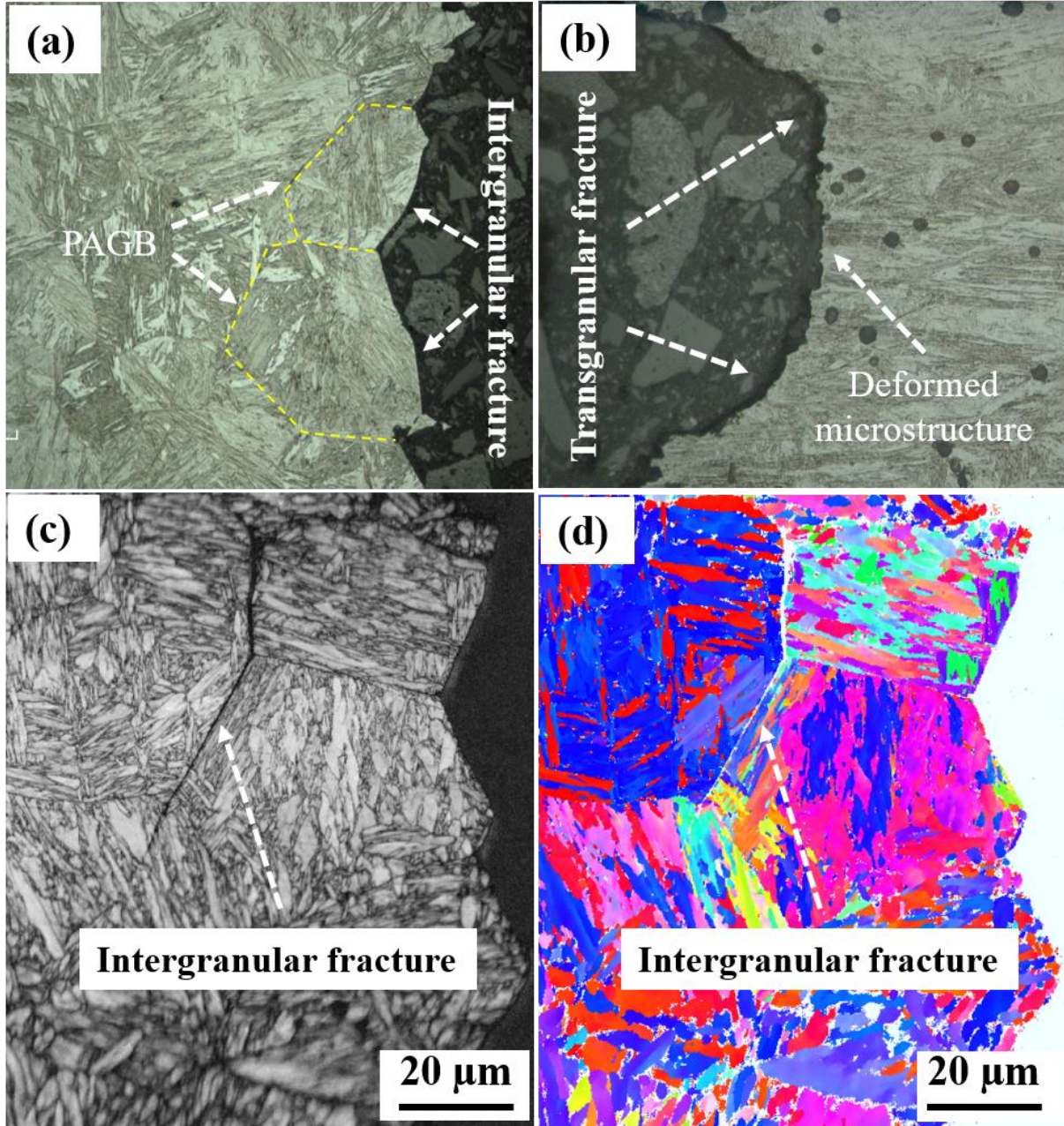


Figure 6.11: Secondary crack analysis of the samples showing (a and b) OM of the crack beneath the fracture surface of G-AWS and G-AWS PB, respectively, and (c and d) EBSD IQ and IPF map showing the intergranular fracture in G-AWS sample.

#### 6.4.3. Microstructural Inhomogeneities

In the previous sections, the mechanical and micro- (fractography) and macro-fracture (crack propagation) behavior of welded samples were assessed before and after PB. In the following, the differences in fracture

behavior will be discussed in terms of microstructural evolution. The OM & SEM microstructures of the AWS sample, which failed in an intergranular fashion, are shown in Figure 6.12. As seen in Figure 6.12a, there is solidified liquation product seen in this weld. The presence of liquation in the spot-welded Q&P 980 steels has been reported before [68]. This phenomenon is attributed to the segregation of Mn, Si, and P, at grain boundaries [164]. Alizadeh-Sh and Marashi [165] observed the liquation during the dissimilar RSW of austenitic/duplex stainless steel. They proposed a grain boundary sweeping mechanism for grain boundary liquation. This mechanism proposes that upon heating, grain boundaries sweep the solute and/or impurity atoms as the grain growth proceeds. Elements that have high surface activity and/or exhibit low solubility in the matrix would have the highest affinity for being swept into and assimilated with grain boundaries. Consequently, during the HAZ grain growth certain solute atoms, Al in their case, assimilated with moving boundaries. Afterwards, these solute atoms acted as melting point depressant elements and preferential melting occurred along the grain boundaries during the HAZ thermal cycle [165].

The same grain boundary liquation mechanism is active here during the RSW. Evidence of liquation can be seen near the fracture path of the AWS sample, as shown in Figure 6.12b. Evidence of small cracks near the fracture path can also be seen in Figure 6.12c, where the crack propagates through grain boundaries. As shown in Figure 6.12d, the EDS elemental analysis across the fracture shows the segregation of Mn and Si, with an average composition of 4.6 and 2.3 wt.%, respectively. Saha et al. [166] pointed out that the liquation is formed in alloys of Al, Mg, and Fe, where a comparatively larger window of solidus and liquidus temperatures exist; therefore, these alloys will exhibit a wider solidification range. While solidifying, the liquid film that had a comparatively lower solidus temperature, will remain in the liquid state along the grain boundaries (liquation). This will lead to the deterioration of inter-grain bonding. It should be worth noting that, the liquation present in the AWS sample solidifies between the grain boundaries, providing a crack-free bond between the grains. This solidified liquation product can further initiate cracking during post-weld loading since the liquation regions are low-strength regions due to the presence of alloying elements, susceptible to stress concentration [68]. According to Li u et al. [68] and

Jing et al. [121], the formation of liquation cracks was due to phosphorous segregation to grain boundaries during the welding. However, in the current study, segregation of phosphorous in this region was not observed, only Mn and Si. Still, cracks formed during CTT originating from the solidified liquation product, as may be seen in the AWS sample (Figure 6.12a).

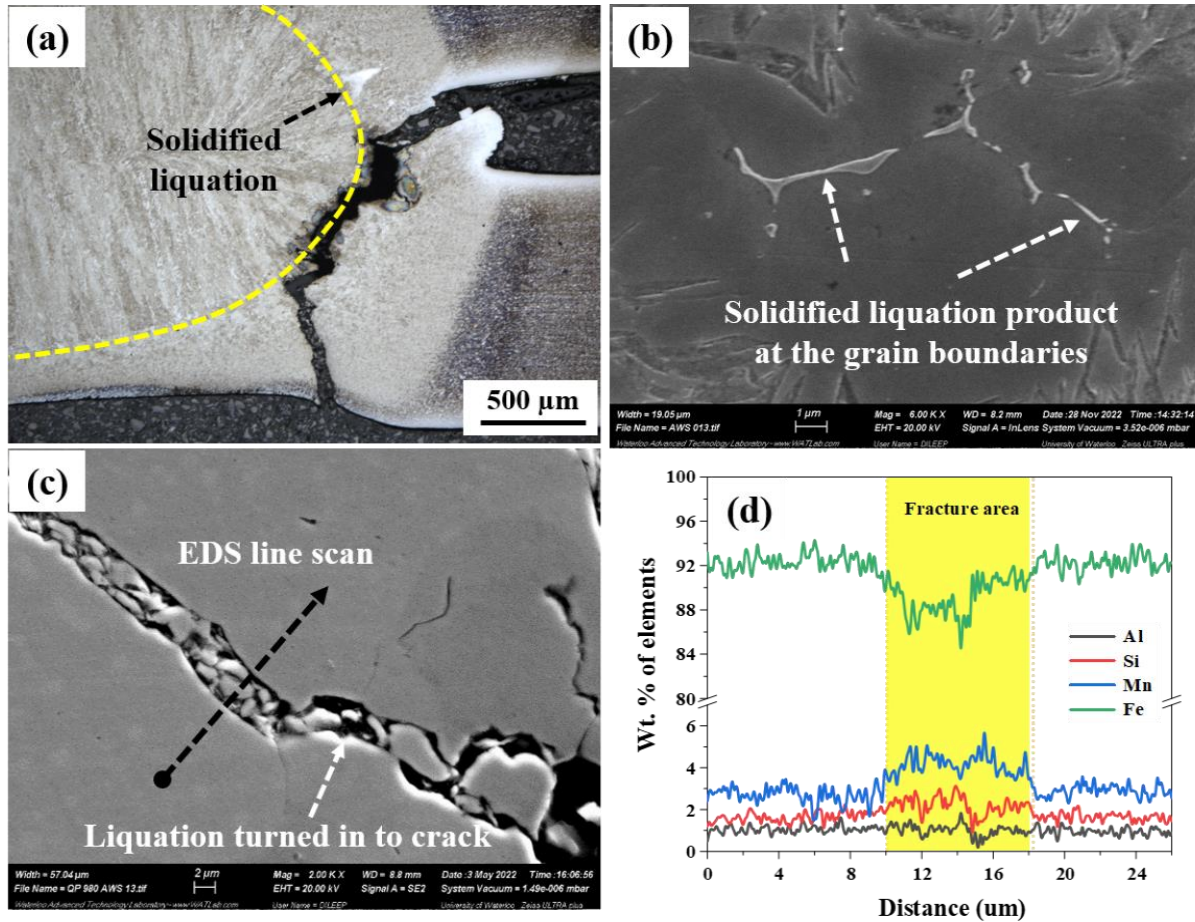


Figure 6.12: Microstructure and elemental analysis of the solidified liquation product present in the AWS sample after the CTT: (a) OM micrograph showing the fracture path and solidified liquation product, (b) SEM micrograph of a typical solidified liquation product at grain boundary after fracture, and (c and d) representative SEM image and the EDS line scan across a solidified liquation, which transformed into a crack during the CTT.

Detailed EBSD analysis of the solidified liquation in the AWS sample can be seen in Figure 6.13. Figure 6.13a shows the IQ map of the weld with FZ, halo ring, and HAZ with a typical solidified liquation product.

The width of the solidified liquation near the fusion boundary is approximately 90  $\mu\text{m}$ . The IPF map shows the presence of smaller grains compared to the surrounding microstructure, in the solidified liquation region (see Figure 6.13b). The phase map shows the presence of austenite grains at the edge of this solidified liquation region (Figure 6.13c). Combining the EDS line scans, and the presence of austenite, it can be interpreted that the austenite grains were stabilized to room temperature due to the presence of high Mn content at the tip of the solidified liquation, which greatly improves the stability of austenite at the lower temperature. As mentioned above, the presence of Mn, and Si along the grain boundaries can pin the movement of grain boundaries, which could be the reason for the presence of smaller grains in the solidified liquation region. During the application of the load, the solidified liquation product can also initiate some micro-cracks, since the lattice mismatch due to the crystallography between the austenite and martensite grain causes stress concentration in its common boundaries [128,129]. It is worth noting that the solidified liquation was not observed in the AWS-PB sample (solidified liquation exists after welding was disappeared after PB treatment). It is believed that PB treatment enhances atomic mobility and grain growth. Heating at 180°C for 27 minutes provides significant time for the solidified liquation product to diffuse out of the grain boundaries into the grain interiors. In other words, Mn, and Si trapped at the grain boundaries can redistribute into the grains by diffusion during heat treatment. Moreover, the solidified liquation also can be consumed by the surrounding grains during the grain growth. These two metallurgical phenomena can eliminate the intergranular planes of weakness (liquation) in the AWS-PB samples. Since, these weak liquation regions, can act as crack initiation sources during post-weld loading, their elimination by the PB process will noticeably improve energy absorption and fracture behaviour.

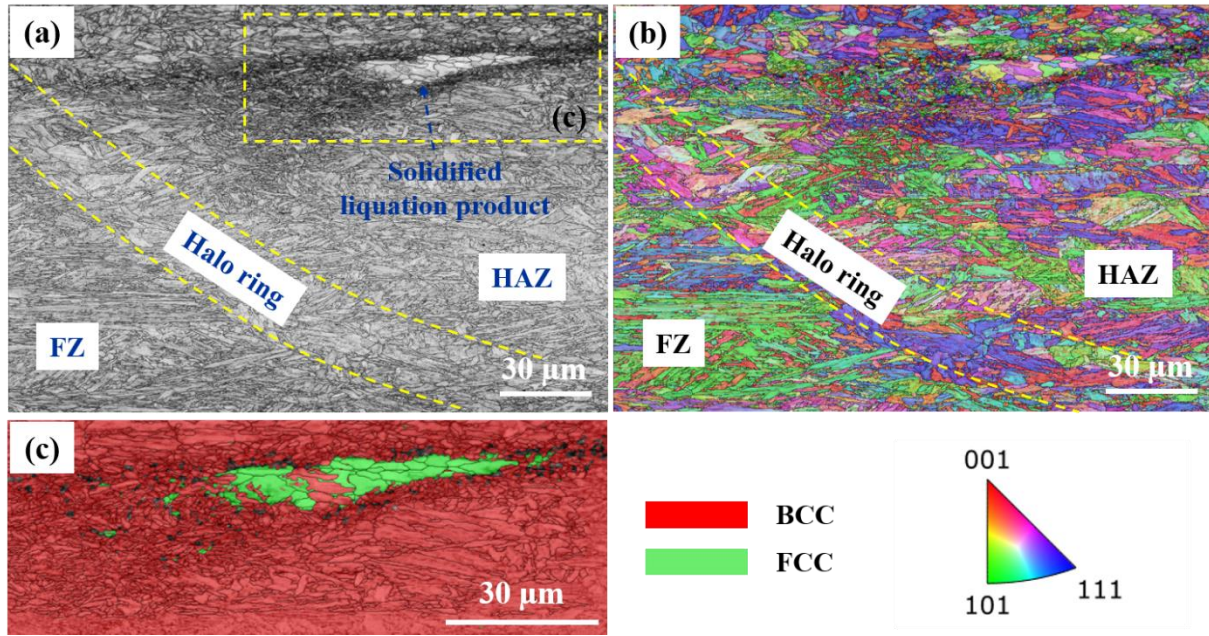


Figure 6.13: Characterization of solidified liquation product in AWS condition (a) IQ map, (b) IPF map, (c) phase map showing austenite in the solidified liquation.

Detailed microstructural analysis of the G-AWS and G-AWS PB samples are shown in Figure 6.14. The IQ and corresponding IPF maps of the G-AWS sample are described in Figure 6.14a and b. The martensitic microstructure is present throughout the matrix for the G-AWS sample (Figure 6.14a). However, disintegrated martensite is dominant in the G-AWS PB sample, as seen in Figure 6.14d and e. It is worth noting that there is no tangible change in the grain size after the paint-baking treatment. Moreover, the microstructure and the grain size after the Gleeble simulation show a good agreement with the actual welded samples. As shown in Figure 6.14c, a comparatively random texture can be seen after welding where a strong component is formed between  $\langle 101 \rangle$  and  $\langle 001 \rangle$  peaks with a weak component between  $\langle 001 \rangle$  and  $\langle 111 \rangle$ . This is the common welding texture with a reduced intensity (1.54). However, after PB (Figure 6.14f), the strong component formed between  $\langle 101 \rangle$  and  $\langle 001 \rangle$ , peaks are moving toward the  $\langle 101 \rangle$  direction which is implying that for most of the grain, their  $\langle 101 \rangle$  direction is parallel to the rolling direction (RD). The texture intensity also increased after PB (from 1.54 to 2.75) which can be due to the fact that the grains with their  $\langle 101 \rangle$  direction slightly deviated from RD of the steel (in the AWS sample), start to reorient and reduced their angle from RD until their  $\langle 101 \rangle$  direction is parallel to  $\langle 111 \rangle$ . This texture is

the favorable orientation for BCC materials. The formation of such texture components is believed to affect the crack propagation during post-welding loading, which will be discussed in the following.

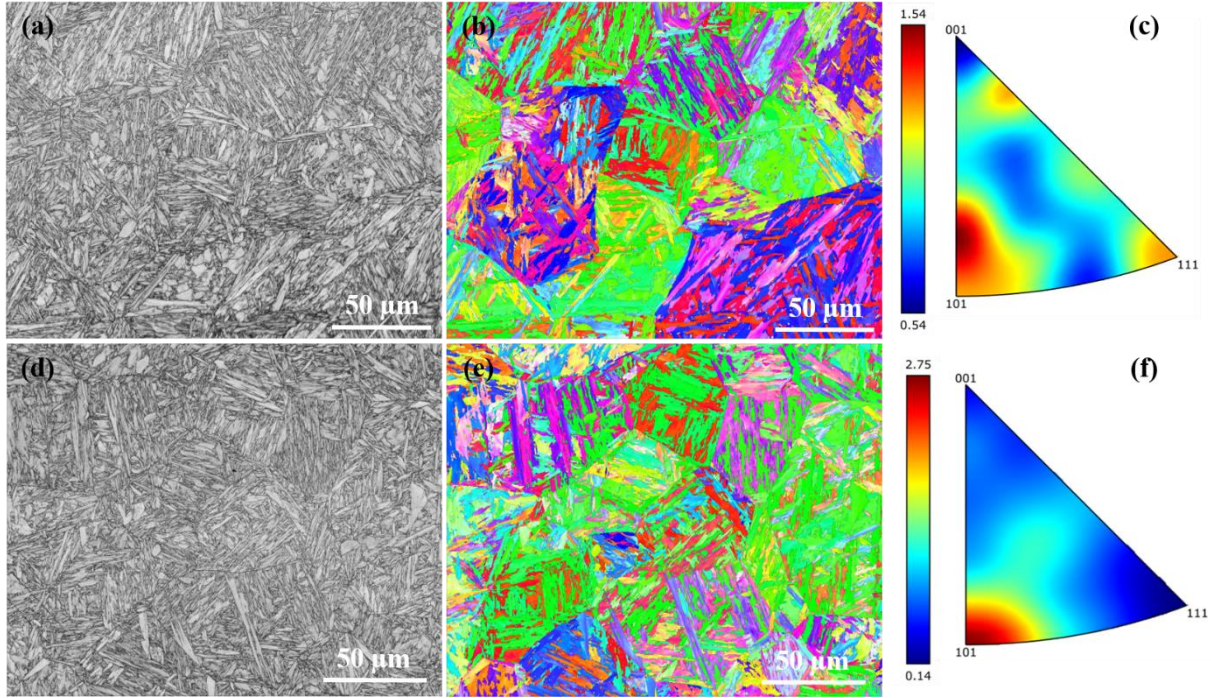


Figure 6.14: IQ, IPF map, and IPFs for (a-c) G-AWS and (d-f) G-AWS PB samples. The IPF in (c) and (f) are in a rolling direction of the steel.

## 6.5. Discussion

### 6.5.1. Effect of PB on Mechanical Properties

As shown in the previous section, the mechanical properties of resistance spot welds of Q&P 980 steels were remarkably improved after the PB treatment. The AWS sample exhibited intergranular fracture and failed through the PAGB (see Figure 6.8 and Figure 6.10), and the load-bearing capacity of the spot weld increased significantly after PB. To investigate the reason for this behavior, a CTT test was interrupted in the sudden increase in load-bearing capacity regime (see Figure 6.15a) and its corresponding cross-section is shown in Figure 6.15b. The cross-section of the interrupted CTT test of the AWS-PB showed that the failure proceeds through the corona bond at the faying surface, and the failure in the interrupted test arrested at the fusion boundary, as shown in Figure 6.15c. The corona bond turned into a crack at the sheet-sheet

interface during cross-tensile loading (where the fracture initiated in both the AWS and AWS-PB samples) and as it may be seen, the cracks blunted at the nugget boundary. A high-magnification SEM image shows the presence of disintegrated martensite (tempered martensite resulting from stage 3 of tempering) at the blunted crack tip (see Figure 6.15d). Crack propagation during loading is indicated by a small drop in the load-displacement curve. However, crack propagation occurs at higher displacements in the paint-baked sample as compared to the AWS sample. As discussed earlier, the higher displacement in the AWS-PB sample before the crack propagation was due to the change in the crack propagation path, which ran through the UCHAZ region rather than through the halo ring. This change in cracking direction results in pull-out fracture in the AWS-PB weld. Furthermore, here the interrupted tests show the blunting of the crack at the fusion boundary. Accordingly, it can be speculated that the reason for the sudden change in the slope of the load-displacement curve after PB (increase in loading) is more likely due to the change in the crack path where the propagation is diverted.

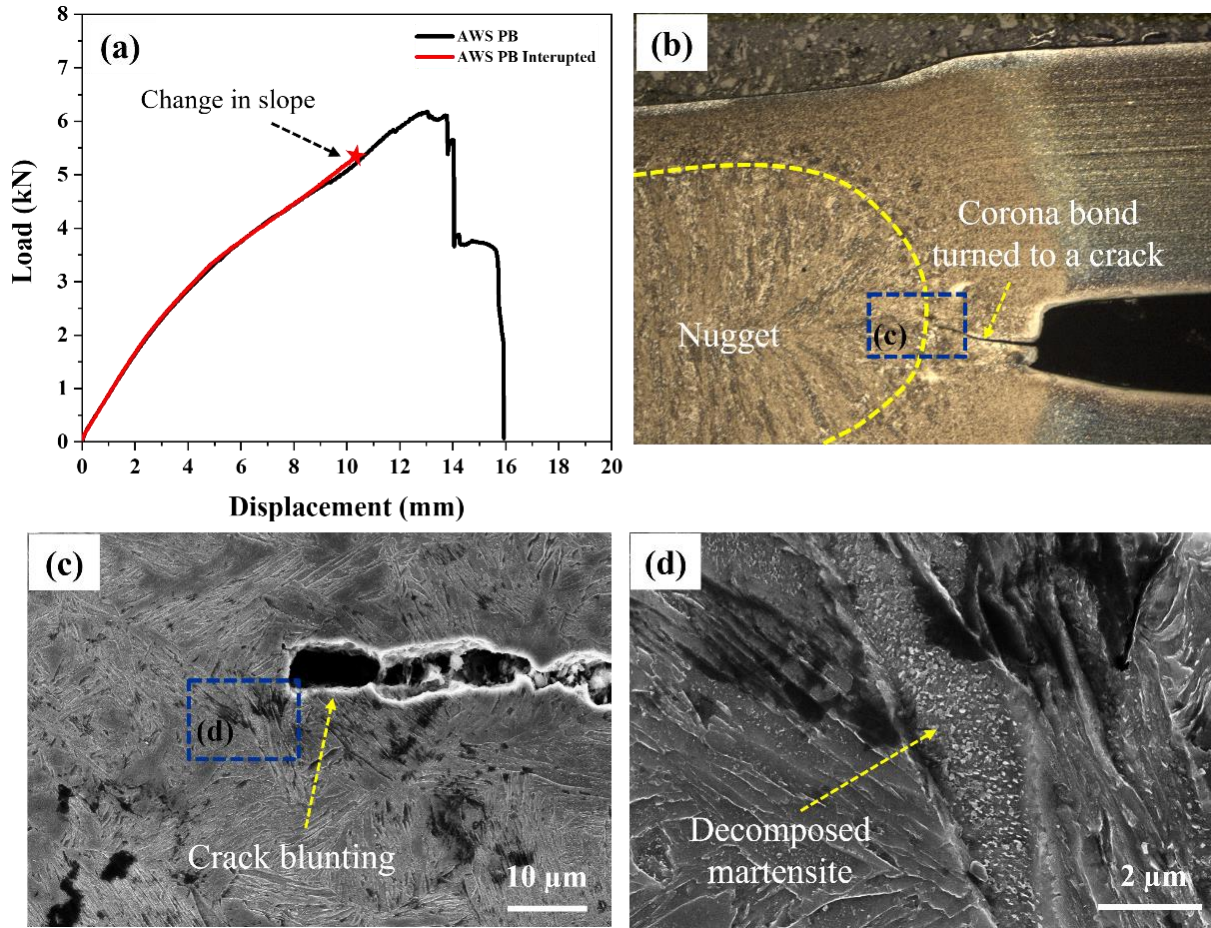


Figure 6.15: (a) load-displacement curve for interrupted CTT showing the sudden increase in the slope, (b) cross-sectional OM image after the interrupted CTT for the AWS-PB interrupted sample, and (c and d) high magnification SEM images showing blunting of crack and presence of decomposed martensite, respectively, for the AWS-PB interrupted sample.

The crack will change its path from the fusion boundary toward the UCHAZ which requires more energy, and therefore an increase in the loading will be seen in the load-displacement curve, after the PB. Here it is worth mentioning that the texture is also another factor playing a key role in diverting the crack propagation [167,168]. As shown in Figure 6.14d, the texture intensity enhanced after the PB where a strong  $\langle 101 \rangle$  texture was seen in RD. It can be inferred that once a crack advances through the microstructure, a large portion of grains have orientations ( $\langle 101 \rangle$  direction along RD) unfavorable for the crack propagation which thereby reduces the crack propagation rate. For further propagation, the load should increase, which will

help the crack to change its path toward regions (toward UCHAZ) with less intensified texture so it can propagate easily. Additionally, along with the grains' orientation, the surrounding microstructure is also another decisive factor in crack propagation behavior.

The mechanical properties of the spot welds are directly related to their local microstructure. It has been reported that the disintegration of martensite (tempered martensite) present in the microstructure, is one of the reasons behind the increase in cross-tensile properties [157]. The detailed microstructure of the Gleeble samples is shown in Figure 6.16. The presence of lath martensite was observed in the G-AWS sample and its corresponding selected area diffraction (SAD) pattern was shown in the inset (see Figure 6.16a). There was no other reflection than the BCC in the SAD pattern. G-AWS PB sample, on the other hand, consisted of a fully decomposed martensite microstructure, that can be confirmed by the corresponding SAD pattern (see Figure 6.16b). The presence of  $\epsilon$ -carbide in the martensitic matrix was indexed from the SAD pattern. This  $\epsilon$ -carbide is assumed to be the reason for the improved CTS after the paint baking.

A significant amount of research has been conducted by different researchers, and they came up with different theories regarding load-bearing improvements after PB. Tumuluru [42] investigated the effect of PB in DP and TRIP steels and explained that the formation of the Cottrell atmosphere of C is the reason for improving the mechanical properties. Although the author tried to explain the reason for the improvement in properties after PB, he failed to clarify the source for the geometrically necessary dislocations (GNDs) density to form this Cottrell atmosphere. After PB, due to the reduced microstructural inhomogeneities, as discussed in section 3.3, a lower GNDs density is expected. Eftekhari Milani et al. [157] reported that the possibility of an increase in the CTS could be due to the metallurgical transformation that happens in martensite or retained austenite. Moreover, they discussed that the high-carbon martensite present in the steel can transform into tempered martensite by virtue of carbide formation during the PB, which leads to significant improvements in both CTS and fracture toughness.

During CTT, two different mechanisms are concurrently competing, the plastic deformation by dislocation multiplication and their further movement, and crack propagation. Whenever the dislocation movement happens during the application of stress, plastic deformation will occur, and that will dominate rather than the crack propagation mechanism in the early stages of the CTT, as seen in Figure 6.15a. Once the hardening enters the recovery stage, due to the dynamic recovery processes (e.g., substructure formation, dislocation annihilation, etc.), the overall dislocation density will increase, but in a significantly slower rate, and the material will not undergo more dislocation multiplication. Then the crack propagation starts. It is widely accepted that martensite has a higher dislocation density than decomposed martensite, which means due to the dislocation multiplications and entanglement, the hardening and recovery stages initiates right away with plastic deformation. However, the  $\epsilon$ -carbides present in the decomposed martensite can interact with dislocations and can absorb more energy by pinning the dislocations and impeding their movement [160]. Therefore, the AWS sample did not have enough room for deformation during CTT; since it had a martensitic microstructure, thereby easily reaching a point where crack propagation dominates within a comparatively lower strain value. AWS-PB, on the other hand, has a microstructure consisting of decomposed martensite with  $\epsilon$ -carbides, which can delay crack propagation by absorbing more energy via delaying the dislocation movement. Accordingly, this will lead to strength and toughness improvement after PB.

It is established that the stacking fault energy (SFE) of ferrite is lower than that of martensite [169]. It can be assumed that the decomposed martensite is considered a transition phase towards the transformation of ferrite and cementite from the fully martensite microstructure. It can be concluded that the improvement in CTS after PB is due to the presence of  $\epsilon$ -carbide in the martensitic matrix, which can pin the movement of dislocations and delay crack propagation. Nonetheless, Speich et al. [170] reported that tempering at temperatures higher than 200°C can activate the transformation of retained austenite into martensite in some particular conditions. However, there was no clear evidence found in their study for transformations happening in retained austenite. More investigations will be done to explore the effect of retained austenite

during the paint-baking process on peak load and toughness improvement, and the micro-mechanisms behind mechanical properties advancement in the presence of such transitional phases, in our future studies.

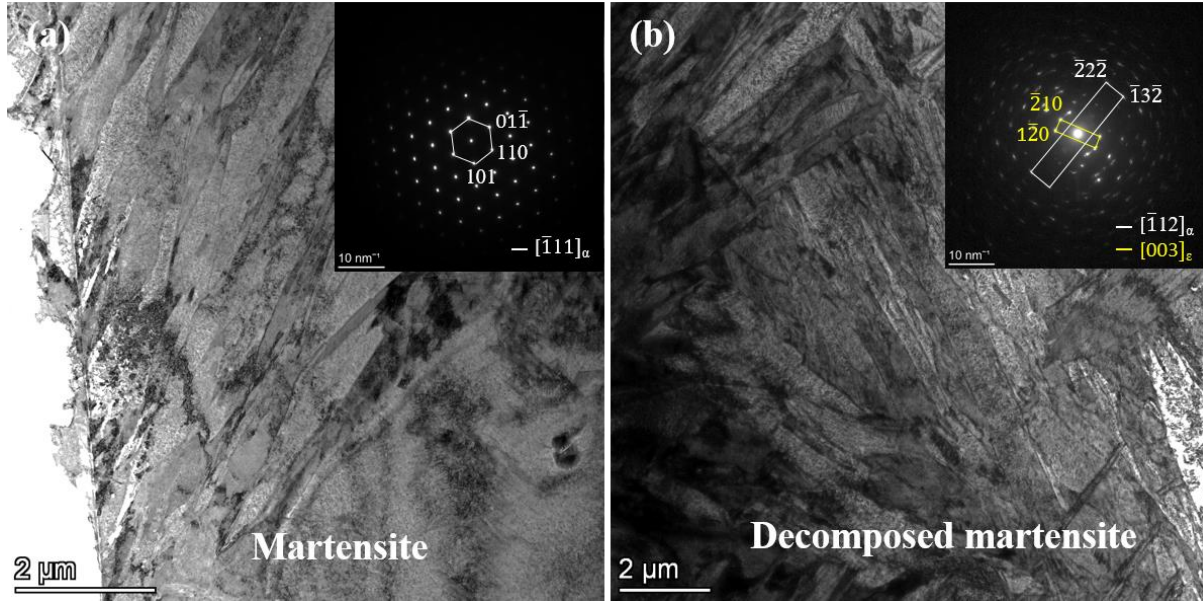


Figure 6.16: TEM images and their corresponding SAD pattern showing the microstructure in (a) G-AWS and (b) G-AWS PB samples.

### 6.5.2. Changes in Local Mechanical Properties

Examination of the AWS-PB load-displacement curve shows that it may be divided into distinct regions. Region 1 displays a linear loading rate, region 2 shows a gradual decrease in loading rate, followed by a sudden increase in loading rate (region 3), and final fracture (region 4). Of most interest, there is an increase in the slope of the load-displacement curve (region 3) above a load of  $\sim 4.5$  kN (Figure 6.17a). To assess the loading behavior during the CTT, the derivative of the load (L) to its displacement (D) was plotted against the sample displacement in Figure 6.17b.

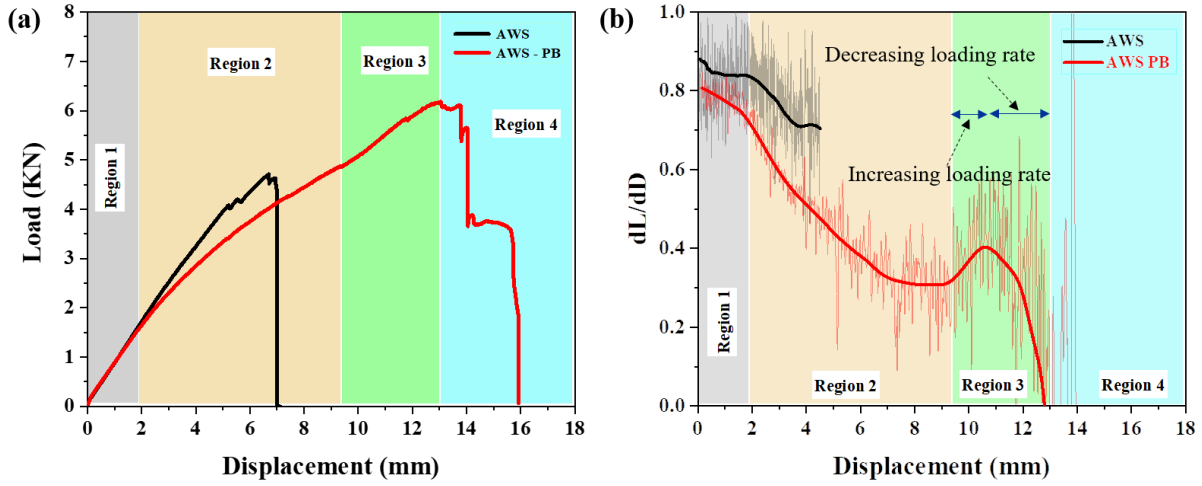


Figure 6.17: Mechanical properties of the spot welds before and after paint baking: (a) CTT, (b) derivative of the load-displacement curve.

There are some similarities between the derivatives of the CTT curve and the Kocks-Mecking work hardening model [171] (work hardening rate, e.g., derivative of the true stress-strain curve, as a function of flow stress) in materials during Mode 1 loading (tensile stress normal to the plane of the crack), to show work hardening behavior [171,172]. Therefore, the derivatives of the load-displacement curve (Figure 6.17b) are used to probe into the loading behavior of AWS and AWS-PB samples during CTT. The representative loading behavior is highlighted by the derivative of the load-displacement curve (filtered with a smoothening function to highlight the overall trend in the data) and matches with the identified regions from the load-displacement curve (Figure 6.17b). The AWS sample solely exhibits the defined region 1 and 2, while the AWS-PB sample shows all four regions. It should be noted that the load-displacement behavior of the AWS-PB sample exhibits a strong resemblance to load-displacement curves (CTS) documented in the literature [94,157]. In region 1, both the AWS and AWS-PB samples exhibit extensive bending of the base material, which is widely accepted in the literature [171]. In region 2 (Figure 6.17a), the two loading curves start deviating, implying the different loading behavior of the samples, resulting from the different microstructures. For both samples, starting in region 2, the deformation localizes around the nugget (where the PB-AWS microstructure is different, from the AWS weld). At a displacement

of 6 mm, the AWS sample fractures. Previous research demonstrates that the fracture surface of the AWS sample exhibits a brittle fracture behavior with an intergranular fracture path [141], aligning well with the load-displacement curve illustrated in Figure 6.17a.

The AWS-PB weld exhibits similar loading behavior to the AWS weld in region 1, although the loading rate of the AWS-PB weld is slightly lower than exhibited by the AWS weld. However, the AWS-PB weld demonstrates distinct behavior in regions 2 and 3 (Figure 6.17a). As discussed, the differences in the microstructure surrounding the nugget in the AWS-PB sample resulted in different loading behavior compared to the AWS weld. During testing of the AWS-PB sample, the load increases gradually in region 2 and entering region 3, a change in the slope of the load-displacement curve is seen (Figure 6.17a). Despite a longer region 2, region 3 is shorter where the sample reaches the peak load, and fractures entering region 4. As can be seen in Figure 6.17b, the loading rate decreased gradually in region 2 until it reached a plateau by the end of this region. Entering Region 3, the loading rate (Figure 6.17b) again initially increased, reached a maximum and then decreased. The reduction in loading rate continued until fracture in region 4. To discuss this unique loading behavior, the microstructure of the AWS-PB needs to be investigated. For this purpose, an interrupted CTT was done, as shown in Figure 6.15a. SEM-EBSD is used to assess the microstructure of the interrupted sample (Figure 6.18a and b).

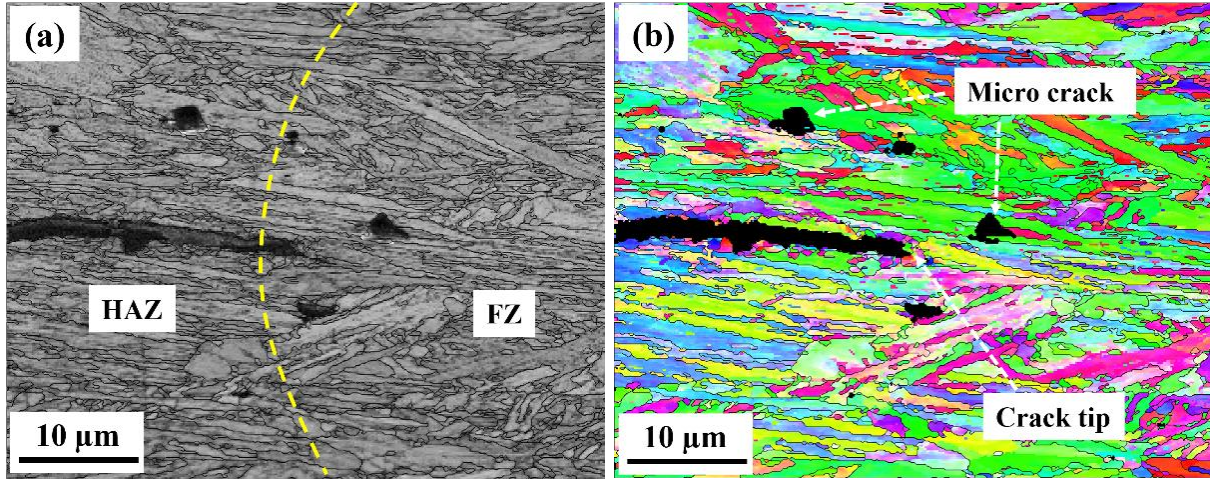


Figure 6.18: EBSD maps show the crack tip and the micro-cracks in the interrupted sample shown in Figure 6.15a, (a) IQ map and (b) IPF map.

It is important to emphasize that the interrupted sample behaves in the same manner as the AWS-PB sample, further confirming the reproducibility of this load-displacement curve. A blended crack in the sheet-sheet interface can be seen in Figure 6.18a, which primarily confirms the strain localization around the nugget area in region 3. The EBSD analysis also reveals the formation of small microcracks near the sheet-sheet interface, confirming that crack tip blunting occurred. By initiation of micro-cracks and blunting of the sheet-sheet interface cracks, the loading rate increases until the newly formed micro-cracks reach the critical propagation energy and start expanding through the microstructure, by which the loading rate will again decrease up to the fracture. Therefore, the improved energy absorption and fracture behavior of the AWS-PB sample can be attributed to this crack tip blunting and formation of micro-cracks, as they would have delayed crack propagation. It's important to emphasize that the weld sample undergoes rotation during the test, which should be taken into account to accurately quantify the actual normal load applied to the sample during testing. A comprehensive study on this aspect has been reported elsewhere [65] which is out of the scope of this work.

Even by understanding crack propagation behavior in the AWS-PB weld, the connection between the increase in loading rate in region 3 (Figure 6.18b) and changes to the dislocation-obstacle interactions

resulting from PB is unclear. To understand the changes in mechanical behavior, local mechanical properties needed to be assessed by focusing on the UCHAZ (Region 3) microstructure. For this purpose, the yield strength (YS) of the UCHAZ region was estimated for AWS and AWS-PB samples using instrumented indentation.[161] The load-displacement behavior shown in Figure 6.19, for AWS and AWS-PB samples does not show any substantial changes, where there is only a slight difference in the YS. The YS of the AWS and AWS-PB samples are measured to be 1248 and 1189 MPa, respectively. The slight decrease in YS observed in AWS-PB sample can be attributed to the presence of softer microstructural features, such as decomposed martensite, in the UCHAZ. Therefore, it seems that PB does not improve the local mechanical properties, e.g., YS, contrary to the improvement seen in the macro-mechanical performance (cross-tension strength). To understand the effect of microstructural changes in UCHAZ (region 3 microstructure) on local tensile properties, including the work hardening behavior, the UCHAZ was recreated using Gleeble and was mechanically tested.

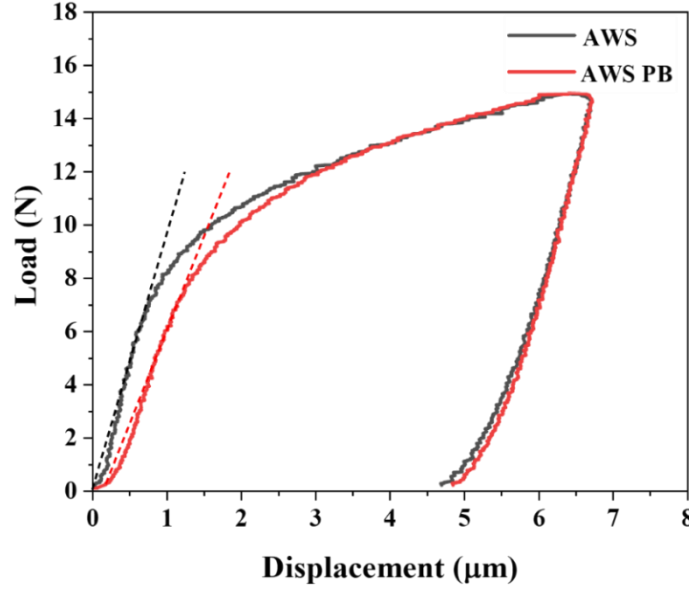


Figure 6.19: Instrumented indentation load-displacement curves for region 3 of AWS and AWS-PB samples.

The uniaxial tensile tests for Gleeble samples are presented in Figure 6.20. The strain hardening rate ( $d\sigma/d\varepsilon$ ) is determined by imposing the Kocks-Mecking model on the experimental data. The true stress-strain curves (Figure 6.20a) show the same broad behaviour of the cross-tension test (Figure 6.17a), where the G-AWS exhibited brittle behavior, showing sudden fracture after yielding, and the G-AWS PB sample had supported large plastic deformation ( $\sim 11\%$  elongation to fracture). However, due to the localization of the strain at the necked area, and the non-uniform plastic deformation behavior, only the data before necking (uniform plastic deformation region) was used to extract work hardening curves (Figure 6.20b). For the identification of the necking strain, Considère criterion [173] is used. The true stress at fracture and fracture strain was calculated using the cross-sectional area of the failed sample. As can be seen in Figure 6.20a, YS is 1278 and 1161 MPa for the G-AWS and G-AWS PB, respectively, which does not show a significant difference. These values align well with the yield strengths obtained from the actual welds (AWS and AWS-PB samples) by performing the micro-indentation (1248 and 1189 MPa, respectively). Hence, it can be assumed that the Gleeble-simulated samples well-represented the microstructure of specific weld regions.

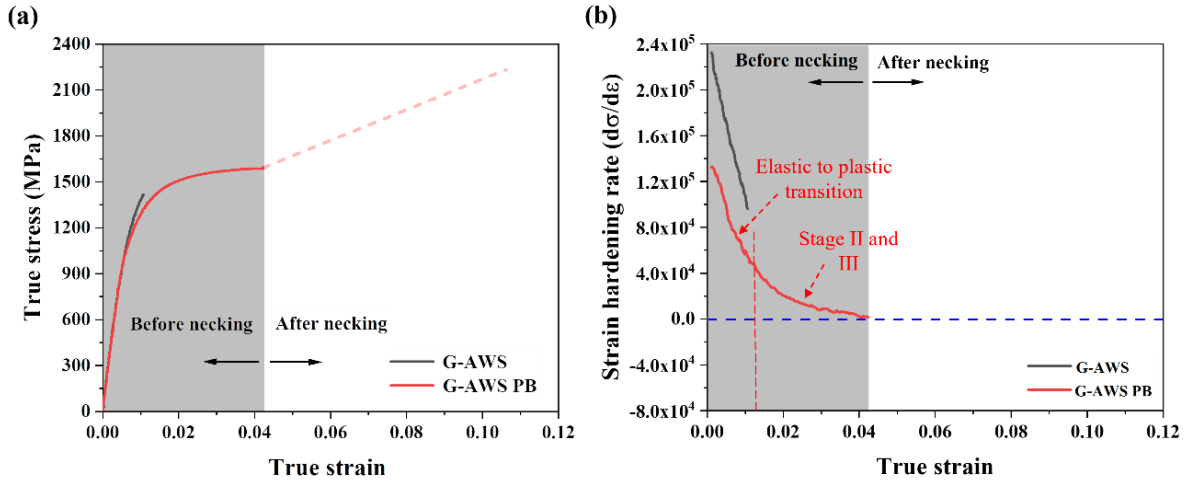


Figure 6.20: (a) true stress-strain behavior of the samples, and (b) derivative of the stress-strain curve showing the strain hardening behavior

From examining the work-hardening behavior of the samples, the G-AWS sample fractured immediately after yielding, and only the elastic to plastic transition is observable with no distinct work-hardening stages (Figure 6.20b). However, the G-AWS PB sample, exhibited Stage II (athermal hardening) and Stage III (dynamic recovery) work hardening after yielding. Stage II is typically accompanied by the persistent interaction of obstacles with dislocations and dislocation multiplications, where the work hardening rate shows a plateau (no further reduction in the work hardening rate)[174]. In Stage III, or dynamic recovery, dislocation substructures form a stage where rate of dislocation multiplication decreases due to the recovery processes, which is associated with a continuous reduction in the work hardening rate, until the fracture [174]. However, these two stages are not distinguishable in the current data. Immediately after the elastic to plastic transition region, the work hardening rate continuously decreases until the start of necking. Compared to the CTT behaviour, it may be seen that the increase in loading rate in region 3 (Figure 6.17b) was not present in the work hardening curve of G-AWB PB. Therefore, it can be concluded that PB does not affect yield strengthening and work hardening behavior (micro-mechanical behavior). However, it does substantially improve the fracture and energy absorption behavior (macro-mechanical performance) [141]. It is important to highlight that, increased elongation and work hardening of UCHAZ enables strain

localization to be continued around the nugget before fracture, offering new areas of strain hardening, leading to the increase in loading rate in the CTS specimens.

The increase in loading rate seen in region 3 of Figure 6.17b is not solely attributed to the changes in the work hardening behaviour of the UCHAZ after PB. Another important factor that needs to be considered regarding the work-hardening behavior is the dislocation density. For post-deformation hardening to occur as a result of PB, both the amount of carbon in the solid solution and the density of dislocation needs to be above a critical limit. However, in most of the studies, the critical dislocation density required to activate the SSA mechanism is absent while those studies primarily focus on the critical carbon content. It is also worth noting that PB leads to the redistribution of solute in the matrix and decreases elemental segregation, leading to a more homogenous microstructure [16,141]. This implies that after PB there will be a low GND density as they are generated from microstructural inhomogeneities and existence of grain boundaries. Therefore, it is expected that GNDs will not have a significant influence on the SSA mechanism in the PB sample, although the carbon content in the solid solution may reach the critical limit. However, this needs a systematic study incorporating characterization techniques and interrupted tests to examine the evolution of dislocation density before and after PB as well as during tensile deformation, which is the topic of an ongoing study.

### 6.5.3. Fracture Macro-mechanism (Intergranular vs. Trans-granular)

It was shown previously that the failure for the AWS sample is a partial thickness (Mode 3) type through the halo ring and it is a pull-out failure (Mode 1) through the UCHAZ in the AWS-PB sample. To reveal the reason behind the intergranular fracture for the AWS sample, a detailed TEM-EDS analysis was conducted on the Gleeble samples. EDS analysis on typical PAGBs of G-AWS and G-AWS PB are shown in Figure 6.21. It can be seen that (Figure 6.21a) the G-AWS sample has a clear segregation of C, Mn, Al, and Cr to its PAGBs. It's also worth noting that P segregation hasn't been observed in the G-AWS condition. Furthermore, PB treatment improves the grain boundary properties by redistributing the alloying elements

to the matrix (See Figure 6.21b) reducing or eliminating their segregation. Hence, the heat treatment improves the grain boundary characteristics by diffusing out C, Mn, Al, and Cr to the grain interior, thereby improving the crack propagation properties by changing the crack propagation path from an intergranular to a transgranular mechanism.

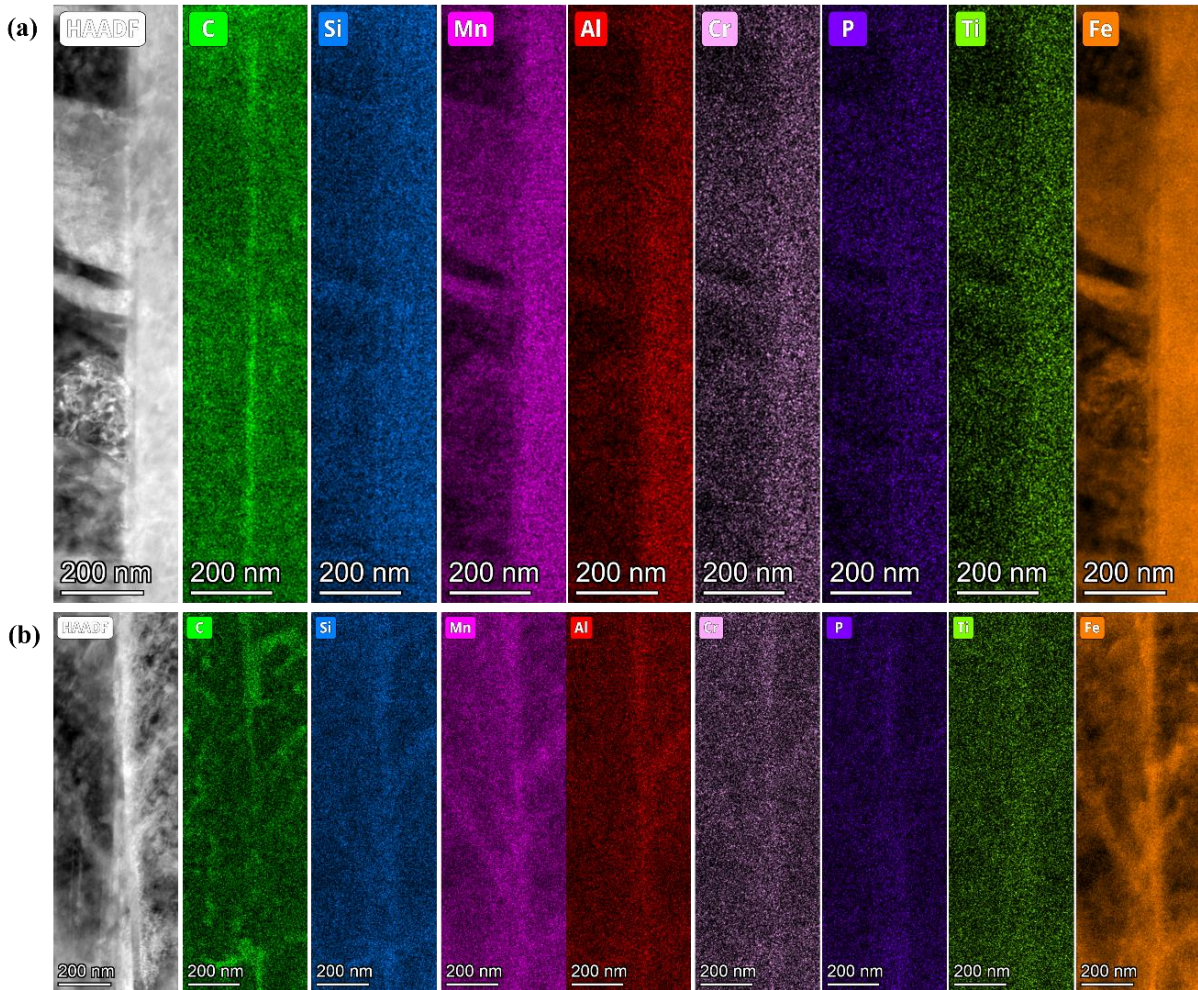


*Figure 6.21: TEM-EDS images showing (a) elemental segregation in the grain boundaries for the G-AWS sample, and (b) no clear/reduced segregation for the G-AWS PB sample.*

It has been reported that during the transformation of austenite into martensite (diffusion-less transformation), the solubility limit of carbon largely exceeds in martensite [97]. This high thermal energy triggers carbon to segregate to the grain boundaries since the grain boundaries are the favorable sites for defects, such as dislocations to nucleate, this segregation further increase the residual stresses [175]. It is

also worth noting that the high carbon content in the grain boundary also can stabilize the austenite during transformation, thereby forming a thin film of retained austenite at the grain boundaries. This presence of solute elements can cause embrittlement of the grain boundaries during loading. The presence of higher amount of Mn in the grain boundaries also implies presence of austenite. Park et al. [54] reported that C and other substitutional alloying elements can segregate to the grain boundary during welding, further deteriorating the fracture behavior. However, PB treatment was found to facilitate the redistribution of C and Mn from the grain boundaries to the grain interiors. Furthermore, the segregation of C and other alloying elements to the grain boundary mainly depends on the activity of each of these elements. We believe that the rate of diffusion and the factors favouring this diffusion are out of the scope of this work and will be addressed in follow-up work.

It is well reported that regardless of the welding schedules, the crack will always start forming at the sheet-sheet interface due to the stress concentration during CTT [61]. Therefore, for both AWS and AWS-PB samples, the preliminary crack initiation mechanism remains the same. However, the microstructure surrounded by the sheet-sheet interface (UCHAZ and FZ) serves a significant role in the crack propagation behavior, thereby determining the mechanical and fracture properties of the spot welds. In general, cracks will only propagate when the stress applied at the crack tip exceeds the material's fracture toughness [75,116]. In the AWS sample, the crack also initiates at the solidified liquation products and propagates through the halo ring. It is understood from the results of the G-AWS sample that the halo ring and the region near the halo (UCHAZ) have the segregation of C, Mn, Al, and Cr, which decreases the grain boundary energy (bonding nature of the grain boundary) thereby experiences a low energy brittle fracture. On the contrary, the PB treatments change the fracture mode to pull out through the UCHAZ by modifying the grain boundary characteristics and altering the crack propagation mechanism from intergranular to trans-granular, due to the evolution of the surrounding microstructure and texture. That means the change in crack propagation path in the PB sample was due to the transformation of martensite to decomposed martensite, with  $\epsilon$ -carbide, and the redistribution of C, Mn, Al and Cr from the grain boundary to the grain

interior. Both microstructural changes, delays the crack propagation and improves the maximum strength and toughness of the PB samples. In other words, the PB treatment enhances atomic mobility, and grain growth. This will allow the redistribution of alloying elements as well as the disappearance of the solidified liquation from the grain boundaries, thereby changes the brittle (for the AWS sample) to the ductile fracture. The delayed crack propagation is also partially supported by the change in the microstructure and texture of the UCHAZ since the toughness of the decomposed martensite is higher than that of the high-carbon martensite (AWS sample). Therefore, PB is shown to be very essential in terms of mechanical and fracture behavior improvement for the welded samples.

## **6.6. Summary**

The effect of paint baking on spot-welded Q&P 980 steel under cross-tension loading was studied. Moreover, Gleeble simulations were done to simulate the upper-critical heat-affected zone in the weld samples, followed by uniaxial tensile testing to gain insights into the effects of PB on material properties and fracture micro- and macro-mechanisms. The following conclusions were drawn from this study:

- 1) Post-weld PB heat treatment significantly improved the load-bearing capacity of the Q&P 980 steel spot welds in the cross-tension loading conditions. The application of PB increases the weld strength (peak load) by 34 and 17% for AWS and DPS samples, respectively. The absorbed energy was improved by the order of 102% and 67%, for AWS-PB and DPS-PB samples, respectively. PB significantly decreased the differences in mechanical properties between the AWS and DPS welds. Unless strength needs to be maximized further, the DPS schedule is not required under normal manufacturing conditions to mitigate sub-optimal joint strength resulting from the halo.
- 2) PB changes the fracture mode from Mode 3 (partial thickness fracture) to Mode 1 (button pull) in AWS welds. However, DPS weld failed with Mode 1 fracture regardless of whether the welds were subjected to PB or not. AWS welds failed in a brittle manner whereas the AWS-PB failed in a ductile manner.

- 3) The presence of solidified liquation regions at the grain boundaries in the AWS sample also serves a role in crack initiation. Cracks can be initiated from the solidified liquation regions during loading due to the presence of retained austenite structures and the strain inhomogeneity between austenite and martensite, culminating in stress concentrations. However, due to the enhanced atomic mobility and grain growth during the PB, the weak solidified liquation regions are consumed by surrounding grains and the redistribution of Mn and Si to the grain interiors. This further improves energy absorption due to the elimination of crack initiation sources.
- 4) Microstructural analysis revealed that PB resulted in the decomposition of the martensite and formation of  $\epsilon$ -carbide, which is associated with an increase in the fracture toughness and is likely the major factor improving the CTS of Q&P 980.
- 5) PB was associated with a change in the micro-texture where an intensified texture with the  $\langle 101 \rangle$  direction of most grains parallel to the rolling direction was observed. This texture was found to be less favorable for crack propagation, increasing the energy required for a crack to continue its movement leading to the change in the crack path which was accompanied by an increase in the loading rate during CTT.
- 6) The presence of segregation of C, Mn, Al, and Cr was observed along the grain boundaries in G-AWS conditions, which was further redistributed throughout the grains after the PB treatments. The presence of C and Mn in the grain boundaries decreases the grain boundary cohesion thereby decreasing the fracture toughness of the grain boundaries.
- 7) Four distinct regions were identified for CTT load-displacement curves after the PB. It was found that regions 2 and 3 involve the localization of deformation at the nugget area where in region 3, the loading rate (derivative of load-displacement) surprisingly increased and then after reaching a maximum, decreased up to the fracture in region 4. It was later shown that this unique loading behavior is due to the crack tip blunting and initiation of new micro-cracks.

- 8) Uniaxial tensile behavior of the simulated microstructure of region 3 (UCHAZ) did not show any significant difference in yield strengthening and work hardening behavior between the G-AWS and G-AWS PB samples. It was found that the G-AWS PB sample experienced Stage II and III work hardening, where these two stages were not distinguishable. However, the G-AWS sample only entered early Stage II right after elastic to plastic transition, before experiencing a premature fracture.

## Chapter 7. Conclusions and Future Recommendations

### 7.1. Conclusions

The current research focused on the characterization and understanding of a localized softened zone in resistance spot welded joints, referred to as the halo ring. For the first time, this study establishes the relationship between the joining material composition on halo formation, characterization of the halo microstructure, proposed halo formation mechanism, and the development of two distinct techniques to mitigate the halo formation. From the initial part of the study, it was observed that single-pulse weld failed through the halo ring under cross tensile loading. Adoption of a double pulse welding schedule led to the elimination of the halo ring, and consequently it modified the failure path to the UHAZ. Detailed understanding of the role of FZ chemical composition on halo formation was investigated by producing dissimilar welds using a sandwich joint geometry. It was observed that welds made with LC steel exhibited a more prominent halo ring at the Q&P steel side, while those with HC steel showed less halo formation. The microstructure of the halo ring is identified as upper bainite, and the C diffusion from the FB to the molten nugget during solidification is the reason for halo formation. Another mitigation technique, PB heat treatment, was explored, with the underlying mechanisms outlined. PB proved effective in eliminating halo effects and liquation by redistributing alloying elements from grain boundaries to grain interiors. In summary, this study presents a novel and practical discovery regarding the halo ring. The following major conclusions were drawn from this study:

1. Resistance spot welds made with single pulse condition recommended by AWS exhibit poor CTS on the order of  $3.1 \pm 0.05$  kN. These welds failed through the transient softened zone along the fusion boundary, called halo ring. The fracture surface analysis indicates a brittle dominant fracture, passing through the fusion boundary. A novel double pulse welding schedule (higher second current pulse with a short cooling time of 30 ms between the current pulses) was implemented, which eliminated the halo ring and improved CTS and absorbed energy by 33 % and 110%, respectively. The double pulse

schedules modified the grain structure of the fusion boundary, led to changes in the crack propagation paths by altering the stress concentration areas. Additionally, it was observed that the halo ring had depleted levels of C, Mn, and Si compared to the surrounding microstructure.

2. In dissimilar welds, the formation of halo was more prominent in welds with a fusion zone that had a lower alloy content (made using LC steel) than the surrounding HAZ, as compared to welds made with a higher alloyed FZ (with HC steel). When the halo from the three joints were compared, the order of softening severity, from highest to lowest, was as follows: LC steel weld, 3-sheet Q&P weld, and the HC steel weld. It was observed that the halo in LC steel weld exhibited a significant depletion of C, Mn and Si elements at the fusion boundary for approximately 75 $\mu$ m. Conversely, no significant depletion of C, Mn, and Si was observed in the HC steel stack-up on the Q&P steel side. The transient softened zone can be modified by alloying with greater and lesser compositions of steel in dissimilar welded joints.
3. In-depth TEM analysis showed the microstructure of halo ring. TEM investigation revealed that the microstructure of the carbon depleted halo ring is identified as upper bainite (bainitic ferrite with needle like cementite). Carbon diffusion from the FB to the molten metal during solidification, driven by the elevated activity at the FZ attributed to its higher temperature is the reason for the halo formation.
4. Post-weld paint baking (PB) heat treatment significantly improved the load-bearing capacity of the Q&P 980 steel spot welds in the cross-tension loading. It was observed that the application of PB increases the weld strength, and absorbed energy by 34 and 102%, respectively and consequently shifted the failure location from halo ring to the UCHAZ. Additionally, PB resolved the presence of liquation's present at the UCHAZ. The enhanced atomic mobility and grain growth during the PB treatment led to the redistribution of alloying elements such as C, and Mn from the grain boundary to

the grain interiors. This redistribution reduced strain localization at the grain boundary, thereby enhancing energy absorption by eliminating sources of crack initiations.

5. Paint bake treatment was also associated with a change in the micro-texture where an intensified texture with the  $\langle 101 \rangle$  direction of most grains parallel to the rolling direction was observed. This texture was known to suppress the crack propagation. Thereby PB treatment eliminates the detrimental effect of the halo ring on mechanical properties.

## **7.2. Areas of Future Research**

Although the present study provided insights into the role of localized microstructure on the mechanical properties of the resistance spot weld, there remain numerous unanswered questions. Future study should focus on the following aspects.

The present research revealed that there was diffusion of alloying elements across the fusion boundary. Subsequent studies could delve deeper into the individual effects of each alloying element's movement across the boundary by isolating the effects of other elements. There is potential to quantify the diffusion of specific alloying elements, particularly C, Mn and Si. This will provide a comprehensive map of alloying elements detailing its individual effects on the halo formation. For instance, studying the pure C diffusion in halo can be achieved by conducting analysis on a low Si and Mn steel. HRTEM analysis coupled with high resolution elemental mapping will provide valuable insights into the carbon diffusion. Additionally, proposing diffusion models alongside experimental observations could offer valuable insights into the applicability of these models to other steel compositions and combinations.

Another area for further research involves adjusting process parameters to prevent halo formation, such as implementing force ramping during the welding process itself. This study has established that cooling of the nugget boundary during the latter stages of welding time contributes to halo formation. However, exploring variations in cooling rate while maintaining constant heat input will provide insights into the

impact of cooling rate on the characteristics of the halo ring. Therefore, adjusting the cooling rate during welding by reducing the electrode force (force ramping) in the latter stages of the welding time could be explored. Various experiments should be conducted to optimize the force and time at which the halo free weld is achieved. Additionally, investigating the use of different electrode geometries for welding, such as flat-faced electrodes, may offer insights, as they could potentially intensify cooling compared to dome-shaped electrodes. A systematic approach involving step-by-step variations in radius of the electrode could be employed to identify the critical electrode geometry that can be used to optimize the halo formation. Following the optimization of these methods, microstructure and elemental composition of the halo ring could be analysed using TEM analysis to validate the results. Furthermore, exploring the feasibility of utilizing different electrode surface coatings to modify heat dissipation through the electrodes could also be considered.

## Chapter 8. Research Contributions.

### 8.1. Journal Publications.

1. **D.C. Ramachandran**, A. Salandari-Rabori, A. Macwan, E. Biro, Effect of material chemical composition on the formation of the transient softened zone in a Gen 3 Q&P steel, Under review, *Metall. Mater. Trans. A* (2024).
2. **D.C. Ramachandran**, A. Salandari-Rabori, A. Macwan, E. Biro, Microstructural evolution and formation mechanism of the transient softened zone along the fusion boundary in resistance spot welding of a 3G advanced high strength steel, Under review, *Scripta Materialia* (2024).
3. **D.C. Ramachandran**, A. Salandari-Rabori, A.R. Midawi, A. Macwan, E. Biro, Effect of paint baking treatment on mechanical properties of resistance spot welded Q&P 980 steel, Under review, *ISIJ International* (2024).
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## 8.2. Conference Papers and Presentations

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2. **D.C. Ramachandran**, A. Macwan, E. Biro, Effect of material composition on the formation of the transient softened zone in Gen 3 Q&P 980 steel, *IIW CIII Intermediate meeting-Paris*, (2023)
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