

The Role of Microstructural Modifications in Improving the Mechanical Properties of Resistance Spot Welded Automotive Steels

by

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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 consists of 8 chapters, including the introduction (Chapter 1) and literature review (Chapter 2). A series of data chapters starting from Chapters 3 to 6 includes published or submitted manuscripts that the candidate authored as the primary author. The manuscripts have been modified to fit the style of the thesis. Majority of the lab work (except where noted), including welding, sample preparations, mechanical testing, characterization and the writing were done by the candidate. The following co-authors have made contributions to the present work, as outlined below:

Professor Elliot Biro supervised the research progress, advised the research direction, reviewed and edited all manuscripts.

Dr. Hassan Ghassemi-Armaki from General Motors, Michigan, USA, acted as industrial supervisor, provided material, financial support, feedbacks on the research, and assisted with editing the manuscripts.

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Abstract

The advent of advanced high-strength steel (AHSS) in the automotive industry has evolved in recent years to satisfy the global demands for lightweight and safer vehicles. These AHSSs offer an attractive combination of strength and ductility, making them ideal for use in vehicle body-in-white structures. However, their weldability and joint performance in-service are crucial to remain competitive for selection in the automotive industry. Joining AHSS is mostly achieved by resistance spot welding (RSW), and it is envisaged to continue for the foreseeable future. Despite its advantages, the rapid cooling rates during the RSW process result in the formation of martensite in the weld fusion zone, which is known to be hard and brittle, thereby resulting in low energy absorption that is undesirable in case of a crash event.

This thesis explores various metallurgical pathways that can be employed during in-situ RSW process to modify the joint microstructure and enhance the energy absorption capability of the weld. For each technique, an understanding of how different in-situ post-weld heat treatment (PWHT) parameters induce microstructural changes was investigated in this work. Furthermore, this research elucidates the microstructural evolution occurring during the non-equilibrium in-situ PWHT process and correlates these changes with the resulting mechanical properties. Grain refinement was found to be the most effective approach to improve the energy absorption capability of the weld compared to tempering, strain hardening, and paint baking processes. The refined prior austenite grain (PAG) structure was accompanied by a refinement of the substructure with high-angle grain boundary that poses more resistance to crack propagation thereby resulting in 89% improvement in energy absorption capability to failure compared to the baseline welds. It was found that the grain refinement is achieved after applying a PWHT current pulse when the edge of the FZ is solidified and in the austenitic region, rather than when the region has transformed martensite – the latter being preferred for tempering. The recrystallization schedule that induces the grain refinement was also achieved at a relatively shorter process time compared to the other PWHT techniques, which is an important criterion for industrial applicability.

Additionally, the PWHT schedules adopted in this research altered the weld failure mode, causing crack propagation to deviate at the edge of the FZ during cross-tension tests. For the welds with grain refinement, the improved energy absorption capability was majorly attributed to the new equiaxed prior austenite grain structure and the change in crystallographic texture from the cleavage (001) plane in the baseline welds to the (101) plane that supports plastic

deformation ahead of the crack tip, thereby retarding the crack propagation. These changes led to ductile failure, in contrast to the brittle failure observed in baseline schedules where cracks propagated into the fully martensitic FZ along the columnar structure. The findings of this research provide a unique perspective on the metallurgical transformations during in-situ RSW PWHT, offering valuable insights to the scientific community. Furthermore, these results inform the automotive industry of the optimal PWHT technique that can be employed in their manufacturing lines, enhancing the performance and safety of AHSS joints in vehicles.

Keywords: *Resistance spot welding (RSW), Advanced high strength steels (AHSSs), Post weld heat-treatment (PWHT), Microstructure, Mechanical properties.*

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Dedication

*Dedicated to my family for believing in me and supporting me
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List of Abbreviations¹

AHSS	Advanced High Strength Steel
ASTM	American Society of Testing and Materials
AWS	American Welding Society
BCC	Body-Centered Cubic
BCT	Body-Centered Tetragonal
BWS	Baseline Welding Schedule
BH	Bake Hardening
BM	Base Metal
CCR	Critical Cooling Rate
CCT	Continuous Cooling Transformation
CGHAZ	Coarse Grain Heat Affected Zone
CT	Cool Time
CTS	Cross-Tension Strength
CTT	Cross-Tension Test
DF	Dual Force
DP	Dual Phase
EBS	Electron Backscatter Diffraction
EPMA	Electron Probe Micro-analyzer
FZ	Fusion Zone
GB	Grain Boundary
GOS	Grain Orientation Spread
GROD	Grain Reference Orientation Deviation
HAG	Homogenization and Grain Refinement
HAGB	High-Angle Grain Boundary
HAZ	Heat-Affected Zone
HNG	Homogenization no Grain Refinement
IF	Interfacial Failure
IPF	Inverse Pole Figure
MFDC	Medium Frequency Direct Current

¹ Only abbreviations mentioned more than once is listed.

MWS	Minimum Weld Size
PAG	Prior Austenite Grain
PB	Paint Baking
PHS	Press-Hardened Steel
PIF	Partial Interfacial Failure
PO	Pull-out
PT	Pulse Time
PWHT	Post Weld Heat Treatment
RecX	Recrystallization Pulse
RSW	Resistance Spot Welding
SCHAZ	Sub-critical Heat Affected Zone
SEM	Scanning Electron Microscope
TEM	Transmission Electron Microscope
TPS	Temper Pulse Schedule
TRIP	Transformation Induced Plasticity
TST	Tensile Shear Test
UCHAZ	Upper-Critical Heat Affected Zone
UTS	Ultimate Tensile Strength
XRD	X-ray Diffraction

"In the realm of automotive manufacturing, the strength of the weld is a testament to the quality and durability of the vehicle." – Robert Wilson,

CHAPTER

1

1. Introduction

1.1 Study Background

Since the 1920s, steel has been the main material used for automobile construction. According to Ducker Worldwide, the steel content in a vehicle as of 2015 is estimated at around 60 % [1]. To reduce the weight of vehicles, car manufacturers must adopt design strategies, which include the development of new lightweight materials that can also reduce fuel consumption and emission of greenhouse gases to the environment [2,3]. However, responding to environmental concerns and safety requirements impose conflict on a vehicle design because lighter vehicles have inferior safety performance during a crash, but they are associated with lower levels of fuel consumption and carbon emissions [4]. Therefore, car manufacturers must balance the requirements of weight reduction and crash safety. These demands have led to the development of Advanced High Strength Steels (AHSS) which allow weight reduction by using thinner sheets while maintaining the part's structural integrity. Moreover, the use of AHSS to replace conventional steel in a car body structure can save costs up to 14% [5,6].

AHSS have continued to gain attention due to their unique properties achieved by microstructural changes, alloying and advanced manufacturing processes [3]. The alloying and microstructural changes result in steels such as dual-phase (DP) steels with ultimate tensile strength (UTS) in the range of 500–1200 MPa [5,7], and press-hardened steels (PHS) with UTS that can exceed 1500 MPa commonly referred to as Ultra-high strength steels (UHSS) [8]. Despite the high strength levels of these AHSS, their usage in the automotive industry still requires them to have a high level of weldability and joint reliability.

Resistance spot welding (RSW) remains the predominant joining technique in the automotive industry, but the process is known to alter the metallurgical attributes of materials which dictates its performance in service. Specifically, the welding process is associated with a rapid cooling rate that promotes the formation of martensite, a microstructure known for its high hardness but low ductility and toughness, leading to increased brittleness [9]. Additionally, the weld joint exhibits a heterogeneous microstructure due to the varying thermal cycles experienced across different zones during welding. The fusion zone (FZ), where the metal has completely melted and solidified, and the surrounding heat-affected zones (HAZ), experience different peak temperatures, which results in the formation of different microstructures in each region. These microstructural variations across the joint lead to varying mechanical property responses when the welded part is subjected to loading conditions. Particularly, the martensite formed in the fusion zone (FZ) of the weld is a brittle structure that can result in low energy absorption capability of the joint, which is undesirable in the event of a crash scenario [10,11].

This is particularly problematic for automotive applications, where high energy absorption is crucial to ensure vehicle crashworthiness. Therefore, it is vital to develop welding schedules to induce metallurgical transformation that can modify the FZ microstructure of the welded joint. The microstructural modification in the FZ of the weld is expected to improve the joint properties and ultimately the performance of the joint in service.

The modification of the FZ microstructure can be achieved through either isothermal or non-isothermal heat treatments. Isothermal heat treatment is typically performed using conventional furnaces, whereas non-isothermal heat treatment can be integrated into the welding process without the need for external equipment. This research employs a non-isothermal pathway achieved via in-situ post-weld heat treatment (PWHT) during resistance spot welding. Since this method is a non-equilibrium transformation process, inducing certain microstructural modifications in the weld requires adequate study. Specifically, it is uncertain how different in-situ PWHT parameters lead to microstructural changes in the weld and how these changes impact the resulting joint performance under quasi-static loading conditions. Therefore, this research will examine various thermal-mechanical cycles to initiate metallurgical transformations and establish correlations between each cycle and the mechanical properties of the weld joint.

1.2 Objectives

Improving the mechanical performance of resistance spot welds will improve overall vehicle crashworthiness, which can improve passenger safety during crash events. The use of well-defined and controlled PWHT schedules can be a feasible means to modify the structure of the welds which dictates the properties and performance in service. This research aims to understand how various post-weld thermo-mechanical treatments affect weld structure and its subsequent mechanical properties. Additionally, the underlying physical metallurgical mechanisms of each PWHT technique during the non-equilibrium RSW process were investigated and correlated to the changes in the energy absorption capability. The specific objectives of this research include to:

1. Modify the process parameter recommendations of American Welding Society (AWS) standards to obtain an acceptable weld free of defects.
2. Develop in-situ post-weld heat treatment (PWHT) schedules to modify the as-welded fusion zone microstructure and maximize weld toughness.

3. Characterize the effect of each developed PWHT schedule on the mechanical properties and failure mechanism of the joint.
4. Establish the connection between the microstructural modifications and the joint properties by examining the metallurgical transformations occurring in the weld.
5. Identify the PWHT welding schedule that yields optimal joint properties and determine the factors contributing to the enhancement of joint properties.

The findings of this research will benefit the academic community and the automotive industry by providing knowledge on the underlying principles to achieve intrinsically safe microstructures that will lead to improved joint quality of AHSS. The microstructural changes will lead to better energy-absorbing welds that will improve passengers' safety in the case of a crash event.

1.3 Organization of the Thesis

This thesis is focused on understanding how metallurgical transformations during in-situ PWHT of welds govern the mechanical performance in service. This thesis has been organized as follows:

- Chapter 1 presents the study background and the objectives of the thesis.
- Chapter 2 presents a detailed and systematic review of the material properties and microstructural features; RSW process variables and the various underlying principles of the metallurgical transformations to be exploited in this study.
- Chapter 3 describes how tempering changes the mechanical properties and failure behaviour of the joints. In addition, the chapter details the influence of voids during PWHT schedule and the effect on joint mechanical properties.
- Chapter 4 focuses on how the recrystallization pulse refines the grain structure of the weld, particularly at the edge of the fusion zone. This chapter provides an in-depth analysis of the microstructural evolution, outlining the sequence of transformations observed with different PWHT parameters.
- Chapter 5 elucidates the dual force technique, with a focus on investigating how force ramping can induce metallurgical changes in the weld structure and evaluates the resulting joint properties.
- Chapter 6 shows the effect of paint baking on the mechanical properties following the application of the optimal PWHT schedule identified from the previous chapters.

Additionally, the microstructure along the crack path was examined to understand the influence of the PWHT schedule and subsequent paint baking.

- Chapter 7 offers a comprehensive discussion to underscore the major factors dictating the changes in mechanical properties. A comparative analysis of the techniques examined in the study is also presented to summarize each metallurgical transformation, energy input and process time requirement needed for industrial applicability.
- Chapter 8 outlines the major conclusions and provides recommendations for future research.

CHAPTER

2

2. Literature Review

2.1 Advanced High-Strength Steels

The steel industry has been developing new AHSS through unique metallurgical processing methods and yields enhanced material properties. These advancements have enabled the automotive industry to meet stringent requirements for safety, reduced emissions, ease of manufacturability and quality at a relatively low cost. The emergence of AHSS with a good combination of strength and ductility satisfies the demands for lightweight and crashworthy vehicles [3,5]. Based on the different development stages, AHSSs can be divided into three generations as outlined in the elongation-tensile strength graph in Fig. 2.1, with unique processing routes and microstructure [12].

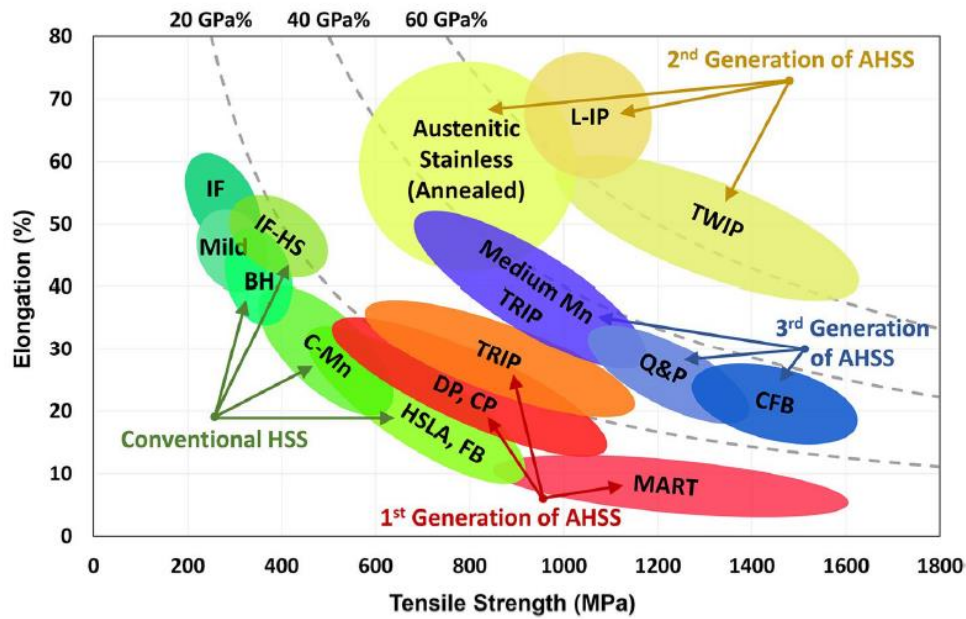


Fig. 2.1: Elongation versus tensile strength of various grades of AHSS [13].

The first generation (1G) of AHSS includes dual phase (DP), complex phase (CP), transformation-induced plasticity (TRIP) and martensitic (MART) steels. These grades of steel were developed using relatively small quantity of alloying constituents and mostly have a multi-phase microstructure that is predominantly ferrite-based with one or more phases such as bainite, martensite or retained austenite. The strength of the 1G AHSSs is typically higher than the conventional mild steels but low ductility still poses a major problem, especially for applications where complex shapes and deformation is required [12,14]. The second generation (2G) AHSSs, which include twin-induced plasticity (TWIP) steels, austenitic stainless steel (AUST.SS) and lightweight steel with induced plasticity (L-IP) were developed as a solution to improve ductility. These 2G steels show an excellent combination of strength and ductility, which is due to the TWIP effect that is achieved in the austenitic structure that is stabilized by

alloying with Mn. However, the high cost of alloying elements significantly increases the production cost of these grades which makes them not economically viable for the automotive industry [5]. Third generation (3G) AHSSs are currently under research and development. They are projected to exhibit improved combinations of strength and ductility compared to the first-generation and lower costs than the second-generation AHSS. Medium-Mn TRIP, quench and partitioning (Q&P), and carbide-free bainite (CFB) steels are examples of the 3G-AHSS grades [7]. Overall, there is still a persistent need to balance the requirements for high strength, high elongation, and reduced cost, especially from alloying. This challenge also extends to ultra-high-strength steels (UHSS) such as PHS, where higher ductility is necessary for formability during hot stamping to achieve complex shapes and enhance energy absorption during crash events. Additionally, these steel grades must possess a high level of weldability along with their mechanical properties to remain competitive for selection in the automotive industry.

2.2 Hot Stamping Process

Of the numerous processing routes for manufacturing AHSS materials and components, hot stamping has become a prominent process to manufacture automotive structural parts. The process is adopted to simultaneously form and quench the steel part, which is required for anti-intrusion automotive components [15]. The hot stamping process is also referred to as press hardening or hot press forming (HPF) and has proven to be effective in producing UHSS for automotive body-in-white structures. Originally patented by Plannja, a Swedish company, the technology was first adopted by Saab Automobile AB in 1984 and has since then grown from only 3 million hot stamped parts in 1987 to over 107 million parts/year in 2007 [16,17].

Hot stamping can be classified as direct hot stamping and indirect hot stamping. During direct hot stamping, the as-received material is heated above the A_{c3} temperature to the austenitic phase, which is usually between (880-950 °C) depending on the chemical composition of the steel and then held in a furnace for about 4-10 minutes to allow complete transformation to austenite. Afterwards, the heated metal is quickly moved to the die to be formed and quenched in a die at a cooling rate of at least 25-30 °C/s until its temperature is below the martensitic transformation finish temperature, M_f [18,19]. Conversely, the indirect process cold forms the metal sheet to approximately 90 – 95% of the final shape in a conventional die set before heating to austenitic temperature and finally quenching in a die [20]. A schematic describing both processes is shown in Fig. 2.2.

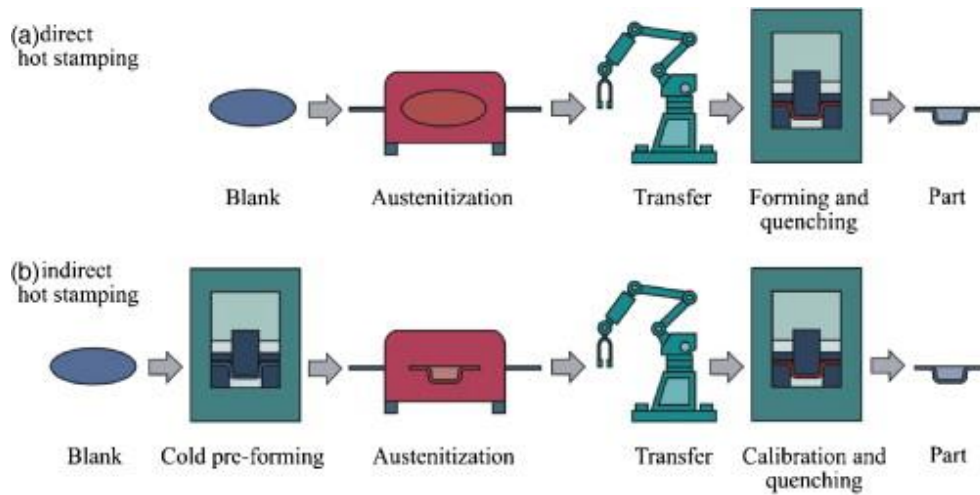


Fig. 2.2: Schematic showing the basic hot stamping process for (a) direct hot stamping (b) indirect hot stamping [16].

2.2.1 Press Hardened Steel (22MnB5)

The use of quenched boron steel has been adopted by the automotive industry to make parts with complex shapes and good anti-intrusion properties [21]. Compared to other Mn-B alloyed steels such as 27MnCrB5 and 37MnB4, 22MnB5 is the most widely used automotive hot-stamped steel. The 22MnB5 has a ferritic-pearlitic microstructure before hot-stamping that transforms into martensite after hot-stamping [22]. The grade name “22MnB5” was adopted based on the alloying constituents of the steel where 22 indicates the approximate carbon content (~ 0.22 wt%) while Mn and B are major alloying elements that enhance hardenability [20]. The use of boron (10-30 ppm) in the alloy plays a major role in the hot stamping process by increasing the hardenability of the steel (i.e. the ease of forming martensite). This occurs by the segregation of boron to the austenite (γ) grain boundary (GB), which delays the nucleation of ferrite at the γ -GB as shown in Fig. 2.3 [23]. It is worth noting that there is a critical amount of boron addition, which results in the formation of BN or borocarbide such as $\text{Fe}_{23}(\text{C},\text{B})_6$ [24]. It has been reported that these phases form early during isothermal treatments. Due to their high diffusion rate to the GB, they act as nucleation sites for ferrite [25]. Thus, the addition of alloying elements such as Ti and Nb acts as strong nitride and carbide formers, scavenging the free C and N in the steel, thereby freeing boron to segregate and occupy ferrite nucleation sites preserving the hardenability effect as illustrated in Fig. 2.3. Additionally, manganese significantly contributes to hardenability by delaying the decomposition reactions of austenite [18].

The use of 22MnB5 has evolved over the years and is now mostly adopted for passenger safety-related anti-intrusion parts and structural reinforcements such as side impact beams, bumpers,

B-pillars, tunnels and roof rails [15,26] as shown in Fig. 2.4. These parts are designed to resist deformation during impact, thereby providing safety for passengers. 22MnB5, which is also referred to as press-hardened steel (PHS1500) was the primary material under investigation in this research.

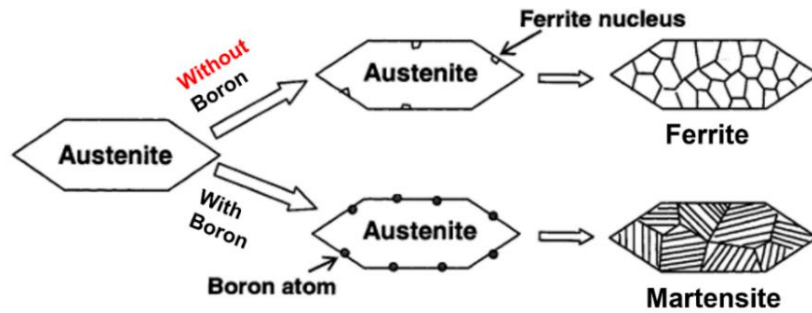


Fig. 2.3: Schematic showing boron hardenability effect on the microstructure of steel [24].

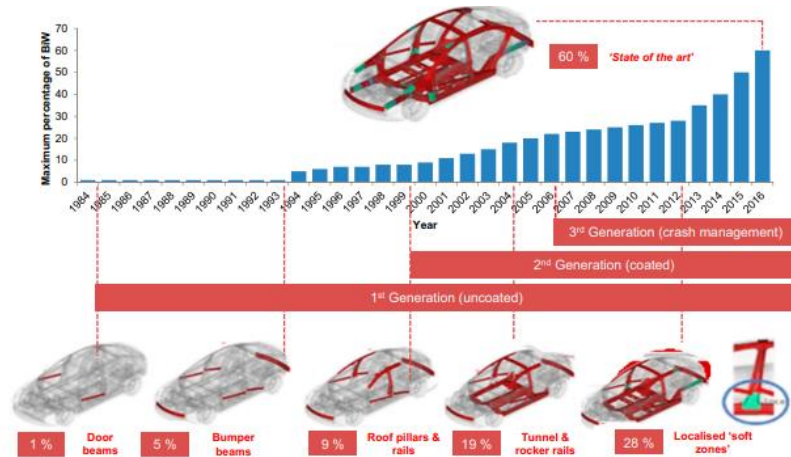


Fig. 2.4: Evolution of the application of hot-stamped sheet to the body-in-white structure of a car [17].

2.3 Resistance Spot Welding (RSW)

Invented in 1877 by Elihu Thomson, resistance spot welding is now recognized as one of the most rapid and economical joining methods for sheet metal [27,28]. The automotive industry has adopted this joining technology because of its unique advantages, which include its reliability, suitability for automation, high production efficiency and low cost. As such, the RSW is used for producing about 3000-5000 spot welds needed in a modern vehicle body-in-white structure [29].

The RSW process involves the simultaneous application of sufficient force and electrical current during a relatively short time. Generally, the RSW process can be described in four stages as summarized in Fig. 2.5. Stage one includes squeezing two or more sheet metals

together between two electrodes by applying a predetermined amount of force. This stage is necessary to allow intimate contact at the faying interface to ensure electrical conductivity across the sheets. During the second stage, current flows through the sheet metals, which results in heat generation at the faying interface due to resistance to the flow of current. The current must be controlled as excess heat generation can result in expulsion (*i.e* the ejection of molten metal from the fusion zone). Stage three consists of the termination of the current and the solidification of the melted zone due to the cooling effect from the water-cooled electrodes. The solidified melted zone is an elliptical-shaped joint between the sheets known as the weld nugget. The release of the electrode from the solidified nugget occurs at the final stage (*i.e* stage 4).

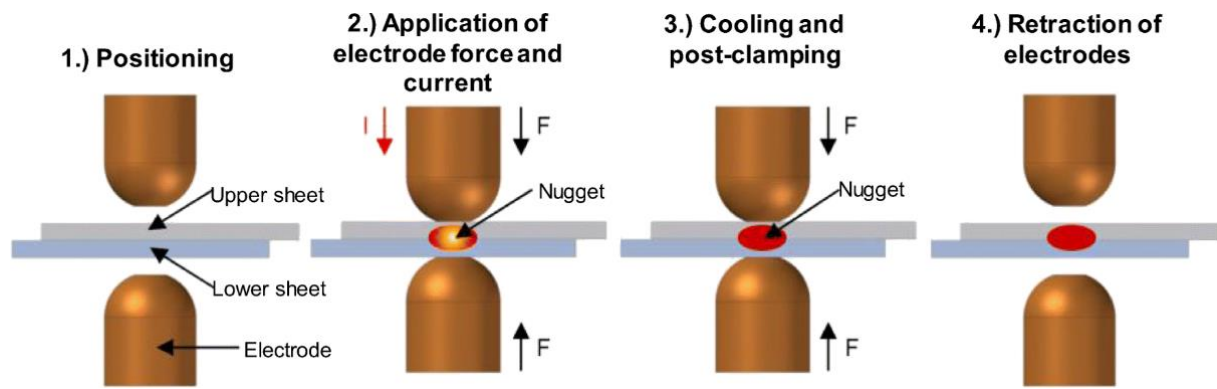


Fig. 2.5: Schematic representation of the stages in resistance spot welding [30].

Acceptable welds are commonly defined by various standards which detail common practices and procedures used for RSW of different materials. According to the American Welding Society (AWS) D8.1M standard [31], an acceptable weld must be equal to or greater than the minimum weld diameter (lower limit) as described in Equation (2.1):

$$d_{min} = 4 \sqrt{t_s} \quad (2.1)$$

Where d_{min} is the minimum weld diameter (mm) and t_s is the material sheet thickness (mm). The minimum weld diameter is set by automotive manufacturers to meet mechanical property and safety requirements. The upper limit according to the AWS D8.9M standard [32] is defined by the appearance of expulsion or severe electrode sticking on test welds. Therefore, the weldable current range is defined by the range between the minimum current (I_{min}) that produces d_{min} , and the maximum current before the appearance of expulsion or electrode sticking as illustrated by the weldable current range graph in Fig. 2.6a. A wide weldability range is preferred over the narrow lobes, because of the greater ease of producing acceptable welds based on the wide range of possible parameter combinations as shown in Fig. 2.6b.

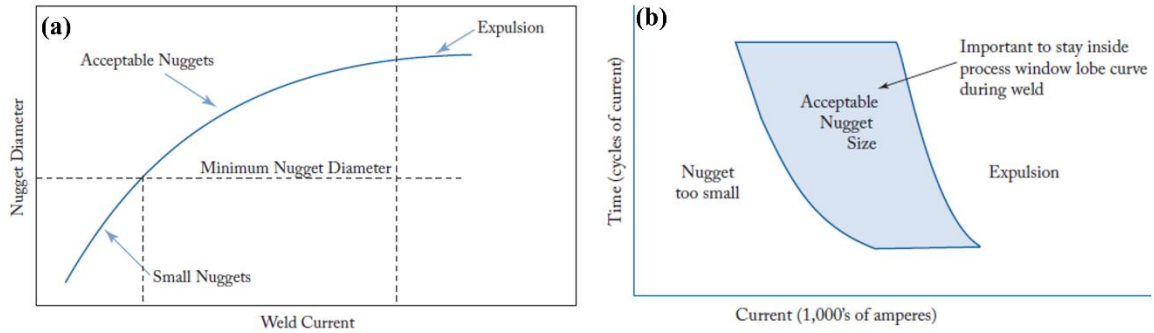


Fig. 2.6: (a) Weldable current range showing the upper and lower limit current (b) weld lobe showing weld time and current parameter combination obtained from resistance spot welding [33].

2.3.1 Principle and process parameters of RSW

Resistance spot welding is a complex multivariable process which involves electrical, magnetic, mechanical, thermal and metallurgical phenomena [34]. However, the dominating factor needed for nugget formation is the amount of heat energy during welding which is based on Joule's law as expressed in Equation (2.2(2.2) [33] :

$$Q = \int_0^t I^2 R dt \quad (2.2)$$

Where Q is the amount of heat energy generated during the process, I is the weld current, t is the weld time and R is the total resistance. The total electrical resistance is the sum of the contributions from all the resistances (bulk and contact resistance) as illustrated in Fig. 2.7a. R_1 and R_5 are contact resistances at the electrode-sheet interfaces, R_2 and R_4 are the bulk resistances of the substrate metal sheets to be welded while R_3 is the contact resistance at the sheet-sheet interface. During RSW, the heat generation is predominantly due to the contact resistance at the faying interface which is dependent on the material, temperature, and welding force used [35].

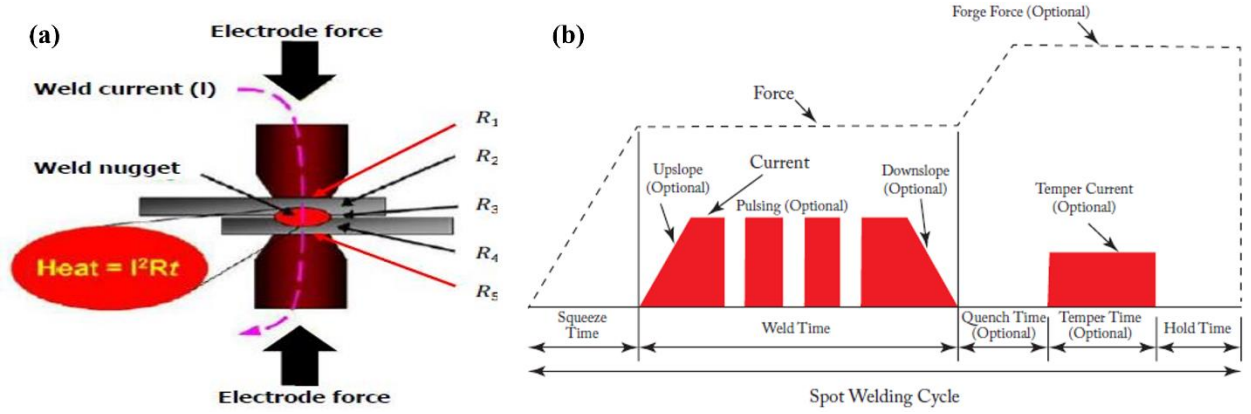


Fig. 2.7: (a) Schematic set-up for resistance spot welding showing the bulk and contact resistance (b) RSW process parameters including additional PWHT pulse [33,36].

The three most important spot-welding parameters are the weld current, weld time as expressed in equation (2) and the electrode force (higher electrode force leads to lower resistivity and heat generation). Other parameters include the squeeze time and hold time as shown in Fig. 2.7b. The squeeze time is the time interval between the initial application of the electrode force on the workpiece and the first application of current while the hold time is the time after welding when the water-cooled electrodes are still applied to the sheet to extract heat from the workpiece thereby cooling the weld. Additional parameters such as quench time, temper time and current represent in-situ PWHT parameters that can be incorporated into the RSW process.

2.3.2 Dynamic resistance curves

The electrical resistance is the primary factor influencing heat generation during the welding process. The composite resistances ($R_1 - R_5$) earlier explained changes with time and is not considered as a constant during the welding process. The acquisition of dynamic resistance is relatively straightforward as it does not necessitate the use of complicated sensors, rather, it is calculated by dividing the dynamic change in weld voltage by the current [37]. Previous studies have demonstrated significant variations in dynamic resistance over time across different material combinations, surface conditions, welding parameters, coating characteristics amongst others [38,39].

An early study by Dickinson et al., [40] delineated six stages in the dynamic resistance curve illustrated in Fig. 2.8 that characterize the resistance spot welding process. In the first stage (I), surface films, oxide layers or other contaminants present on the work piece surface break down. These surface contaminants serve as insulators and the initial contact resistance as the current is applied will be high leading to high heat generation based on Joules law. As the heat increases, these contaminants are broken resulting in a sharp drop in resistance. In stage II,

more metal-to-metal contact exists as asperities soften leading to an increased contact area for current flow and a decrease in resistance. Simultaneously, the electrical resistivity increases due to increase in temperature for the bulk resistance leading to a competition between the two mechanisms. The competition between the two mechanisms determines the α -minimum position. Eventually, the increase in contact area will be overcome by the increasing temperature effect, leading to an overall increase in resistance. Heating of the workpiece occurs in stage III resulting in an increase in temperature and resistivity. Thus, an overall increase in resistance occurs as contact resistance has been mostly eliminated and local melting begins. Three mechanisms occur at stage IV: (i) increase in resistivity due to an increase in temperature; (ii) decrease in resistance due to the formation of a molten pool as the liquid has a lower resistivity than the solid (iii) increased softening and collapse that shortens the path for current flow decreasing resistance. The β -point represents temperature stabilization resulting in nugget growth and mechanical collapse, thereby leading to a decrease in resistance. The final stage (V) is dominated by the growth of the nugget beyond the β -point resulting in a decrease in resistance as the liquid has a lower resistivity. The occurrence of expulsion in stage VI results in a drop in resistance.

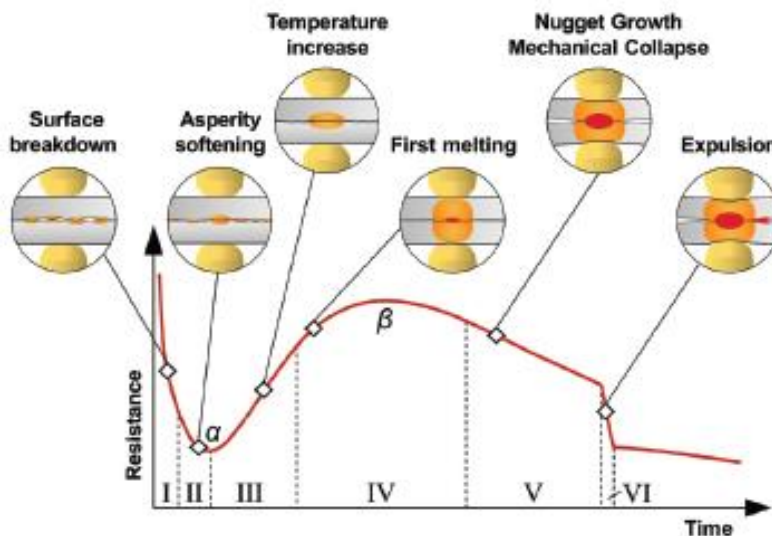


Fig. 2.8: Theoretical dynamic resistance curve showing the five stages during resistance spot welding [40,41].

The description made by Dickinson et al., [40] has been well accepted in the scientific community, however, recent research has proposed an updated evolution of resistance during RSW process. Ighodaro et al., [42] mentioned that there is a stage 0 (surface heating), where there is an initial rise in resistance at the beginning of the process. The initial rise in resistance is dependent on the coating type and applied current as shown in Fig. 2.9 for an as-received

and hot stamped steel. Recently, Xia et al., [41] found that the main factor that dictates the resistance trend is the average sheet temperature and electrode/sheet contact diameter rather than the nugget growth process as shown in stage V. Nonetheless, Xia's interpretation is still a subject of research. It is worth noting that the dynamic resistance curves are also used for weld quality control [43], characterizing nugget growth [34,44], and to determine the magnitude of expulsion [45,46]. However, no work has explored this technique to predict phase transformations in spot welds, particularly during in-situ PWHT.

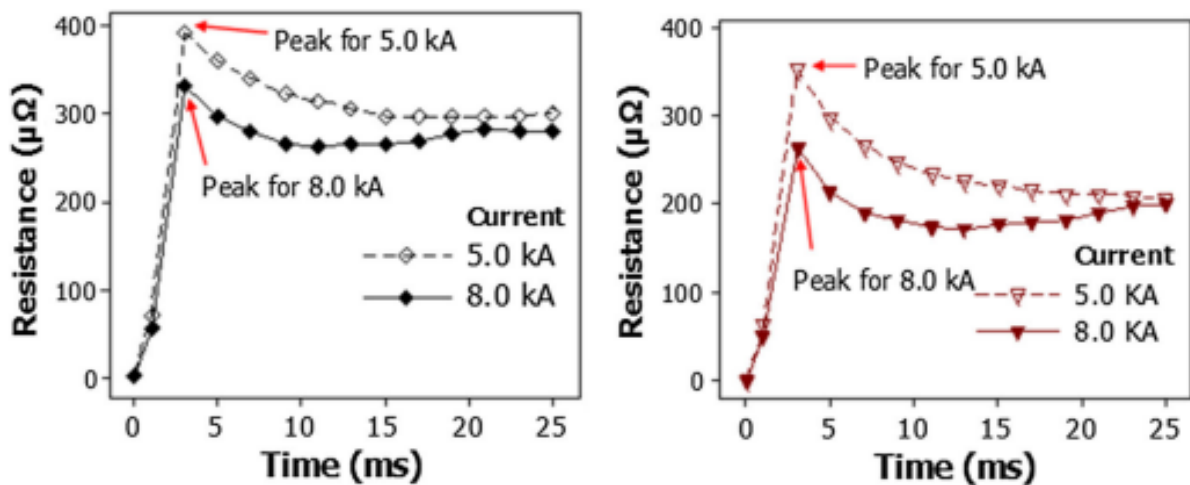


Fig. 2.9: Early-stage dynamic resistance for (a) hot-stamped Al-Si steel (b) Hot-stamped galvanized steel. Initial resistance peak varies with welding current and coating type [42].

2.4 Evaluation of resistance spot weldability

After welding, assessing the properties of the joint can either be done qualitatively or can be quantified via destructive or non-destructive testing methods. Destructive testing, such as tensile test is mostly used to obtain the load bearing capability of the joint and the energy absorption capability. Non-destructive methods such as online resistometry technique [28,34,40], acoustic microscopy [47] and x-ray scans [48] have also been used to examine nugget development and internal structures such as voids. The major criteria used to evaluate the quality of spot welds are the physical features, metallurgical attributes, mechanical performance and the failure mode of the joint [49]. The following subsections will expand on each of these metrics.

2.4.1 Physical features

The physical features of spot welds refer to the visible attributes revealed via cross-sectioning or separation of the joint. Several physical features such as the fusion zone size, voids in the FZ, shape of the notch (i.e., interface where the two sheets being welded come into contact),

and electrode indentation depth can be identified as shown in Fig. 2.10. The nugget diameter, which is the diameter of the weld nugget at the sheet/sheet interface has been regarded as one of the most important physical features in evaluating the quality of spot welds [29]. The nugget diameter has also been identified to be directly related to the load-bearing capacity and energy absorption capability of the joint [50]. Other geometric features associated with the process such as the shape of the notch and the indentation depth have been identified as regions with high-stress concentrations, which may affect joint performance during loading [29,51].

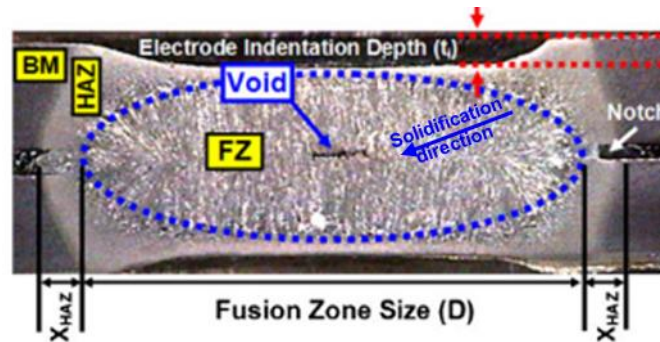


Fig. 2.10: Macrostructure of resistance spot welds showing the physical features [29].

The presence of voids in the FZ of a weld shown in Fig. 2.10 is another critical physical feature. Vijayan et al. [52] mentioned that voids are formed as a result of interference between axially dendritic growth with a higher solidification rate (in the thickness direction) compared to the interdendritic feeding (growing in the diametric direction of the nugget) during the final stage of solidification. Another explanation could be due to the formation of gas pores due to the vapourization of the coating during the welding schedule creating vapour pockets or bubbles that are trapped within the molten pool. This phenomenon was observed by Chen et al. [26] where the evolution of pores in uncoated and Al-Si coated PHS was studied. No pores were observed in the uncoated PHS while a smooth surface and circular void, indicating gas pores, were observed in Al-Si coated PHS. It is worth mentioning that voids reduce the effective volume of the nugget (*i.e* difference between the nugget volume and void volume), which may affect the load-bearing capacity of a joint [52].

2.4.2 Metallurgical features of the joint

The heat generation during the RSW process creates a variation in the thermal history and microstructure from the base metal to the FZ of the joint. An illustration of the metallurgical mechanisms within various regions of the joint is shown in Fig. 2.11a while the resulting microstructures after the RSW process are shown in Fig. 2.11b. The FZ experiences the highest

temperature ($T > T_{\text{liquidus}}$) during RSW and predominantly consists of a martensitic phase. The martensitic transformation is due to the high cooling rates associated with the RSW process which is in the order of 10^3 to 10^5 °C/s [53].

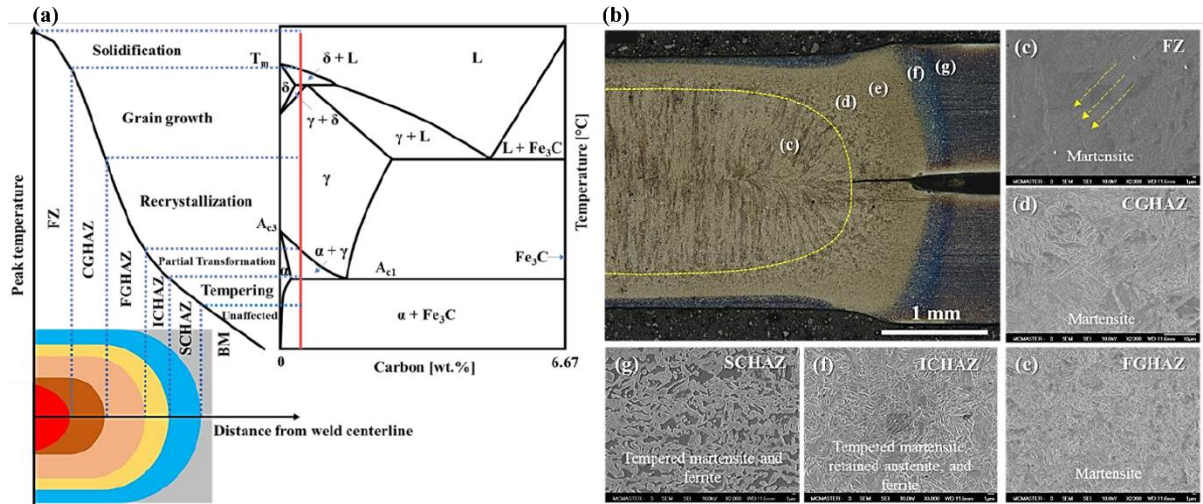


Fig. 2.11: (a) Temperature distribution of the different regions in a spot welded joint with corresponding temperatures on an equilibrium Fe-C phase diagram [54] (b) Optical microscopy image of the different regions in a spot welded joint; Scanning electron microscopy (SEM) images of (c) FZ (d) CGHAZ (e) FGHAZ (f) ICHAZ (g) SCHAZ [55].

It is worth noting that the temperature in the heat-affected zone (HAZ) is below the T_{liquidus} and the peak temperature in the HAZ decreases with increasing distance from the FZ. The HAZ can be divided into four regions namely the coarse-grained HAZ (CGHAZ), fine-grained (HAZ), inter-critical HAZ (ICHAZ) and sub-critical HAZ (SCHAZ), as shown in Fig. 2.11(d-g) respectively. The CGHAZ experiences a temperature range that is less than the liquidus temperature but greater than the A_{c3} temperature (i.e $A_{c3} < T < T_{\text{liquidus}}$). The temperature regime in the CGHAZ provides sufficient thermal energy for austenite grain growth compared to the temperature at the fine-grained HAZ (FGHAZ), which is also greater than the A_{c3} (i.e $T > A_{c3}$) but at a lower temperature for grain growth. The temperature at the inter-critical HAZ (ICHAZ) is within $A_{c1} < T < A_{c3}$ and mostly consists of martensite and ferrite after the RSW process while the sub-critical HAZ (SCHAZ) experiences $T < A_{c1}$. Typically, the temperature at the SCHAZ is sufficient to promote martensite tempering leading to a reduction in hardness while the base metal (BM) microstructure remains unaffected by the welding process [29,55–57].

2.4.3 Mechanical performance

Evaluation of the mechanical properties of resistance spot welds is usually obtained under static/quasi-static, fatigue and impact loading conditions. Common testing geometry of single

spot welds includes the tensile-shear test (TST), cross-tension test (CTT) and coach peel test (CPT). These tests (TST, CTT and CPT) evaluate the load-bearing capacity of the joint when subjected to shear, tensile and bending loads respectively as shown in Fig. 2.12(a-c) [29]. In recent years, combined loading conditions of spot welds have also been examined via testing KS-II coupons [58] test while component testing of spot welds has also been assessed through the Caiman test [59]. Generally, a load-displacement curve can be acquired from these tests as shown in Fig. 2.12d and the following parameters can be obtained:

- Peak Load, P_{max}
- Elongation at peak load, L_{max}
- Energy at peak load, W_{max} obtained by calculating the area under the curve to peak load
- Failure energy at fracture, E_{max} obtained by calculating the under the curve to failure

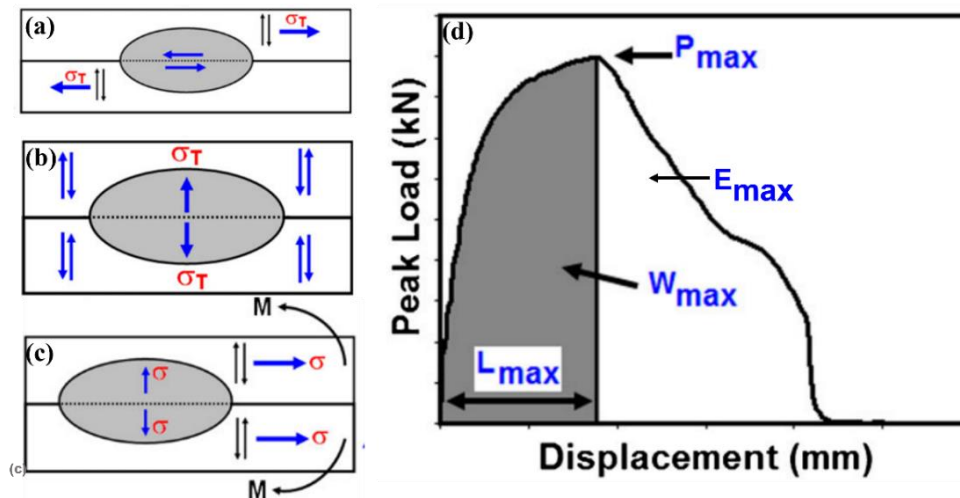


Fig. 2.12: Schematics describing loading condition for (a) TST (b) CTT (c) CPT; and (d) load-displacement curve showing extracted parameters [29].

Spot welds show varying responses under various loading conditions. In other words, a discrepancy in the peak load and/or energy absorption capability of spot welds is expected when subjected to different loading conditions. Pouranvari et al., [60] have identified a couple of other factors that control the mechanical performance of spot welds. Some of those factors include the FZ size, sheet thickness, microstructural features, welding parameters, and post-weld treatments such as tempering, paint baking amongst others.

2.4.4 Failure mode

Resistance spot welds exhibit different failure modes and features after loading. Three failure modes are commonly cited in the literature, namely: interfacial failure (IF), partial interfacial failure (PIF) and pull-out (PO) failure modes as shown in Fig. 2.13. These failure modes have

been associated with the load-bearing capacity and energy absorption capability of the resistance spot welds [29]. Interfacial failure is observed by the propagation of failure through the FZ as shown in Fig. 2.13a and is usually considered undesirable since the failure occurs rapidly. However, recent studies have shown that IF failure of a joint during the TS test can demonstrate satisfactory load-bearing capacity [61]. Hence, the load-bearing capacity of the AHSS spot welds during the TS test has been recommended as the primary focus for evaluating the quality of spot welds rather than the failure mode [62]. In the case of PIF, the failure initiates and propagation of cracks first goes through the FZ and then is redirected through the sheet thickness direction as shown in Fig. 2.13b. Pullout failure occurs when the weld nugget is pulled out of one sheet and remains as a button on the other sheet as shown in Fig. 2.13c. Generally, the PO failure mode is preferred because of higher plastic deformation and energy absorption capability, which results in the most satisfactory mechanical properties. It is worth noting that a weld can transition from IF to PO failure mode depending on the weld nugget size, metallurgical characteristics and geometrical factors [63,64]. Understanding and controlling these factors are essential for optimizing the mechanical performance of spot welds, ensuring they exhibit the desired failure mode under various loading conditions.

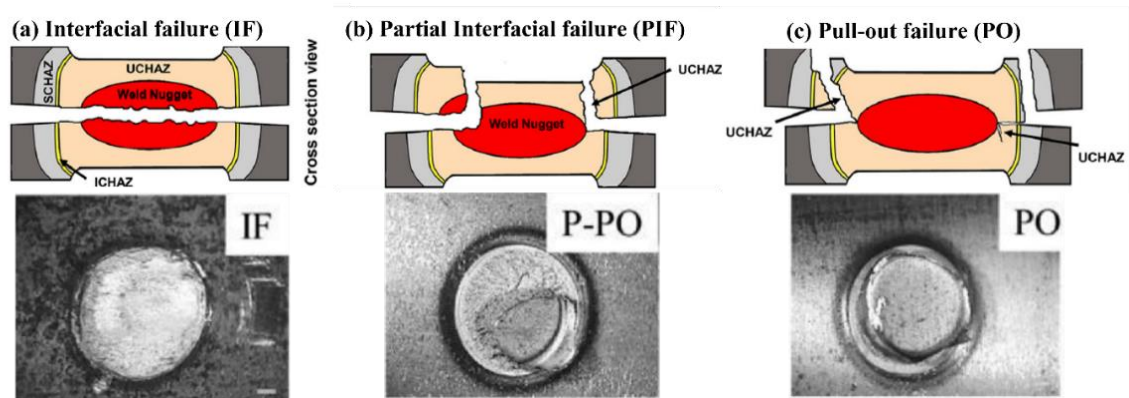


Fig. 2.13: Schematics of the major failure modes of RSW: (a) Interfacial failure (b) partial interfacial failure (c) pull-out failure [51,58].

2.5 Martensite Hierarchy

In 1895, the term “martensite” was named after a German metallurgist, Adolf Martens, which followed the traditional nomenclature such as ferrite, pearlite and cementite introduced by former metallographs [65]. Martensitic transformation is a diffusionless process that results in a change in crystal structure from γ face-centered cubic (FCC) to a body-centered tetragonal (BCT) structure, without altering the composition in an iron-carbon system [66]. It is mostly denoted as α' , as it is derived from the body-centered cubic (BCC) ferrite (α). However, the

solubility limit of carbon in the BCC structure is exceeded, and the lattice undergoes strain to accommodate the excess carbon. The strained lattice transforms into a BCT structure where the “c” parameter of the unit cell is greater than the other two “a” parameters. The degree of tetragonality (c/a) is known to increase with an increase in carbon content [67,68].

Martensite formation occurs by a shear or displacive mechanism, wherein the atoms move cooperatively and almost simultaneously in contrast to the atomic movement observed in diffusion-based transformations [67]. The displacive mechanism is achieved through rapid cooling or quenching to avoid diffusion-dependent transformations such as pearlite. However, the exact cooling condition to form martensite in steel is dependent on the carbon content, as well as other alloying constituents [69]. Martensite starts to form in a given alloy at the martensite start temperature, M_s and is mostly completed at the martensite finish temperature, M_f . The M_s temperatures can be estimated based on the empirical calculation as shown in Equation (2.3) [70]. In general, the M_s temperature decreases as the carbon content increases as shown in Fig. 2.14a.

$$M_s(^{\circ}\text{C}) = 551 - 474C - 33\text{Mn} - 11\text{Si} - 17\text{Cr} - 17\text{Ni} - 21\text{Mo} - 11\text{W} \quad (2.3)$$

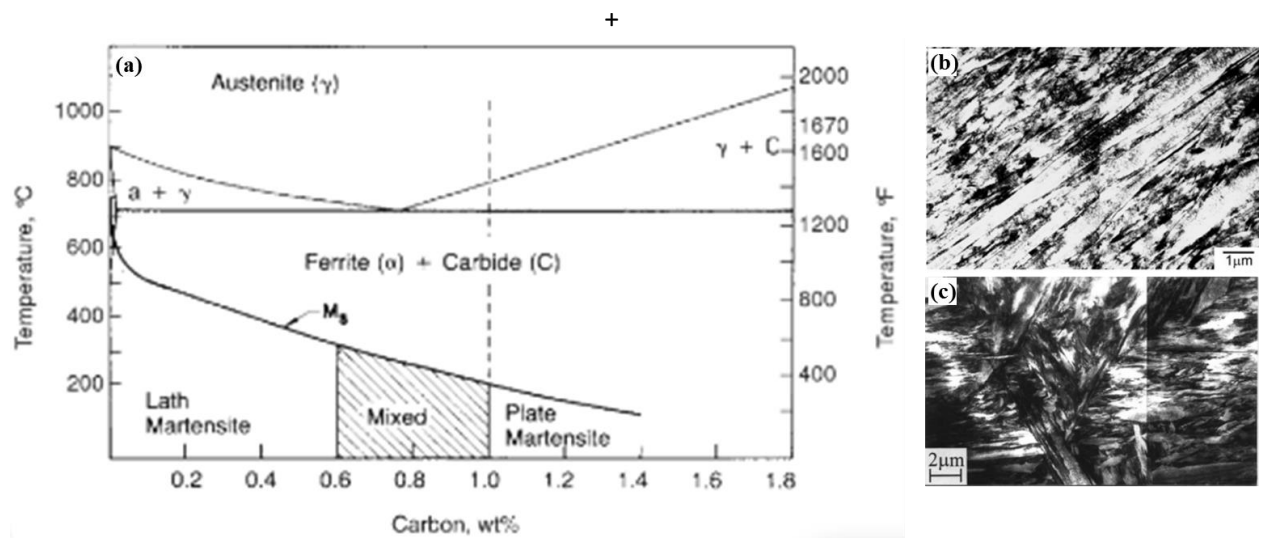


Fig. 2.14: (a) Ranges of lath and plate martensite formation in iron-carbon alloys [5,6] (b) TEM image of lath martensite in 0.20 wt% C steel [71] (c) TEM image of plate martensite in 0.6 wt% steel [72].

Martensite in carbon steels tends to manifest in one of two morphologies- lath and plate martensite or a mixture of both as illustrated in Fig. 2.15b and c. These morphologies differ in their structure, unit shape and formation process. It has been reported that the lath martensite mostly forms in low to medium carbon steel compared to the plate-shaped martensite that forms in high-carbon steels (>0.6 wt %) [73]. Martensite is known to have a distinct morphological

hierarchy where a single parent austenite grain can evolve into 24 lath martensite variants (V1-V24). These variants under ideal conditions are grouped into 4 packets with each comprising six variants having the same closed-packed parallel plane relationship with the austenite [74,75]. These variants organize themselves into variant pairs in structures known as “blocks” which are separated by high-angle grain boundaries as shown in Fig. 2.15a and b, while the misorientations across lath boundaries are low angles that vary within a few degrees [76]. Since the block is the smallest unit with high-angle boundaries, the block size is considered as the effective grain size of martensite. It is worth noting that the different degree of misorientation angle of various substructure boundaries contributes to the strength of the lath martensite [77,78].

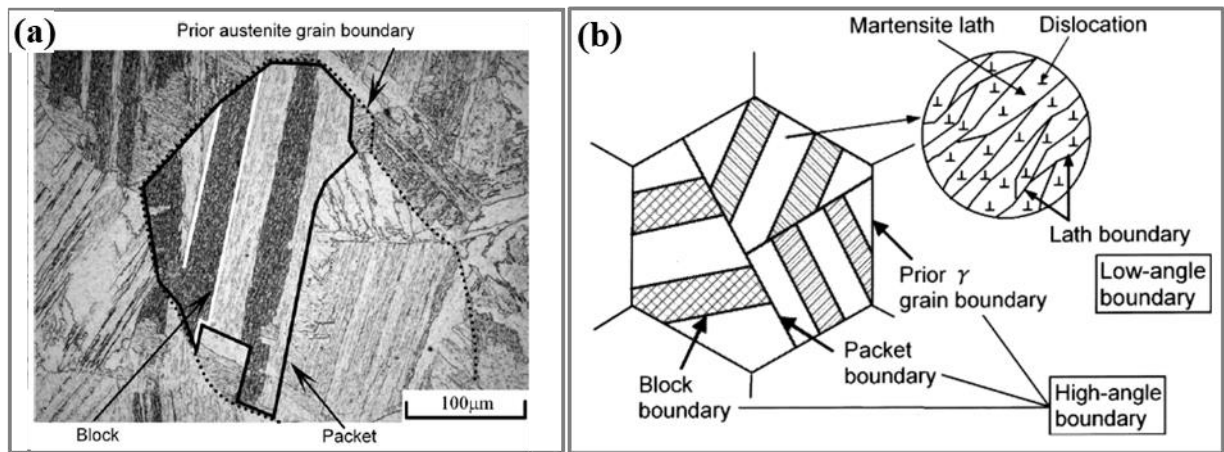


Fig. 2.15: Optical micrograph showing the martensite hierarchy from PAG, block and packets [72] (b) schematic illustration of martensite hierarchy showing various boundaries [79].

2.6 Mechanical properties of PHS spot welds and related challenges

The mechanical properties of PHS spot welds are influenced by a complex interplay of factors mostly from the welding parameters which determines both the physical features (such as the weld size, indentation amongst others) and the microstructural characteristics. PHS base metal has a martensitic microstructure after hot stamping as established in section 2.2, likewise, the FZ of PHS forms martensite due to the rapid cooling rates inherent with the RSW process. The martensitic phase has high hardness which can lead to increased brittleness and more susceptibility to cracking when subjected to loading [80]. Particularly, PHS spot welds are known to show low ductility and toughness, resulting in reduced energy absorption during crash events, which is a concern for automotive safety.

The energy absorption capability of spot welds can be traced to the ability of a structure to retard crack propagation during loading. It has been established in the literature that there is

competition between the stress concentration associated with the weld notch, which promotes failure from the martensitic coarse-grained UCHAZ, and the strain localization related to the HAZ softening, which helps failure from the SCHAZ [51]. It has been shown that necking in SCHAZ occurs and crack preferentially propagates along the tempered martensite microstructure in that region resulting in pull-out failure mode [51,81,82] as shown in Fig. 2.16a. However, for PHS spot welds, the stress concentration at the notch dominates and the crack tends to propagate from the notch at the sheet-sheet interface during loading as shown in Fig. 2.16b and c. Thus, the microstructure in front of the notch is more important in improving PHS weld toughness. Recent studies have also observed a softened region along the FZ boundary of PHS welds known as “halo ring” [80,83,84] as shown in Fig. 2.16(d-f). It was reported that the crack propagates along the softened region along the fusion boundary due to shear localization at the softened region, and not localised thinning in the softened HAZ (SCHAZ) [84]. This halo ring has been reported to deteriorate the weld load bear capability and result in brittle failure [55]. More studies have been conducted to eliminate the halo ring and it was found that both post-pulsing [55] and paint-baking [85–87] are effective in eliminating the halo ring and intergranular failure.

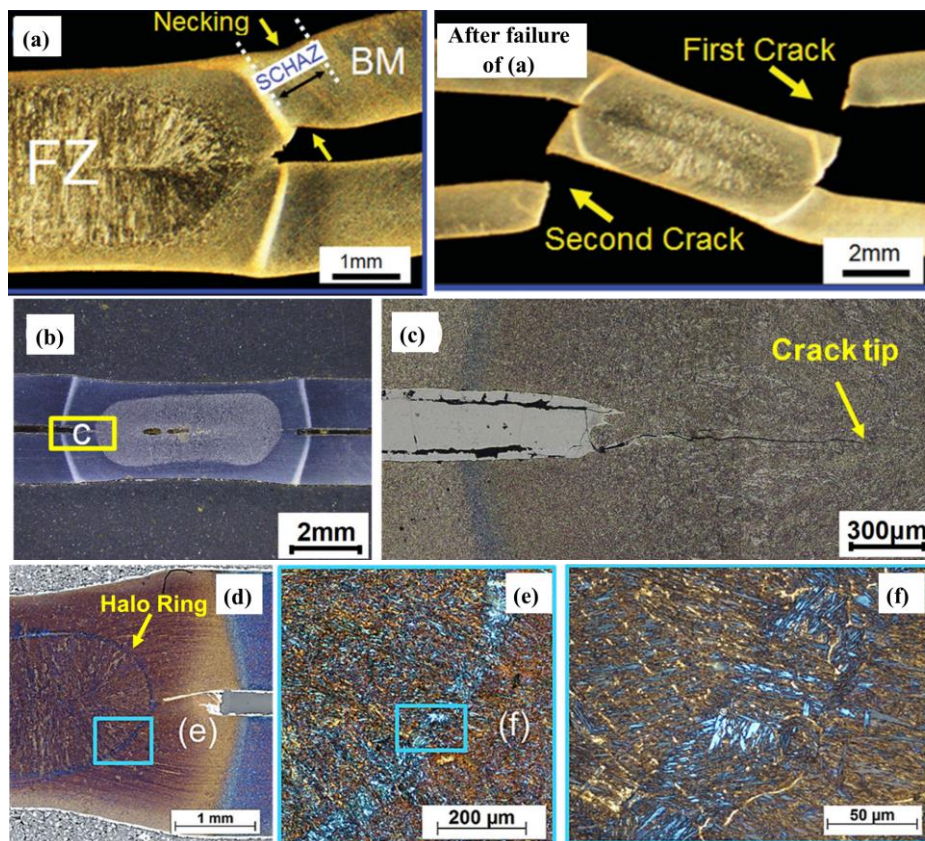


Fig. 2.16: (a) Necking of the SCHAZ in MS1400 [81] (b,c) Crack propagation from notch for PHS welds [26] (d,e,f) halo ring at the fusion boundary of PHS welds [80].

A few other studies have primarily focused on improving the weldability of PHS, with limited research on microstructural modification aimed at enhancing the weld mechanical properties. For instance, Chen et al., [88] found that using a stepped current pulse schedule retards expulsion, thereby enabling the growth of the nugget size. Although they observed a 12% increase in TS strength, no microstructural changes were reported, but rather only an increase in nugget size. In a follow-up study, the same authors [89] applied a tempering pulse after the stepped current pulse and observed a reduced TS strength of 8% after the post-weld schedule, as shown in Fig. 2.17a. However, they noted a change in failure mode from IF to PO, as shown in Fig. 2.17b and c. It should be noted that there was no microstructural tempering evidence in this study, and the selected welding parameters were significantly long (cool time of 2s and post-weld time of 1s), which is undesirable for the automotive industry. Therefore, optimizing the post-weld parameters for effective tempering is crucial. Moreover, other pathways to improve the properties of PHS welds need to be explored. The following sections will examine various metallurgical pathways that can be used to modify the weld microstructure for improved mechanical performance.

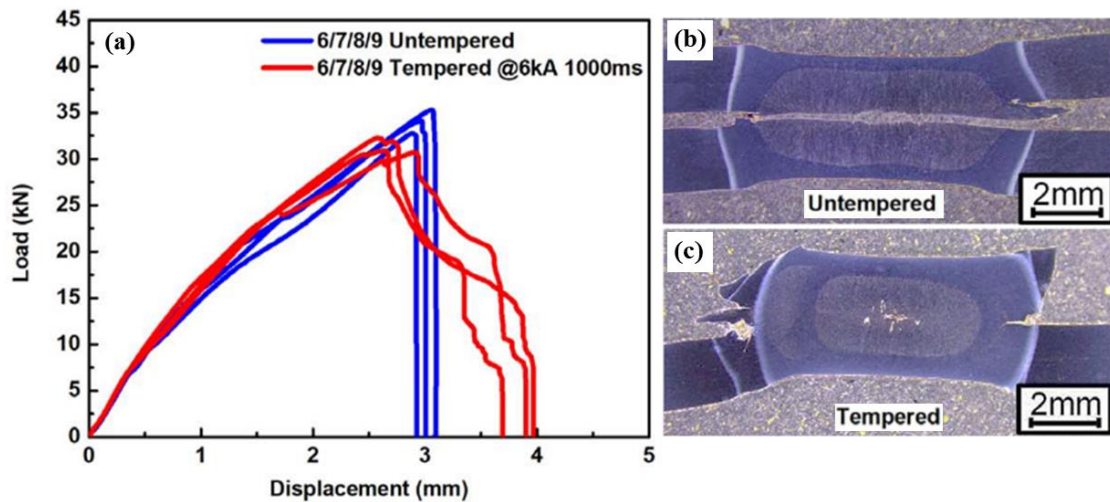


Fig. 2.17: (a) Load-displacement curves after tensile shear testing of PHS spot welds; failure mode (b) before tempering (c) after tempering [89].

2.7 In-situ PWHT: Tempering

Tempering heat treatment is a well-known metallurgical phenomenon, which has been adopted to reduce the hardness of martensite and improve ductility. By controlling the tempering parameters, a balanced combination of strength and toughness can be achieved, making the steel more suitable for a wide range of applications. Martensite tempering has been described to occur in four stages depending on the temperature regime as described in the literature

[90,91]. In the first stage (~80-180 °C), segregation and redistribution of carbon atoms occur at lattice defect along with the formation of carbon clusters and transitional carbides such as ϵ -carbide ($\text{Fe}_{2.4}\text{C}$). It is worth mentioning that the formation of ϵ -carbide occurs for steel with carbon content greater than 0.2 wt.%. At temperatures between 200-300°C (second stage), the retained austenite which is sometimes present at the interlath area of the martensite structure decomposes into ferrite and cementite. In the third stage (250 to 350 °C), segregated carbon and transitional carbides transform into cementite (Fe_3C). At higher temperatures close to the A_{C1} (stage four), cementite becomes metastable and the formation of other alloy carbides (such as Mo, W, Nb if present in the steel) occurs.

In the case of spot welds, tempering is known to be effective in alleviating the problem of brittle fracture by reducing weld hardness and improving weld ductility when done using the appropriate thermal cycle [92,93]. Tempering can be performed in two ways. First is isothermal tempering which involves slow heating to a particular temperature, long holding time at that temperature and slow cooling; second is non-isothermal tempering which is commonly adopted during welding. Non-isothermal tempering involves rapid heating, and negligible holding time at tempering temperature followed by rapid cooling [94]. Whichever approach is used, the tempering temperature must be below the A_{C1} transformation temperature to avoid the formation of austenite. Moreover, a sufficient temperature must be attained to allow the precipitation of solute carbon atoms from the martensitic phase [95].

During RSW, tempering can be achieved by applying an additional post-weld current pulse after the weld nugget has solidified by holding the electrodes in place long enough to sufficiently quench the martensite thereby eliminating the need for any additional equipment [95]. Hence, the cool time between the main pulse and the tempering pulse must be adjusted so that the temperature of the weldment is below the martensite finish temperature (M_f) before applying the tempering pulse. Typically, the time needed for the completion of martensitic transformation reported in the literature ranges from (1000-2000) ms [48,96–98], which was obtained either by finite element (FE) simulation [97], analytical model [53,70] or experimental procedures [93]. It is worth noting that the selection of cool time, tempering current and temper time during in-situ RSW post-pulsing is dependent on the chemical composition and/or material thickness [99].

Optimizing the post-weld parameters to achieve effective tempering for different AHSS grades has been studied by various researchers. Nikoo et al. [100] reported that three outcomes are

possible depending on the post-weld current level: (i) tempering of martensite at $T < A_{c1}$, thereby reducing its hardness; (ii) refining of martensite packets at $T > A_{c3}$, thereby improving toughness; and (iii) remelting of the nugget at $T > T_{\text{liquidus}}$ with a possible reduction in grain boundary impurity segregation. The reduction in hardness has been a consistent observation due to the decomposition of the martensitic structure. However, Saha et al., [91] have shown that at a lower tempering temperature below the A_{c1} , the cementite in bainite present in the base material can dissolve and form other alloy carbides (MC) that result in secondary hardening. The secondary hardening has also been reported to occur in the HAZ of resistance spot welds [61,101]. Fig. 2.18a and b show the scanning electron microscope (SEM) and transmission electron microscope (TEM) bright field image of a tempered martensite microstructure while Fig. 2.18c shows the formation of other alloy carbides that lead to secondary hardening.

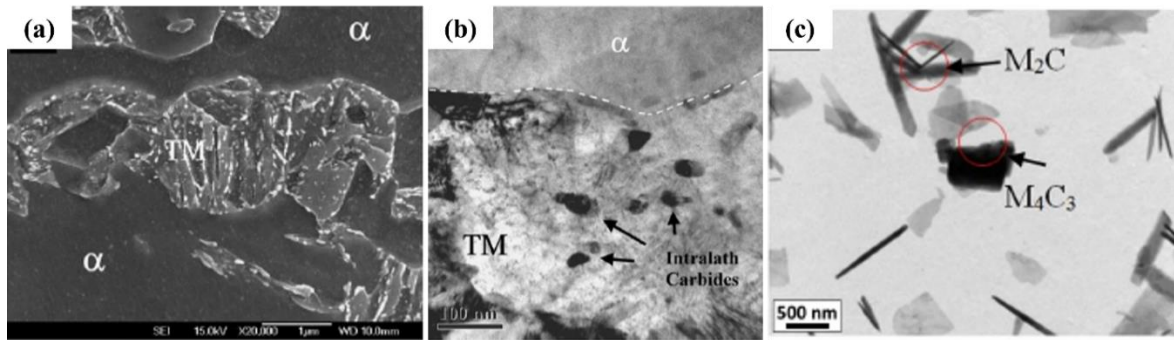


*Fig. 2.18: (a) SEM images showing tempered martensite structure (TM); Bright-field image of (b) precipitated intralath carbides and (c) alloy carbides leading to secondary hardening in DP steel [91,94] *It should be noted that these carbides were found after isothermal tempering at 500 °C for 0.5s.*

2.7.1 Effect of tempering on FZ hardness

The hardness of the FZ depends on the microstructural features, which are directly linked to the heat input and thermal cycle during the in-situ RSW tempering [48,92]. It is well known that the occurrence of martensite tempering is accompanied by a reduction in hardness due to the diffusion of carbon atoms from the supersaturated martensitic phase [95,102]. The decreased hardness can be observed when the temperature of specific regions across the joint (for instance the FZ or SCHAZ) is heated below the A_{c1} . Hence, the effectiveness of tempering the FZ can be related to the hardness of the region [53].

During in-situ RSW tempering, it is imperative to optimize the process parameters (i.e post-weld current and time) so that on reheating the temperature is below A_{c1} to achieve effective tempering. Gould and Chuko [99] developed a hardness map by varying the tempering current

and time of DP steels designated as DP690 MPa and DP960 MPa. It was observed that the hardness varied from 32 to 42 Rc depending on the temper current and time. A distinct C-curve within 68 and 82% of the expulsion current showed a range of reduced hardness values (33-39 Rc), as shown in Fig. 2.19a. However, it was observed that above 82% of the expulsion current, the microstructure retransforms to austenite. Upon quenching after the current is terminated, martensite is formed, resulting in higher hardness value above 40 Rc. On the other hand, below 62% of the expulsion current, the hardness value (i.e 40-42 Rc) were similar to those observed above the C-curve. At this stage, the temperature on reheating was insufficient for carbon diffusion from the supersaturated martensite. In addition, it can be seen from Fig. 2.19a and b that the optimal tempering parameters for both DP steels with a slight difference in chemical composition vary tremendously. Hence, optimizing the temper current and time for different grades of AHSS is vital in attaining effective tempering.

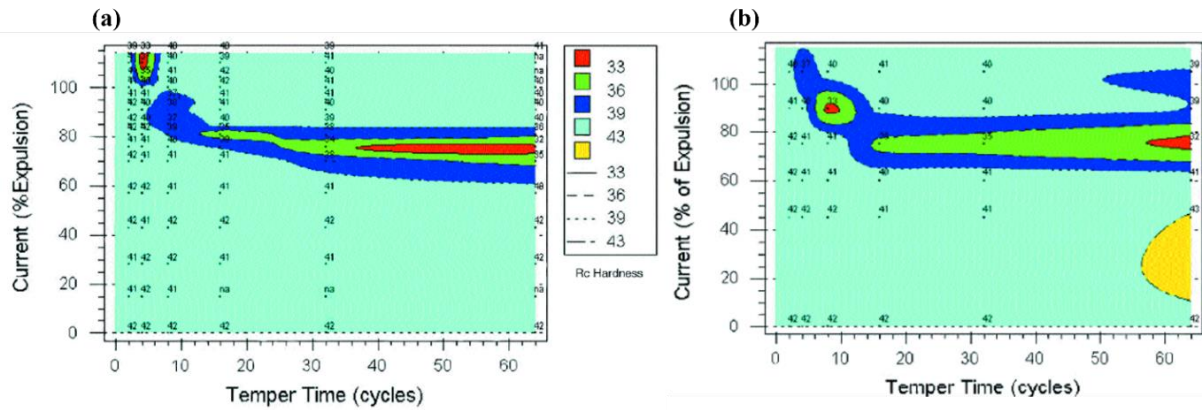


Fig. 2.19: Temper diagram showing the hardness in the FZ of resistance spot welds of (a) DP 690 MPa (0.87 mm) and (b) DP960 MPa (0.87 mm) [53].

2.7.2 Effect of tempering on mechanical properties and failure mode

Researchers have often shown the improved energy absorption capability of spot-welded AHSS after tempering the FZ. For instance, Stadler et al. [103] reported that tempering led to a soft and tough FZ which resulted in a remarkable improvement in cross-tension strength (CTS) up to 107%. Majid et al. [95] reported a 102% increase in CTS after tempering and suggested the improvement to be due to Ashby-Orowan strengthening by nano-sized carbides in the FZ. Similar results related to the formation of nano-sized carbides ($M_{23}C_6$) at the center and edge of the FZ have also been reported after in-situ tempering [70]. Soomro et al. [92] have shown that the optimum tempering condition for DP590 spot welds results in 62% and 62.3% improvement in the peak load and energy absorption capability respectively. After tempering of the FZ, the weld failure mode transitions from IF mode to PO mode after CTT.

Based on the reviewed literature, a major finding was related to the selected test (i.e. CTT) for evaluating the mechanical response of the joint after tempering. The majority of tempering studies reported improvement in CT strength (CTS) after tempering as summarized in Table 2.1 with only a few on TS properties.

Table 2.1: Previous work on in-situ RSW tempering of AHSS showing changes in mechanical properties under tensile loading and the various failure modes

Single Pulse					Tempering Pulse					
Material/ thickness	Weld Schedule	Peak Load (kN)	Energy Absorb ed (J)	Failure Mode	Optimal Temper Pulse	% Increase in Peak Load	% Increase in Energy Absorbed	Failure Mode	Test type	Ref
TBF Steel / 1.34 mm	380 ms 5.5 kA	4.0	8.8	PIF	380 ms 4.5 kA	107.5	37.4	PO	CT	[10 3]
DP 590 / 1.8 mm	560 ms 7.5 kA	10.6	161.2	PO	560 ms 7.5 kA	62.0	62.3	PO	CT	[92]
TRIP700 / 1.5 mm	300 ms 6 kA	6.3	72.0	PO		60.0	50.0	PO	CT	[10 0]
Q&P 1180 / 1.2 mm	320 ms 6 kA	4.0	--	PIF	500 ms 4 kA	70.0	--	PO	CT	[93]
Q&P 980 / 1.0 mm	320 ms 7 kA	4.7	47.0	PO	320 ms	10.2	37.5	PO	CT	[10 4]
Non- coated 22MnB5 / 1.4 mm	375 ms 6 kA	6.5	--	PIF	375 ms 5 KA	30.0	--	PO	CT	[96]
Q&P 1180 / 1.2 mm	320 ms 6 kA	4.0	--	IF	500 ms 4 kA	6.0	10.0	IF	TS	[93]
Coated PHS / 1.4 mm	360 ms 5/6/7 kA	25.3	--	PO	1000 ms 6 kA	5.5	--	IF	TS	[89]
Coated PHS / 1.4 mm	360 ms 5/6/7 kA	34	--	IF	800 ms 6 kA	-8.8	--	PO	TS	[89]

**Note: the optimum properties from all these studies had a minimum cool time of 400 ms [92] and ranged to 2000 ms [89] before applying the tempering pulse.*

Recently, Ting et al. [93] showed an increase of 70% in CTS with only a 6% increase in TS strength after tempering of Q&P1180 steel as shown in Table 2.1. They demonstrated that the TS peak load is dependent on the hardness and the size of the nugget, which is in agreement

with Chen et al., [89] finding that reported a 5% change in TS strength after tempering PHS welds. Likewise, an extensive study by Pouranvari [105] with different AHSS has shown that increased fracture toughness achieved by tempering does not improve the TS properties of welds. Hence, the CTT has been recommended to be more sensitive to the presence of a brittle microstructure compared to the TS test and has been adopted by most researchers to evaluate the joint tempering response [103,106,107].

2.8 In-situ PWHT: Recrystallization

From a broader perspective, the mechanism of recrystallization is vital in efficient microstructural design due to the possibility of stress relaxation and the nucleation and growth of new strain-free grains [108]. This process is known to significantly improve a material mechanical property by reducing internal stresses, enhancing ductility, and increasing toughness [109]. The recrystallization mechanism has been adopted as a means to refine the grain structure in the HAZ during multi-pass welding [110] and has recently been explored by applying a post-weld pulse current during the RSW process [9,111,112]. Specifically, researchers have used this concept to refine the microstructure at the edge of the FZ after solidification, by re-austenitizing the region at a temperature above the Ac_3 but below the melting point [113].

Most studies on the recrystallization phenomenon during RSW process have shown the formation of two distinct zones within the nugget with different grain morphologies. EftekhariMilani et al. [9] referred to the zone close to the periphery of the nugget as the “annealed zone” while Chabok et al. [112] called it the “rex-zone” as shown in Fig. 2.20. The formation of the Rex-zone in the weld nugget can be attained by the non-uniform temperature distribution in the nugget during the post-weld schedule. During cooling after the main weld pulse, the edge of the FZ is solidified while the inner part of the nugget remains liquid. Applying the post-pulse schedule leads to a solid state transformation at the edge of the FZ which is accompanied by the migration of prior-austenite grain (PAGs) boundary and rearrangement of dislocation configurations that results in the formation of the “rex-zone” [10]. It is noteworthy to mention that the size of the PAG can significantly dictate the mechanical properties and failure behaviour of the spot-welded joint. Overall, finer PAG has shown better mechanical performance and the grain size is governed by the applied heat input [114,115].

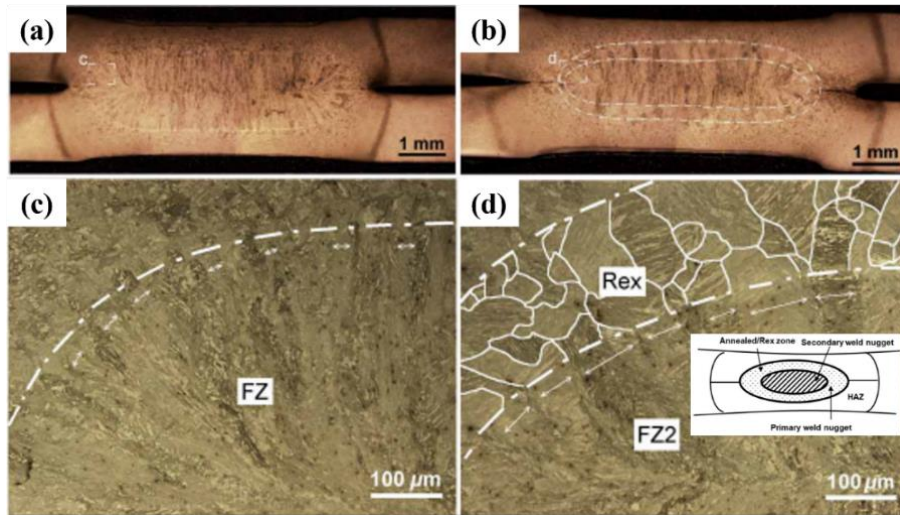


Fig. 2.20: Optical microscopy of single pulse weld (a,c) and post-weld schedule showing the recrystallized “Rex-zone” in the fusion zone (b,d) [10].

To evaluate the mechanical properties after the formation of the rex-zone, researchers have used the CTT to assess the joint response to tensile loading and understand the role of the local microstructural change at the edge of the FZ. Chabok et al. [112] showed that post-pulsing enhanced the cross-tension strength and energy absorption capability of the weld compared to the single pulse weld schedule. They reported a 27% increase in peak load and a 36% increase in energy absorption capability. The improved mechanical performance in this study was attributed mainly to lower hardness and residual strain in the rex-zone. More interestingly, the crack propagation path was observed to propagate through the rex-zone and then through the CGHAZ, which served as obstacles to crack propagation as shown in Fig. 2.21b compared to the single pulse condition in Fig. 2.21a. Similarly, Soomro et al. [113] showed that introducing a second pulse increased the energy absorption capacity of the weld by 48.79% due to the refined grain morphology from a columnar structure to quasi-equiaxed grains of ferrite and martensite in spot-welded HSLA350 steel.

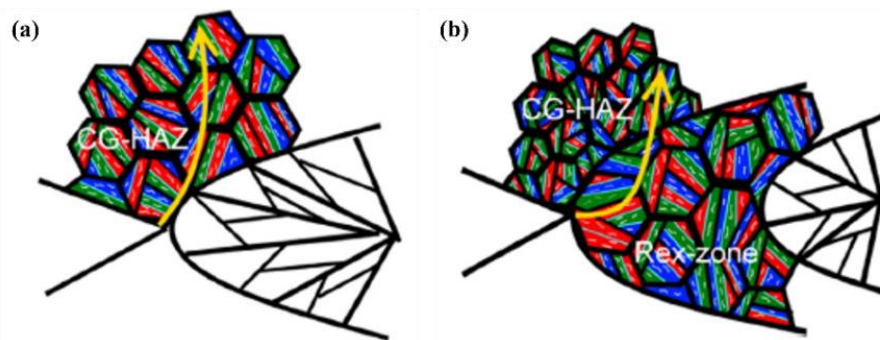


Fig. 2.21: Schematic showing the crack propagation path in (a) single pulse weld and (b) post-pulse weld with rex-zone [112].

The major advantage of this technique in terms of manufacturing and applicability to the automotive industry is the overall production time. In comparison to the temper pulse schedule which requires a cool time of approximately 1000 ms, most studies that used the recrystallization pulse employed a cool time in the range of 40 ms [10,112,115] to 200 ms [113]. The cool time provides an indication of the microstructure before the application of a post-pulse schedule. Though Stadler et al. [116] recently studied the effect of various cool times on the resulting post-pulse properties, however, in the author's opinion, more work still needs to be done to understand the microstructural evolution with various cool times. It is envisioned that this might be an arduous task considering the rapid cooling rates experienced during the RSW process, nonetheless, a detailed analysis of the microstructure with different cool times will give more insight into understanding the recrystallization mechanism during post-pulsing.

2.9 In-situ PWHT: Slow Cooling

It is well known that the high cooling rates (on the order of 10^3 to 10^5 °C/s) associated with the RSW process promote martensitic transformation in the FZ [53]. The hard martensitic phase impairs the toughness of the nugget by providing an easy path for crack propagation [48]. Another pathway to improving the energy absorption capability of the weld is by tailoring the thermal cycle during the RSW process such that martensitic transformation is avoided. This can be achieved by the addition of in-situ PWHT pulse(s) to the weld schedule to cool the weld below the critical cooling rate (CCR). The CCR is the minimum cooling rate above which there is insufficient time for C and Fe diffusion, which would otherwise allow the formation of softer phases such as pearlite or bainite. When the cooling rate exceeds the CCR, the steel will transform entirely to martensite, which is an unstable microstructure with high dislocation density [117]. In other words, the cooling rate must cross the nose of the continuous cooling transformation (CCT) curve for a particular steel, as shown in Fig. 2.22 (CCT for PHS), to prevent a structure from being entirely martensite. The CCR for different steels can be obtained based on the chemical composition of the steel (in wt%) as expressed in equation (2.4) [118]:

$$\log CCR = 7.42 - 3.13 C - 0.71 Mn - 0.37 Ni - 0.34 Cr - 0.45 Mo \quad (2.4)$$

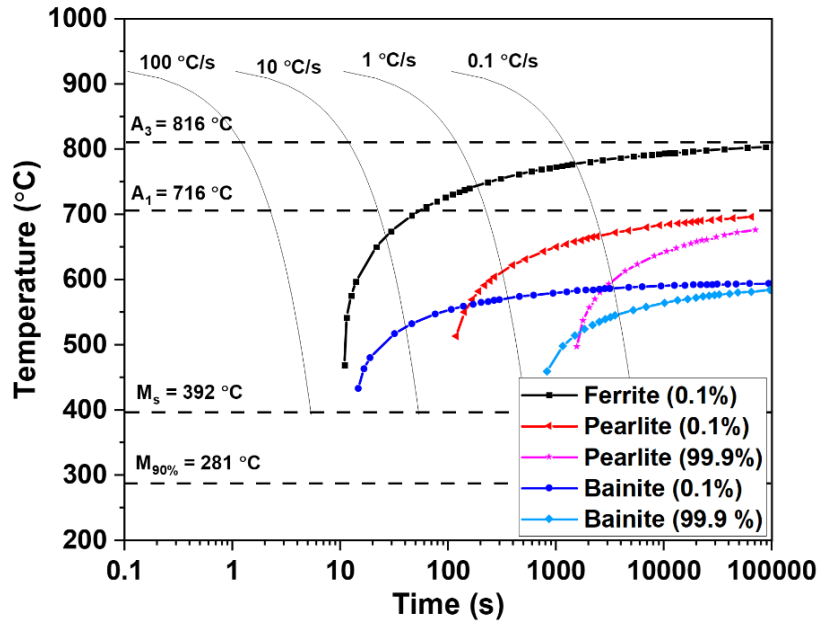


Fig. 2.22: Continuous cooling transformation (CCT) curve for PHS calculated from JMATPRO® with CCR around 27 °C (Unpublished work).

Khan et al. [119] explored this technique to slowly cool the FZ of a spot-welded DP600 AHSS. Numerical simulations were performed to simulate the weld thermal cycle and the results were analyzed to understand the effect of pulsing on the microstructure and mechanical properties. Emphasis was placed on cooling time within the critical temperature range of transformation (i.e., between 800 °C to 500 °C, denoted as $\delta t_{8/5}$) across different regions of the weld joint when applying the PWHT pulse. It was found that the transformation time ($\delta t_{8/5}$) decreases from the HAZ to the center of the FZ with an increase in weld current, as shown in Fig. 2.23a. SORPAS simulation results predicted the optimal slow cooling pulse of 4 kA for 20 cycles that will result in softening of the FZ. Thus, the formation of a 100% martensitic phase can be prevented thereby allowing the formation of softer phases. However, a discrepancy between the predicted optimal slow cooling pulse (from the simulation) and the measured hardness values experimentally was observed and it was commented that $\delta t_{8/5}$ may not be the best choice for the alloy studied and welding process. It was stated by the authors that the high alloying elements in AHSS may shift the temperature range for transformation (i.e., between 800 °C to 500 °C), which was the focus in their work as it is usually considered for conventional steels. The optimal weld quality in terms of failure load and energy absorption in tensile shear mode was observed as a function of the post-weld current as shown in Fig. 2.23b. Overall, there was no significant difference in the mechanical properties up to a secondary pulse current of 4 kA, while further increase in current deteriorated the mechanical properties under shear loading conditions. It was also reported that all samples failed with an interfacial fracture mode.

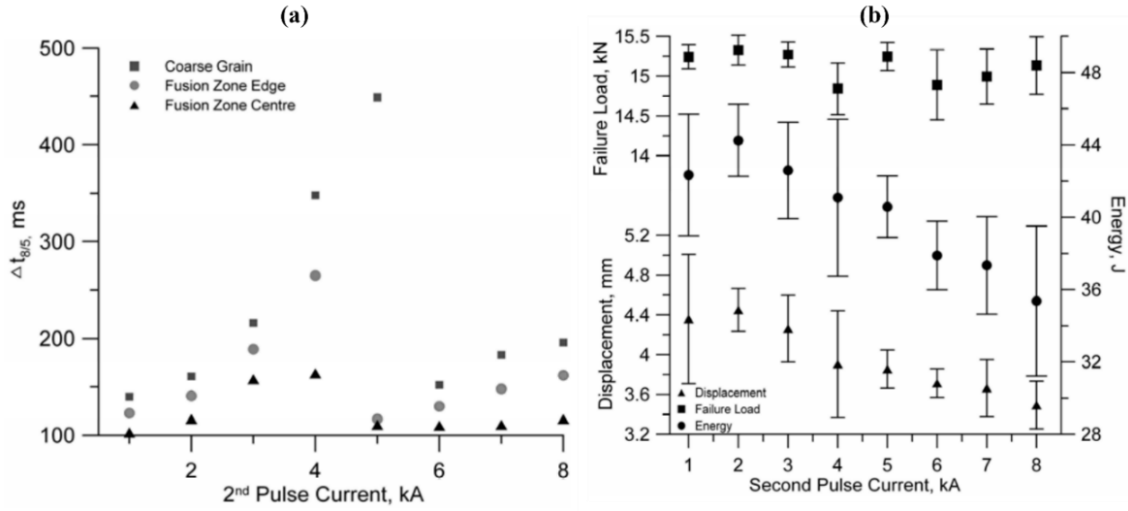


Fig. 2.23: (a) Time between 800°C and 500°C ($\delta t_{8/5}$) for various weld zones after slow cooling post-weld pulsing (b) Failure load, displacement and energy absorbed for varying second pulse current [119].

As observed in Khan et al. [119] study, the concept of slow cooling may be metallurgically feasible, but additional work is required to obtain the optimal PWHT parameters to yield the required microstructure and improve mechanical performance. Though the claim of the effect of alloying elements on the temperature range for transformation is plausible, however, additional work needs to be done to fully comprehend the preferred temperature range for cooling to obtain the desired microstructure. Therefore, developing process maps with various post-weld parameter combinations as in the case of tempering (as in Fig. 2.19) might reveal the reason for the discrepancies observed in the hardness and $\delta t_{8/5}$ in the different regions of the weld. Additionally, characterizing the microstructural features after different post-weld pulses will be integral in developing the relationship between the microstructure and mechanical properties which was omitted in Khan et al., [119] study.

2.10 Bake Hardening Cycle

Bake hardening (BH) also known as paint baking (PB) is a processing step that takes place during the production of a vehicle body structure after paint application. Typically, the welded body structure undergoes a finishing process where it is painted followed by a baking process at temperatures ranging between 150-200 °C for about 20 mins [120–122]. The BH process cures the paint on the surface of the automotive body after welding, likewise, metallurgists have discovered the process to be a metallurgical approach that strengthens the materials. Particularly, the BH process has led to an increase in yield strength when performed under the right temperature and time conditions [123]. Nonetheless, the chemical composition

(particularly the content of alloying elements), presence of mobile dislocations, baking temperature and time also dictate the BH response (BHR) in certain AHSS [120,124,125].

The metallurgical concept of BH, which leads to an increase in yield strength in ultra-low carbon steel has been attributed to the formation of a “Cottrell atmosphere” wherein interstitial atoms (usually C) diffuse to pre-existing dislocations [126–128]. This process occurs because the thermal energy in the system promotes the diffusion of interstitial atoms, with relatively small atomic radii, to dislocation core sites. The dislocations are then pinned by segregated carbon atoms which impede the dislocation motion thereby resulting in an increased yield strength [129]. Tumuluru [125] suggested the formation of a similar interstitial atmosphere around dislocations and grain boundaries in the HAZ of a DP spot weld after BH, however, no direct evidence of such was provided. Similarly, previous works have shown considerable improvement in cross-tension strength after BH spot welds but the rationale behind such improvement was not attributed to the Cottrell atmosphere. Instead, the improved mechanical performance of spot welds after BH has been attributed to martensite tempering and/or martensite morphology modification [125,130] and reduction in the weld residual stresses [131].

It should be noted that BH of PHS at 170 °C for 20 mins has been reported in the literature [132] but there is no published work on the application of PB to PHS spot welds as well as PHS spot welds subjected to in-situ PWHT. In this research, the BH mechanism will be explored after determining the optimal in-situ PWHT schedule to further improve the mechanical properties of the joint. Hence, this study will explore similar BH parameters in [132] and examine its effect on the changes in microstructural features and mechanical properties of the PWHT PHS joint.

2.11 Summary of literature review and research gap identification

The microstructure of the weld has been identified as one of the indices that dictate the performance of resistance spot welds. However, the welding process is known to alter the microstructural feature leading to the formation of a martensitic structure with low energy absorption capability. Thus, modifying the microstructure to yield better weld performance in service has to be explored. An economically viable method to modify the microstructure is in-situ post-weld heat treatment because the technique can be incorporated directly into the manufacturing line as it does not require additional equipment. Nonetheless, adequate design

of process parameters is essential to achieving the required microstructural changes that can improve mechanical performance.

The understanding of the physical metallurgy in selecting the optimal cool time needs to be studied in more detail. In the case of tempering, it is quite clear that the martensite phase is required before tempering. However, there has not been a clear consensus on the phase transformation considering the non-equilibrium condition during in-situ recrystallization PWHT pulse. Specifically, the welding parameters have not been optimized in a way that reveals the metallurgical transformations occurring during in-situ PWHT and thus, needs to be studied extensively. Moreover, there is no study on the formation of a recrystallized zone in PHS welds which can potentially improve the energy absorption capability as well as the effect of the microstructural changes on crack propagation.

The studies reviewed in this chapter majorly exploited the thermal energy from the post-weld current pulse to trigger microstructural changes in the FZ. However, other RSW parameter such as the welding force has not been explored as an alternative route for microstructural changes. It is hypothesized that the intensification of welding force during the cooling cycle can increase the strain energy in the weld thereby promoting dynamic recrystallization. This technique hereafter referred to as “dual force” (i.e., the force intensified which is different from the initial welding force) has not been well explored as a pathway to improving the mechanical performance of the joint.

The effectiveness of these PWHT schedules has not been well examined, particularly considering the additional processing time which is of high importance to manufacturers as the addition of long process time will serve as a barrier to the implementation to the developed technique. Thus, this work intends to consider designing feasible techniques that will yield the desired microstructure for improved joint properties taking into account the applicability of the technique in any automotive manufacturing line. Lastly, the role of PB on welds subjected to PWHT is still unknown. As these welds are subjected to PB after joining of the body-in-white structure, it is imperative to evaluate the mechanical properties of the weld following the PB schedule.

CHAPTER

3

3. Influence of Voids During In-Situ Tempering ²

² This chapter is composed of a published research paper as outlined in the statement of contributions. **O.T. Betiku**, M. Shojaee, O. Sherepenko, A.R.H. Midawi, A.M. Chertov, H. Ghassemi-Armaki, R.G. Maev, E. Biro, Optimizing post-weld performance of press-hardened steel resistance spot welds by controlling fusion zone porosity, Weld. World. 66 (2022) 1733–1746.

3.1 Overview

Resistance spot welding of PHS produces a martensitic microstructure in the fusion zone (FZ) exhibiting brittle failures with low energy absorption capacity in the case of a vehicle crash event. Typically, the martensitic microstructure impairs the toughness of the fusion zone leading to interfacial failure (IF) which is perceived to be undesirable for crashworthiness. This chapter focuses on the tempering of the martensite in the FZ as a means to improve weld toughness. However, differential tempering was observed across the nugget diameter in this study. The differential tempering was attributed to the presence of pre-existing irregular voids formed during the welding cycle which altered the current flow at the weld center and periphery. Findings revealed that reducing the voids in the nugget leads to effective tempering and improved mechanical properties of the joint when performed under the right temperature and time regime. The optimal tempering condition showed an improved cross tension peak load of 36%, and 33% in the energy absorption capacity. The improved cross tension performance was attributed to the tempered martensitic structure in the FZ which promoted crack deflection leading to a pull-out failure mode.

3.2 Background

Spot welds exhibit different failure modes when loaded to failure and can show low energy absorption due to the martensitic structure in the FZ after welding. Mostly, the intrinsic notch associated with the RSW process serves as a point with high stress concentration and is liable to crack propagation when subjected to loading [107]. Thus, the microstructure in front of the notch is important in dictating how the crack propagates. Previous study has shown that the overall performance of AHSS in vehicle structures relies on the strength and toughness of the welded joints [84]. Hence, it is imperative to assess and improve the weld toughness of PHS joints used in vehicle body-in-white structures.

Tempering is known to be effective in alleviating the problem of brittle fracture and improving weld toughness when done using the appropriate thermal cycle [92,93]. Tempering can be performed in two ways. First, isothermal tempering which involves slow heating to a particular temperature, long holding time at that temperature and slow cooling; second, non-isothermal tempering which is commonly adopted in manufacturing processes such as welding. Non-isothermal tempering involves rapid heating, negligible holding time at tempering temperature followed by rapid cooling [94]. In the case of RSW, tempering can be achieved by applying an additional post-weld current pulse to heat the martensitic structure below the A_{c1} temperature. The additional pulse is usually applied after the weld nugget has solidified by holding the

electrodes in place long enough to sufficiently quench the martensite thereby eliminating the need for any additional equipment [95]. It is worth mentioning that the effectiveness of the tempering process is dictated by several factors such as tempering temperature, tempering time, heating rate, steel chemical composition and steel thickness [91,94]. Hence, certain AHSS can require different tempering temperatures or time depending on their chemical composition and/or thickness [53].

Though some works have explored non-isothermal tempering on RSW in other types of AHSS such as transformation induced plasticity (TRIP) steels [106,133], dual-phase (DP) steels [94] and isothermal tempering of martensitic steels [134–136], there is still a dearth of information on non-isothermal tempering of spot-welded PHS as only a handful of studies have addressed it until now [89,97,98]. Earlier work by Hou et al. [98] has shown heterogeneous transformations in the FZ after in-situ tempering, however, the rationale behind such changes was not further investigated. More specifically, the response of spot-welded PHS joints to different combinations of tempering temperatures and time is still not fully understood and the effects of shrinkage voids, a common imperfection in RSW of PHS, during post-weld heat treatment (PWHT), has not been reported in the literature. It is believed that the presence of a void in the nugget is not detrimental to the weld strength provided the void is not located at the sheet/sheet interface and the effective nugget diameter is greater than a critical diameter of $5t^{1/2}$, where t is the sheet thickness [52]. However, the presence of voids, which is highly resistive compared to the rest of the nugget, could affect the current flow in subsequent post-weld in-situ tempering process. This work investigates the effects of in-situ tempering during RSW of PHS and how the presence of voids in the FZ influences the post-weld tempering treatment and the resulting properties.

3.3 Materials and Methods

3.3.1 As received material

The material used for this research is an Al-Si coated PHS received in the hot-stamped condition from Auto/Steel Partnership (A/SP). The material is also commercially referred to as Usibor-1500®, which is a brand name from the manufacturer. The chemical composition of the as-received material is outlined in Table 3.1 while the carbon equivalent of the investigated steel was calculated using the formula proposed by Yurioka et al. [137]. The sheet thickness of the PHS was 1.4 mm and each side of the sheet had an average coating thickness of 35.0 μm

$\pm 0.7 \mu\text{m}$ as shown in Fig. 3.1a. The microstructure of the base metal after hot stamping is shown in Fig. 3.1b which is a predominantly martensitic.

Table 3.1: Chemical composition (wt%) of the investigated PHS

Material	C	Mn	Si	Cr	Al	Ti	P	B	S	Mo	C _{eq}
PHS	0.21	1.15	0.26	0.17	0.04	0.031	0.01	0.0029	0.003	0.0017	0.47

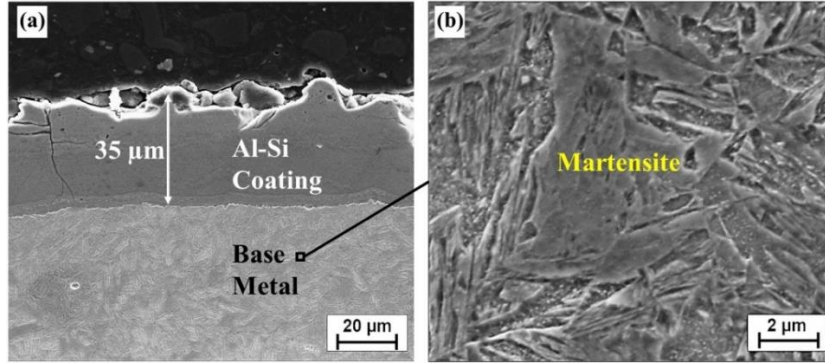


Fig. 3.1: SEM images of (a) Al-Si coated PHS (b) microstructure of PHS after hot-stamping.

Base metal mechanical properties were measured by performing uniaxial tensile tests at a crosshead speed of 1 mm/min according to the ASTM E8 [138]. The average tensile properties evaluated after five repetitions are reported in Table 3.2 and the error represents a 95% confidence interval.

Table 3.2: Average tensile properties of PHS in the rolling direction.

Material	YS (MPa)	UTS (MPa)	T.E (%)	U.E (%)
PHS	1228 $\pm$ 14	1623 $\pm$ 13	6.89 $\pm$ 0.50	4.66 $\pm$ 0.15

*YS: yield strength; UTS: Ultimate tensile strength; TE: total elongation UE: uniform elongation.

3.4 Methodology

3.4.1 Development of spot weldability lobe and baseline weld schedule (BWS)

Resistance spot welding was done using a medium-frequency direct current (MFDC) RSW FANUC robot with a C-type spot welding gun and a Rexroth Bosch weld controller. The welding equipment operates on 60 Hz which makes a cycle to be 16.67 ms. Cu-Cr electrodes RWMA type B 5/8" with a flat face diameter of 7 mm subjected to a constant water-cooling rate of 6 L/min were used for welding all specimens. The baseline welding parameters as shown in Table 3.3 were selected according to the AWS D8.9 [32] standard. The weldability lobe was developed with the AWS weld time of 20 cycles (334 ms) in addition to 25% below (i.e., 15 cycles $\sim$ 250.5 ms) and above (i.e., 25 cycles $\sim$ 417.5 ms) the recommended weld time. For

each of the weld times, the weld current was varied from 5.5 kA to 9 kA to obtain the minimum weld size (MWS), and maximum welding current ($I_{\max}$). The minimum welding current ($I_{\min}$) is the current that results in the MWS which is 4.73 mm (obtained from $4\sqrt{t}$, where t is the sheet thickness); while the $I_{\max}$ is defined by the minimum current that results in expulsion.

Table 3.3: Resistance spot welding process parameters according to AWS D8.9 standard

Material	Electrode force (kN)	Squeeze time (ms)	Total weld time (ms)	Hold time (ms)	Number of pulses	Cool time between pulses (ms)	Weld current (kA)
PHS	4.9	167	334	167	2	33	5.5 to 9.0

The weldability lobe showing the $I_{\min}$ and $I_{\max}$ for PHS is shown in Fig. 3.2a. The weld time of 334 ms shows a weldable current range of 2.5 kA while the longer time of 417 ms shows a lower $I_{\max}$ resulting in a lower weldable current range of 2.1 kA. Generally, a welding current range above 1.0 kA is regarded by the industry to have satisfactory weldability. For this research, a fixed nugget diameter of 6 mm was set as a baseline which was obtained at a current above the $I_{\min}$ and below the $I_{\max}$. To determine the welding current that forms a 6 mm nugget diameter, weld sizes from varying currents were measured using the chisel test as shown in Fig 3.2b. The chisel test results were further verified by the average nugget diameter from three weld cross-sections. A welding current of 8.0 kA resulted in an average nugget diameter of 6.08 mm $\pm$ 0.06 mm as shown in the inset in Fig. 3.2b, which was selected as the baseline current for further investigation. The etched cross-section of the weld made with a current of 8.0 kA showing the FZ diameter, HAZ and BM is shown in Fig. 3.3 and is hereafter referred to as the baseline weld schedule (BWS).

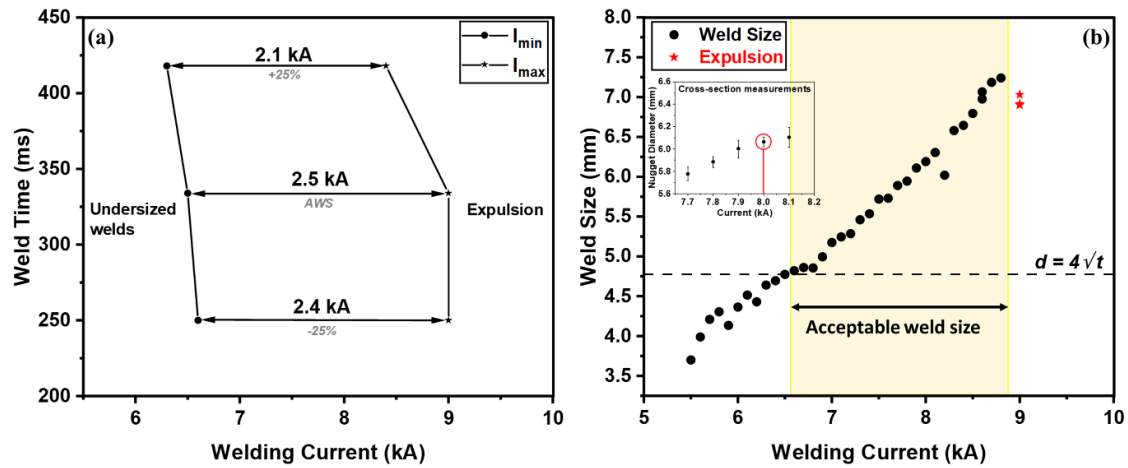


Fig. 3.2: (a) Weldability lobe for PHS for 3 welding times (b) Weldable current range for PHS with weld time of 334 ms (graph inset shows weld cross-section measurement with 8.0 kA selected as baseline current).

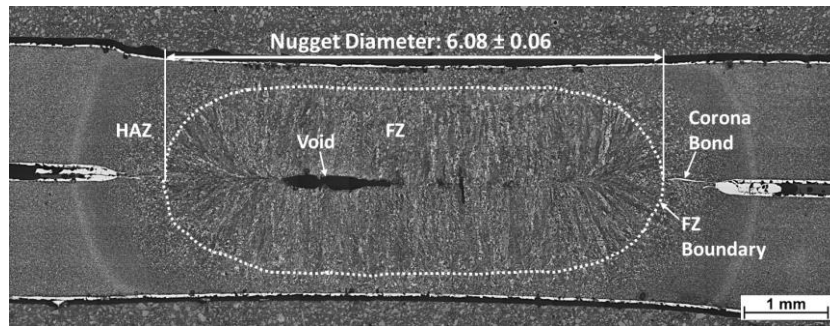


Fig. 3.3: Optical micrograph of a cross-section of the weld with 8.0 kA current; Baseline weld schedule (BWS).

3.4.2 Temper Pulse Schedule (TPS)

An additional pulse was applied after the BWS to modify the microstructure of the FZ and improve the mechanical properties of the joint. In this study, three different durations of temper pulse were selected: 4 cycles (67 ms), 8 cycles (134 ms) and 10 cycles (167 ms). As for the temper pulse current, 8 different currents including 3.2 kA, 4 kA, 4.8 kA, 5.6 kA, 6.4 kA, 8.0 kA, 8.8 kA and 9.6 kA were employed corresponding to (40 - 120) % of the baseline current to develop the tempering process map. A temper pulse time of 67 ms as highlighted in Fig. 3.4b was used for further investigations to study the influence of voids on tempering outcomes. The cooling time between the second pulse of the BWS and temper pulse was 60 cycles (1000 ms), which was obtained from simulation (nodal thermal history). The 1000 ms cool time was selected to ensure that the martensitic transformation is completed *i.e* the nugget temperature decreased below martensite finish (M_f) temperature before applying the temper pulse [70].

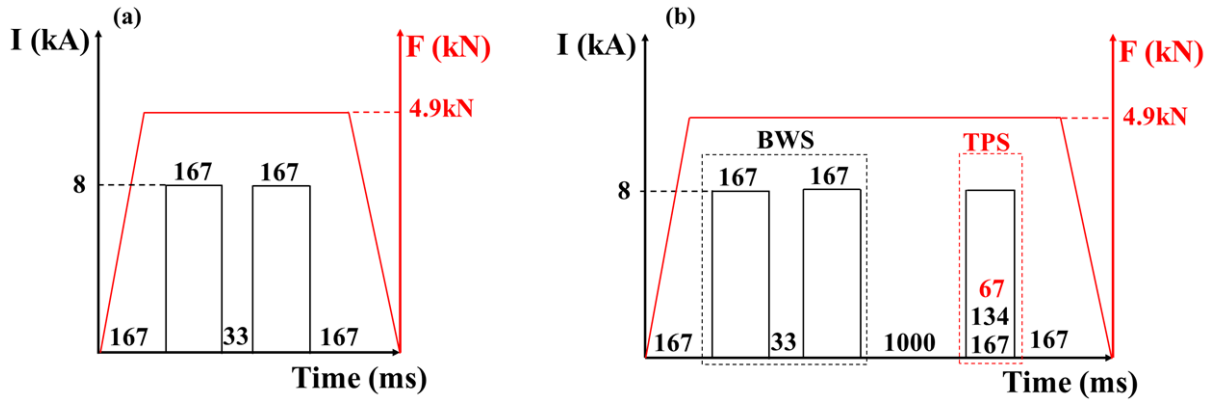


Fig. 3.4: Schematic representation of (a) baseline weld schedule (BWS), and (b) BWS followed by temper pulse schedule (TPS).

3.4.3 Hardness Measurement

Hardness measurements were performed on polished samples using an automated Clemex microhardness machine with a Vickers Indenter under a load of 200 gf and a dwell time of 10 s. The spacing between adjacent indents was 150 μm which is greater than three times the diagonal of each indent as specified by AWS D8.9M standard [32]. The average hardness values at the weld center and periphery were first obtained by averaging 40 indents at the weld center (black indents on Fig. 3.5) and 12 indents at the nugget periphery (red indents on Fig. 3.5). The error bars for the hardness measurements represent 95% confidence interval. Thereafter, the hardness maps (region indicated by the dotted line in Fig. 3.5) were plotted for the BWS and selected temper pulse conditions with a horizontal and vertical distance of 300 μm between consecutive hardness lines.

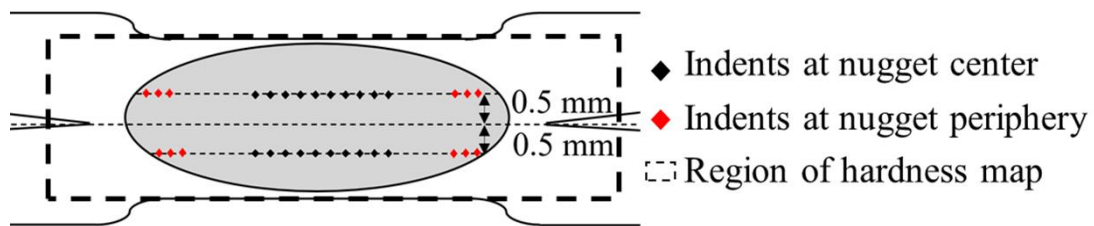


Fig. 3.5: Region of interest for hardness indents within the center and periphery of the FZ.

3.4.4 Cross tension test

The mechanical performance of the joint was evaluated through cross-tension (CT) tests according to AWS D8.9M standard [32]. The dimensions of a CT specimen after welding are schematically illustrated in Fig. 3.6. All mechanical tests were performed at a constant crosshead speed of 10 mm/min using a 100 kN MTS frame universal tester. The force-displacement data was obtained from the test while the energy absorbed was obtained by

integrating the area under the curve to failure. All reported data from the mechanical tests represent an average of three repetitions while the error bar indicates a 95% confidence interval. An additional sample was tested and interrupted during the test at 90% of the average peak load of the initial three tests to understand the crack path during loading.

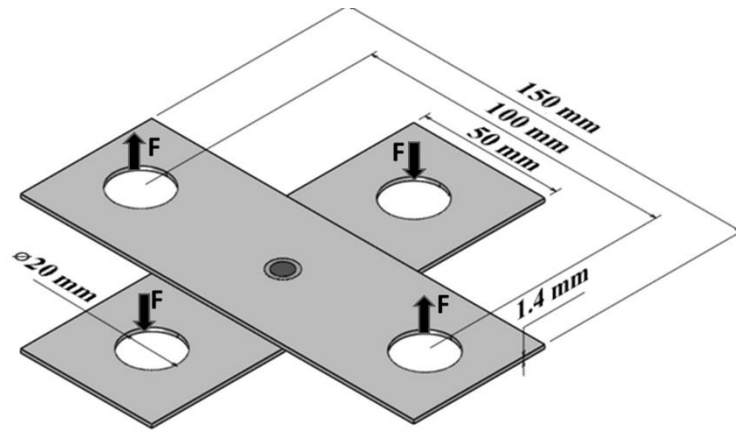


Fig. 3.6: Schematic representation of cross-tension test sample.

3.4.5 Microstructural examination

After the RSW process, samples were sectioned and prepared using standard metallography procedures for microstructural observations. Welds were sectioned along the weld centerline using a SiC cutting disc on a Struers' precision cutter. Afterwards, grinding and polishing of samples were performed using diamond paste up to 1 μm while etching was done using 2% Nital (98 % ethanol + 2 % nitric acid). Microstructural analysis and fractography were done using a Zeiss scanning electron microscope (SEM).

3.4.6 Void analysis

Voids in the FZ were analyzed using a scanning acoustic microscope (Tessonic AM1103) which uses a collimated ultrasonic beam to inspect the weld structure. The specimen and the lens were immersed into water, which serves as a couplant for the ultrasound propagation [47]. Data were collected in the form of ultrasonic C-scan, when reflections from a certain depth of the weld structure are collected to form a 2D image. To obtain a higher resolution scan showing the void morphology and its surrounding region, X-ray scans with a resolution of 5 $\mu\text{m}/\text{pixel}$ were performed on a ZEISS Xradia Versa 520 microfocus x-ray using a presetting of 140 kV and exposure time of 4 seconds.

3.4.7 Numerical simulation of the welding process

The thermal history of the RSW process was simulated using a commercially available finite element (FE) Simulation and Optimization of Resistance Projection and Spot welding process

(SORPAS) software V13.95. The welding process was simulated using a FE-model of over 4000 nodes. The material properties of the Al-Si layer and the PHS were obtained from the software database. The welding parameters used during the simulations were similar to the parameters used in the experiment and temperatures obtained were from nodal analysis at the weld center and periphery. The effects of the presence of voids in the FZ during the post-weld treatment were simulated using a cylindrical shape with a diameter of 1 mm, and height of 0.22 mm, which corresponds to the average experimental measurement of void sizes in 20 samples.

3.5 Result and Discussion

3.5.1 Effect of temper pulse on the FZ hardness

The effectiveness of the temper pulse was initially analyzed from the average hardness distribution at the weld center in the FZ. The average hardness at the FZ for the BWS was 507 HV, which is similar to the base metal hardness of 500 HV after hot stamping. The process window for tempering RSW PHS with various temper pulse times and currents is shown in Fig. 3.7. The hardness values after tempering were classified into three regions. Region 1 represents the parameter space where an increased hardness value in the FZ was observed at a low temper pulse current. The current was assumed to be insufficient to effectively temper the martensite within this parameter space. A possible explanation for the increased hardness with the low temper pulse current could be due to secondary hardening where below the A_{c1} , cementite dissolves leading to the formation of other metastable alloy carbides [91]. However, verification of the alloy carbides is beyond the scope of the present study. The second region (*i.e* region 2) shows the combination of parameters space where a reduction in hardness is observed after applying the temper pulse. A possible reason for the reduced FZ hardness could be related to the decomposition of martensite during the tempering pulse [70,95]. Region 3, which is the higher tempering current region showed an increased hardness above the second region. The increased hardness can be attributed to reaustenitization and the formation of a martensitic structure upon cooling. The tempering time of 67 ms was studied in detail since effective tempering was attainable at a shorter time (spike tempering) [139] compared to the conventional tempering which requires longer temper pulse times up to 167 ms, as shown from the process map in Fig. 3.7. The tempering time of 67 ms was selected as it can achieve effective tempering in a shorter time which reduced the overall process time.

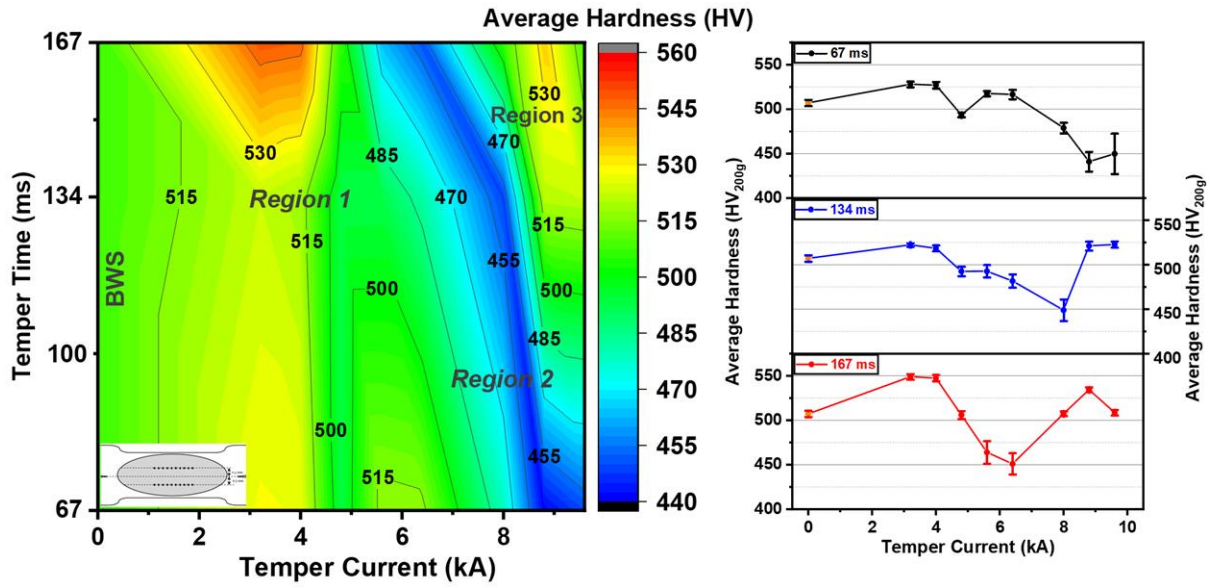


Fig. 3.7: Process map of different combinations of temper pulse schedule indicating the average hardness value in the FZ.

Hardness mapping was carried out throughout the weld region to gain a better understanding of the effect of tempering on the global FZ properties. The hardness maps of the FZ showed that the TPS used for this study resulted in a significant difference in the hardness between the central region and periphery of the FZ compared to the BWS condition as shown in Fig. 3.8. The average hardness at the center and periphery of the FZ was carried out using the hardness pattern in Fig. 3.5 and the average hardness values at both regions are shown in Fig. 3.9a. An interesting observation in the average hardness distribution was made when increasing temper current from 8.0 kA, to 8.8 kA and finally to 9.6 kA. Within this current range, the average hardness values at the weld periphery show a transition in hardness from a relatively soft (363 HV) to hard (520 HV). At 8.0 kA, the weld periphery exhibited softening while the center remained untempered and at 9.6 kA, the center exhibited softening with a hard periphery. The difference between the hardness at the center and periphery are denoted by δ_{HV} as shown in Fig. 3.9b with $+\delta_{HV}$ at 8 kA of 115 HV and $-\delta_{HV}$ at 9.6 kA of -70 HV. At 8.8kA, tempering seemed more effective with average hardness at the center and periphery within (441-445) HV range. Overall, the average hardness at the nugget center was reduced by 6%, 15% and 12.7% by applying a post-weld current of 8.0 kA, 8.8 kA, and 9.6 kA respectively, compared to the BWS. Despite the hardness drop with the addition of the temper pulse, a heterogenous nature in the hardness distribution at the weld center and periphery was observed for the parameters studied.

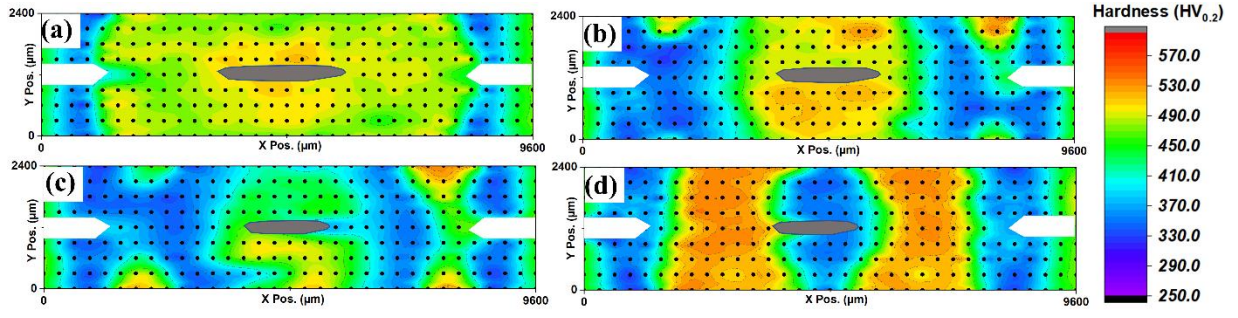


Fig. 3.8: Hardness maps for (a) BWS; and tempering at 67 ms with (b) 8.0 kA (c) 8.8 kA (d) 9.6 kA.

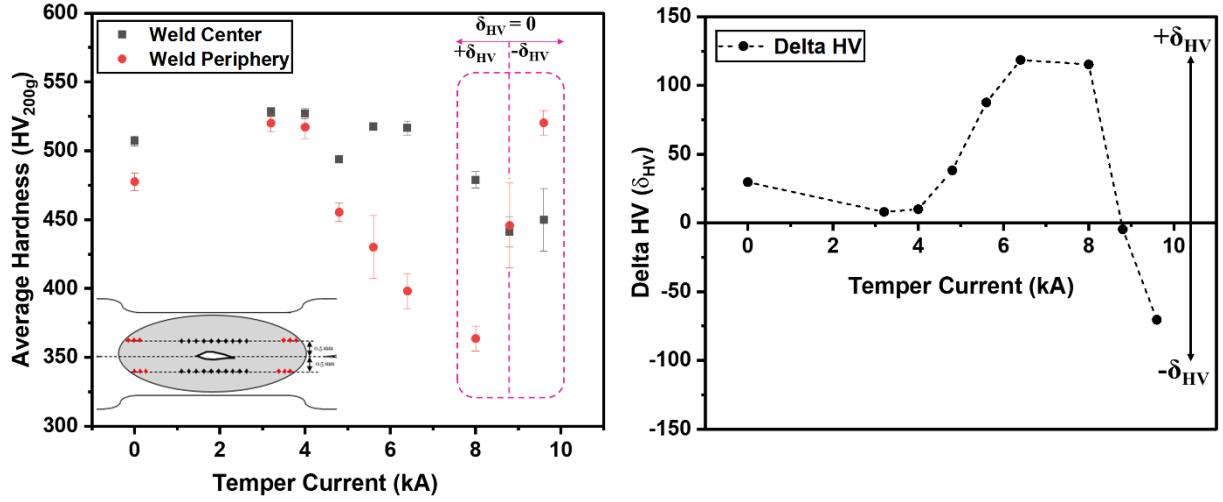


Fig. 3.9: (a) Average hardness at the nugget center and periphery with temper time of 67 ms (b) hardness difference between the FZ center and periphery denoted as $+\delta_{HV}$ and $-\delta_{HV}$.

3.5.2 Influence of voids during tempering

The hardness results shown in Fig. 3.8 and Fig. 3.9 indicate temperature heterogeneities within the nugget which led to effective tempering of either the FZ periphery or the central region of the FZ depending on the applied temper pulse schedule. As it is known that Joule heating is the mechanism responsible for the heat generation during the post-welding pulses [92], it can be assumed that the variation in tempering response, as seen in Fig. 3.8 and Fig. 3.9, could be due to heterogeneous current flow through the FZ. The non-homogeneous current flow in the FZ can be attributed to the presence of voids formed at the sheet/sheet interface. These voids which were formed during the weld schedule (*i.e* when melting and solidification occurs) affect the current flow and heat transfer during the temper pulse schedule resulting in temperature heterogeneities in the nugget. Previous researchers have suggested that the voids are formed as a result of interference between axially dendritic growth with a higher solidification rate (in the thickness direction) compared to the interdendritic feeding (growing in the diametric direction of the nugget) during the final stage of solidification [52,140]. In other words, their findings

presume that the shortage of liquid feeding to the nugget center during solidification leads to shrinkage voids. Another explanation could be due to the formation of gas pores from the coating during the welding schedule which migrate to the sheet/sheet interface during cooling. This phenomenon was observed by Chen et al. [26] where the evolution of pores in uncoated and Al-Si coated PHS was studied at different welding times. No pores were observed in the uncoated PHS while a smooth surface and circular void, indicating gas pores, were observed in Al-Si coated PHS. Based on these formation mechanisms, it is assumed that the latter is the dominating factor leading to void formation in coated PHS. Overall, it was observed that the morphology of these voids are unpredictable (*i.e* irregular in shape) and could severely affect the temperature distribution within the nugget.

To better understand the temperature heterogeneity in the FZ due to the presence of voids, SORPAS FE-analysis shows the temperature distribution at the nugget center and periphery during the tempering schedule. Since the voids were formed during the BWS and remain after solidification prior to tempering, the influence of the voids during the tempering schedule is shown in Fig. 3.10 (a -c). The temperature history was extracted from the black dot (0.5 mm above the faying surface at the weld center) and red dots (0.5 mm above the faying surface at the weld periphery). It is worth noting that the temperature prediction by SORPAS most likely deviates from the exact weld temperature, nevertheless, it was observed that the presence of the voids has a significant effect on the temperature distribution during tempering. As shown in Fig. 3.10a, tempering without the presence of a void at 67 ms and 8.8 kA could result in a somewhat homogenous temperature in the nugget with the center and periphery attaining a maximum temperature of 480 °C and 474 °C respectively. On the other hand, the presence of voids at the sheet/sheet interface with the same tempering parameter reduces the temperature at the nugget center while the periphery reaches a relatively higher temperature as shown in Fig. 3.10b. In this case of 8.8 kA with voids, the temperature at the center reaches a peak of 405 °C while the periphery increases to 533 °C. The temperature difference explains the variation in the hardness distribution in the nugget whereby the weld periphery appears to be effectively tempered (lower hardness value) than the center at 8.8 kA. On the contrary, at a higher current of 9.6 kA, the periphery was overheated attaining a peak temperature of 674 °C while the center temperature only reached 505 °C, as shown in Fig. 3.10c, which is in agreement with the hardness map in Fig. 3.8d.

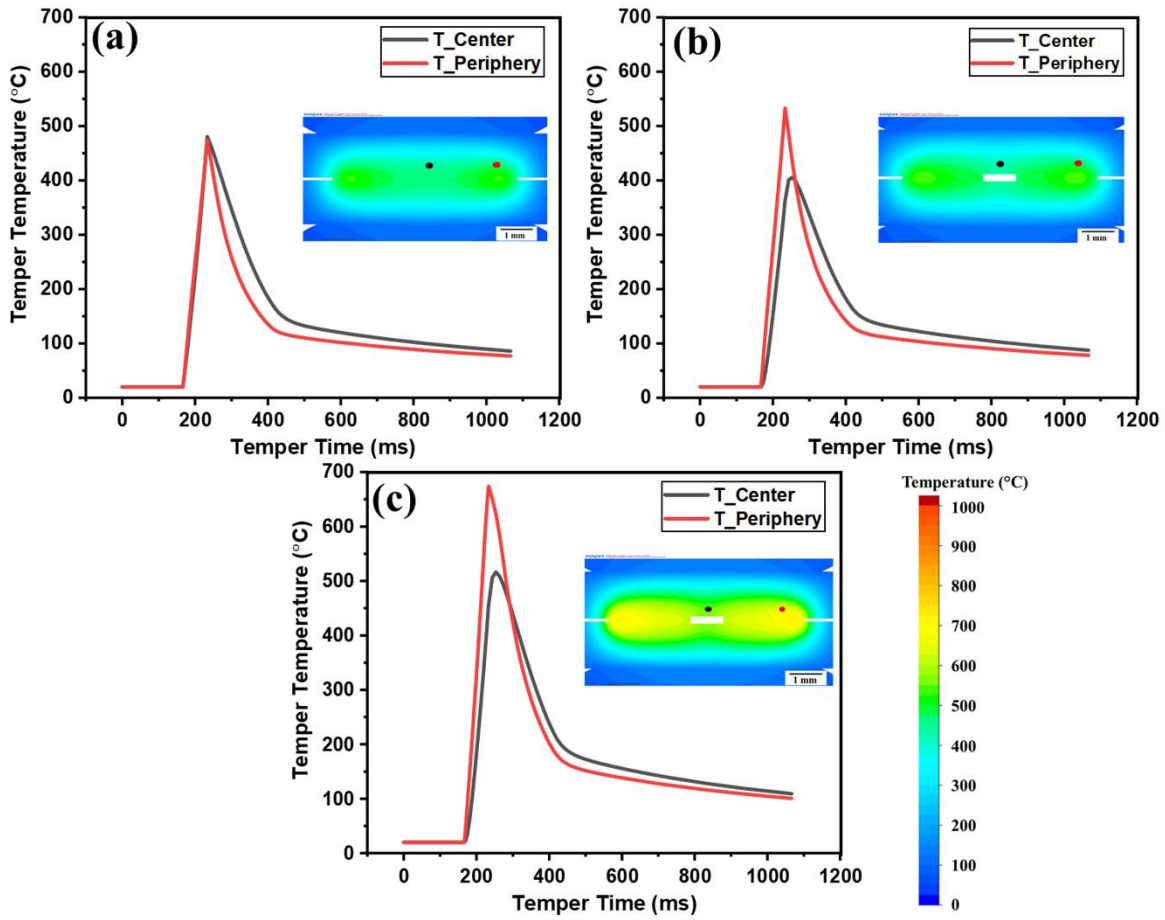


Fig. 3.10: SORPAS simulation showing temperature distribution at the nugget center and periphery for (a) no void condition and tempering at 8.8 kA; (b) void condition and tempering at 8.8 kA (c) void condition and tempering at 9.6 kA.

3.5.3 Void reduction using pre-pulse

To attain a homogenous temperature in the nugget during post-weld treatment, it is imperative to minimize and/or eliminate the voids. A few researchers have attempted to minimize the voids formed during welding. Lin et al. [141] employed a force ramping schedule which led to consistency in improving mechanical properties. However, cross-sections showed that the voids were still present at other angular sections (blind sections) of the nugget. Longer weld time has also shown promising results in reducing voids [142], however, further studies report that this promotes the formation of a locally softened region known as the “halo ring” [80]. Since gas pores are assumed to be the responsible void formation mechanism in Al-Si coated PHS, as described by Chen et al. [26], this study employed a short pre-pulse of 20 ms and relatively high current (16 kA) before the weld schedule to remove the coating thereby reducing the possibility of gas pores formation during solidification. It should be noted that the pre-pulse

only displaced the coating without the formation of any nugget, and there was no cooling time between the end of the pre-pulse and the welding pulse.

Once the pre-pulse was integrated into the welding schedule, the dynamic resistance curves of welds made with and without a pre-pulse were compared to determine if pre-pulsing affects the RSW process. A comparison of these curves showed a significant difference with the application of a pre-pulse. For welds with a pre-pulse, the resistance at the beginning of the welding process was $75\ \mu\Omega$ lower than the resistance of the BWS welds. The measured difference in resistance as shown in Fig. 3.11a is an indication of removal of the Al-Si coating which is known to have a high resistivity [143]. Interestingly, the welds made with pre-pulse showed a difference in resistance of $56\ \mu\Omega$ (at 1400 ms) during the tempering schedule compared to the BWS welds as shown in Fig. 3.11b indicating less resistance to heat generation due to void reduction, thereby resulting in a better current flow through the effective nugget area. On the other hand, the presence of voids in the BWS welds reduces the effective nugget area and the heating dynamics of the nugget resulting in higher dynamic resistance as shown in the highlighted section during tempering on Fig. 3.11b.

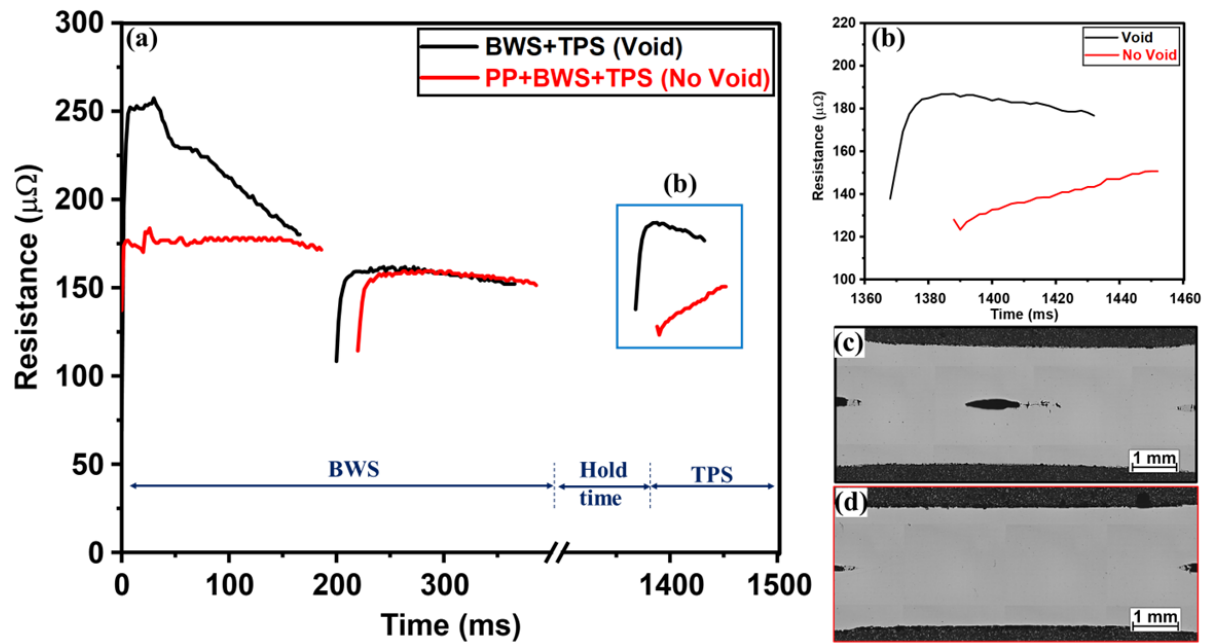


Fig. 3.11: (a) Dynamic resistance curves for tempered welds with and without pre-pulse (b) highlighted section of TPS schedule (c) cross-sections for void condition without pre-pulse (d) reduced void condition with pre-pulse.

Additionally, metallography cross-sections of the joints welded with pre-pulse showed that the voids were significantly reduced compared to welds made without prepulse as shown in Fig. 3.11c and d. It is worth mentioning that the void morphology in the FZ is unpredictable,

which results in irregularities in the shape and size. The cross-section at the weld centreline in Fig. 3.11d shows the absence of voids, however, cross-sections only provide a planar examination of the weld nugget. Hence, it is likely that voids may exist at some blind sections away from the cross-section plane. Therefore, non-destructive scans (ultrasonic and x-ray) with a better resolution were performed to confirm that pre-pulsing reduced the voids in the welds. For the ultrasonic scans, the bulk solid material does not reflect acoustic waves to the probe and is shown as a dark area on the image. In contrast, the white region represents interference of the ultrasonic waves by the voids as shown in Fig. 3.12a and c. Based on the AWS D8.1 standard, the length of the void should be less than 25% of the nugget diameter to be accepted. In this case, the welds made without a pre-pulse show a large cavity with an average diameter of 1.52 mm (approximately 25% of nugget diameter of 6 mm) and some irregular cracks as shown in the x-rays scans in Fig. 3.12b. Vijayan et al. [52] characterized such cavities to include two areas: a smooth spherical non-dendritic region (the void) and an irregular dendritic crack (the surrounding). In comparison to the welds made with a pre-pulse, it was observed that the voids were significantly reduced by 60% (average diameter of 0.60 mm) though the surrounding dendritic cracks remain as shown in Fig. 3.12d. The surrounding dendritic cracks have connecting dendritic structures and can allow a partial flow of current thereby reducing the inhomogeneous temperature distribution during the tempering schedule [140].

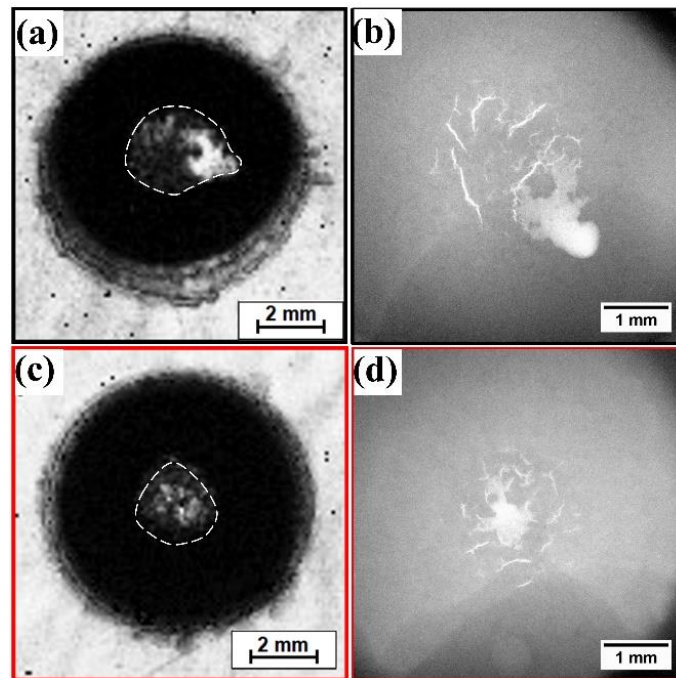


Fig. 3.12: Ultrasonics scans of tempered weld showing void morphology (a) without pre-pulse; and (c) with pre-pulse. X-ray scans of tempered weld showing void morphology (b) without pre-pulse; and (d) with pre-pulse.

3.5.4 Joint properties after void reduction

3.5.4.1 Hardness

After the temper pulse, the hardness at the center and periphery of the FZ depends on the microstructural features which are directly linked to the heat input and thermal cycle during the in-situ RSW tempering [92]. The FZ hardness with the reduced void condition obtained by applying a pre-pulse to the initial AWS schedule (now referred to as BWS) is shown in Fig. 3.13a. Compared to average hardness after tempering with the presence of voids in Fig. 3.9a, the general trend shows that the average hardness in both regions decreases with an increase in the temper pulse current. Interestingly, the average hardness difference between the center and periphery (δ_{HV}) shows a reduced scattering of the hardness in the FZ after tempering as shown in Fig. 3.13b. It was also observed that effective softening occurred at 8.0 kA and 8.8 kA temper pulse current at the center and periphery of the FZ as shown in Fig. 3.14b and c indicating a homogenous temperature in both regions during the tempering schedule. However, the average hardness at 9.6 kA suggests re-austenization and formation of martensite on cooling as the hardness at the center increased much faster than the hardness of the periphery.

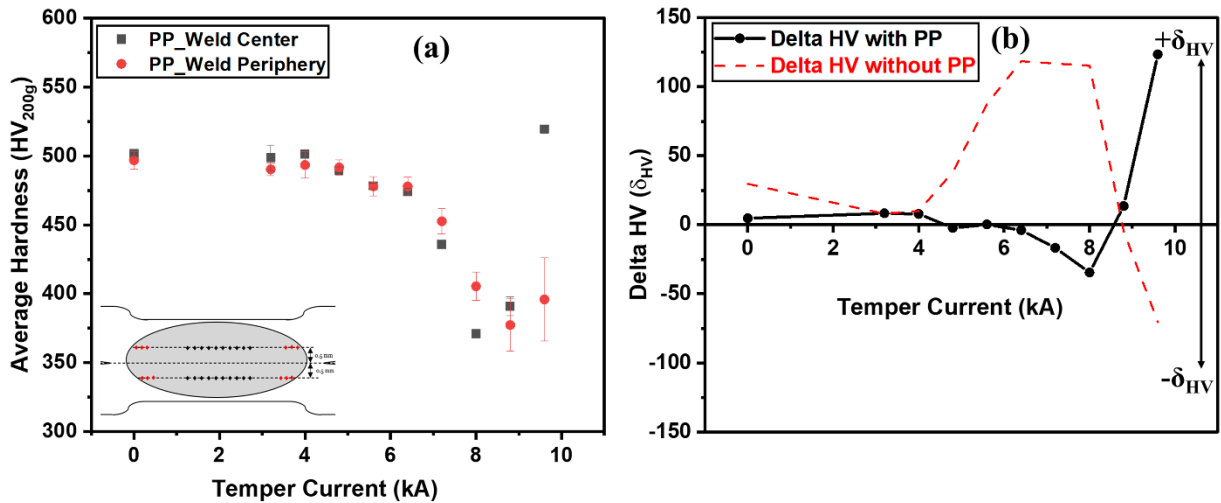


Fig. 3.13: (a) Average hardness at the nugget center and periphery with reduced void using a pre-pulse and temper time of 67 ms (b) hardness difference between the FZ center and periphery denoted as $+\delta_{HV}$ and $-\delta_{HV}$.

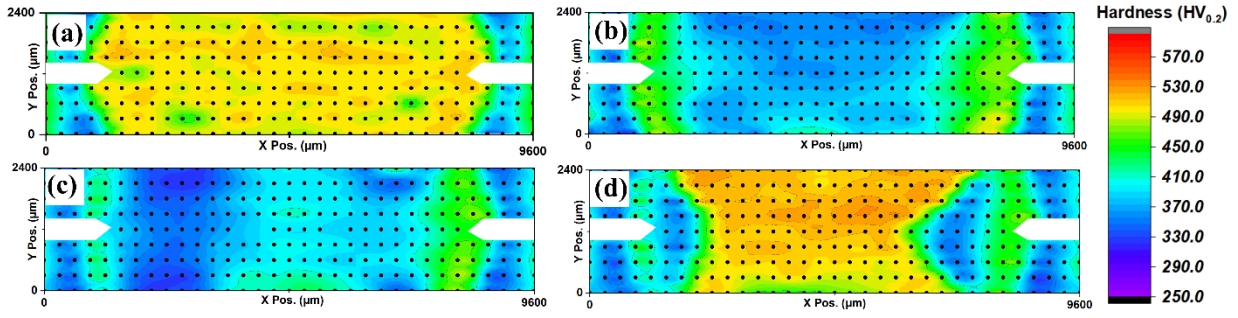


Fig. 3.14: Hardness maps for the reduced void condition with pre-pulse for (a) BWS; and tempering at (b) 8.0 kA (c) 8.8 kA (d) 9.6 kA.

3.5.4.2 Mechanical properties

A comparison of the mechanical properties in terms of peak load and energy absorption capability after tempering with voids and reduced void conditions are shown in Fig. 3.15a and c. The cross-tension test was selected over the tensile shear test because of its sensitivity to the presence of brittle microstructure at the joint interface [106,107]. The tested specimens were for the conditions that showed a significant change in the average hardness values at the periphery of the FZ and the reported CTS values. Compared to the strength of the BWS welds in the reduced void condition having a CTS of 6.25 kN, an improvement in the peak load was observed when PWHT was applied. The improvement in the strength of the PWHT welds was 32.4% (8.27 kN), 36.1% (8.51 kN) and 16.5% (7.28 kN) for the temper pulse currents of 8.0 kA, 8.8 kA and 9.6 kA respectively. The area under the load-displacement curve was calculated, which is representative of the failure energy of the welds. It was observed that the temper pulse led to improved absorbed energy to failure during the CT test. The energy absorbed to failure for the BWS welds in the reduced void condition was 31.11 J, which was improved by 27.66 % (39.72 J), 33.13% (41.43 J) and 21.62% (37.85 J) for the temper pulse currents of 8.0 kA, 8.8 kA and 9.6 kA, respectively.

Reports suggest that the load-bearing capacity and energy absorption of RSW under CT loading depend mainly on the weld nugget size and failure mode [92]. Since there was no significant difference in the nugget diameter, it is most likely that the improved CT properties could be attributed to the increased resistance of the tempered microstructure to crack propagation during loading, compared to the BWS welds. Stadler et al. [103] reported that tempering the weld nugget in a TRIP-aided bainitic ferrite (TBF) steel both softened and toughened the microstructure thereby improving mechanical properties. Analysis of the tempered microstructure showed increased fractions of high angle grain boundary which promotes crack deflection. However, assessing the validity of this claim for tempered PHS weldments requires

further characterization, which is a subject to be considered in a latter chapter. Another plausible argument for the increased energy absorption capacity for the tempered weld at 8.8 kA could be related to the significant hardness drop in the nugget indicating a ductile nugget with the capacity to resist brittle failure. This softening of the FZ favors improved toughness compared to a brittle martensitic microstructure observed in the BWS condition.

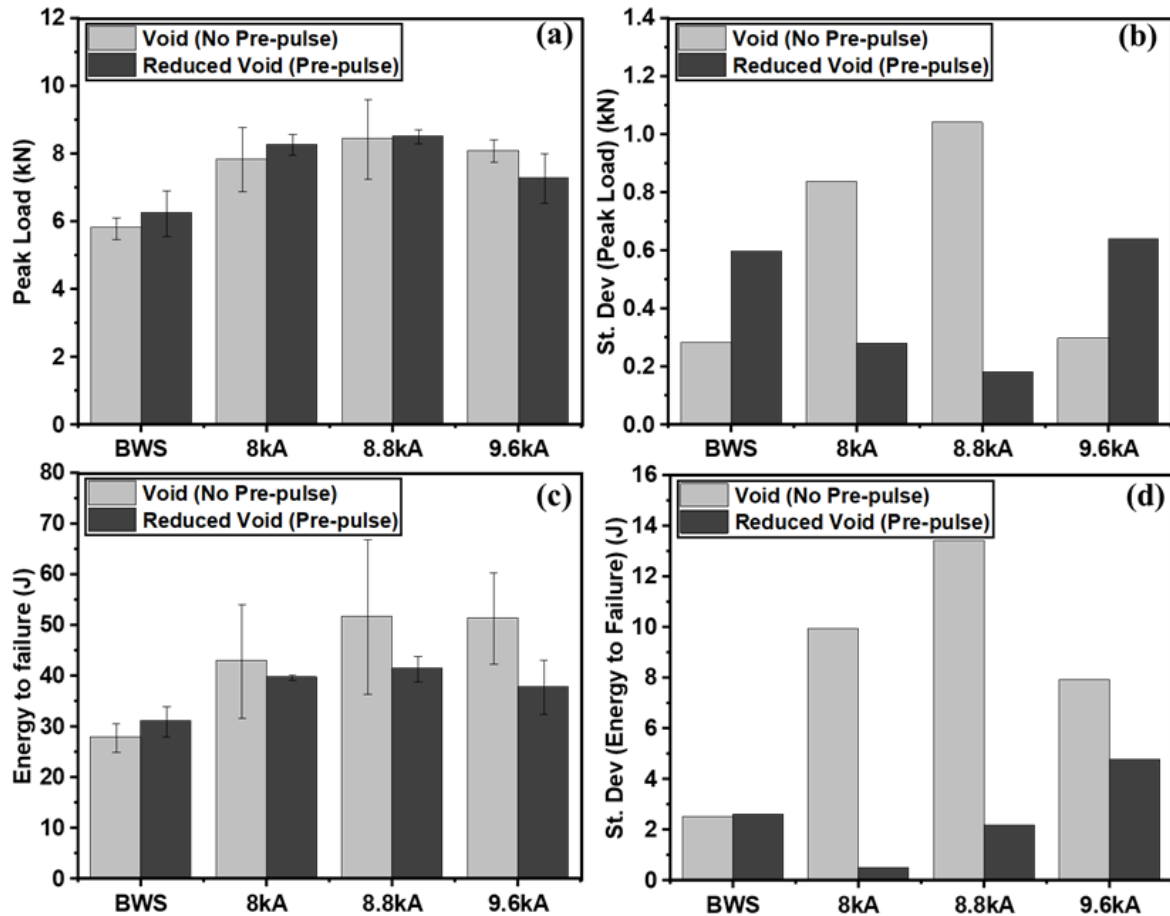


Fig. 3.15: (a,c) Peak load and energy absorption capacity comparing the BWS to different temper pulse currents; (b,d) standard deviation in peak load and energy absorption capacity of BWS and temper pulse with voids and reduced voids during cross tension (CT) test

Interestingly, when comparing the strength and failure energy of welds with voids and reduced void conditions, the presence of voids did not significantly affect the properties in the BWS condition. However, it was observed that the presence of voids drastically increased the scattering in the measurement (standard deviation) of tempered welds. The standard deviation of the mean peak load and failure energy measurements when comparing the tempered welds made in the void and reduced void conditions are shown in Fig. 3.15 b and d. The increased standard deviation in the measurement of the welds containing voids implies that there will be less reliability of the in-use strength of such welds compared to the welds with reduced voids.

This increased scattering in measurement may be linked to the unpredictable morphology of the voids which impacts the temperature distribution and the effectiveness of the FZ tempering. Hence, reducing the effect of voids during tempering is key to obtaining consistent mechanical properties of tempered welds.

3.5.5 Failure Mode

The failure mode during CT test was investigated for tempered welds with reduced void conditions that showed minor deviation in the mechanical properties in Fig. 3.15 b and d. The failure mode observed for the BWS as shown in Fig. 3.16a was a partial interfacial failure (PIF) otherwise known as an interfacial fracture with button pull according to AWS D8.1M [144] fracture mode 5. In this failure mode, the crack initiates at the notch tip and propagates into the FZ. Thereafter, the crack propagation path is redirected in the through-thickness direction as shown from the interrupted test in Fig. 3.16b. Liu et al. [145] described the stages and mechanism of this mode as crack initiation, crack propagation and instantaneous fracture corresponding to the separation of weldment due to crack diversion. For the BWS case, the fracture surface shows cleavage features indicating a brittle and instantaneous failure through the sheet thickness as shown from the fractography in Fig. 3.17b.

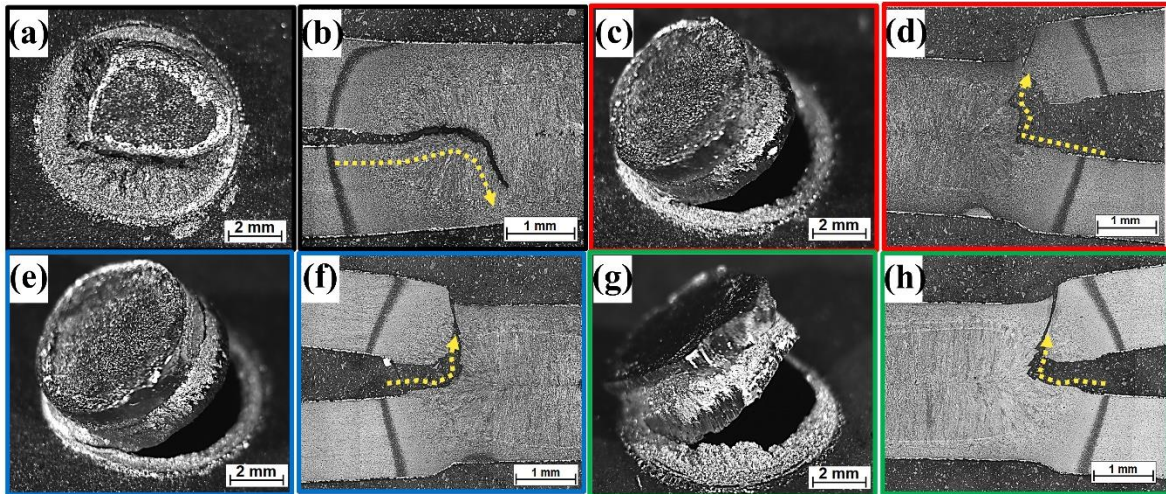


Fig. 3.16: Stereographic images and metallographic cross-sections showing the failure modes and crack propagation paths from interrupted CT tests for BWS (a and b); tempering at 8.0 kA (c and d); 8.8 kA (e and f); and 9.6 kA (g and h).

The temper pulse samples showed pullout failure mode as shown in Fig. 3.16 (c-h) corresponding to fracture mode 1 according to AWS D8.1M [144]. It was observed that tempering resulted in crack blunting, and deviated crack propagation to the through-thickness direction at the fusion boundary compared to the BWS schedule. For the optimal temper pulse

schedule welded at 8.8kA, the crack initiates at the notch and propagates towards the upper critical heat affected zone (UCHAZ), close to the FZ boundary as shown in Fig. 3.16f. Similar findings by Park et al. [128] revealed that such crack propagation in the coarse grain HAZ (CGHAZ) proceeds along the boundaries of the prior austenite grains. The fracture surface at 8.8 kA in Fig. 3.17d showed dimples features indicating a ductile failure in contrast to the cleavage facet observed for the BWS in Fig. 3.17b. Overall, these results indicate tempering as an effective pathway to increase the energy required for crack propagation during loading in the CT mode.

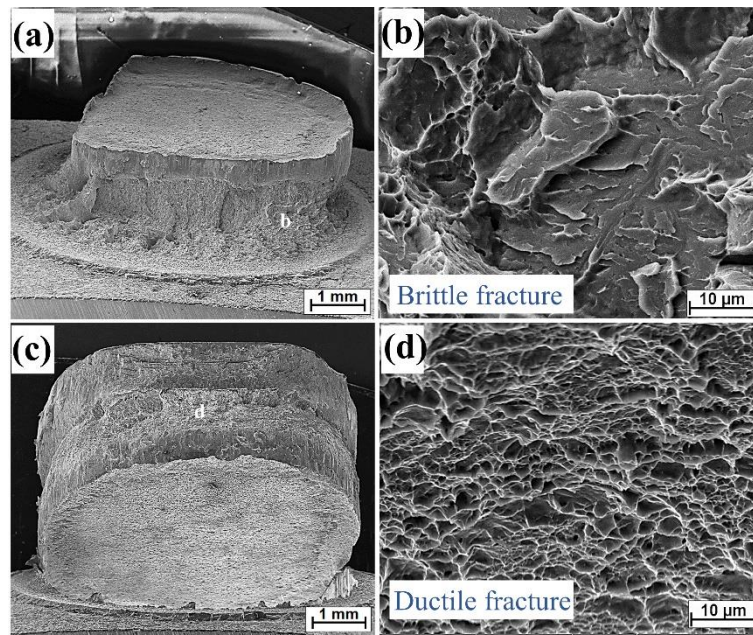


Fig. 3.17: SEM fractography images of BWS (a and b); and temper pulse at 8.8 kA (c and d) after cross-tension tests.

3.5.6 Microstructural examination

The microstructural features dictated by the thermal cycle during the RSW process govern the resulting FZ hardness, mechanical properties and failure modes of the joint. The microstructural features at the weld center and periphery for the BWS and the TPS at 8.8 kA after reducing the voids are shown in Fig. 3.18. The red boxes on the hardness maps indicate where the SEM images were taken. The weld periphery for the BWS in Fig. 3.18a shows an island of martensite which is typically expected due to the rapid cooling rates inherent in the RSW process. At the weld center (Fig. 3.18b), packets of lathy martensitic structure were observed which has been reported to have a low-angle boundary [94]. The martensitic structure shows relatively low energy absorption and offers less resistance to crack propagation.

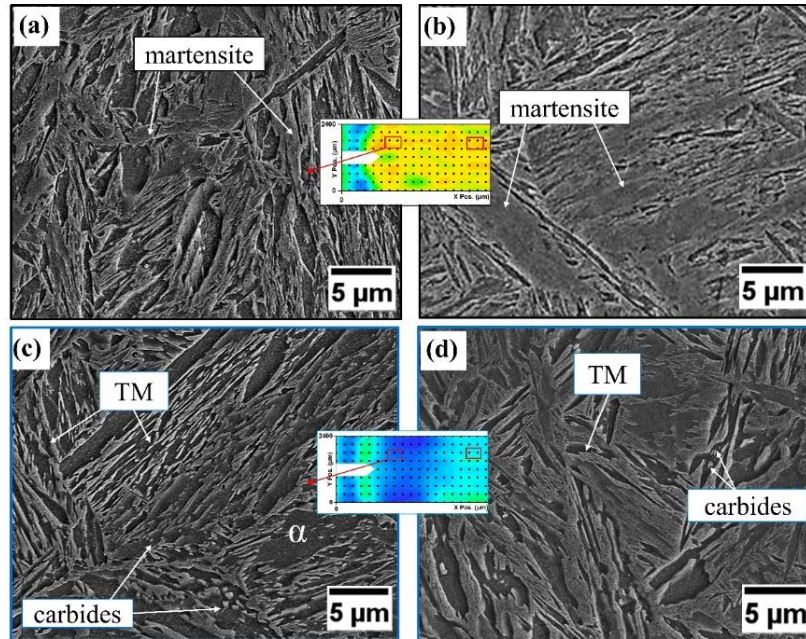


*Fig. 3.18: Microstructural features at weld periphery and center for BWS (a and b); and temper current of 8.8kA (c and d). *TM: Tempered martensite.*

When the nugget center and periphery of the tempered sample were observed (Fig. 3.18c and d), it was seen that the packets of lathy martensite structure disappeared indicating the migration or disintegration of the prior lath boundaries. The tempered microstructure attained from 8.8 kA tempering current showed precipitates of carbides in the α matrix which are more prevalent at the periphery of the weld. These carbide precipitates have been studied extensively and reports suggest that finer carbides precipitate during non-isothermal tempering with preferred locations within laths and interlath regions rather than at prior austenite or block boundaries [79]. The finer precipitates observed in the tempered sample suggest that the FZ temperature was sufficient for the disintegration of the martensitic packets existing from the BWS condition. It is noteworthy that the size and morphology of the precipitated carbides in the tempered condition at 8.8 kA can delay recovery process by pinning dislocation movement [94]. The precipitated carbides observed in the tempered microstructure support the failure mode and crack propagation path observed in Fig. 3.16f where a barrier to crack propagation was observed at the nugget periphery which could be attributed to impediment of dislocations by precipitated carbides.

3.6 Summary

This chapter has explored different temper time and currents combinations to develop an optimal tempering schedule for PHS spot-welded joints. It was observed that tempering of welds with reduced voids in the FZ led to improved peak load and failure energy with more consistency while tempering of welds with voids affects the effectiveness of the process and increases scattering in measurement of the resulting mechanical properties. Based on the experimental work, the following conclusions can be drawn:

1. Applying a post-weld tempering schedule leads to the decomposition of the martensitic laths in the BWS condition which were transformed to a tempered martensitic microstructure with carbide precipitates that deviates the crack propagation path at the edge of the FZ.
2. Voids formed during the weld schedule affect the temperature distribution in the nugget during post-weld tempering thereby resulting in the variation of hardness at the weld center and periphery.
3. Applying a pre-pulse proved effective in reducing the average size of the voids by 60%. Welds with reduced voids are preferred to achieve effective tempering due to the temperature homogeneity attainable in the FZ during PWHT.
4. The optimal tempering schedule of 4 cycles and 8.8 kA after void reduction showed an improved cross-tension peak load of 36 % and 33 % increase in energy absorption compared to welds not subjected to heat treatment. Similarly, the failure mode transitioned from a PIF mode in the BWS condition to a PO mode after applying the optimal post-weld tempering schedule.
5. The reduction of voids in tempered welds by applying a pre-pulse leads to a reduced standard deviation in the mean peak load and energy absorption capacity resulting in a more consistent mechanical performance of tempered welds. Hence, reducing voids in welds is essential for effective post-weld heat treatment.

CHAPTER

4

4. In-situ Grain Refinement in Spot Welds: A Paradigm of the Metallurgical Transformation and Mechanical Properties³

³ This chapter is composed of a published research paper as outlined in the statement of contributions. **O.T. Betiku**, D.C. Ramachandran, A. Ghatei-kalashami, C. DiGiovanni, O. Sherepenko, H. Ghassemi-Armaki, E. Biro, Improving the mechanical performance of press-hardened steel resistance spot welds via in-situ grain refinement, J. Mater. Process. Technol. (2023) 118122.

4.1 Overview

From the previous tempering study, it was observed that the microstructure at the edge of the FZ is crucial in improving the mechanical properties and failure mode of RSW welds. This chapter focuses on refining the grain structure specifically at the edge of the FZ around the notch, which is known to be the preferred site for crack initiation and propagation [9,115]. However, the metallurgical transformation sequence to achieve in-situ grain refinement during the non-equilibrium RSW process needs to be established. Hence, this chapter investigates how various in-situ PWHT parameters (particularly cool time and post-weld time) dictate the transformation occurring in the weld. Findings revealed that applying the post-weld current when the edge of the FZ is austenitic (after a short cool time) and below the melting point prompts in-situ grain refinement from columnar to a recrystallized equiaxed structure. Meanwhile, after a longer cool time, when the edge of the FZ cools below the M_s temperature, reheating the edge of the FZ above the austenite start temperature results in martensite reversion to austenite with the grain structure similar to the initial epitaxial structure after cooling. The result indicates that in-situ grain refinement can be achieved through a PWHT schedule that involves reheating the edge of the FZ to initiate austenite recrystallization. Moreover, the formation of new equiaxed grains from the austenite recrystallization process effectively retards crack propagation during cross-tension test resulting in 89% improvement in the energy absorption capability compared to the baseline condition. Therefore, optimizing in-situ PWHT parameters to initiate austenite recrystallization at the edge of the FZ is critical to enhancing joint performance and overall crashworthiness.

4.2 Background

The martensitic microstructure formed in the FZ as a result of the rapid cooling ($\sim 10^3$ to 10^5 °C/s) inherent with the RSW process exhibits relatively low toughness when subjected to loading condition as shown in chapter 3. Likewise, the rapid cooling also results in the formation of a columnar structure from the epitaxial solidification towards the weld centerline [56]. The columnar grain structure is known to also promote low-energy crack propagation leading to a reduced mechanical performance and an undesirable brittle failure [10,146]. Thus, it is imperative to modify the grain structure to improve the performance of spot welds, which is a criterion to enhance crashworthiness and the structural integrity of a vehicle.

Earlier studies by Hernandez et al., [111] observed the formation of quasi-equiaxed grain morphology in the FZ after applying a PWHT current. The findings of that work suggested the possibility of recrystallization occurrence in weld nugget, however, there was no detailed description of the how the changes occurred. Subsequently, Chabok et al., [112] employed a

post-weld pulse scheme on dual-phase (DP) steels and observed a sub-division of the FZ. The outer part of the FZ was referred to as the recrystallized zone also known as the “rex-zone”. The rex-zone had equiaxed prior austenite grains (PAG) containing martensite packets while the inner part of the FZ had a columnar grain structure. It was established that the rex-zone (formed after the post-weld pulse) and the inner part of the FZ exhibit different features as the rex-zone was reported to pose more barriers to crack propagation since the crack is forced to navigate several equiaxed PAGs in the rex-zone. In contrast, Sajjadi-Nikoo et al., [106] reported the formation of an equiaxed zone in the inner part of the nugget while the outer region remains unaffected. Despite the improvement in mechanical properties reported in these studies, the underlying microstructural evolution was not detailed with each PWHT schedule employed for each study.

In addition to tempering and changes in the grain morphology in the FZ, PWHT has also been used to redistribute segregated alloying elements at the edge of the FZ. Eftekharimilani et al., [9] reported that the application of a PWHT pulse led to the homogenization of alloying elements such as phosphorus and manganese at the edge of the FZ in a third-generation (3G) 1 GPa ultimate tensile strength AHSS. The reduced segregation of alloying elements after PWHT was reported to improve the mechanical properties of the joint, which is in agreement with the findings of Sawanishi et al., [11]. Though Stadler et al., [103] did not observe any difference in phosphorus distribution after a PWHT pulse, they reported softening of the sub-critical HAZ (SCHAZ) in a transformation-induced plasticity (TRIP)-aided bainitic ferrite (TBF) steel. The softening of the SCHAZ has been reported to be beneficial in reducing stress concentration around the edge of the FZ (the location where the fusion boundary meets the faying surface) and promoting failure outside the weld during cross-tension test [112]. Nonetheless, it is worth mentioning that failure within the FZ under mode 1 loading is mostly dependent on the fracture toughness of the weld [147], as well as the characteristics of the martensite hierarchical microstructure such as the size of the block, packet and ultimately PAG [148]. According to Zhang et al., [149], a refined finer PAG size contributes to enhancing the strength of a martensitic steel.

A major similarity of previous studies investigating in-situ recrystallization is the short cooling time (40-160) ms between the weld schedule and the PWHT pulse. The short cooling time is more beneficial for industrial applications in terms of minimizing the overall production time compared to the longer cooling time (1000-2000) ms needed to achieve martensitic tempering in PHS earlier reported by Chen et al., [89]. Stadler et al., [116] compared different cooling

times and obtained 50 ms to be the optimal time to modify the elongated cast-like structure in an as-welded 1.18 mm thick medium Mn-steel. However, the chemical composition and thickness of different AHSS grades could alter the optimum cooling time and post-weld parameters (pulse current and time) needed to achieve effective PWHT [53]. Likewise, post-weld current influences the thermal cycle at various regions of the weld and can either improve or deteriorate the mechanical properties [10]. Therefore, it is important to optimize the process parameters to transform the microstructure at the edge of the FZ to an equiaxed grain morphology so that the temperature on reheating is above the A_{C3} but below the melting point [111].

Though post-pulsing has been adopted as an effective method to modify microstructures and improve mechanical properties in spot welds, the correlation between various post-weld parameters and microstructural changes has not been reported. Specifically, it is unknown how different in-situ post-weld heat treatment parameters alter the grain morphology of PHS spot welds which can be pivotal to improving the joint performance in service. This work examines the influence of a recrystallization pulse on the microstructural features and the consequent changes in the mechanical performance of spot-welded PHS.

4.3 Material and Methods

4.3.1 Material

This study was carried out on Al-Si coated 22MnB5, otherwise referred to as PHS. The sheet thickness of the PHS was 1.4 mm and each side had an average coating thickness of $35.0\ \mu\text{m} \pm 0.7\ \mu\text{m}$ measured from 9 samples as shown in Fig. 4.1a and b. The chemical composition and base metal mechanical properties was reported in chapter 3. The PAGs of the base metal are shown in Fig. 4.1b with an average grain size of $7.13\ \mu\text{m} \pm 0.6\ \mu\text{m}$ obtained using linear intercept method (which will be detailed in section 4.3.2). The PHS sheet was prepared by shearing 500 x 500 mm coupons which were hot stamped. The hot stamping was performed by heating the coupons in a furnace to 930°C and holding for 5 mins to achieve austenitization. After homogenization of the austenitic phase, the material was stamped in a water-cooled die resulting in a fully martensitic microstructure as shown in Fig. 4.1a. The surface of the hot stamped sheet was cleaned with ethanol before the welding process.

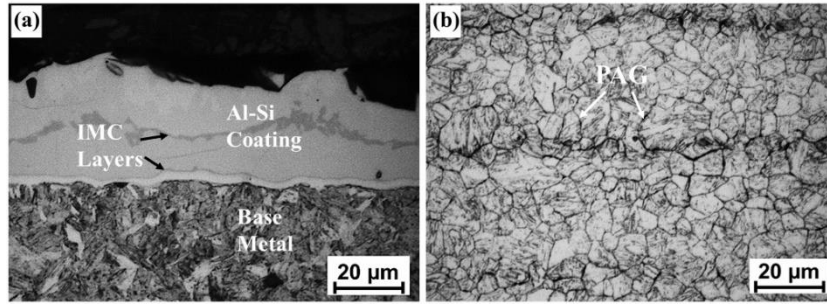


Fig. 4.1: (a) Optical microscope of base metal with Al-Si coating; (b) Grain structure of base metal showing PAG (c) SEM images of base metal with Al-Si coating (d) microstructure of PHS after hot stamping showing the martensitic microstructure.

4.3.2 Resistance spot welding

Welds were made according to the specifications of AWS D8.9M [32] standard as shown in Fig. 4.2a and earlier described as baseline welding schedule (BWS). A welding current of 8.0 kA resulted in an average nugget diameter of 6.05 ± 0.03 mm and was selected as the baseline current. To study the effect of the post-weld schedule, the cool time (CT) which is the time between the weld pulse and post-weld pulse was varied from (40-300) ms while the post-weld time (PT) was varied from (167-334) ms as shown in Fig. 4.2b. It should be noted that a short pre-pulse of 20 ms and 16 kA was used for all welds to remove the coating at the faying surface. This pre-pulse (studied in section 3.5.3 of chapter 3) reduces the formation of voids, which would otherwise lead to a heterogenous current distribution of the post-weld current.

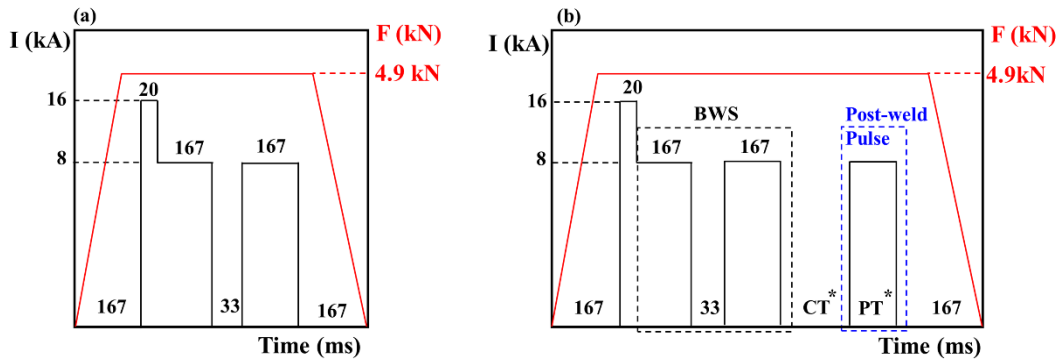


Fig. 4.2: Schematic representation of (a) baseline weld schedule (BWS), and (b) BWS followed by post-weld pulse. *CT is the cool time which was varied from (40-300) ms while PT is the post-weld time varied from (167-334) ms to develop the process map.

4.3.3 Microstructural examination

Samples were prepared using standard metallography procedures for both optical and electron microscopy investigations. Welds were sectioned along their diameter followed by grinding and polishing with the final polishing step using 1 μ m diamond suspension. Etching was done

using 2% Nital (98% ethanol + 2% nitric acid) to measure the nugget diameter, while an etchant consisting of 4% saturated aqueous picric acid, 0.5 g of a wetting agent (sodium dodecylbenzene sulfonate) and 3 drops of HCl was used to reveal the PAG boundaries. A Zeiss optical microscope was used to obtain images at the edge of the fusion zone in front of the notch. The average grain size was measured using the linear intercept method conforming to ASTM E112 [150]. For the linear intercept method, eight lines were drawn per micrograph (two micrographs per condition) both in the horizontal and vertical directions with each line intercepting a minimum of six grains. The grain aspect ratio (GAR) which is the ratio of the major axis to the minor axis of the grains was obtained for a minimum of ten grains per condition. Further detailed microstructural analysis of the grain morphology and fracture surface was observed using a JOEL JSM 7000F field emission scanning electron microscope (SEM) equipped with an electron backscattered diffraction (EBSD) detector. Data acquisition during EBSD scans was obtained at a working distance of 15 mm with a step size of 0.5 μm operating at 20 kV. The EBSD data was analyzed using the MTEX toolkit in MATLAB [151] while the PAG boundaries were reconstructed using ARPGE software [152].

4.3.4 Hardness Measurement

The hardness measurements were carried out across different regions including the FZ, HAZ and BM using an automated Clemex microhardness machine with a Vickers indenter. A load of 200 gf was applied for a dwell time of 10 s. The measurements were performed using an indentation pattern conforming to SEP 1220-2 [153] and the distance between indents was 200 μm , which is greater than three times the diagonal of each indent as stated in ASTM E384 [154]. The average FZ hardness was obtained by averaging 15 indents across the diagonal in the fusion zone. Additionally, twelve indents were averaged to obtain the hardness at the front of the notch similar to where the microstructure was obtained as shown in Fig. 4.3. The error bars for the hardness measurement represent the 95% confidence interval.

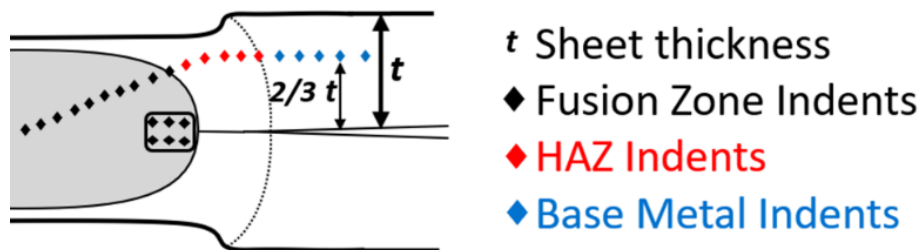


Fig. 4.3: Schematic illustrating various regions for hardness measurements.

4.3.5 Mechanical test

The mechanical performance of the joint was determined by cross-tension test according to the AWS D8.9M [32] standard. Welds were made on specimens with a dimension of (150 x 50) mm similar to the geometry in chapter 3 (Fig. 3.6) for cross-tension test using the BWS, and a selected post-weld pulse parameter as shown in Table 4.1. The mechanical tests were performed on a 100 kN MTS frame universal tester at a constant crosshead speed of 10 mm/min. The average peak load was estimated by averaging three repetitions, while the energy absorbed to failure was obtained by integrating the area under the force-displacement curve. The error bars presented indicate a 95% confidence interval. The crack propagation path during tensile loading was observed by interrupting the cross-tension test at 90% of the average peak load for each welding condition, thereafter, welds were cross-sectioned, etched and observed by optical microscopy.

Table 4.1: Resistance spot welding parameters for the BWS and optimized PWHT schedule.

Weld Schedule	Squeeze time (ms)	Weld Pulse 1 (ms)	Cool time 1 (ms)	Weld Pulse 2 (ms)	Weld and post pulse Current (kA)	Cool time 2 (ms)*	Post Pulse time (ms)*	Hold time (ms)
BWS	167	167	33	167	8	-	-	-
PWHT Schedule	167	167	33	167	8	100	167	167

**In addition to the BWS and optimal PWHT schedule, mechanical testing of other CT of (40, 200 and 300) ms and PT of (167, 250 and 334) ms were conducted.*

4.3.6 Numerical simulation of the welding process

The welding parameters were simulated to obtain the thermal history of the RSW process. The simulation was done using a commercially available finite element (FE) Simulation and Optimization of Resistance Projection and Spot-welding process (SORPAS) software V13.95. The RSW process is simulated in SORPAS with four numerical models such as the electrical, thermal, metallurgical and mechanical model [155]. The material properties of the Al-Si PHS were obtained from the software database. The FE model consisted of over 4000 nodes with refined mesh in the FZ. Simulations were used to obtain the state of the nugget after different cool times before the post-weld pulse.

4.4 Result and Discussion

4.4.1 Process map

The development of a process window is a key element in understanding process efficiency with different parameter combinations. The process map developed in this study combined different cool times (CT) (i.e the time before applying the PWHT pulse) and post-weld times (PT) at a constant current of 8 kA. The average grain size showed a range of size distributions at the edge of the FZ. The average grain size for the welding schedule with a CT of 100 ms and PT of 167 ms was 36 μm and varied to 82 μm for the weld schedule with a CT of 300 ms and PT of 334 ms, as shown on the process map in Fig. 4.4a. Likewise, the grain morphology for different parameter combinations showed a variation in the grain aspect ratio as shown on the map in Fig. 4.4b. Overall, it was observed that at a CT between 75-100 ms and PT between 167-200 ms, the grain morphology was mostly equiaxed at the edge of the FZ with an aspect ratio between 1.0 and 1.2. In contrast, a longer pulse time shows either an equi-columnar or elongated columnar grain structure with an aspect ratio between 1.75 and 2.0. The schematic of the region of interest (i.e at the edge of the FZ) and optical micrographs of the different grain morphology for equiaxed, equi-columnar and elongated columnar grains are presented in Fig. 4.4 (c-f).

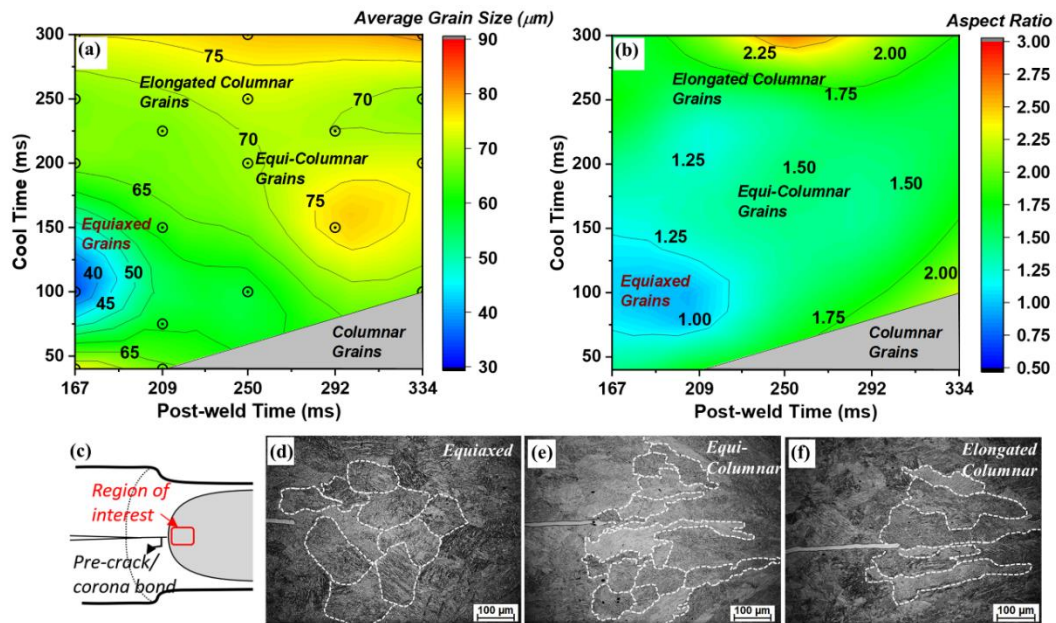


Fig. 4.4: (a) Process map showing the average grain size at the edge of the FZ with various parameter combinations of CT and PT; (b) aspect ratio map; (c) schematic illustrating the region of grain analysis; Region of interest optical micrograph with different grain morphologies for (d) equiaxed grains (e) equi-columnar and (f) elongated columnar grains.

From the process map, it was observed that a longer CT from 150-300 ms predominantly showed mostly equi-columnar (a mixture of equiaxed and columnar grains) and elongated columnar grains dominating the microstructure at the edge of the FZ. It is believed that after a longer CT, more heat was dissipated from the weld, which is indicative of a lower temperature at the edge of the FZ before the application of the post-weld pulse. Hence, the thermal energy in the weld was insufficient for the formation of new equiaxed grains all through the structure at the edge of the FZ. In addition, the longer CT could allow the diffusion of alloying elements like Mn which retards the migration of the grain boundaries by solute drag effect thereby delaying recrystallization [156].

4.4.2 Dynamic resistance curves during the PWHT schedule

Dynamic resistance curves have become a major tool to monitor transient spot welding process control variables and characterize phases in real-time [41]. The dynamic resistance curves were extracted from the welding process (which includes the BWS, different cool times and the post-weld time) to better comprehend the effect of different cool times on the recrystallization mechanism occurring at the edge of the FZ. The dynamic resistance during the BWS is presented with the black line for the first 387 ms as shown in Fig. 4.5a which is inclusive of the pre-pulse applied for 20 ms. The trend in dynamic resistance during the BWS is well known as Dickinson et al., [40] have explained its important features such as the α -trough and β -peak points and how it relates to material changes during the welding cycle. The dynamic resistance curve during the BWS cycle was in-line with the reported literature. However, various trends in resistance were observed for the different PWHT parameters.

When the dynamic resistance curves were compared for the schedules made with various CT from 40-300 ms, the resistance showed different behaviours at the start of the post-weld pulse. It was observed that the resistance reduced from 153 $\mu\Omega$ at the end of the weld schedule to 141 $\mu\Omega$ after 40 ms CT at the start of the post-pulse and slightly increased to 147 $\mu\Omega$ during the post-weld pulse as shown in Fig. 4.5. Similarly, after 100 ms CT, the resistance reduces even more to 131 $\mu\Omega$ at the beginning of the post-pulse schedule and increased to 140 $\mu\Omega$ as the post-pulse was applied. For both cases, the reduction in resistance at the start of the post-pulse was due to the reduction in weld temperature during cooling before the application of the post-pulse current which is in agreement with Stadler et al., [116] findings.

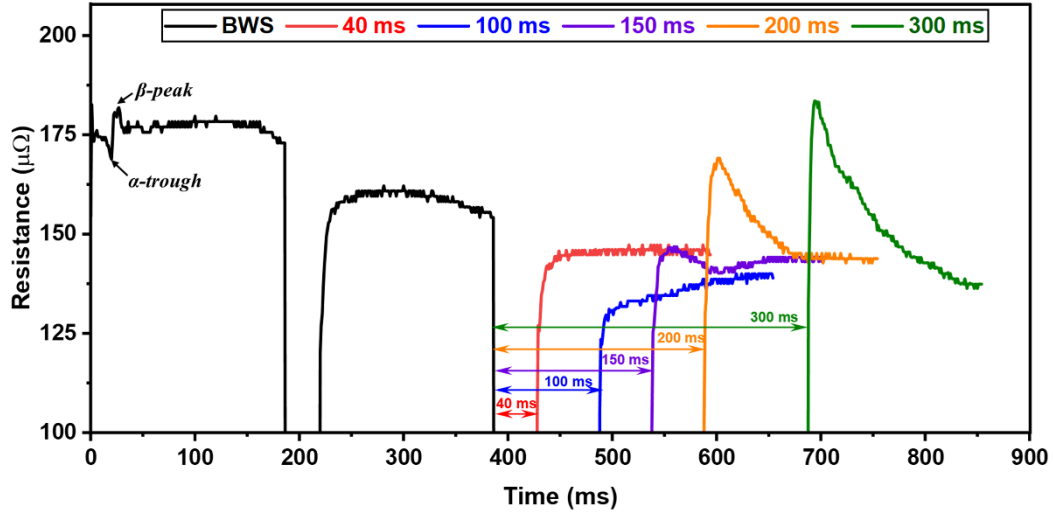


Fig. 4.5: Dynamic resistance curves extracted after the welding process showing the resistance for the BWS and post-pulse schedules.

At a CT from 150-300 ms, a different trend in the dynamic resistance was observed. The resistance at the beginning of the post-pulse after 150 ms, 200 ms and 300 ms increased to 145 $\mu\Omega$, 166 $\mu\Omega$ and 181 $\mu\Omega$, respectively. It is presumed that at each of these conditions, two mechanisms were competing. First, there is the decreased electrical resistivity with decreasing temperature, and second, there is the higher electrical resistivity of the solid phase compared to the liquid [34]. As temperature will decrease as CT increases, it is expected that the overall temperature of the nugget will cool thereby decreasing resistivity. However, solidification also progresses with increasing CT which decreases the phase fraction of liquid and increases the solid phase in the nugget thereby increasing resistivity. It was observed from simulations that after 150 ms, the nugget is completely solid and at a lower temperature as shown in Fig. 4.6. Hence, the resistance to the current flow from the solid metal is believed to be the dominating factor, leading to the increased dynamic resistance observed after CT greater than 150 ms compared to the 100 ms CT where the last solidifying liquid occurs. Additionally, it was observed that the resistance decreases as the post-weld current is applied indicating the growth of the liquid metal with a lower resistivity. The decrease in resistance during the PT has been reported to be due to the increasing size of the molten region and cross-sectional area available for current flow [40].

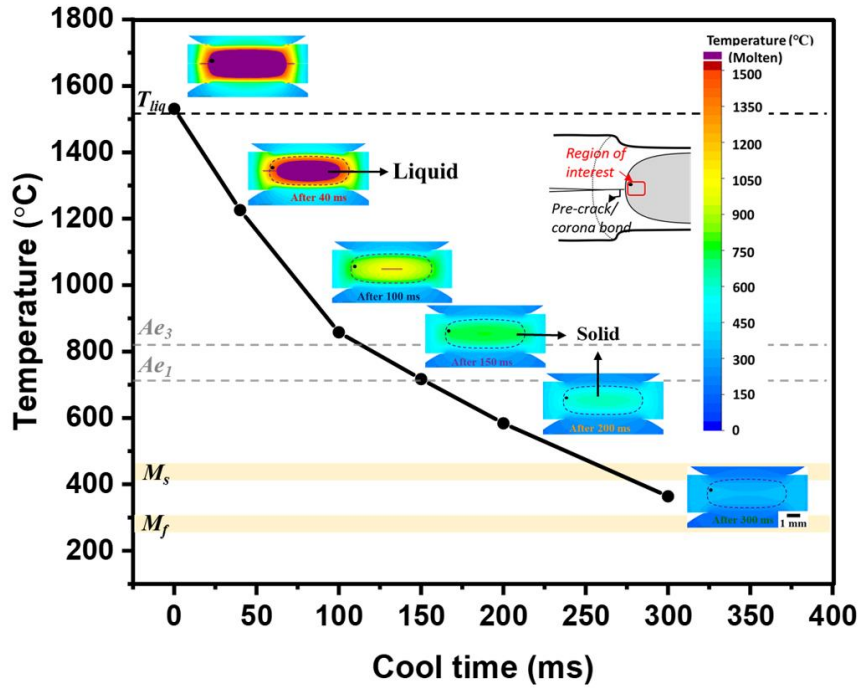


Fig. 4.6: Temperature at the edge of the FZ after selected CT. The image insert indicates the state of the nugget after the CT of 40 ms; 100 ms; 150 ms; 200 ms and 300 ms before the post-weld current. *Black dots indicate the node where the temperature was extracted.

Based on the temperature history shown in Fig. 4.6, the temperature at the edge of the FZ after a CT of 300 ms was 364 °C which is below the martensite start temperature, M_s . The reported M_s for PHS ranges between (402-439)°C, and is dependent on cooling rates, deformation and initial PAG size [157]. Moreover, Shiga et al., [158] showed that the M_s temperature increases up to 30 °C due to stress-induced transformation caused by the welding process. The mechanism of stress-induced transformation has been explained in detail by Olson and Cohen [159]. Thus, a solid-state transformation must have occurred after a CT of 300 ms resulting in a high fraction of martensite which inherently has more lattice defects (dislocations, lath boundaries) that could contribute to the overall increase in resistivity [160]. In other words, the phase transformation occurring during the cooling period also contributes to the increase in resistance at longer CT as shown in Fig. 4.5. The trend in the dynamic resistance observed in this study for the longer CT is contrary to the findings of Stadler et al., [116], where the dynamic resistance decreased at the start of the PWHT pulse with increasing CT. However, it is worth mentioning that the investigated steel in that study (i.e., medium Mn-steel) was different from this current work.

The result of the dynamic resistance and temperature profile with the various PWHT schedules can be correlated with the observed changes in grain size and morphology as earlier shown in

Fig. 4.4a and b. It was seen that the grain refinement with a lower aspect ratio occurred at a short CT between 75-100 ms and PT of 167 ms. During this time, the post-pulse was applied when the edge of the FZ was still austenitic and would have been reheated below the melting point. Meanwhile, at low CT of 40 ms and high PT, reheating led to remelting and the longer CT of 300 ms led to a transformation to martensite before reheating. It is believed that the CT of 150 ms and 200 ms with a short PT of 167 ms resulted in partial recrystallization with an aspect ratio of 1.25. The observed grains were equi-columnar as shown in Fig. 4.4e as some of the grains were still elongated indicating that the thermal energy was insufficient for complete recrystallization. Overall, the use of dynamic resistance curves can be a useful in-situ tool to understand the state of the nugget before applying a post-weld pulse schedule.

4.4.3 Microstructural evolution during in-situ PWHT

Microstructural evolution is integral to understanding the sequence of transformation induced by the PWHT schedule. It has been established that a long CT of 300 ms resulted in the formation of martensite and an unrefined grain structure after the PWHT pulse. The unrefined grain structure observed with a CT of 300 ms was due to the diffusionless phase transformation, resulting in a martensitic (α') microstructure on cooling. On reheating, the martensite then reverted to austenite (γ') retaining the same structure of the martensite (and prior austenite grains), as illustrated in the schematic in Fig. 4.7a. This martensite-austenite reversion process sequence has been reported by Sajjadi-Nikoo et al., [106] during rapid reheating of TRIP steel and the process is known to be a displacive transformation according to Nakada et al., [161]. After the final cooling, martensite is again formed, retaining the morphology of the PAG structure with a higher aspect ratio (>2) as shown in Fig. 4.4b.

In the case of a CT of 100 ms, the edge of the FZ is at a sufficiently elevated temperature for the austenite (γ) to be stable. Thus, reheating during the post-weld pulse for 167 ms provides energy for the nucleation of new-austenite grains (γ_2) within the structure. The nucleation of new grains mostly occurs at the prior γ boundaries [162]. The new-austenite grains then grow in all directions to reduce the overall grain boundary surface energy as shown in Fig. 4.7b. After the PWHT current pulse, the edge of the FZ cools and martensite is formed within the new refined PAG structure with a lower aspect ratio. Therefore, both recrystallization on reheating and phase transformation to martensite after the final cooling occurred in the welds made using a CT of 100 ms and PT of 167 ms resulting in a refined grain structure at the edge of the FZ.

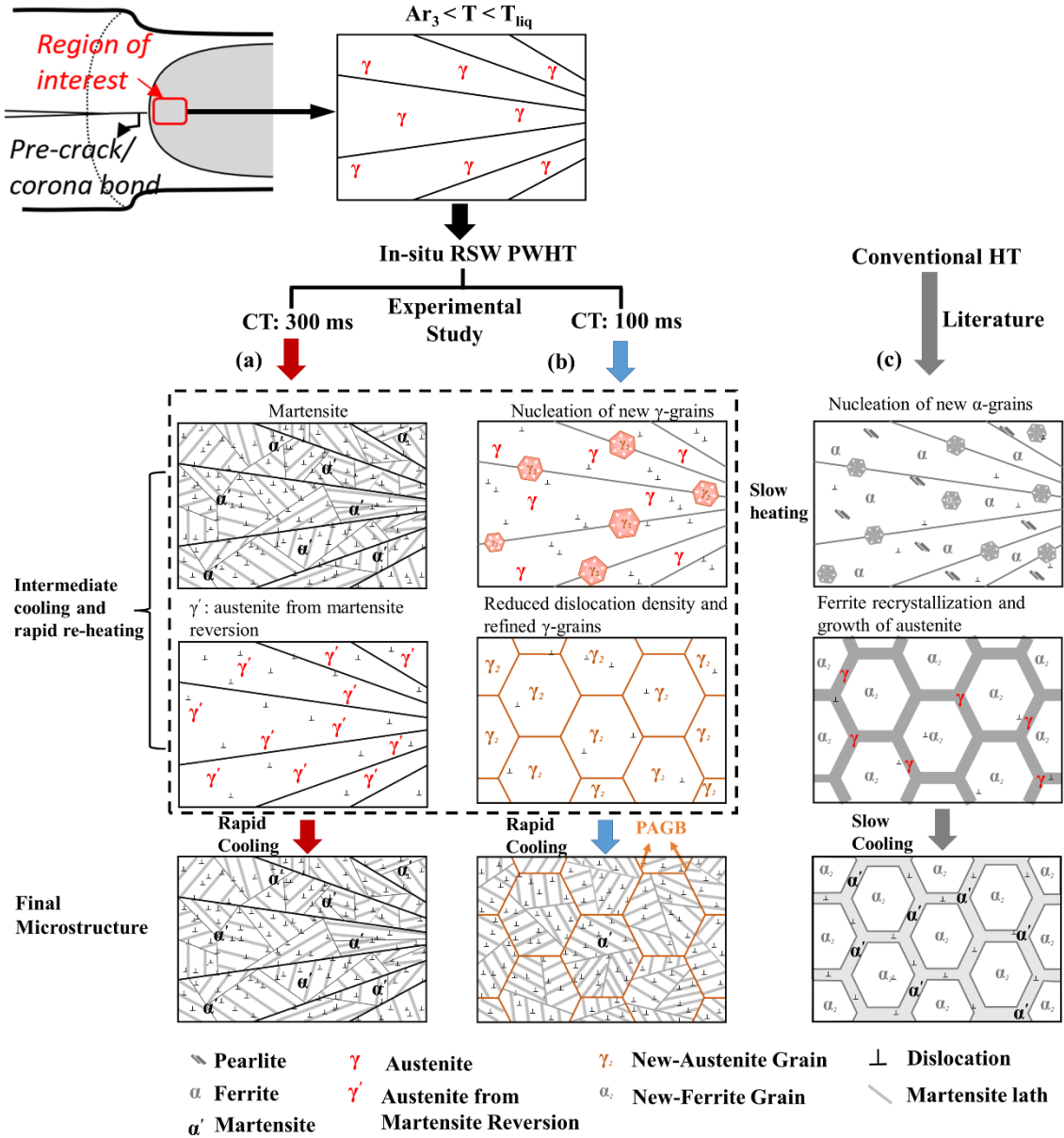


Fig. 4.7: Schematic representation of the phase transformation and grain refinement occurring at the edge of the FZ with a CT of (a) 300 ms (b) 100 ms during the RSW process; and (c) slow cooling; microstructural evolution for slow cooling summarized from Bandi et al., [163].

It is worth mentioning that previous work on conventional heat treatment (HT) described the occurrence of an overlap between ferrite recrystallization and austenite formation when the material was heated from room temperature to above the Ac_1 . Bandi et al., [163] showed that during slow heating (7 °C/s), the recrystallization of polygonal ferrite grains is completed while the growth of austenite occurs at the recrystallized ferrite grain boundaries as shown in Fig. 4.7c. The austenite then transforms to martensite at a relatively slower cooling rate (~150 °C/s). The details of the transformation mechanism and microstructural evolution have been described extensively by Bandi et al., [163] and supports the assertion made by Chbihi et

al., [156]. Though the findings of these earlier studies are plausible, they did not account for the high heating or cooling rates existing during the RSW process. In the case of the RSW process with a higher heating rate ($\sim 7,800\text{ }^{\circ}\text{C/s}$)⁴ and cooling rate ($\sim 4,100\text{ }^{\circ}\text{C/s}$)¹, the recrystallization of new austenite grains was achieved by reheating when the edge of the FZ is austenitic to prevent the retention of the original epitaxial structure. Therefore, optimizing the CT is imperative to achieving in-situ grain refinement of spot welds.

Based on the findings from this study, applying the post-weld pulse in the austenitic phase proves to be more effective for grain refinement than a longer CT which forms martensite before the post-weld pulse. This is evident from the changes in grain morphology as will be discussed in section 4.4.4 and the changes in mechanical properties comparing different CT and PT in Fig. 4.8. From Fig. 4.8a, it can be seen that the shorter CT of 40 ms (where remelting of the edge of the FZ occurred after applying the PWHT current) has comparable peak load with the BWS condition, as statistical analysis showed no significant difference. Likewise, the long CT of 300 ms and PT of 250 ms with a higher grain aspect ratio of 2.7 (from Fig. 4.8b) statistically have comparable peak load with the BWS condition. It is worth mentioning that the formation of small grains with large aspect ratio (indicated by the gray-area in Fig. 4.8b) will result in a high surface area to volume ratio as such will be thermodynamically unstable to form.

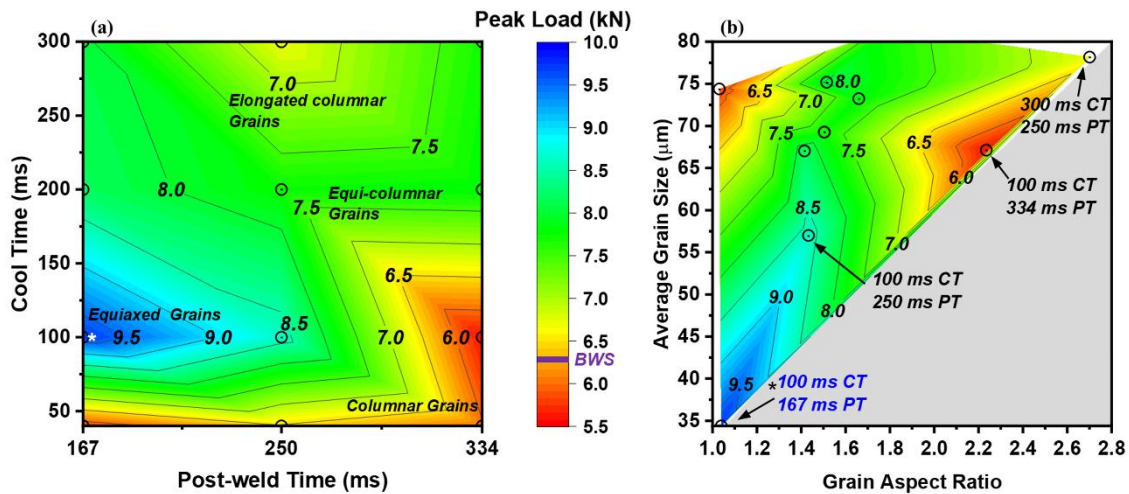


Fig. 4.8: Peak load of BWS and selected PWHT schedules (a) Changes in microstructure (grain size and morphology) with peak load. *condition shows the optimal PWHT schedule (CT: 100 ms; PT 167 ms) which is described in detail in the following sections.

⁴ Heating and cooling rates were calculated from SORPAS simulation for 1.4 mm PHS sheet.

With a CT of 200 ms where it was suggested that partial recrystallization occurred with equi-columnar grain morphology, a 24% increase in peak load was observed. However, with 100 ms CT and 167 ms PT, the peak load is increased by 54% as reheating from the PWHT current resulted in a lower grain size ($35\ \mu\text{m}$) and aspect ratio (1.03) at the edge of the FZ as shown in Fig. 4.8b. Increasing the PWHT time to 250 ms at 100 ms CT led to grain growth as seen on the map in Fig. 4.8b and a reduced peak load compared to the 167 ms PT while at 334 ms PT, melting occurred at the edge of the FZ on applying the PWHT current resulting in comparable peak load with the BWS. Thus, further analysis was based on the CT of 100 ms with a pulse time of 167 ms denoted as the PWHT schedule in Table 4.1.

4.4.4 Microstructural changes at the edge of the FZ

The microstructure of selected parameters for mechanical testing (that is the BWS condition and PWHT schedule at a CT of 100 ms with a PT of 167 ms) are shown in Fig. 4.9a and b. The BWS condition shows a primary nugget with a columnar grain structure which was elongated along the solidification direction from the edge of the FZ towards the center of the nugget as shown in Fig. 4.9a. It is important to state that the images were taken at the edge of the FZ in front of the corona bond as represented by the schematic superimposed on the BWS condition in Fig. 4.9a. After the BWS pulse and a CT of 100 ms, the edge of the FZ is solidified while the center remains liquid. On account of applying the post-weld current for 167 ms, the solid-state region at the edge of the FZ undergoes a recrystallization process that is associated with the rearrangement of dislocation networks and migration of PAG boundaries [10]. This leads to an equiaxed grain structure at the edge of the primary nugget while the grain structure of the molten secondary nugget remains columnar after solidification as shown in Fig. 4.9b.

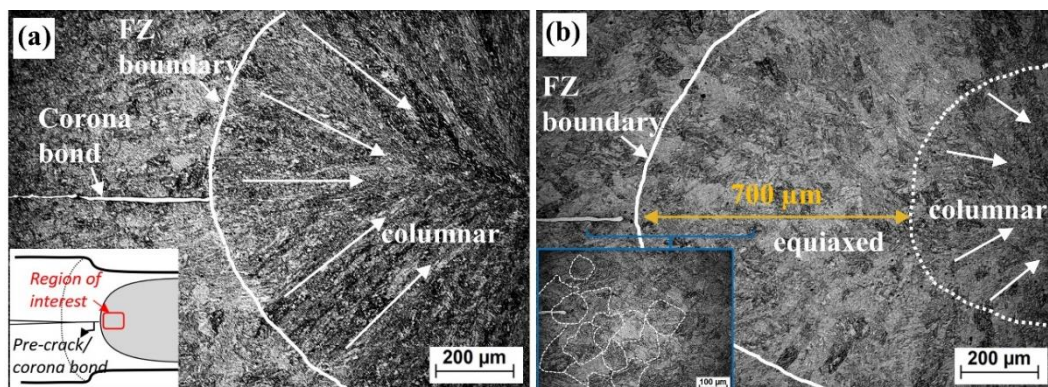


Fig. 4.9: Optical microscope image of the edge of the FZ for (a) BWS; (b) PWHT schedule with CT of 100 ms and PT of 167 ms.

To further visualize the grain morphology at the edge of the FZ, the inverse pole figure (IPF) maps were obtained at the edge of the FZ for both the BWS and the refined zone of the PWHT schedule as shown in Fig. 4.10a and d. It was observed that the PAG morphology was elongated along the solidification direction for the BWS as shown in Fig. 4.10b indicating an unrefined structure after cooling. In contrast, a refined grain morphology mostly equiaxed was observed for the PWHT schedule as shown in Fig. 4.10e. The refined equiaxed grain morphology is attributed to the growth of the recrystallized PAG in all directions. The equiaxed structure is also maintained after the PWHT pulse on cooling. It is worth mentioning that the decrease in the PAG size results in a reduction in the packet and block size of martensite [148]. Likewise, Zhang et al., [149] have shown that the refinement of prior austenite grains (particularly the blocks and packet) follow the Hall-Petch relationship and controls strength and toughness in an as-quenched martensitic steel. The PAG morphology for the BWS condition in this study was elongated in the radial direction of the weld and wide up to 170 μm but was refined after the PWHT pulse resulting in a decrease in average PAG size ($35 \mu\text{m} \pm 6 \mu\text{m}$). Thus, it is believed that the refined PAG grain structure after the PWHT schedule contributes to the changes in mechanical properties as will be discussed in the weld cross-tension properties in section 3.6.

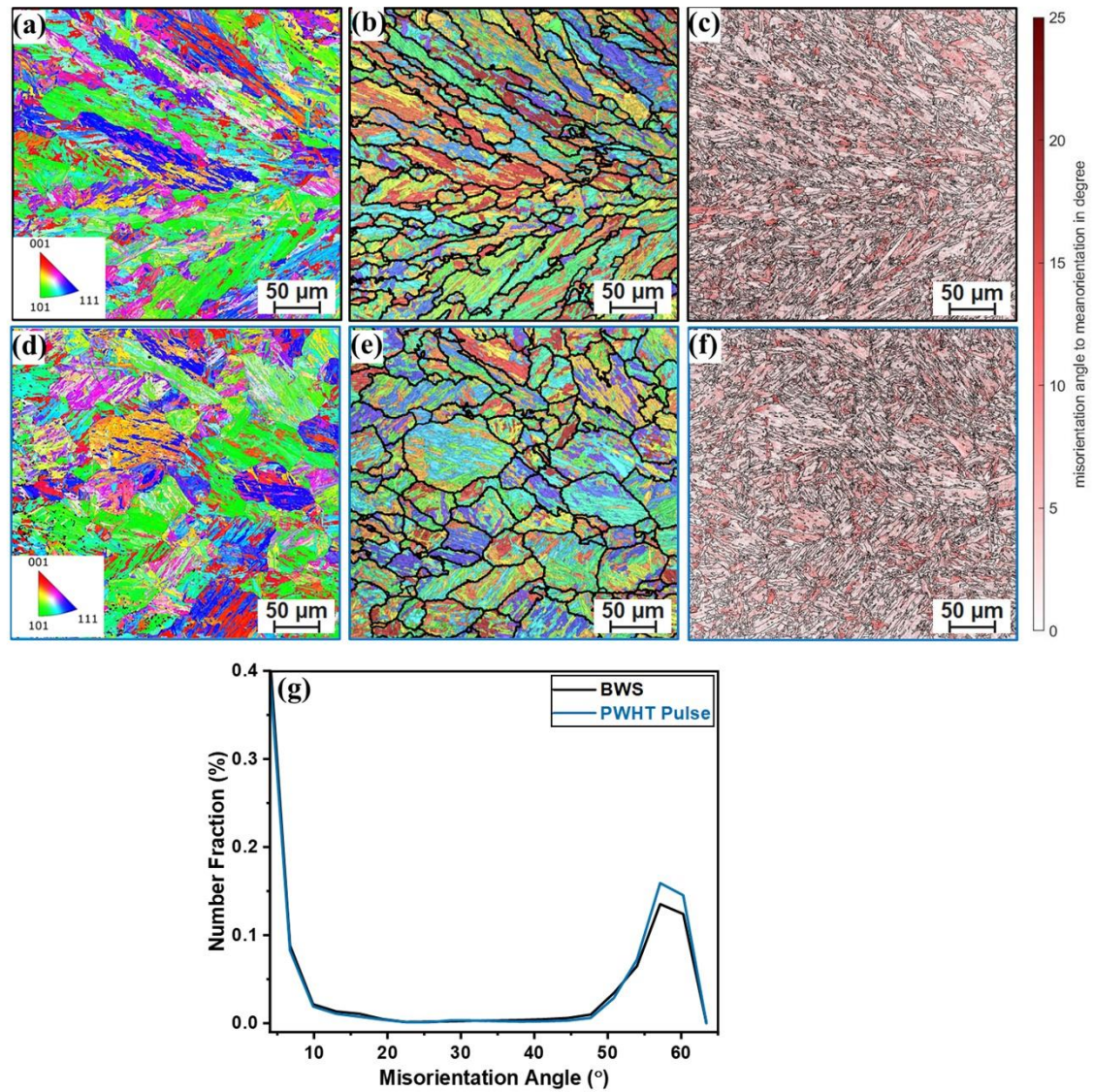


Fig. 4.10: IPF maps at the edge of the FZ for (a) BWS; (d) PWHT pulse; reconstructed maps showing PAG boundaries in black lines for (b) BWS (e) PWHT pulse; GROD maps for (c) BWS, (f) PWHT pulse; and (g) misorientation angle distribution of BWS and PWHT pulse (CT of 100 ms; PT of 167 ms).

The grain reference orientation deviation (GROD) shows the strain in the microstructure by calculating the misorientation between a reference point in a grain and other points [164]. This implies that an increase in the local misorientation of a point in a grain with respect to other points indicates a higher local residual strain in the lattice. The GROD maps for the BWS and PWHT pulse condition indicate a high level of strain in the microstructure and there was no remarkable difference in internal strain after applying the PWHT pulse as shown in Fig. 4.10c and f. The high strain observed in both conditions is associated with the martensitic transformation because of the rapid cooling experienced during the RSW process. Nonetheless,

it is worth mentioning that the internal strain in martensite can be minimized by Ni alloying [74].

In general, grain boundaries (GBs) are mostly characterized by low angle GB (LAGB) and high-angle GB (HAGB), where 15° is commonly used as a transition between LAGB and HAGB [165]. Interestingly, the misorientation distribution between the grains reveals that the PWHT pulse leads to a higher fraction of HAGBs (41% for boundaries $> 45^\circ$) with the peak at 57° compared to the BWS condition (37% for boundaries $> 45^\circ$) as shown in Fig. 4.10g. A high misorientation of GB ($> 50^\circ$) strongly deflects crack propagation thereby improving mechanical properties [112].

4.4.5 Microhardness

Hardness measurements were done to understand the microstructural changes across the different regions of the weld for the BWS and PWHT schedule as shown in Fig. 4.11a. It was observed that there was no significant difference in the average FZ hardness for the BWS (495 ± 5 HV) compared to the PWHT schedule ($495 \text{ HV} \pm 4$ HV). Similarly, the average hardness value at the edge of the FZ (i.e front of the notch) for the BWS condition was 471 ± 10 HV and 475 ± 5 HV for the PWHT schedule, indicating no significant difference according to the student's t-test. It should be noted that though the PWHT used in this study changed the grain morphology at the edge of the FZ from columnar to equiaxed, there was no significant hardness difference as both grain structures contained martensite which was formed after the rapid cooling schedule. Nonetheless, it is worth mentioning that both conditions (BWS and PWHT schedule) show softening at the SCHAZ as shown in Fig. 4.11b similar to previous findings by Sherepenko and Jüttner [80], however, the degree of softening in the PWHT schedule was more prominent. The hardness of the SCHAZ was measured to be 305 HV for the PWHT schedule and 321 HV for the BWS condition. The hardness drop in the SCHAZ with respect to the base metal is known to be due to martensite tempering in that region [166]. It has been reported that softening in the HAZ can reduce stress concentration at the edge of the nugget during tensile loading, which is integral to the mechanical properties and failure mode of the joint [112]. Hence, it was presumed that the degree of softening could be imperative to the joint response to loading conditions, however, further investigation in this study (as will be discussed in the failure mode section) showed that failure did not occur within the softened region of the joint.

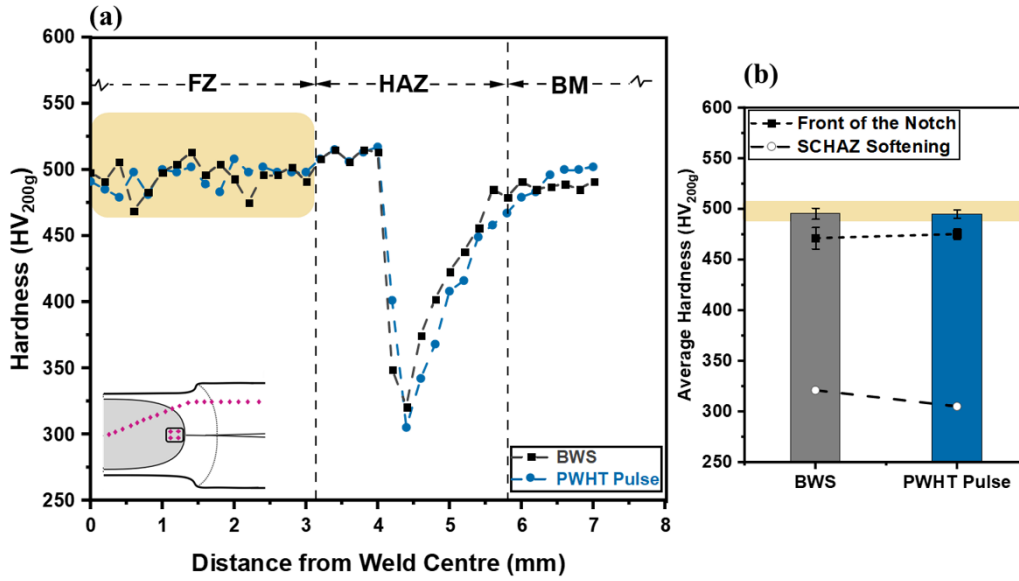


Fig. 4.11: Microhardness map from the weld center to base metal for BWS and PWHT schedule with CT of 100 ms and PT of 167 ms (b) Average hardness of FZ, SCHAZ and edge of FZ for the BWS and PWHT schedule (CT of 100 ms; PT of 167 ms).

4.4.6 Cross-tension properties

The influence of different grain morphologies obtained from the BWS and PWHT schedule on the mechanical properties of the joint was examined via the cross-tension test. The load-displacement curves in Fig. 4.12a indicate a significant increase in the peak load for the PWHT schedule compared to the BWS condition. The average peak load for the PWHT schedule was 9.74 kN which was 54% higher than the BWS with an average peak load of 6.32 kN as shown in Fig. 4.12b. The energy absorbed to failure shows the same trend with a more remarkable improvement of 89% (58.93 J) for the PWHT schedule compared to 31.11 J for the BWS condition. It is worth mentioning that the average nugget sizes for the selected conditions were $\sim 6 \text{ mm} \pm 0.06 \text{ mm}$, hence, the improved mechanical performance cannot be attributed to the effect of weld size variation. Likewise, since the strain maps in Fig. 4.10c and d show no notable difference, the observed changes in mechanical properties can be attributed to the refined grain structure at the edge of the FZ as earlier shown in Fig. 4.10b and e. In addition, it should be noted that previous studies have attributed increase in mechanical performance by post-pulsing to other reasons. For instance, Chabok et al., [10] stated that a post-weld pulse reduced the residual stress in the recrystallized region resulting in slow crack propagation within this region. Moreover, Eftekharimilani et al., [115] have attributed increased mechanical performance by post-pulsing to reduced phosphorus segregation at the weld edge due to the homogenization of alloying elements. However, there was no evidence that the crack path followed the phosphorus-segregated region in the weld. The topic of failure progression along

the phosphorus segregated region is the focus of a follow-up study that will be discussed in Chapter 6.

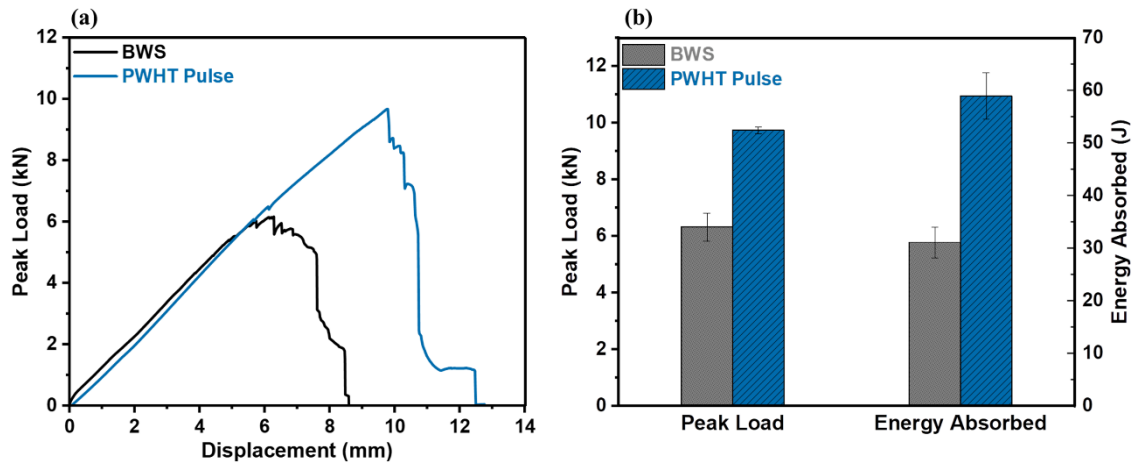


Fig. 4.12: (a) Load-displacement curves for BWS and PWHT schedules (b) Average peak load and absorbed energy to failure for BWS and PWHT schedules (CT of 100 ms; PT of 167 ms).

4.4.7 Failure Mode

The cross-sections of the BWS and the optimal PWHT schedule, interrupted during the cross-tension test are shown in Fig. 4.13. The macroscopic image of the failure mode for each condition is superimposed on the cross-sections. The BWS condition failed in a partial interfacial failure (PIF) mode and it was observed that the crack initiated at the notch and propagated into the fully martensitic nugget along the columnar grains parallel to the fusion line. The crack path then deviated towards the through-thickness direction once the grain direction changed near the middle of the nugget as shown in Fig. 4.13a. It is well known that the columnar grains are the predominant microstructure obtained after the RSW process and it has been reported to promote low-energy crack propagation leading to reduced mechanical performance [146].

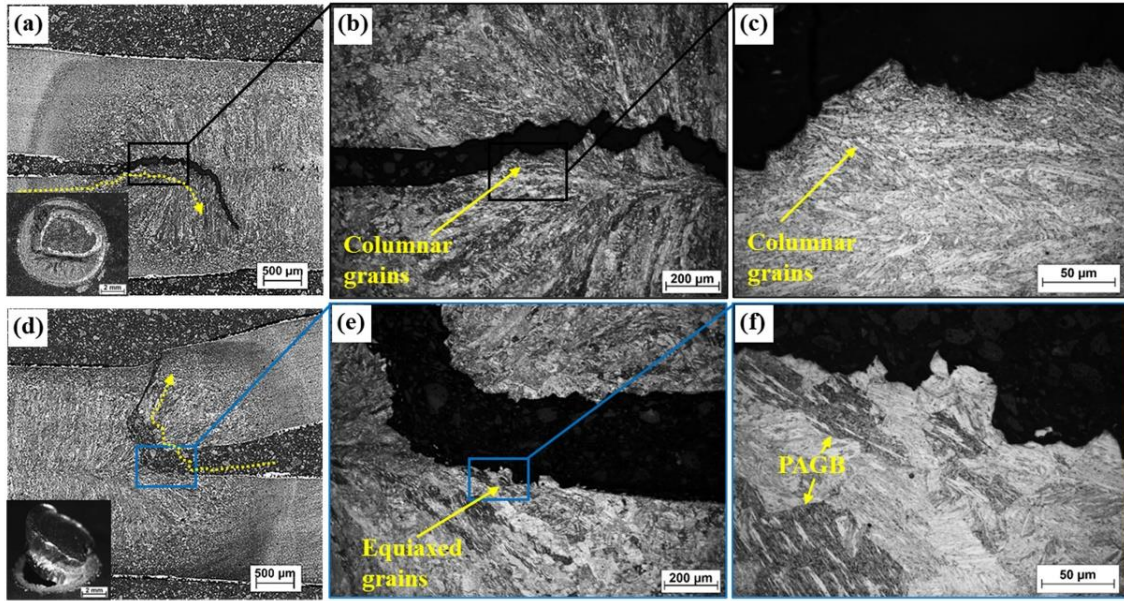


Fig. 4.13: Cross-section of interrupted cross-tension test and failure mode for (a) BWS and (d) PWHT Schedule; optical microscopy of crack propagation path for (b) BWS and (e) PWHT Schedule; (c,f) high magnification images of grain morphology for selected areas in (b) and (e)

The PWHT schedule showed a different crack propagation path compared to the BWS condition. In this case, the crack also initiated at the notch tip and propagated along the sheet-sheet interface to a short distance into the recrystallized equiaxed zone at the edge of the FZ, as shown in Fig. 4.13d. In contrast to the crack propagation path for the BWS schedule, the crack was observed to be strongly retarded as shown with the irregular crack path along the equiaxed PAGs in Fig. 4.13f, which was then directed toward the sheet thickness. The crack propagation path supports the improved energy absorption capability of the PWHT schedule in Fig. 4.12 as more energy was required for the crack to propagate along the several equiaxed PAGs with a higher fraction of high-angle boundaries. It is believed that the refined grain structure poses a greater barrier to crack propagation than the columnar grain structure which is in agreement with Chabok et al., [112] findings.

The fractography analysis for the BWS condition revealed cleavage features indicating brittle fracture within the fully martensitic nugget as shown in Fig. 4.14 (a-c). On the other hand, the fracture surface after post-pulsing exhibits the presence of dimples indicating microvoids due to ductile failure mode as shown in Fig. 4.14 (d-f). The fracture behaviour related to void initiation-growth-coalescence ahead of a non-stationary crack tip for ductile fracture has been detailed by [167]. Though the crack slightly propagates into the nugget for the PWHT condition, however, the microvoids observed from the fractography imply that the crack

propagation in the recrystallized equiaxed zone was in a ductile mode before the redirection through the sheet thickness. Overall, the resistance of the nugget to crack propagation indicates the toughness of the nugget and can be related to the button size [93]. The measured average button size and plug ratio (ratio of button size to nugget) for the PWHT schedule were $6 \text{ mm} \pm 0.04 \text{ mm}$ and 1, respectively. On the other hand, the measured average button size and plug ratio for BWS was $5.03 \text{ mm} \pm 0.22 \text{ mm}$ and 0.84, respectively. The difference in button size indicates that the crack propagates around the FZ boundary in the PWHT schedule compared to the BWS where the crack easily propagates into the martensitic nugget. Hence, it can be deduced that the formation of a recrystallized equiaxed zone at the edge of the FZ can retard the crack propagation from the notch thereby significantly improving the toughness of the nugget.

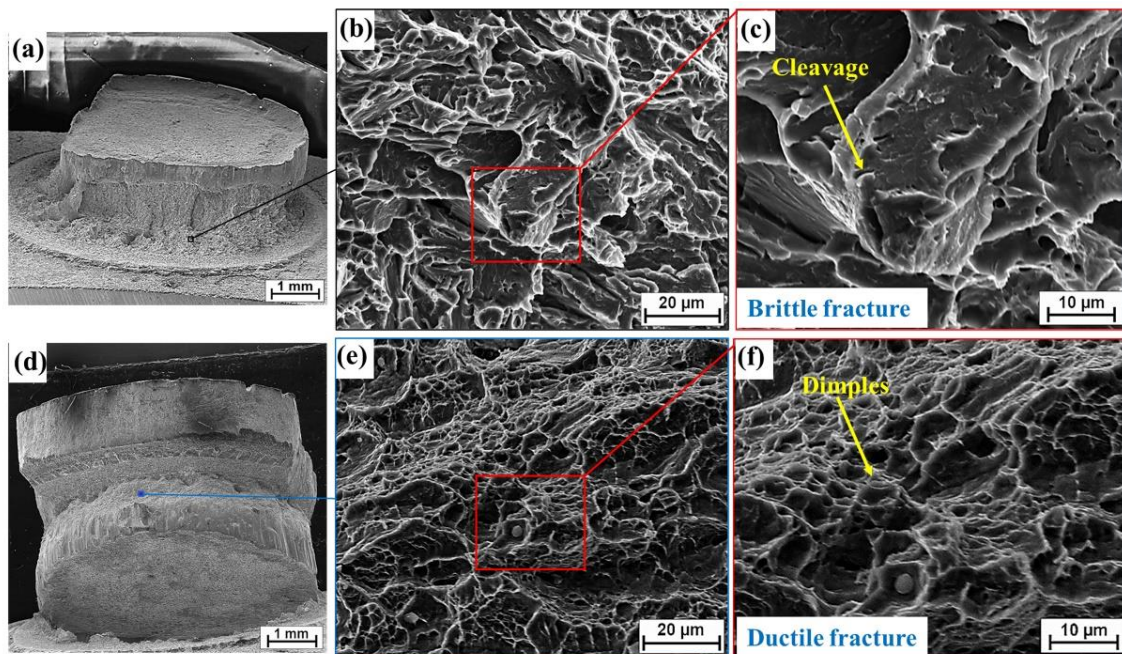


Fig. 4.14: Fractography of BWS (a,b and c); and PWHT schedule (d, e and f).

The schematic in Fig. 4.15 summarizes the changes in the grain morphology at the edge of the FZ with the columnar grains dominating the BWS and a recrystallized zone with equiaxed PAGs for the PWHT schedule. In the BWS condition, the welds fail in the PIF mode as the crack propagates along the weak interface of the columnar grain boundaries as shown in Fig. 4.15a. On the other hand, the PWHT schedule led to a different crack propagation path. As shown in Fig. 4.15b, the crack propagation is retarded in the recrystallized equiaxed zone resulting in significant improvement in energy absorption capability.

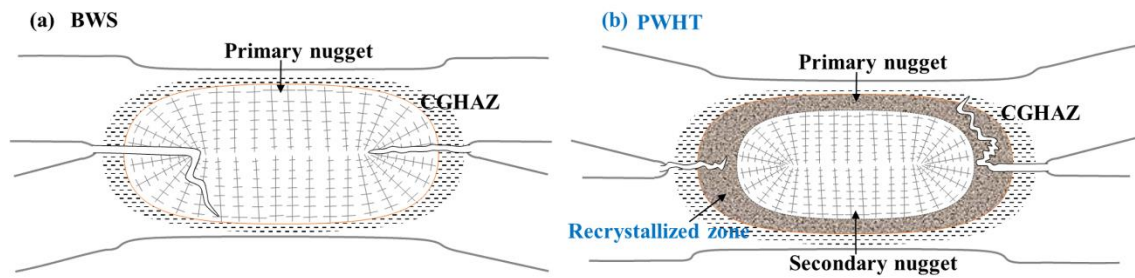


Fig. 4.15: Schematic representation of the crack propagation path for (a) BWS with crack propagating along the columnar grain boundaries and (b) PWHT schedule showing crack propagation along the refined grain structure in the recrystallized zone.

4.5 Summary

In this study, an in-situ post-weld heat treatment schedule has been optimized to induce austenite recrystallization at the edge of the FZ which modified the grain structure and improved the mechanical performance of PHS spot welds. By applying the optimized PWHT parameters, it was observed that a recrystallized zone was formed with equiaxed PAGs compared to the BWS condition with columnar grains elongated along the highest heat dissipation direction. The following conclusions can be drawn from the study:

1. The cool time and heat input during the PWHT schedule dictates the grain size and morphology at the edge of the FZ. Longer CT leads to more heat dissipation from the weld and insufficient thermal energy for recrystallization while longer PT results in grains with a higher aspect ratio. The optimal CT and PT (100 ms and 167 ms) resulted in the growth of new equiaxed grains at the edge of the FZ.
2. By applying the PWHT schedule when the edge of the FZ is austenitic and below the melting point, recrystallization is promoted, which refines the grain structure. When the PWHT current is applied after the edge of the FZ has transformed to martensite, austenite is formed by martensite reversion on reheating and the original epitaxial structure is retained after cooling.
3. The optimal PWHT schedule with a CT of 100 ms and PT of 167 ms resulted in remarkable improvement in mechanical properties. Compared to the BWS condition, the PWHT schedule led to an increase in the peak load and energy absorption by 54% and 89% respectively.
4. The crack propagation path for the BWS condition was along the columnar grain structure resulting in a low-energy absorbing weld with a high amount of cleavage features indicating a brittle failure mode. Crack propagation was retarded at the edge of

the FZ due to the equiaxed grains structure for the PWHT schedule and the failure surface indicated a ductile failure mode.

5. From the current study, the different grain morphology and the misorientation between grains at the edge of the FZ were the dominating factors responsible for the improved mechanical performance of the joint.

CHAPTER

5

5. In-situ dual force: A novel pathway to improving the mechanical properties of resistance spot welds⁵

⁵ This chapter is composed of a published research paper as outlined in the statement of contributions. **O.T. Betiku**, A. Ghatel-Kalashami, H. Ghassemi-Armaki, E. Biro, In-situ dual force: A novel pathway to improving the mechanical properties of resistance spot welds, Materials Science and Engineering A (Under Review).

5.1 Overview

Although in-situ post-weld heat treatment (PWHT) has been a viable method to modify resistance spot weld microstructure and improve joint mechanical properties, prevailing methodologies (as studied in previous chapters) only employ post-weld current to initiate microstructural transformations. This study uses in-situ dual force (DF) and a PWHT current pulse to induce microstructural changes specifically at the edge of the fusion zone (FZ), a region prone to crack propagation. After a short cooling period following the welding cycle, the application of strain energy from the DF and thermal energy from the PWHT current resulted in the formation of new equiaxed grains via austenite recrystallization. The energy absorption capability of the weld improved by 39% after the DF schedule and 85% when DF was combined with a PWHT current. The changes in mechanical properties resulted from strain hardening induced by the DF schedule, while grain refinement from the combined DF and PWHT current schedule led to the deviation of cracks at the edge of the FZ. In contrast, the crack propagated directly into the FZ along the columnar structure in the as-welded condition. The novel application of in-situ dual force extends beyond the conventional PWHT and offers a promising avenue to trigger microstructural changes in the weld which can improve mechanical performance and overall crashworthiness.

5.2 Background

Resistance spot welding is known to exhibit high cooling rates on the order of 10^3 to 10^5 °C/s, which results in the formation of a martensitic microstructure [53], epitaxial solidification towards the weld centerline [56], and the possibility of alloying elements segregation to grain boundaries [168]. Each of these effects has been reported to influence the mechanical properties and failure behaviour of the joint when subjected to loading. For instance, earlier work in Chapter 3 [48] have shown that the fully martensitic fusion zone exhibits relatively low energy absorption capability when subjected to loading. Likewise, it is known that the directional solidification in the FZ leads to a columnar grain structure that promotes low-energy crack propagation resulting in reduced mechanical performance [146,169]. Furthermore, segregation of phosphorus at the columnar grain boundaries has been reported to deteriorate the joint mechanical properties and result in interfacial failure [11,115,168,170].

To address these issues, researchers have mostly adopted various in-situ post-weld heat treatment techniques. The in-situ tempering pulse that adopts a post-weld current has shown to be effective in modifying the martensitic microstructure in the FZ and improving the energy

absorption capability of the joint as shown in Chapter 3 [48]. Applying a PWHT current pulse has also been used to refine the columnar grain structure to an equiaxed grain structure which retards crack propagation at the edge of the FZ as shown in Chapter 4 [169]. The grain refinement is achieved when the edge of the FZ is austenitic before applying the PWHT current to cause a solid-state transformation. Hence, the temperature on reheating during the PWHT schedule should be above the A_{C3} temperature but below the melting point. Furthermore, the application of a PWHT current has also been employed to redistribute segregated alloying elements in the FZ and previous works have attributed the homogenized structure to improved joint performance [9,171].

Despite progress made with employing in-situ PWHT schedules to improve joint performance, most research has only focused on employing a post-weld current pulse to initiate solid-state transformations and homogenization of alloying elements at the edge of the FZ. There is a lack of work on exploring other RSW parameters (such as welding force) as only Lin et al., [141] mentioned that profile force was effective in eliminating voids and improving mechanical properties of a transformation-induced plasticity (TRIP) steel without detailing the metallurgical transformation induced by the profile force. This work employs a profile force (hereafter referred to as dual force) to apply compressive strain as an additional driving force to trigger microstructural changes in the weld. In addition, a combination of the strain energy from the force intensification and thermal energy from a PWHT current was also explored to investigate the resulting changes in microstructure and mechanical properties. This study presents the microstructural and mechanical properties changes of PHS spot welds driven by the dual force and PWHT current pulse schedules.

5.3 Experimental procedure

The investigated material in this study was an Al-Si coated PHS received in the hot-stamped condition with a fully martensitic microstructure as shown in Fig. 5.1 and sheet thickness of 1.4 mm similar to the base metal in previous chapters. RSW was carried out using a medium-frequency direct current (MFDC) RSW robot with a C-type transformer spot welding gun and a Rexroth Bosch weld controller. Welding was done using the welding parameters schematically shown in Fig. 5.2 and a Cu-Cr electrodes with a flat face diameter of 7 mm. The welding schedule in Fig. 5.2a represents the as-welded condition with the squeeze, weld and hold time selected according to AWS D8.9 [32] standard. The term “*in-situ dual force*” indicates the force was change during the welding process to achieve the required microstructural changes. It should be noted that the welding force was reduced in this study

from 4.9 kN (recommended by AWS) to 3 kN to account for the force ramping and is referred to as-welded in this chapter. Moreover, the maximum threshold for welding of steel in the industry is 5 kN which served as the upper force limit for this study. The pre-pulse of 20 ms and 16 kA was added to ablate the coating prior to the weld schedule and reduce the void formation in the FZ. The process map was developed based on the resulting mechanical properties for varying cool time (50 ms, 100 ms and 150 ms) and dual force (4 kN, 4.5 kN and 5 kN) to determine the optimal DF schedule. Additionally, different currents (from 4 kA - 8 kA) were used to determine the optimal DF with PWHT current schedule. The optimal dual force (DF) schedule is shown in Fig. 5.2b while the optimal DF with a PWHT current pulse is shown in the schematic in Fig. 5.2c. The conditions in Fig. 5.2b and c were studied in detail to understand the microstructural changes in the weld. The resulting average weld sizes for all conditions used was $\sim 6.08 \pm 0.06$ mm.

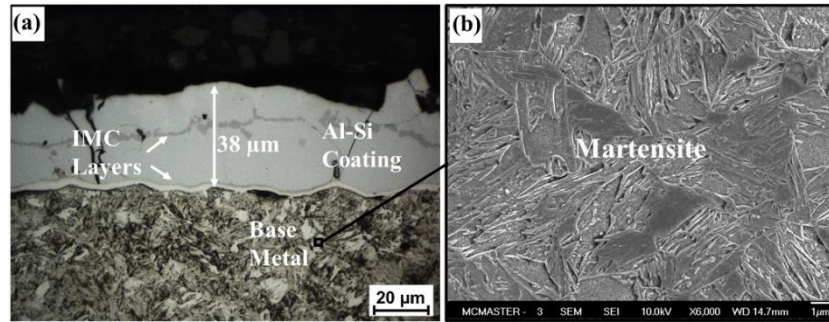


Fig. 5.1: (a) Optical microscopy image of Al-Si coated PHS (b) SEM image of base metal.

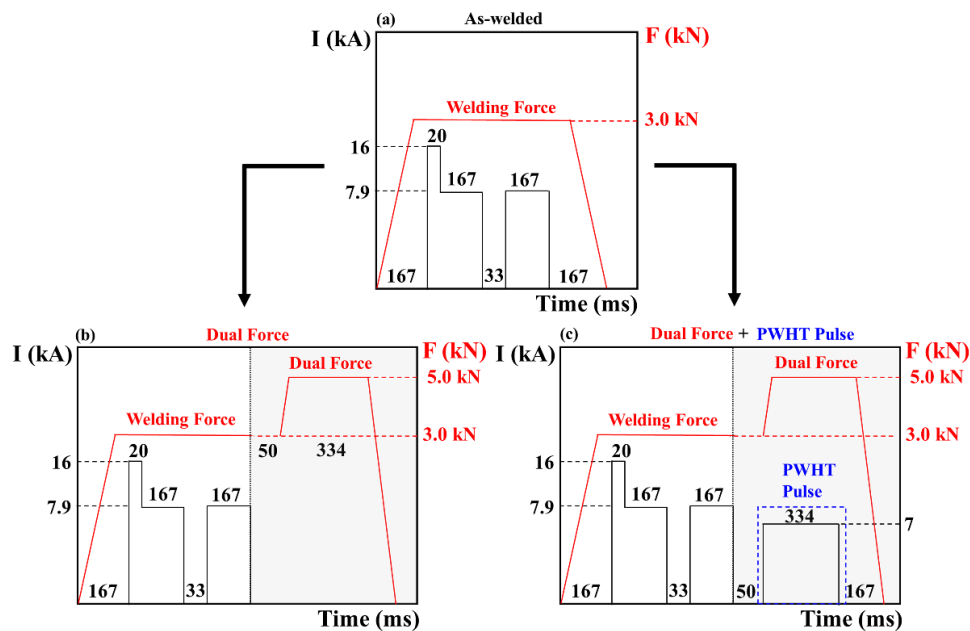


Fig. 5.2: Resistance spot welding schedule for (a) as-welded condition (b) dual-force (DF) schedule (c) DF with PWHT pulse schedule.

Microstructural analysis was performed at the edge of the FZ, which is known to be susceptible to crack propagation. Welds were cross-sectioned, polished down to 0.05 μm colloidal silica and etched with 2% Nital solution. Afterwards, optical microscopy images were taken using an Olympus BX51M optical microscope. A JOEL Scanning Electron Microscope (SEM) equipped with an electron backscattered diffraction (EBSD) detector was used to examine the failure surface and obtain maps at the edge of the FZ. The EBSD maps were obtained at a working distance of 15 mm with a step size of 0.5 μm operating at 20 kV. Prior austenite grain boundaries (PAGB) were reconstructed using APRGE software [152] while grain level analysis was done using MTEX toolkit [151]. The average PAG and block width reported were for five grains extracted the EBSD map for each condition, while the errors represent a 95% confidence interval. Quantitative elemental distribution was obtained at the edge of the FZ on a 300 x 300 μm^2 area using an electron probe microanalyzer (EPMA) with a spot size of 1 μm and a dwell time of 100 ms. Line scans were obtained along the diagonal of the maps. XRD measurements were performed at room temperature using a Bruker D8 Discover XRD equipment with a Cu-K α (λ : 0.154 nm) radiation source operating at 40 mA current and 40 kV voltage. The XRD patterns were obtained in the 2 θ range of 20° and 100°. Post-processing of peak widths was done using the Origin software.

Mechanical properties of welds were evaluated using the standard cross-tension test (CTT). The CTT was done according to AWS D8.9 [32] standard on a 150 x 50 mm specimen similar to the geometry used in our previous study [48]. Five repetitions were done for each condition and the error bars represents a 95% confidence interval. The weld hardness was measured using Clemex microhardness machine with a Vickers indenter under a load of 200 gf and a dwell time of 10 s. A Nanovea-M1 instrumented indentation tester was used to estimate the local yield strength at the edge of the FZ and UHAZ of the different weld schedules. Five indents were done in each region and the average is reported with the error bars showing the 95% confidence interval. A nearly flat tip indenter of 100 μm diameter was used following the procedure suggested by Midawi et al., [172] while the depth of the indenter was measured using a laser sensor. The yield strength was calculated using Equation (5.1) as described in [172]:

$$\sigma_y = \frac{F_s}{3.28 \times \pi \times a^2} \quad (5.1)$$

Where σ_y is the local yield strength in MPa, F_s (in N) is the force corresponding to the point of tangent to the load-depth curve, and "a" is the radius of the indenter (50 μm). Finite element

simulation was done using Simufact ® V2024.1. The thermo-mechanical property of the base metal was extracted from JMATPRO® V12.4 while the numerical models including the contact interactions are detailed in [173].

5.4 Results

5.4.1 Development of weld schedules and process map

5.4.1.1 Baseline mechanical properties

A comparison of the new baseline condition (with force of 3.0 kN at 7.9 kA) was made with the initial BWS condition (4.9 kN at 8.0 kA). The average peak load of the BWS (with 4.9 kN) is 6.25 ± 0.67 kN while that of the new force (3.0 kN) is 6.57 ± 0.64 kN, indicating no significant difference based on students' t-test. Likewise, there was no significant difference in the energy absorbed to failure for the BWS (31.12 ± 2.95 J) and the new force of 3 kN (35.31 ± 4.05 J) as shown in Fig. 5.3. Nonetheless, for the DF study, the new baseline was taken as the new force of 3.0 kN.

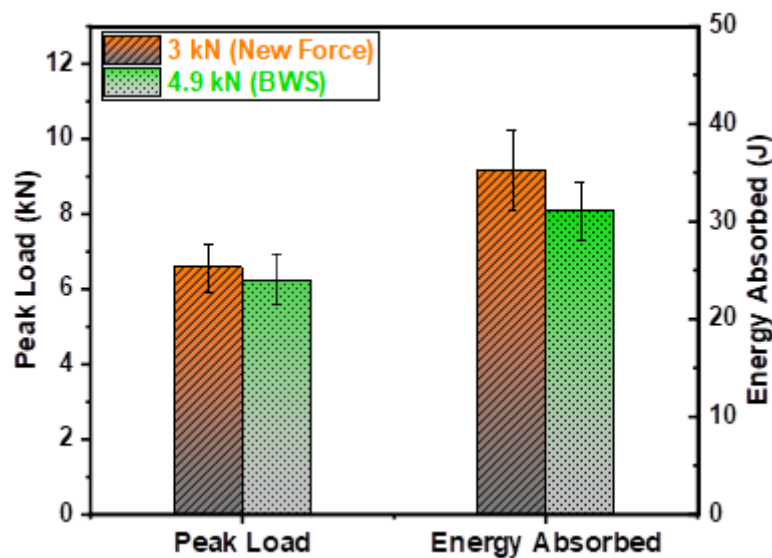


Fig. 5.3: Peak load and energy absorption capability of new welding force of 3.0 kN at 7.9 kA compared with MAWS (4.9 kN at 8.0 kA). *Force 3.0 kN was taken as new baseline condition in the dual force study.

5.4.1.2 Process map development for DF and DF with PWHT

The mechanical properties of the joint were evaluated using the cross-tension test to understand the response of the joint to the various process parameters. In this case, the cool time, and the dual force were varied to obtain the process parameters that result in the optimal joint mechanical properties. Fig. 5.4a and b show peak load and energy absorbed to failure respectively after a dual force time of 334 ms with varying cool time and dual force. The results

showed significant changes in the peak load and energy absorption capability as the DF is increased from 4 to 5 kN. For instance, the optimal properties (peak load of 8.27 ± 0.25 kN; energy absorption capability of 48.99 ± 2.13 J) were achieved with a short cool time of 50 ms and the maximum dual force of 5 kN.

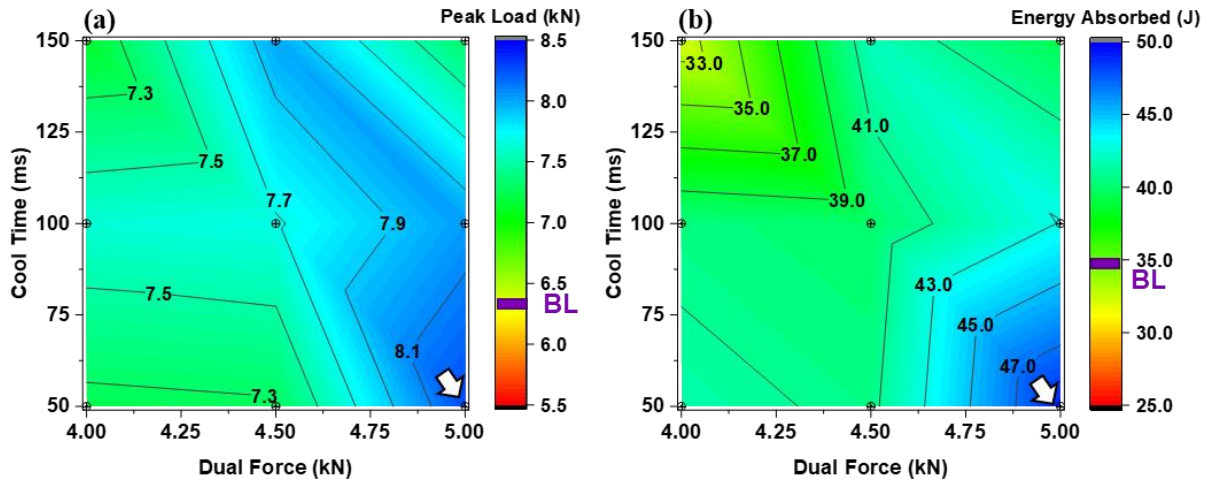


Fig. 5.4: (a) Peak load map and (b) energy absorbed map with fixed dual force time of 334 ms. *Optimal dual force schedule was obtained at a force of 5 kN; cool time of 50 ms and dual force time of 334 ms.

The dual force with PWHT schedule (hereafter referred to as DF+PWHT) was designed by introducing an additional current pulse to the dual force schedule and retaining the force intensification from 3 kN to 5 kN. The current pulse was also varied from 4 kA to 8 kA to obtain the optimal temperature that induces microstructural changes and promotes optimal joint performance. The cool time of 50 ms and DF time of 334 ms were kept constant in this study as adopted from the initial DF and shown in Fig. 5.2b. The mechanical properties after cross-tension test for the DF+PWHT conditions show an increasing trend with an increase in the current as shown in Fig. 5.5. The mechanical properties peaked at the PWHT current of 7 kA with an average peak load of 10.26 ± 0.59 kN and energy absorption capability of 65.22 ± 4.85 J. After a PWHT current of 7kA (lower than the initial welding current), the mechanical properties drop significantly. Compared to the PWHT current of 7 kA, the peak load drops by 30% (7.87 ± 0.59 kN) while the energy absorbed drops by 43% (45.52 ± 5.47 J) at a PWHT current of 8 kA. The optimal DF+PWHT condition shows an improvement of 56% in peak load and 85% in the energy absorption capability to failure compared to the as-welded condition with peak load of 6.58 ± 0.64 kN and energy absorption capability of 35.31 ± 4.05 J. Hence, further analysis was based on the optimal DF schedule shown in Fig. 5.4 and the optimal DF+PWHT schedule as shown in Fig. 5.5.

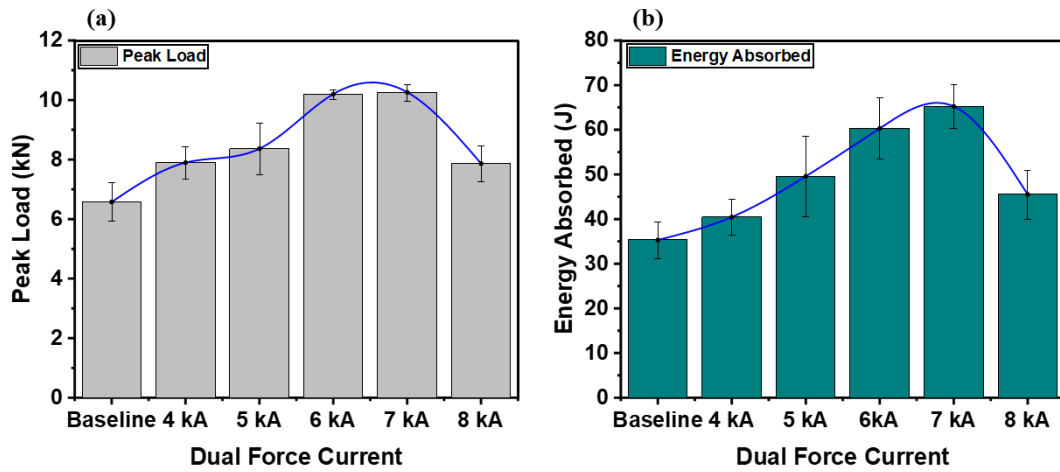


Fig. 5.5: (a) Peak load and (b) energy absorption capability to failure of baseline and DF+PWHT schedules with varying PWHT current.

5.4.2 Mechanical properties for optimal DF and DF with PWHT schedule

The microhardness maps for each weld schedule are shown in Fig. 5.6. The average FZ hardness for the as-welded, DF and DF with PWHT condition was 480 ± 4 HV, 481 ± 5 HV and 482 ± 3 HV respectively. This indicates that there was no significant difference in the average FZ hardness for each weld schedule based on the student's t-test. The hardness value at the sub-critical HAZ (SCHAZ) was ~ 300 HV which is due to martensite tempering that region. It is also noticeable from Fig. 5.6d that the DF with PWHT condition experienced more softening at the SCHAZ, and it is believed that the PWHT current further tempered the martensite in the SCHAZ compared to the as-welded condition. In the case of the DF schedule, a relative increase in hardness was observed at the upper critical HAZ (UCHAZ) compared to the as-welded and DF with PWHT conditions.

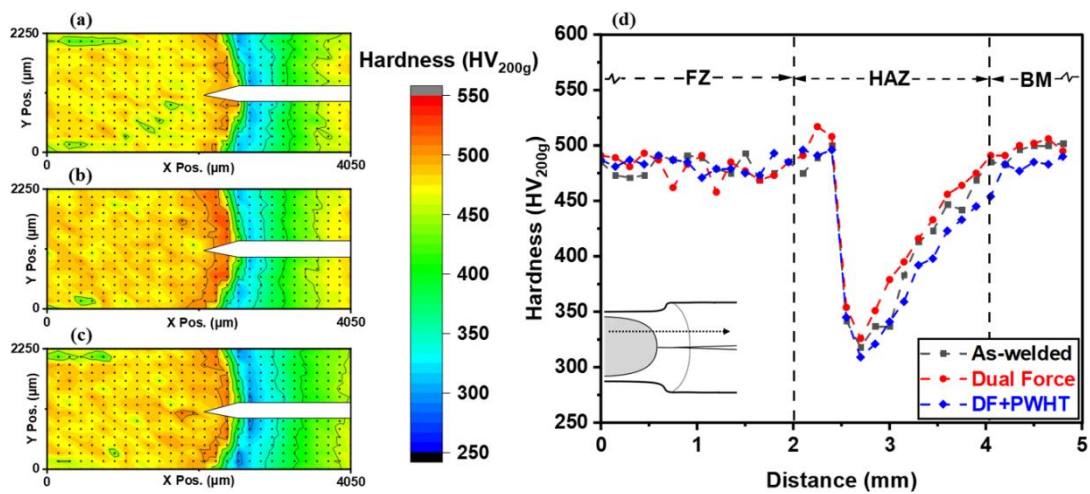


Fig. 5.6: Microhardness maps for (a) as-welded condition (b) dual-force schedule (c) dual force with PWHT pulse schedule; (d) horizontal line scans across maps showing hardness within different weld regions.

The effect of the different weld schedules on the tensile properties was examined using the cross-tension test which is known to be more sensitive to the presence of a brittle microstructure at the edge of the FZ [107]. Compared with the strength of the as-welded condition with a peak load of 6.58 ± 0.64 kN, the DF and DF with PWHT schedule showed an improvement in the average peak load by 25% (8.28 ± 0.24 kN) and 56% (10.26 ± 0.28 kN) respectively. The area under the load-displacement curve in Fig. 5.7a was calculated, which is representative of the failure energy of the welds. It was observed that the post-weld schedules led to an improved energy absorption to failure after the CT test. The energy absorbed to failure for the as-welded condition was 35.31 ± 4.05 J which was improved by 39% (48.99 ± 1.74 J) and 85% (65.23 ± 4.85 J) after applying the DF and DF with PWHT schedules, respectively. It is worth mentioning that there was no significant difference in the average nugget diameter for the three conditions, which strongly influences the load-bearing capacity of the weld. Thus, the improved mechanical properties can be attributed to microstructural changes in the weld, induced by the different weld schedules.

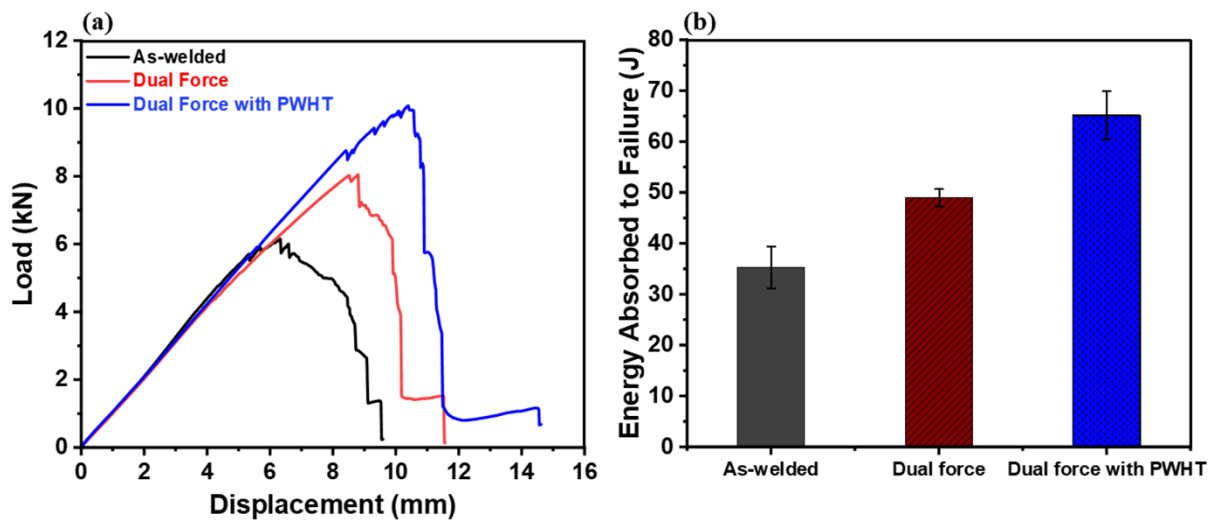


Fig. 5.7: (a) Load-displacement curves and (b) energy absorption capability to failure for the various weld schedules.

5.4.3 Fusion zone microstructure

The weld cross-section at the edge of the FZ for the as-welded, DF and DF+PWHT conditions are shown in Fig. 5.8. The structure of the as-welded condition has a typical solidification structure, evident by the columnar structure aligned along the highest heat dissipation direction as shown in Fig. 5.8a. The columnar structure is also shown in the EBSD IPF map in Fig. 5.8d and the reconstructed prior austenite grain (PAG) boundary in Fig. 5.8g. With the application of the DF on cooling, there was no apparent difference in the weld structure from the optical

micrographs in Fig. 5.8b compared to the as-welded condition. However, the EBSD maps in Fig. 5.8e and h show changes in the PAG size as some grains were less elongated compared to the as-welded condition. For the DF with PWHT current pulse welds, an equiaxed grain structure was observed at the edge of the FZ as shown in Fig. 5.8c, while the center of the FZ showed a columnar grain structure similar to the solidification structure in the as-welded condition. This equiaxed grain morphology in the FZ indicates that a solid-state transformation occurred at the edge of the FZ during the combined force intensification and reheating from the DF and PWHT current, respectively, after a cool time of 50 ms. The solid-state transformation led to the nucleation of new equiaxed grains via austenite recrystallization mechanism detailed in the authors' previous study [169]. The new equiaxed PAGs are fully martensitic owing to the rapid cooling experienced in the RSW.

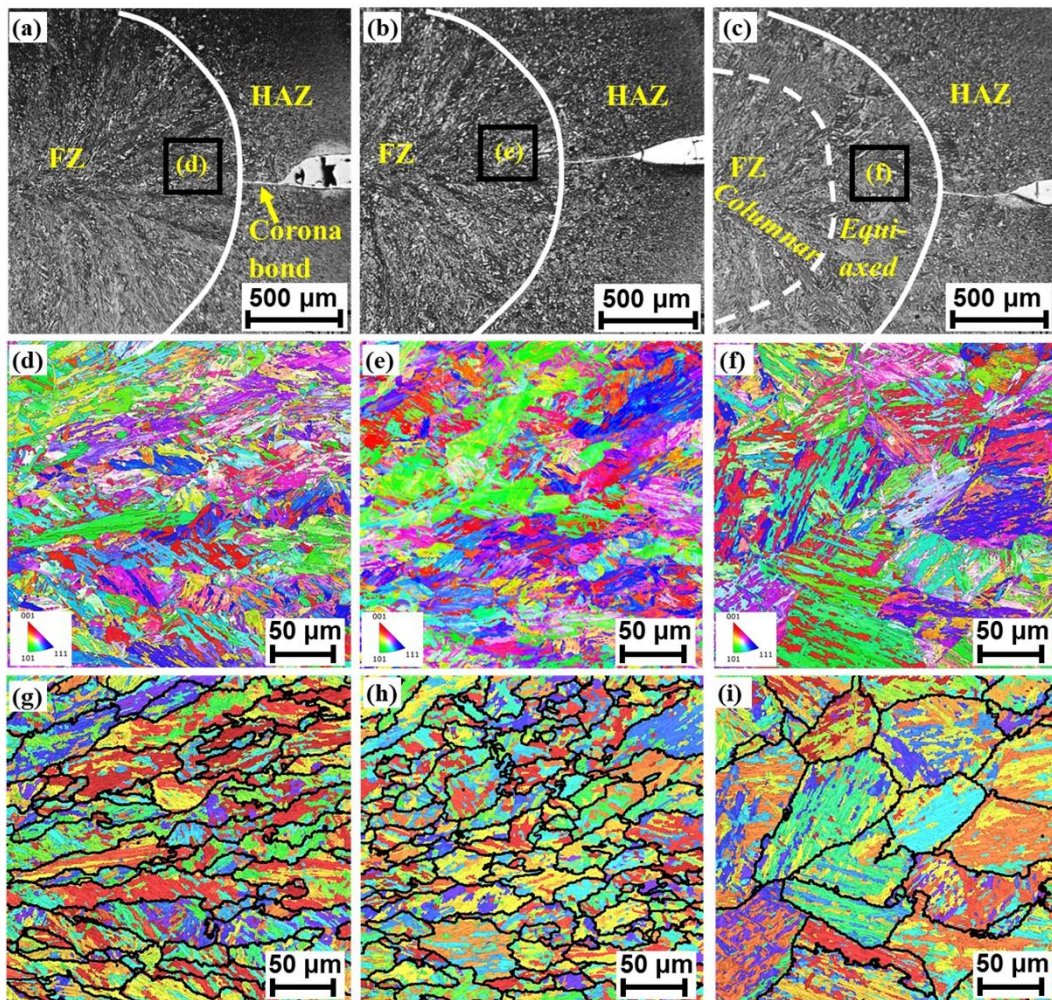


Fig. 5.8: Optical microscopy image and EBSD IPF maps at the edge of the FZ for (a,d) as-welded condition (b,e) DF schedule (c,f) DF with PWHT pulse schedule. (g,h,i) Reconstructed prior austenite grains for each condition. *PAGB are indicated with black lines.

5.5 Discussions

5.5.1 Microstructural evolution and its effect on mechanical properties

The final weld microstructure is directly influenced by the thermal history associated with the three weld schedules. To further understand the transformations occurring at the edge of the FZ, finite element (FE) simulation showing the temperature history of the weld is shown in Fig. 5.9. For the as-welded condition, the weld is heated above the melting point of the PHS and subsequently cooled rapidly, leading to martensitic transformation and retention of the epitaxial solidification structure as shown in Fig. 5.8g. Though the force was intensified for the DF schedule, a rapid temperature drop at the edge of the FZ was observed (as shown by the red line in Fig. 5.9d) indicating insufficient thermal energy to induce complete grain refinement. Conversely, after a cool time of 50 ms, the edge of the FZ is below the liquidus and above the A_{e3} temperature (see Fig. 5.9b and d) indicating a transformation to austenite has occurred. Thus, intensifying the force induces strain at the solidified region within the austenitic phase while applying the PWHT current maintains the temperature above A_{c3} . The combined effect of the strain and thermal energy facilitates the nucleation of new austenite grains which is accompanied by the rearrangement of dislocation networks and migration of PAG boundaries [10,169]. It is worth noting that the recrystallization process occurs very rapid and within a few milliseconds [174,175]. The new-austenite grains then grow in all directions to reduce the overall grain boundary surface energy as shown in Fig. 5.8i.

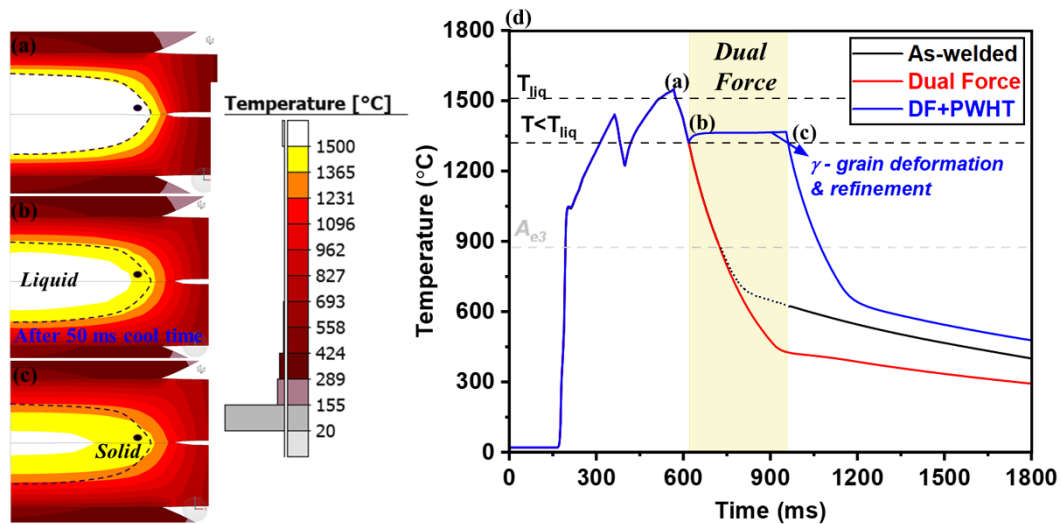


Fig. 5.9: Simulated temperature history for the as-welded, DF and DF with PWHT schedule. The micrographs (a, b and c) correspond to the points on (d).

The hardness of the FZ shown in Fig. 5.6 did not show significant changes with the three welding schedules considered, hence, the changes in CT strength cannot be attributed to

tempering. Similarly, even though the grain morphology at the edge of the FZ for the DF with PWHT schedule changes from columnar to an equiaxed structure, all the welds have a fully martensitic microstructure on cooling and showed similar hardness values. Previous findings have also shown no significant hardness difference after changing the grain morphology in the FZ of spot welds [115,169]. Nonetheless, it is well known that a refined PAG structure contributes to improving the strength of martensitic steel [149]. Thus, it is believed that the refined grain structure at the edge of the FZ for the DF with PWHT current which is evident in Fig. 5.8i contributes to the changes in the joint mechanical properties as shown in Fig. 5.7.

To further examine the microstructural changes induced by the different weld schedules, grain-level analysis was conducted to analyze the substructure features, particularly the block width, as previous studies highlighted the significance of block boundaries as the most effective grain boundary for strength in lath martensite [77,176]. Fig. 5.10(a-c) shows selected PAG grains from the three weld schedules used in this study. The average PAG size for the as-welded condition was $258 \pm 72 \mu\text{m}$ and the grains were predominantly elongated with grain aspect ratio (GAR) i.e., ratio of major to minor axis of 6. Following the DF schedule, the average PAG size decreased to $130 \pm 23 \mu\text{m}$, accompanied by a GAR of 3.4. It was observed that after the DF schedule, certain grains deviated from the elongated grain morphology observed in the as-welded condition (as shown in Fig. 5.8h), which suggests partial recrystallization occurred at certain regions at the edge of the FZ after intensifying the force. Nonetheless, the majority of the grains remained elongated, albeit with a 50% reduction in PAG size compared to the as-welded schedule, as depicted by the representative grain in Fig. 5.10b. In the case of DF with PWHT schedule, the average PAG size was further reduced to $102 \pm 6 \mu\text{m}$ with a GAR of 1.5, indicating that majority of the grains were equiaxed as the strain and thermal energy from the weld schedule was sufficient to induce complete grain refinement at the edge of the FZ.

The average block width for the DF and DF with PWHT schedule was $3.01 \pm 0.41 \mu\text{m}$ and $2.45 \pm 0.26 \mu\text{m}$ respectively, representing reductions of 45% and 55% compared to the average as-welded block width of $5.43 \pm 1.22 \mu\text{m}$ as shown in Fig. 5.10g. This result indicates a substantial refinement in the substructure with both the DF and DF with PWHT schedules. Previous research has established that a decrease in the PAG size results in a reduction in the block size [162,177], which is consistent with the findings observed in this study. The refined block width inherently contributes to an increase in high-angle boundary density, which effectively impedes crack propagation. It is worth mentioning that martensite substructure refinement has mostly been achieved via thermal cycling [114,162,178] and alloying [74,179],

however, this study presents a novel approach to achieving similar refinement using in-situ dual force, particularly within the RSW domain.

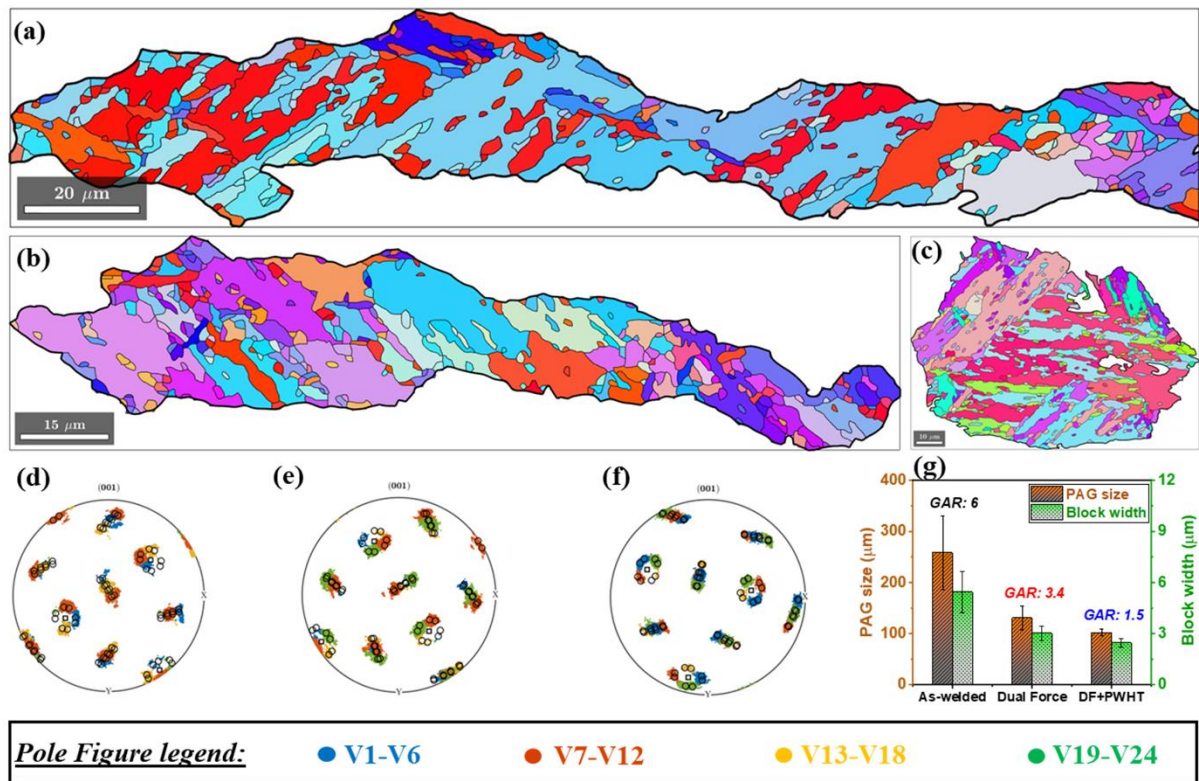


Fig. 5.10: (a) Grain-level EBSD result for (a) as-welded (b) DF and (c) DF with PWHT schedule; Pole figure for each PAG grain used to index martensitic variants for (d) as-welded (e) DF and (f) DF with PWHT schedule; (g) PAG size, block width and grain aspect ratio (GAR) for the three weld schedules.

The thermomechanical process employed for the three weld schedules can result in different variants formation after the martensitic transformation. It is well reported that 24 lath variants (V1-V24) can form within a single parent austenite grain undergoing a cubic-to cubic transformation, where the variants are grouped into 4 packets with each comprising of six variants having the same closed-packed parallel plane relationship with the austenite [74,75]. However, factors such as grain size, accumulated strain, and alloying can influence the ideal formation of the 24 variants and the martensite hierarchy [75]. Since the alloying elements for the material in the study are similar, it was imperative to investigate the effect of the changes in grain size and strain on the resulting martensitic hierarchy, which can dictate the resulting joint properties. From the (001) pole figure (PF) in Fig. 5.10(d-f), it was observed that the selected grains for the three weld schedules show the 24 variants, represented by the different colours in the PF legends. This result indicates that neither the strain nor changes in grain size from the different weld schedules changed the martensite hierarchy. This is plausible because

the measured PAG grain sizes in this study are coarse enough to accommodate the multi-variant transformation without constraining the substructure growth. This result is in agreement with Hidalgo and Santofimia's [180] findings that highlighted that coarse austenite grain contain approximately equal number of all 24 variants, while a reduction in the number of variants is accentuated as the PAG size decreases. Additionally, it has also been reported that a lack of space within a grain can result in the absence of variants [181]. Although no changes were observed in the selected grain variants, it is believed that the changes in the PAG grain size and the refined block size contribute to the improved energy absorption capability for both the DF and DF with PWHT schedule as earlier shown in Fig. 5.7b compared to the as-welded condition.

5.5.2 Elemental distribution

An EPMA scan of the phosphorus (P) distribution at the edge of the FZ was conducted to study the effect of each weld schedule on alloying element segregation as previous studies suggest that phosphorus segregation can be detrimental to the mechanical properties of AHSS [168,182]. As shown in Fig. 5.11a, the measured phosphorus distribution in the as-welded condition shows the segregation of phosphorus within the interdendritic solidification structure. The interdendritic phosphorus segregation is more evident from the spikes on the line scans in Fig. 5.11d. Similar results were observed for the DF schedule (Fig. 5.11b and e) as there was insufficient thermal energy to redistribute the phosphorus from the interdendritic structure on cooling. Phosphorus segregation acts as an impurity that can hinder the grain boundary migration thereby preventing grain refinement as earlier shown in Fig. 5.8h. With the DF and PWHT schedule, phosphorus segregation has been completely removed and a more homogenous structure is observed at the edge of the FZ as shown in Fig. 5.11c and f.

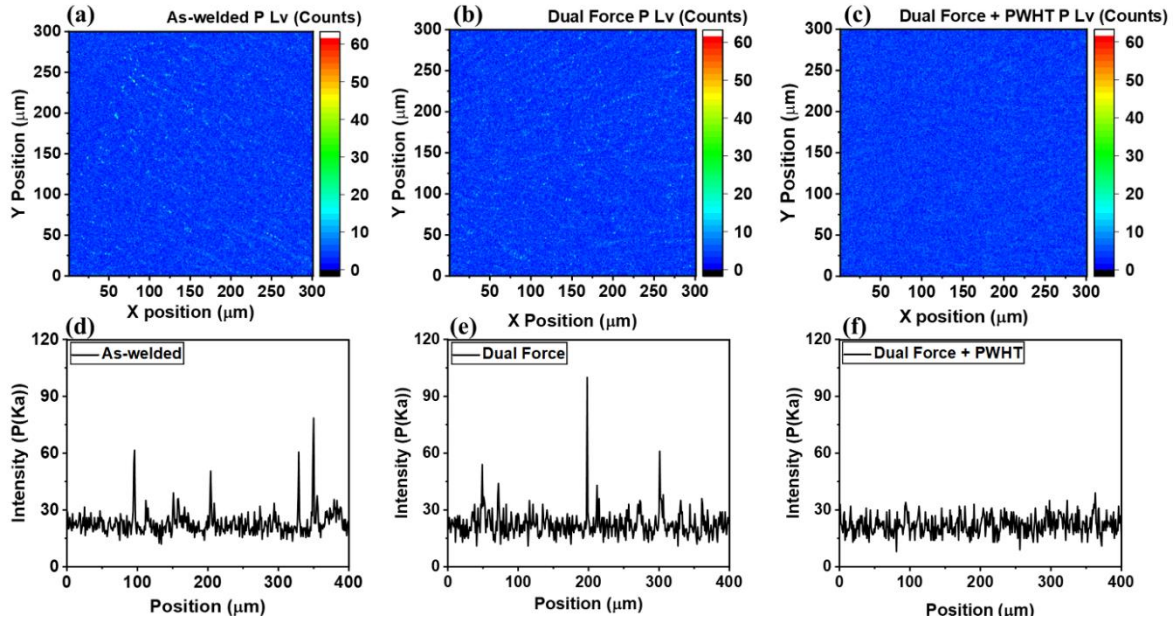


Fig. 5.11: EPMA maps and line scans showing phosphorus distribution at the edge of the FZ for (a) as-welded condition (b) dual-force schedule (c) dual force with PWHT pulse schedule. *Line scans were taken along the diagonal of the mapped area.

Phosphorus redistribution from the interdendritic structure occurs during the PWHT pulse as the temperature is relatively high thereby increasing the diffusivity of phosphorus. The diffusivity of phosphorus in austenite (D_P^γ) is given by equation (5.2) [183]:

$$D_P^\gamma = 0.01 \times 10^{-4} \exp \left\{ -\frac{184200}{RT} \right\} \quad (5.2)$$

Where R is the gas constant and T is the absolute temperature (K). From the temperature profile in Fig. 5.9, the temperature at the edge of the FZ during the PWHT current of 7 kA was maintained at approximately 1360°C (1633K). Consequently, the calculated diffusivity of phosphorus in austenite at this temperature would be $1.28 \times 10^{-12} \text{ m}^2/\text{s}$. The high P interdendritic region had an average width of $1.08 \pm 0.12 \text{ } \mu\text{m}$, and the analysis of phosphorus concentration profile, estimated using the homogenization equations outlined by Porter and Easterling [184] showed an exponential decrease (up to 92%) in phosphorus concentration over a duration of 334 ms when the PWHT current was applied. In the case of the DF schedule (without PWHT current), the segregation increased by 0.05% compared to the as-welded schedule. These findings support the phosphorus homogenization observed in the EPMA results in Fig. 5.11c. Conversely, the rapid temperature decrease led to reduced phosphorus diffusivity for both the as-welded and DF schedules, resulting in insufficient energy for phosphorus homogenization within the interdendritic structure.

It has been reported that the segregation of P in ferrite (α) cooling at a rate of 100 K/s is insignificant, but can occur at ferrite grain boundaries during the transformation from austenite (γ) to α [183]. In the as-welded and DF schedule welds, P segregation occurs because there was less boundary mobility during the γ to martensite (α') transformation, which impairs P mobility as the temperature drops rapidly. Conversely, in the DF+PWHT schedule welds, the boundary is mobile during the austenite recrystallization process as the PWHT current is applied, and the temperature is sufficient to facilitate the redistribution of P due to higher diffusivity. Therefore, the reduced diffusivity of phosphorus, caused by the rapid temperature drop, combined with the limited boundary mobility during the γ to α' transformation in the as-welded and DF schedule welds, results in P segregation during solidification. Furthermore, it is commonly believed that phosphorus segregation deteriorates nugget toughness and results in embrittlement [11,115,182]. This is evident from the mechanical properties result (shown in Fig. 5.7), where even though a reduced grain size was observed for the DF schedule, the presence of P segregation (shown in Fig. 5.11b) contributed to the inferior joint properties compared to the DF+PWHT schedule with a more homogenized structure. However, despite the segregation of phosphorus in the DF welds, a notable improvement in mechanical properties was observed compared to the as-welded condition, which can be attributed to the refined PAG size and block width.

5.5.3 Microstrain and dislocation density analysis

To gain a comprehensive understanding of the changes in microstructural phase, microstrain and dislocation density in the FZ due to the different weld schedules, XRD analysis was conducted. The peaks in the XRD spectrum shown in Fig. 5.12a correspond to a body-centered-cubic structure indicating the FZ microstructure for the three schedules are fully martensitic without the presence of retained austenite. The crystallite size (D), microstrain (ε) and dislocation density (ρ) were calculated from the XRD data using equation (5.3), (5.4) and (5.5) respectively. Details of the method of analysis have been reported elsewhere [185,186].

$$\varepsilon = \frac{\beta}{4 \tan \theta} \quad (5.3)$$

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (5.4)$$

$$\rho = \frac{3\sqrt{2\pi}(\varepsilon^2)^{1/2}}{Db} \quad (5.5)$$

$$\Delta\rho = \left(\frac{\rho_{Dual\ force} - \rho_{as-welded}}{\rho_{as-welded}} \right) \times 100\% \quad (5.6)$$

Where θ is the peak position, K is the Scherrer constant taken as 0.94, β is the FWHM, and b is the magnitude of burgers vector (0.286 nm).

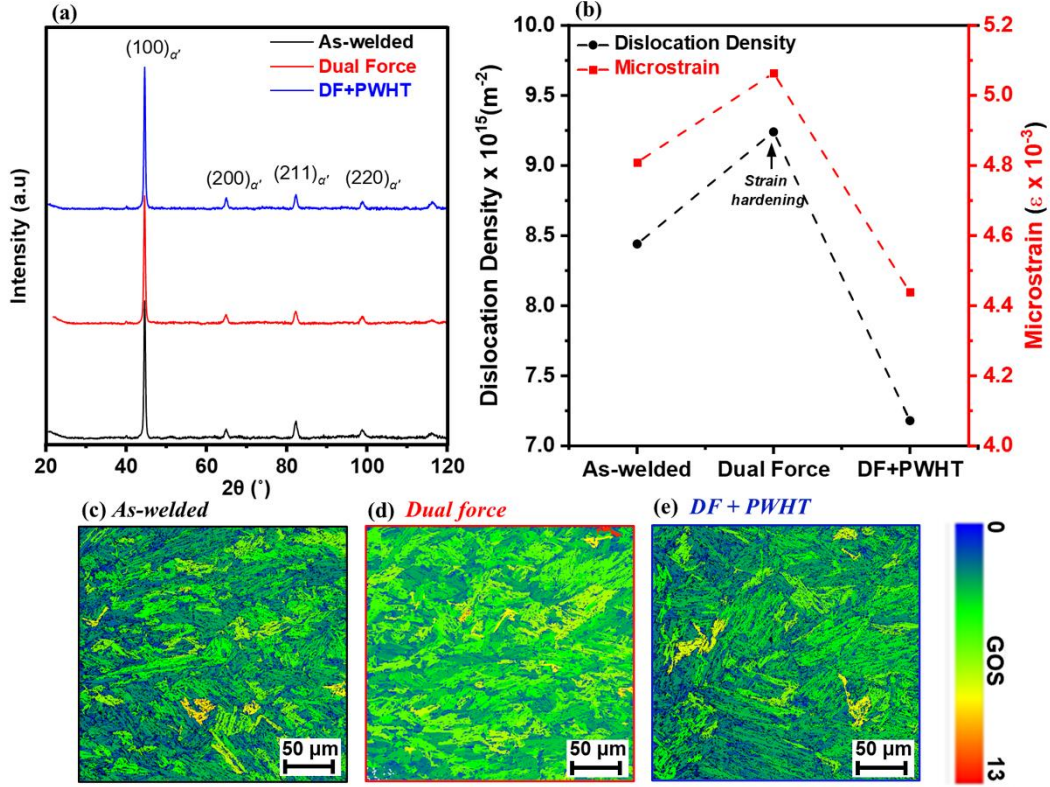


Fig. 5.12: (a) XRD spectrum of the as-welded and post-weld schedules showing a fully martensitic BCC structure (b) computed dislocation density and macrostrain for the as-welded and post-weld schedules; grain orientation spread map (GOS) at the edge of the FZ showing internal strain comparison for (c) as-welded (d) dual force (e) DF+PWHT schedule.

The calculated dislocation densities for the as-welded, DF, and DF+PWHT schedules were $8.44 \times 10^{15} \text{ m}^{-2}$, $9.24 \times 10^{15} \text{ m}^{-2}$, and $7.18 \times 10^{15} \text{ m}^{-2}$ respectively. The dislocation density for the as-welded condition which originates from the plastic accommodation of transformation strain in martensite and is similar to the values reported in the literature [102] for water-quenched PHS. The DF schedule resulted in an increased dislocation density by 9.4% as calculated using equation (5.6), which is believed to be due to the force intensification on cooling, prompting strain hardening. These dislocations (mostly geometrically necessary dislocations) are generated near interfaces such as grain boundaries or substructure boundaries due to the strain gradient from the DF schedule [87]. Moreover, as the DF schedule induces strain to the austenite matrix, the dislocations generated in austenite are inherited into

martensite [187]. Thus, it is believed that the dislocation generation must have surpassed dislocation annihilation as the force was intensified from the austenitic to the martensitic phase on cooling, resulting in an overall net increase in dislocations.

Though there was force intensification for the DF+PWHT schedule, the dislocation density is 14% lower than the dislocation density for weld in the as-welded condition. In the DF+PWHT case, dislocation annihilation occurred within the structure via thermally activated glide since the temperature at the edge of the FZ was maintained at a high temperature $\sim 1360^{\circ}\text{C}$ during the force intensification. In other words, the dislocations had sufficient kinetic energy during the PWHT phase which enabled their interaction and facilitated the elimination of some dislocations within the structure. The strengthening effect (σ_{ρ}) due to dislocation density (ρ_D) on martensitic steels can be calculated using the equation (5.7) proposed by Young and Bhadeshia [188]:

$$\sigma_{\rho} = 0.38\mu b\rho_D^{1/2} \quad (5.7)$$

Where μ is the shear modulus (80 GPa) and b is the Burgers vector. Based on the calculation in equation (7), the strengthening effect of measured dislocation densities at the edge of the FZ were 799 MPa, 836 MPa and 737 MPa for the as-welded, DF and DF+PWHT schedules respectively. A recent study by Nima et al., [189] showed that dislocation strengthening is the predominant factor contributing to the overall yield strength of martensite, ranging from 53 to 65% of the total yield strength. In comparison, boundary strengthening contributes around 20% and solid solution strengthening contributes between 10% and 15%. Hence, the local increase in the dislocation density at the edge of the FZ in addition to the refined block width in the DF condition is believed to contribute to the global mechanical properties of the joint during loading as shown earlier in Fig. 5.7.

The grain orientation spread (GOS) maps in Fig. 5.12c-e show the internal strain in the microstructure by calculating the average deviation in orientation between each point in a grain and the average orientation of the grain. In other words, a high mean GOS value indicates a higher internal strain in the microstructure [74,164]. As shown in Fig. 5.12d, the GOS map of the DF schedule showed a more severe orientation heterogeneity within the microstructure as compared to the as-welded and the DF+PWHT schedule in Fig. 5.12c and e respectively. The overall increase in the mean GOS in the DF schedule indicates a higher strain within the structure, which is supported by the quantitative analysis from the XRD data in Fig. 5.12b. The

increased microstrain in the DF schedule can also be attributed to increased force which generates more strain and dislocations in the microstructure on cooling compared to the as-welded and DF with PWHT schedule.

5.5.4 Local yield strength estimation

In the DF schedule, a notable 25% increase in the load-bearing capacity was observed as shown in Fig. 5.7. Moreover, it was established that force intensification increased dislocation density at the edge of the FZ (refer to Fig. 5.12b). Thus, the DF schedule is believed to promote strain hardening at the edge of the FZ, resulting in increased joint mechanical properties. To verify this claim, the instrumented indentation technique was used to obtain the local mechanical properties (specifically yield strength of martensite) at the edge of the FZ.

The yield strength at the edge of the FZ after the DF schedule was 1428 ± 13 MPa, and 1423 ± 13 MPa at the UHAZ, as shown in Fig. 5.13. This indicates a significant difference in yield strength of 13% (1267 ± 14 MPa) and 7% (1328 ± 13 MPa) at the edge of the FZ and UHAZ respectively, compared to the as-welded schedule. These findings suggest that the DF induced strain at the edge of the FZ, resulting in increased dislocation density that enhanced the load-bearing capacity of the weld. Previous studies have reported similar findings of increased spot weld strength due to increased dislocation density around the failure location which was achieved by pre-straining the weld [190]. However, the strain-hardening effect achieved by the in-situ DF schedule in this study has not been reported until now.

The strain hardening observed within these regions (i.e. edge of FZ and UHAZ) following the DF schedule implies that during loading, the yielding of the material is delayed to a higher stress level. In other words, the weld can resist more deformation before crack propagation, resulting in an overall increase in the global load-bearing capacity as shown in Fig. 5.7. Nonetheless, it is worth mentioning that as the crack begins to propagate after yielding is exceeded and a critical dislocation density is attained, the refined PAG structure (including grain substructure) poses more barriers as the crack will need to navigate several high-angle boundaries which requires more energy. The load-displacement curve in Fig. 5.7a encompasses both the deformation and crack propagation stages, but the precise delineation of these stages would require plasticity models, which is beyond the scope of the current study. For the DF with PWHT schedule, a significant decrease in the local yield strength at the edge of the FZ (950 ± 16 MPa) and UHAZ (1025 ± 14 MPa) compared to the as-welded and DF schedule. In this case, the dislocation density is reduced and yielding occurs earlier. Notwithstanding, the

grain refinement leads to a tougher microstructure in front of the notch which effectively resists crack propagation and improves the global absorbed energy of the weld.

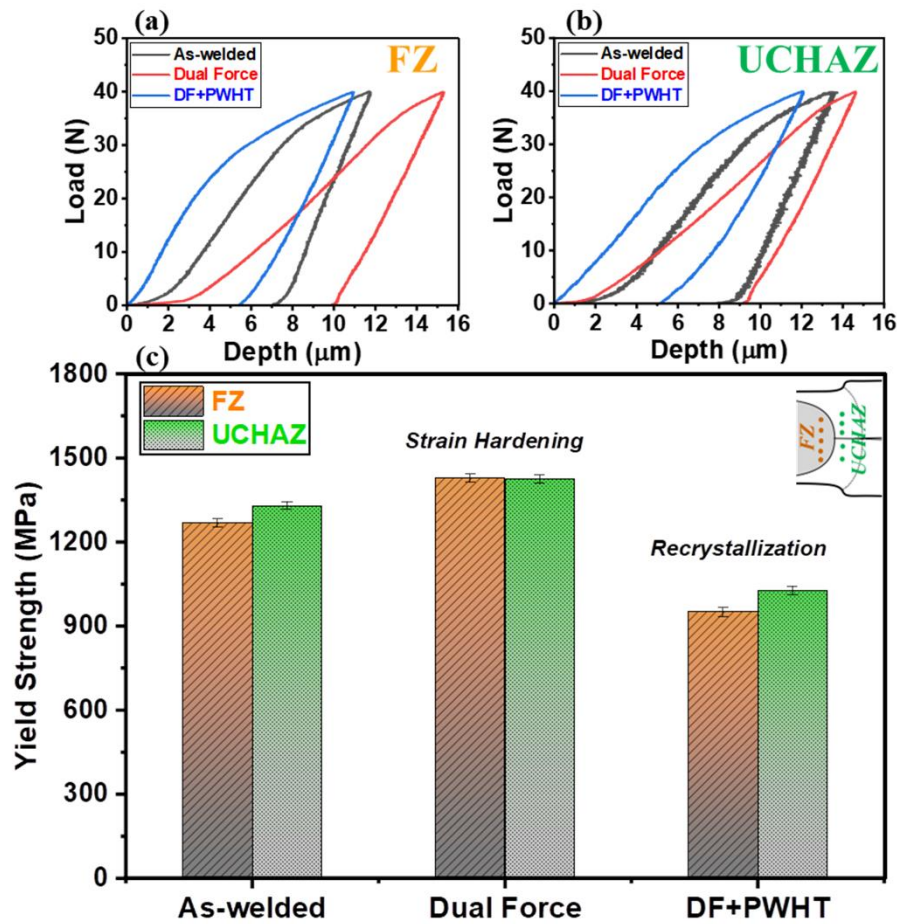


Fig. 5.13: Load-depth curves from the instrumented indentation for (a) FZ (b) UCHAZ; (c) summarized estimated yield strength for as-welded, DF and DF with PWHT schedules.

5.5.5 Toughening mechanism controlling failure progression during CT test

Interrupted tensile tests were carried out for each weld condition to observe the crack path during weld failure, which is shown in Fig. 5.14 (a-c) with the inset showing the load-displacement curves from the interrupted tests. For the as-welded condition, the crack propagated directly into the FZ along the columnar grain structure which is known to show low resistance to crack propagation [146]. The crack path is then redirected through the thickness direction resulting in a partial-pullout failure mode as shown in Fig. 5.14d. Previous work has shown that cracks tends to initiate and propagate along columnar grain boundaries when the tensile stress at the crack tip is perpendicular to the columnar grain boundary within the epitaxial region [191]. Thus, the crack tends to maintain a straight trajectory upon penetration into the epitaxial zone, owing to the elongated grain structure along a specific direction. This is evident from the straight crack path through the sheet thickness in the FZ as shown in

Fig. 5.14a. This failure type within the martensitic FZ is dominated by cleavage features from the fractography in Fig 14g indicating a brittle failure.

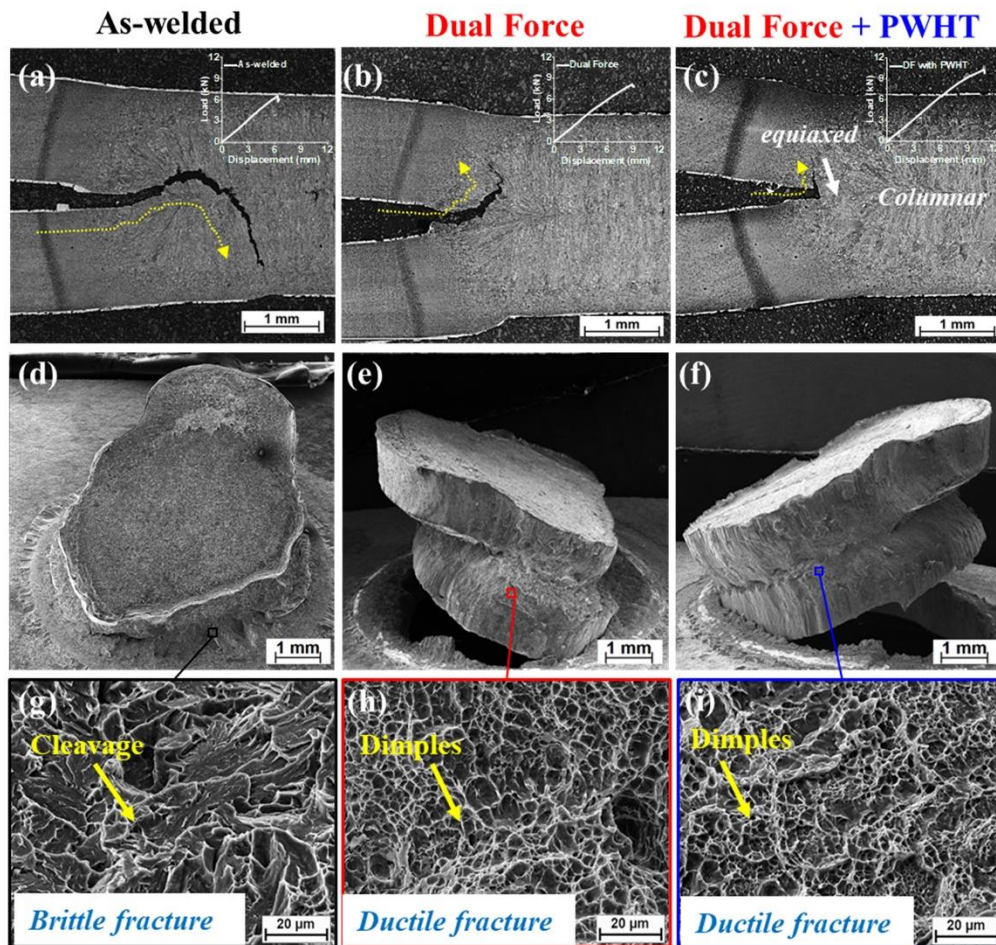


Fig. 5.14: Optical microscopy images for interrupted test samples showing (a) as-welded (b) DF (c) DF with PWHT; SEM images of failure mode for (d) as-welded (e) DF (f) DF with PWHT; fractography for (g) as-welded (h) DF (i) DF with PWHT schedules.

Interestingly, after applying the DF schedules, the crack is redirected at the edge of the FZ as shown in Fig. 5.14b. For the DF schedule, the crack propagates a little distance into the FZ but is redirected towards the upper critical HAZ (UCHAZ) and subsequently through the sheet thickness. It is worth mentioning that the region of crack deviation at the edge of the FZ is where there was an observed increased dislocation density. Hence, the local increase in the dislocation density which is supported by the increase in yield strength and load-bearing capacity leads to more deformation of the weld thereby delaying yielding compared to the as-welded schedule. Once yielding and plastic deformation occur, the failure mechanism transitions from a dislocation-strengthening mechanism to crack propagation. Thus, the increased yield strength due to strain hardening allows more dislocation interaction, delaying

the crack propagation at the edge of the FZ. The fracture surface for the DF schedule is dominated by dimple features indicating the failure was ductile.

In the case of weld schedules combining the DF and PWHT, it was observed that the crack path deviated at the edge of the FZ where the transition from the columnar to equiaxed grain structure occurred. In particular, the equiaxed PAGs at the edge of the FZ strongly retards the crack propagation towards the FZ and get redirected towards the HAZ. Guo et al [192] have shown that the PAG boundary has a higher interface energy with a deflection angle higher than 60° . As shown in Fig. 5.14c, the equiaxed PAG morphology with a high-angle grain boundary hinders the straightforward crack propagation compared to the as-welded condition. Thus, the equiaxed PAG morphology with varying orientations poses more barrier to crack propagation which improves the energy absorption capability of the weld. The fracture surface revealed dimples features, indicating a ductile failure mode compared to the cleavage features observed in the as welded condition.

The schematic in Fig. 5.15a summarizes the crack propagation paths and the microstructural features for the as-welded schedule where the crack propagates along the columnar structure in the FZ. The change in crack path due to strain hardening from the DF is shown in Fig. 5.15b while the two distinct grain morphology in the FZ achieved with the DF and PWHT schedule is shown in Fig. 5.15c. In the latter schedule, the crack is strongly deviated due to the recrystallized PAG formed at the edge of the FZ.

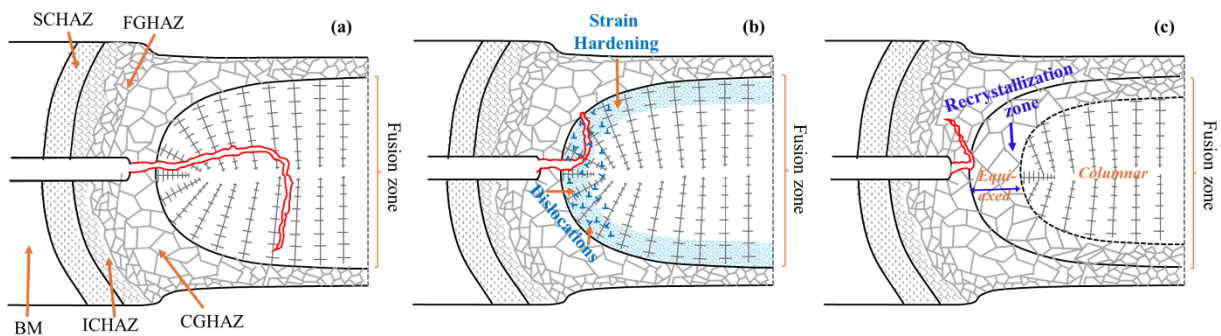


Fig. 5.15: Schematic representation of crack propagation path after interrupted cross-tension test for (a) as-welded (b) dual force and (c) DF+PWHT schedule.

5.6 Summary

The mechanical properties of PHS spot welds have been improved by employing a dual force (DF) schedule and a DF combined with a PWHT current. The improved mechanical properties achieved with the dual force schedule was primarily attributed to strain hardening effect at the edge of the FZ, while refinement of PAG size and morphology through austenite recrystallization resulted in the increased energy absorption capability of the weld for the DF with PWHT schedule. The following conclusions can be drawn based on the results:

1. The peak load and energy absorption capability to failure was enhanced by 25% and 39% respectively after applying the DF schedule compared to the as-welded condition. These mechanical properties were further enhanced by 56% and 85% respectively, after the DF was combined with a PWHT current schedule.
2. The DF schedule induced a 9.4% increase in dislocation density at the edge of the FZ which resulted in a 13% increase in the local yield strength of martensite compared to the as-welded schedule. The increase in the strength of martensite delayed yielding to a higher load, meanwhile, the reduced PAG size and block width contributed to retarding the crack propagation even though there were no apparent changes in the grain morphology compared to the as-welded schedule.
3. A transition from columnar to equiaxed PAG morphology occurred at the edge of the FZ by employing the combination of DF with PWHT schedule. The PAG size and block width was reduced by 60% and 55% respectively and was accompanied by a change in the grain aspect ratio compared to the as-welded condition. The refined grain structure, morphology and substructure strongly hinder crack propagation resulting in improved energy absorption capability after the CT test.
4. Crack propagation paths were notably influenced by the different weld schedules. For the DF schedule, the crack propagated slightly into the FZ but deviated at the edge where strain hardening occurred as there was more yielding ahead of the crack tip. For the DF with PWHT schedule, the crack path was deviated where grain refinement occurred at the edge of the FZ which posed more resistance to crack propagation.

CHAPTER

6

6. Comprehensive discussion on the factors dictating the mechanical properties after in-situ PWHT and the role of paint baking⁶

⁶ This chapter is composed of a research paper as outlined in the statement of contributions. **O.T. Betiku**, H. Ghassemi-Armaki, E. Biro, Understanding the metallurgical factors controlling the mechanical properties of resistance spot welds, to be submitted to Metall. Mater. Trans. A.

6.0 Overview

In previous chapters, it has been established that certain metallurgical transformations can result in improved joint mechanical properties. These transformations including changes in grain structure, redistribution of alloying elements, and changes in weld texture can occur concurrently when there is sufficient driving force to initiate the transformation. However, the specific contribution of each mechanism to the enhancement of joint mechanical properties remains unknown. This chapter aims to delineate the metallurgical contributions achieved during the in-situ PWHT schedules employed in the previous chapters. Additionally, the effect of paint baking (PB) following the in-situ PWHT schedules on the mechanical properties was studied to understand the specific contribution of the PB schedule. This chapter also provides a comprehensive comparison of each technique studied in this thesis, highlighting the underlying metallurgical principles and their industrial applicability.

6.1 Background

Non-isothermal heat treatment, otherwise known as in-situ PWHT has been adopted to modify the microstructure of resistance spot welds in previous chapters. The in-situ PWHT requires precise control of post-weld parameters such as the cool time, post-weld time and post-weld current to induce specific microstructural changes. Previous chapters have demonstrated how these parameters can be controlled to achieve martensite tempering, PAG and substructure refinement, as well as phosphorus homogenization in the weld. It was observed that these transformations resulted in improved mechanical properties. Since most of these techniques require a PWHT current, it is envisaged that an overlap between each microstructural modification may occur concurrently. For instance, the PWHT current that induces grain refinement can also promote homogenization of the alloying element, since the thermal energy from the PWHT current is sufficient to redistribute segregated alloying elements. Likewise, grain refinement from columnar to equiaxed grains is associated with the formation of new grains and grain boundary migration thereby altering the texture, which is another important factor that can dictate the mechanical properties of the joint.

Recent studies by Chen et al [193] showed that in-situ PWHT led to both grain refinement in the FZ and tempering of martensite close to the FZ boundary. Although these changes occurred in the FZ, the crack path was directed towards the SCHAZ where martensite tempering occurred. The authors attributed the improved properties to the formation of equiaxed martensite and martensite tempering but did not identify the dominant mechanism. Similarly,

EftekhariMilani et al, [115] observed that double-pulse welds showed reduced PAG size, smaller block width, and reduced phosphorus segregation compared to single-pulse welds. The authors concluded that the negative influence of phosphorus segregation on the mechanical properties was more pronounced than the positive influence due to grain size reduction. However, there was no direct evidence to confirm that the crack propagated along the P-segregated rich regions and that the homogenization effect was indeed superior to grain size reduction.

Despite these valuable findings, there has been no comprehensive study delineating the specific influence of various microstructural changes (invoked via in-situ PWHT) to improved joint performance. Thus, it is important to understand the relative contribution of each microstructural change induced by post-pulsing on the mechanical properties of the weld. Particularly, analyzing the microstructure along the crack path can provide detailed insights into the interaction between these microstructural changes and the crack propagation path. This study systematically examines the direct influence of each microstructural change in the weld subjected to different PWHT schedules. Additionally, this chapter investigates the effect of paint baking—an additional low-temperature treatment performed during automotive assembly to harden the paint—on the resulting properties after the PWHT schedule.

6.2 Experimental method

The material used for this study was the PHS from previous chapters with similar chemical composition, sheet thickness and coating characteristics. The PAGs of the base metal are shown from the reconstructed EBSD map in Fig. 6.1a with an average grain size of $7.13\ \mu\text{m} \pm 0.6\ \mu\text{m}$, while the SEM image in Fig. 6.1b, and XRD pattern in Fig. 6.1c shows the martensitic microstructure of the base metal with a BCC phase.

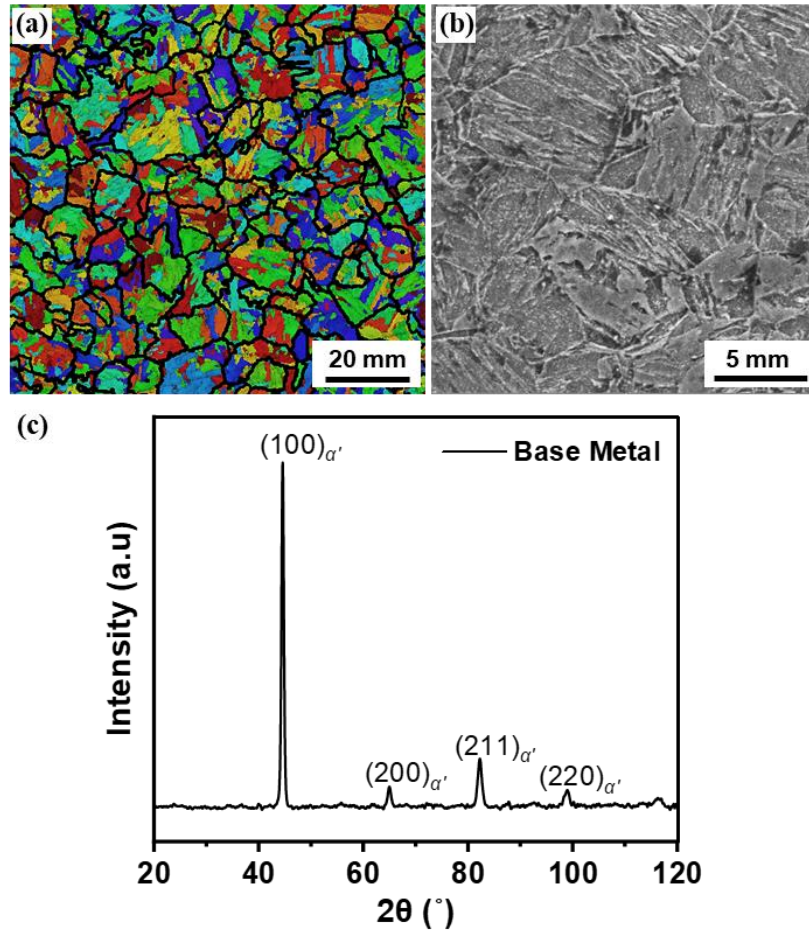


Fig. 6.1: (a) EBSD maps showing grain structure of base metal; (b) SEM image of base metal showing martensitic microstructure (c) XRD of base metal showing BCC martensitic phase.

The baseline weld schedule (BWS) remained the same as in previous chapters with a weld current of 8.0 kA, shown in Table 6.1, which resulted in an average nugget diameter of 6.05 ± 0.03 mm. In addition to the BWS (with no homogenization and no grain refinement), weld schedules were systematically developed to promote only homogenization with no grain refinement referred to as HNG, and both homogenization and grain refinement (referred to as HAG). In addition to the schedules in Table 6.1, another batch of welds was created with the BWS and with the weld schedule with both homogenization and grain refinement and was subjected to the PB step in a furnace at 175 °C for 27 minutes as shown in Fig. 6.2. The PB parameter was selected to replicate industrial practice as was recommend by project partners.

Table 6.1: Resistance spot welding parameters used for the study.

Weld Schedule	Squeeze time (ms)	Weld Pulse 1 (ms)	Cool time 1 (ms)	Weld Pulse 2 (ms)	Weld Current (kA)	Cool time 2 (ms)	Post Pulse Current (kA)	Post Pulse time (ms)	Hold time (ms)
BWS	167	167	33	167	8	-	-	-	-
HNG	167	167	33	167	8	300	7	334	167
HAG	167	167	33	167	8	50	7	334	167

HNG: Homogenization No Grain refinement; HAG: Homogenization And Grain refinement

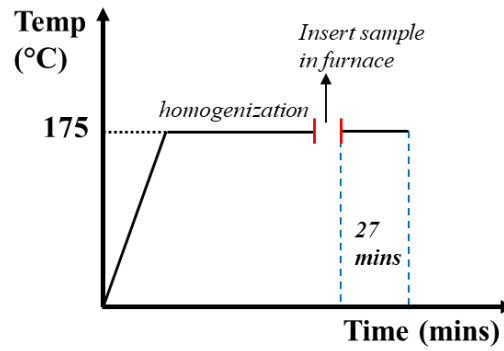


Fig. 6.2: Paint baking cycle (selection was made based on industrial conditions) used after welding.

Cross tension test was performed at a constant crosshead speed of 10 mm/min using a 100 kN MTS frame universal tester. The force-displacement data was obtained from the test while the energy absorbed was obtained by integrating the area under the curve to failure. All reported data from the mechanical tests represent an average of five repetitions while the error bar indicates a 95% confidence interval. An additional sample was tested and interrupted during the test at 90% of the average peak load of the initial three tests to understand the crack path during loading.

Welds were cross-sectioned, polished down to 0.05 μm colloidal silica for electron microscopy analysis. A Scanning Electron Microscope (SEM, JSM7001F) equipped with an electron backscattered diffraction (EBSD) detector was used to examine the failure surface and obtain maps at the edge of the FZ for the weld schedule studied including interrupted test samples. The EBSD maps were obtained at a working distance of 15 mm with a step size of 0.5 μm operating at 20 kV. Prior austenite grains boundaries (PAGB) were reconstructed using MTEX toolkit [151]. Quantitative elemental distribution was obtained at the edge of the FZ on a 300

x 300 μm^2 area using an electron probe microanalyzer (EPMA) with a spot size of 1 μm and a dwell time of 100 ms. Line scans were obtained along the diagonal of the maps. XRD measurements were performed at room temperature using a Bruker D8 Discover XRD equipment with a Cu-K α (λ : 0.154 nm) radiation source operating at 40 mA current and 40 kV voltage. The XRD patterns were obtained in the 2θ range of 20° and 100° . Post-processing of peak widths was done using the Origin software.

6.4 Result and Discussion

6.4.1 Mechanical properties after CT test

The mechanical properties for each of the weld schedule is shown in Fig. 6.3a and b. The peak load of the BWS is 6.32 ± 0.49 kN as reported in previous chapters while HNG welds had an average peak load of 7.75 ± 0.43 kN which is 23% improvement compared to the BWS. The peak load for HAG was 9.37 ± 0.16 kN, which is 48% improvement compared to the BWS. The energy absorption capability to failure follows the same trend as the average energy absorbed to failure for HNG weld was 42.25 ± 4.39 J while HAG was 57.51 ± 3.65 J. This represents a 36% and 85% increase for HNG and HAG welds respectively compared to the welds made with the BWS schedule. It is believed that certain factors contribute to the changes in mechanical properties induced by the different weld schedules. These factors will be discussed in the following sections.

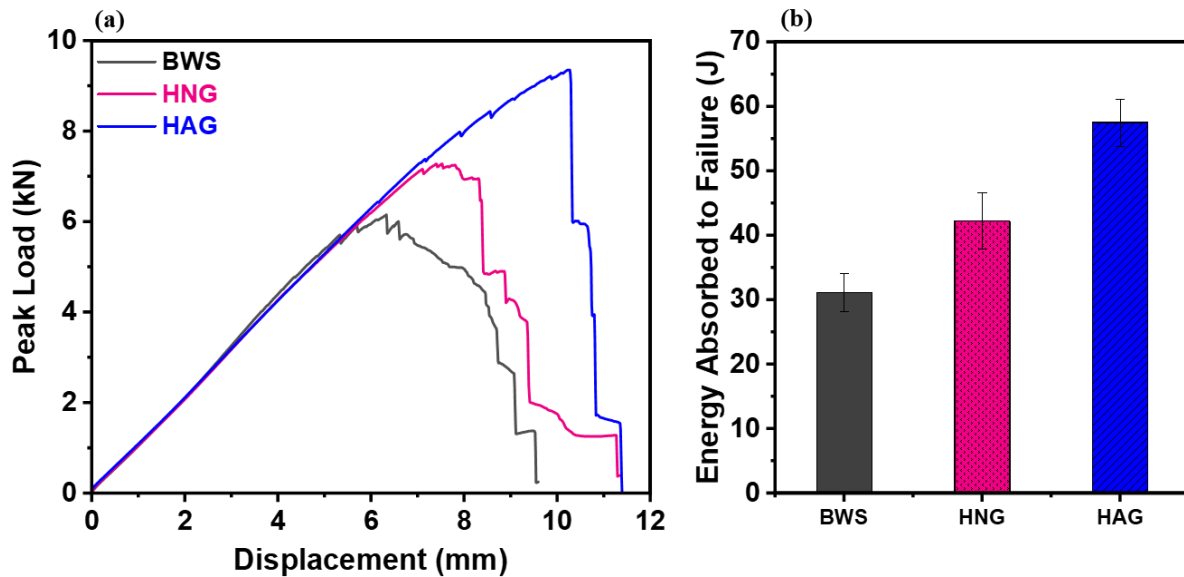


Fig. 6.3: (a) Load-displacement curve and (b) average energy absorbed to failure for BWS, HNG and HAG welds.

6.4.2 Factors governing the mechanical properties response

6.4.2.1 Process-structure-properties relationship

The welding process adopted in this study resulted in distinct grain structure and mechanical properties. As shown in Fig. 6.4a and d, the grain structure from the BWS is columnar oriented in the direction of heat dissipation, which is similar to previous BWS EBSD maps presented in chapters 4. More importantly, within the reconstructed maps in Fig. 6.4 (g-i), each colour represents a single packet that forms along the same habit plane within a given PAG. For the BWS welds, most of the PAGs transform into one or two packets, with no PAGs showing the presence of all four packets. Additionally, it was observed that the substructures are predominantly oriented in one direction, as they typically transform into one or two packets within the grain structure.

The majority of grains for the HNG welds also exhibit only one to two packets, similar to the variant distribution of the welds made with the BWS. The only exception to this was the largest elongated grain, which accommodates three packets within the grain (indicated by the different colours within the grain in Fig. 6.4h). On the other hand, HAG welds designed to induce grain refinement, show grains with up to four packets with substructure orientated differently within a PAG. For instance, the highlighted grain in Fig. 6.4i show four packets represented by the different colours, and it is evident that each packet within the grain has a different orientation. According to Khan et al., [74] in their study on martensitic hierarchy, structures that show a full distribution of four packets with 24 variants indicate an ideal martensitic transformation with minimal strain. The (001) pole figures in Fig. 6.4 j-l confirm the martensitic variants relative to selected grains from each weld schedule. HAG welds exhibit a strong indication of the formation of all the variants compared to the selected grains for HNG and the BWS. Therefore, the changes in the grain structure from columnar to equiaxed (extensively discussed in Chapter 4) combined with the various variant distributions formed after the welding process contribute to the changes in mechanical properties shown in Fig. 6.3.

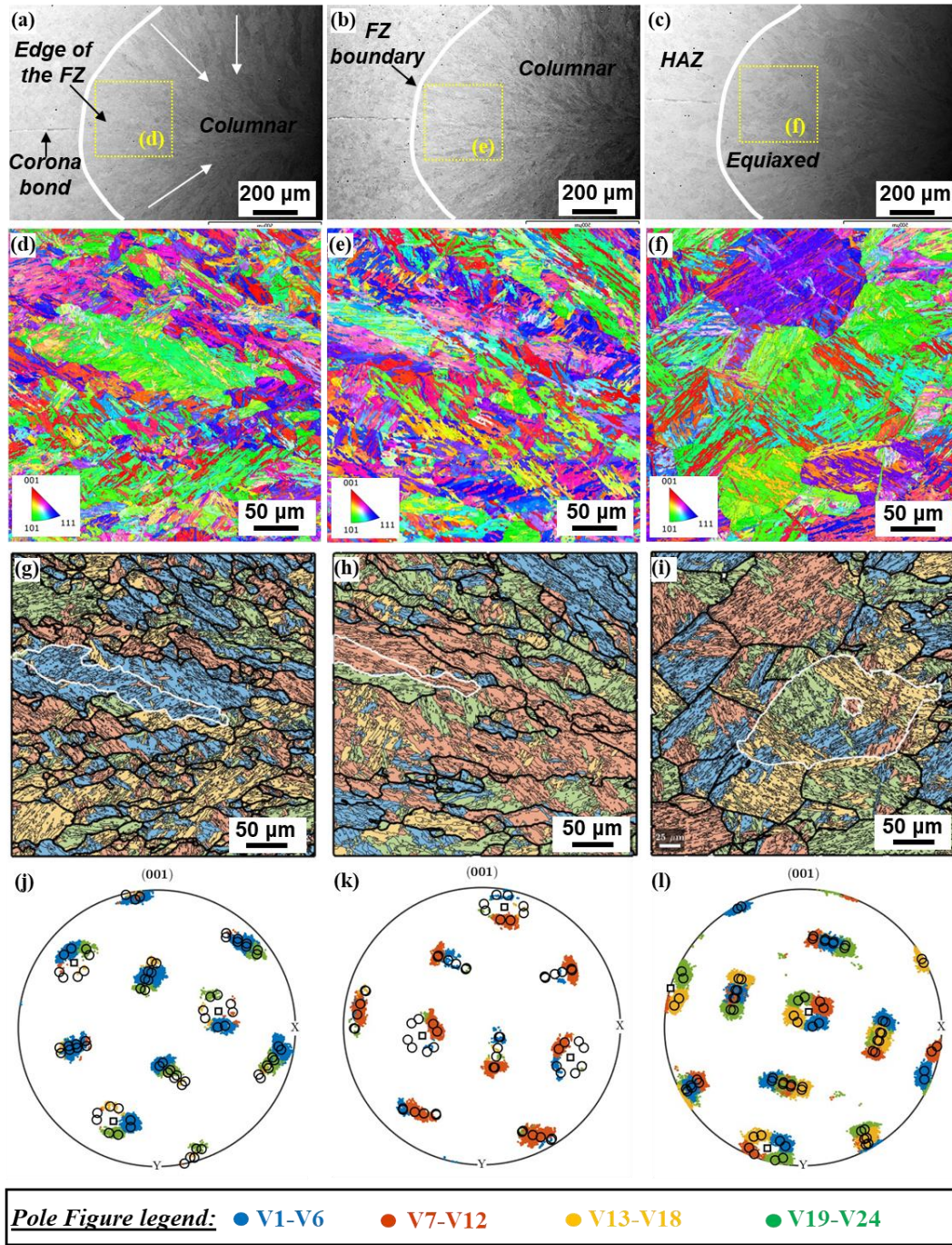


Fig. 6.4: SEM images of (a) BWS (b) HNG (c) HAG; IPF maps for (d) BWS (e) HNG (f) HAG; Reconstructed PAG boundary maps with black lines indicating the PAG boundaries for (g) BWS (h) HNG (i) HAG; Pole figure for each highlighted PAG grain used to index martensitic variants for (j) BWS (k) HNG and (l) HAG.

6.4.2.2 Influence of weld microstructure on crack path propagation

In the previous chapters, it has been established that the microstructure ahead of the notch (i.e., the pre-crack region) plays a critical role in determining the resistance to crack propagation in the joint when subjected to cross-tension tests (i.e., opening mode). For instance, the tempered martensite formed in Chapter 3 impedes crack propagation into the FZ resulting in a ductile

failure. In contrast, the brittle failure observed in the BWS welds was attributed to the crack propagating through the fully martensitic FZ. In this chapter, all welding schedules resulted in a martensitic microstructure similar to that described in Chapter 4. However, the variations in martensitic morphology and hierarchical structure along the crack path significantly influence the resistance to crack propagation.

For both the BWS and HNG welds, the crack typically advances along the columnar structure as shown in Fig. 6.5a and b, which exhibits low resistance to crack propagation [191]. Additionally, the substructures in these welds are orientated in a specific direction (see Fig. 6.4g and h), thus, minimal energy is required for the crack to proceed as it does not encounter varying HAGBs within a grain. In contrast, the HAG welds which were designed to induce grain refinement at the edge of the FZ exhibit a different behaviour. It was seen that the crack path in the HAG weld deviates at the edge of the FZ towards the UCHAZ as shown in Fig. 6.5c. The crack tip becomes blunted as it tries to propagate into the FZ as shown in Fig. 6.5c. The blunting of the crack at the edge of the FZ indicates that the new equiaxed structure resist rapid crack propagation into the FZ. This resistance was not seen for weld schedules (i.e., the BWS and HNG welds) without grain refinement. It should be noted that the region along the crack path (i.e., the UCHAZ) for the HAG welds also has an equiaxed PAG morphology and a smaller PAG size, as the temperature at the edge of the FZ is higher than the UCHAZ [194]. Consequently, the crack in the HNG weld is forced to frequently change direction within the equiaxed structure as it encounters different grain and substructure orientations as shown in Fig. 6.5f, thereby enhancing the overall toughness of the joint compared to the columnar structure in Fig. 6.5d and e.

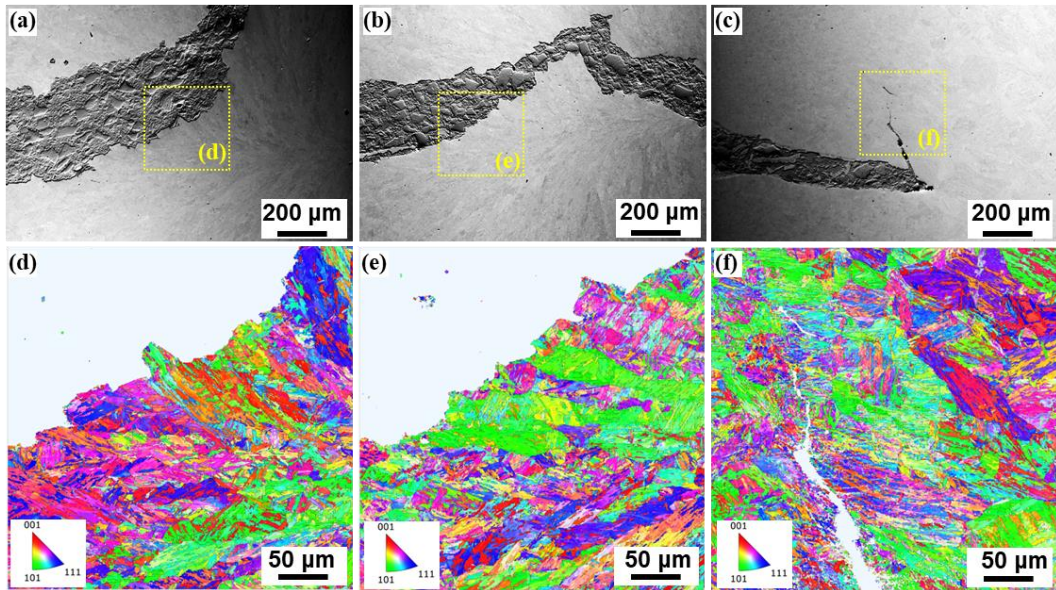


Fig. 6.5: SEM image along crack propagation path for (a) BWS (b) HNG (c) HAG; IPF maps along failure path for (d) BWS (e) HNG (f) HAG.

6.4.2.3 Influence of phosphorus homogenization on mechanical properties

The mechanical properties of HNG weld showed an improvement of 36% in peak load and 42% in energy absorption capacity compared to the BWS schedule. However, as shown in Fig. 6.5, the crack path remains similar, which is along the columnar structure. This observation suggests that another factor other than the grain structure must have contributed to the changes in mechanical properties. Previous literature has attributed improvement in mechanical properties to the homogenization of phosphorus at the edge of the FZ. Specifically, Eftekharmillani et al., [115] claimed that the effect of phosphorus homogenization surpasses the influence of reduced grain size at the edge of the FZ. To further investigate the phosphorus homogenization effect, EPMA maps along the crack path were conducted to determine whether phosphorus segregation deteriorates properties or if its homogenization enhances the weld mechanical performance.

As shown in Fig. 6.6 a-c, there is no clear evidence of phosphorus segregation along the crack path. Although some spikes on the line scans were observed for the BWS welds, the spikes cannot be conclusively linked to P segregation or a potential deterioration of the mechanical properties. For schedules 1 and 2 welds, the P distribution along the crack path is more homogenous. It is important to note that while previous studies have demonstrated P segregation and homogenization after post-pulsing [115], the welds were scanned after welding and there was no report of a direct correlation of the crack propagation along the P-rich regions. In addition, the reported P segregation from the study was in steels with higher P content than

PHS. Contrary to reports in the literature, the results from this study reveal that the changes in mechanical properties are not linked to either P segregation or homogenization. Thus, the improved mechanical properties in HNG and 2 welds must be attributed to other factors not directly related to P distribution along the crack path.

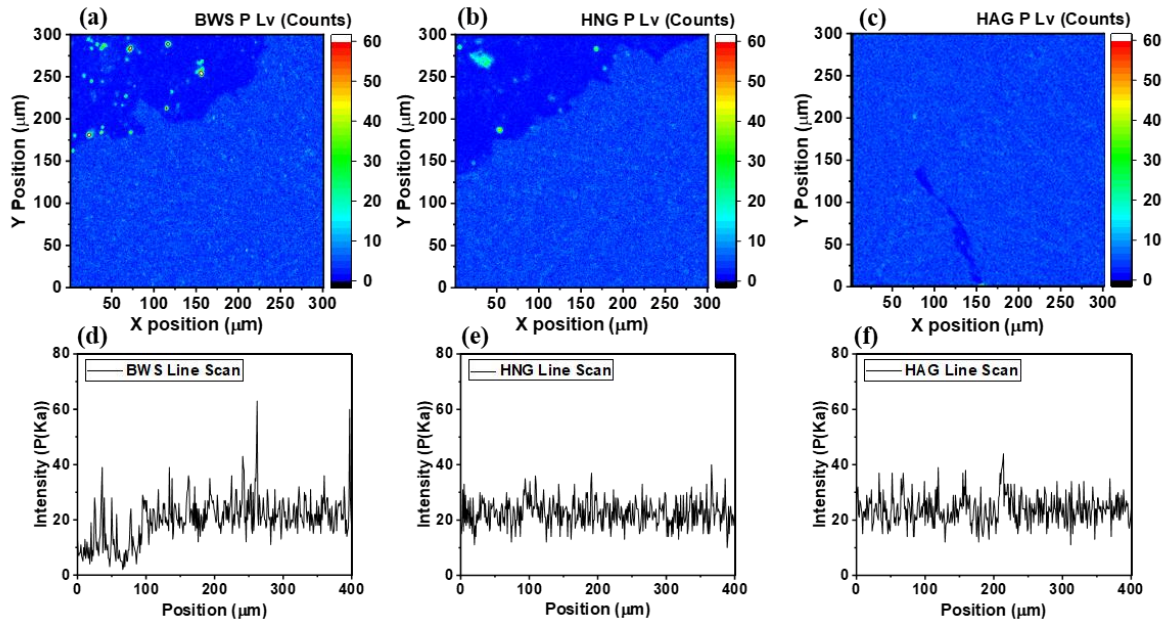


Fig. 6.6: EPMA maps and line scans showing phosphorus distribution at the edge of the FZ for (a) BWS (b) HNG (c) HAG. *Line scans were taken along the diagonal of the mapped area.

6.4.2.4 Influence of texture on crack propagation and mechanical properties

Heydari et al., [195] emphasized that crack propagation is also influenced by crystallographic texture, which is dependent upon the starting and deformation texture. Therefore, it is important to determine the influence of texture in dictating how crack advances. The texture for the three weld schedules before the cross-tension test, after the welding (starting texture) is shown in Fig. 6.7(a-c), and after the cross-test, the deformation texture is shown in Fig. 6.7 d-f. The weld made with the BWS shows a dominant (001) texture, which is perpendicular to the loading direction as shown in Fig. 6.7a. In other words, the majority of grains have their (001) planes vertical to the loading direction. It is well-documented that BCC metals tend to cleave along the {001} planes due to minimal plastic deformation near the crack tip compared to the {110} planes with the highest atomic density [196–198]. This assertion is supported by the extensive work of Suzudo et al., [199] that observed a brittle fracture with a modest amount of dislocation emission from the crack tip of cleavage along {001} planes. Additionally, the deformation texture does not undergo noticeable changes as the (001) texture is only slightly shifted towards the (001) and (111) poles (Fig. 6.7d) as some grains could rotate to accommodate deformation.

Consequently, the failure for the BWS was pure cleavage without crack blunting or significant plastic deformation, as evidenced by the cleavage features from the fracture surface in Fig. 6.8a and d.

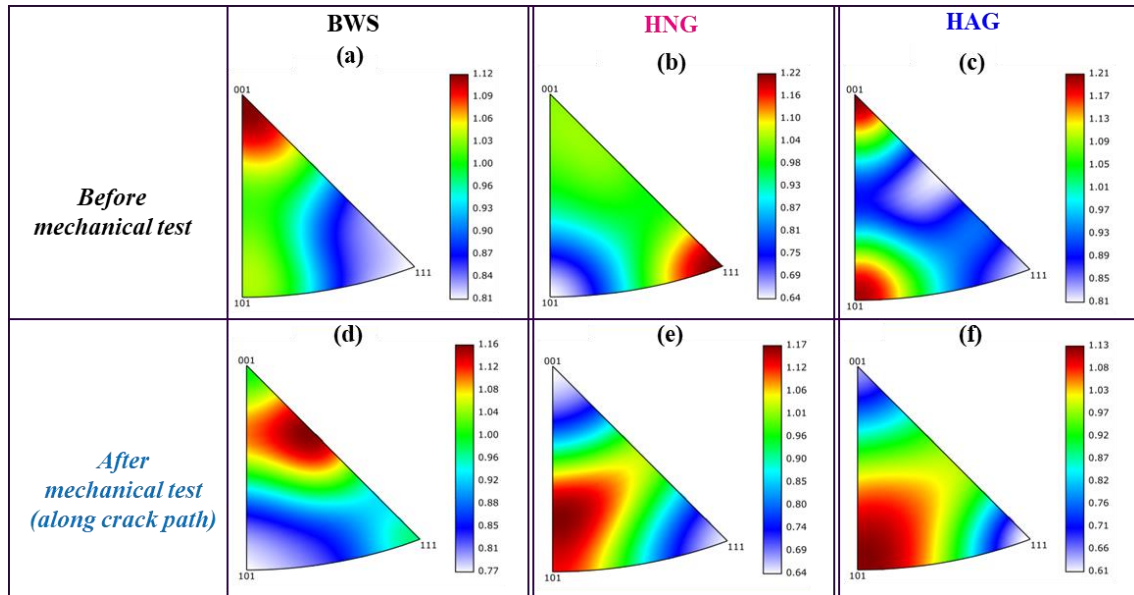


Fig. 6.7: IPF texture maps before mechanical testing for (a) BWS (b) HNG (c) HAG; IPF texture after mechanical testing for (d) BWS (e) HNG (f) HAG.

Cleavage cracks can be blunted by the emission of dislocations, as established in previous literature [198,200]. In the case of HNG welds, a dominant (111) texture was observed after welding, and it is notable that no dominant (001) texture can be seen, which is responsible for a brittle cleavage fracture upon further loading as in the BWS welds. The {111} plane in the BCC structure has the highest surface energy and requires more energy to create new surfaces, which leads to the emission of dislocations before crack propagation [201]. After mechanical testing, there is a shift from the (111) pole to the (101) pole, where (101) planes of most grains are perpendicular to the loading direction. By the activation of the BCC {110} $\langle 111 \rangle$ slip system for most grains, the emission of dislocations occurs before crack propagation to accommodate the deformation which reduces the propagation rate of cracks. Thus, the slip occurring in the HNG weld due to the change in texture likely improved the mechanical properties of the joint. This is further evidenced by the fracture surface shown in Fig. 6.8b and e, which shows mixed fracture particularly some dimple features indicative of ductile failure ahead of the crack propagation. It should be noted that HNG welds also exhibited columnar structure, but the change in texture could be due to the phase transformation during reheating. However, verifying this claim would require additional work which is beyond the scope of this research.

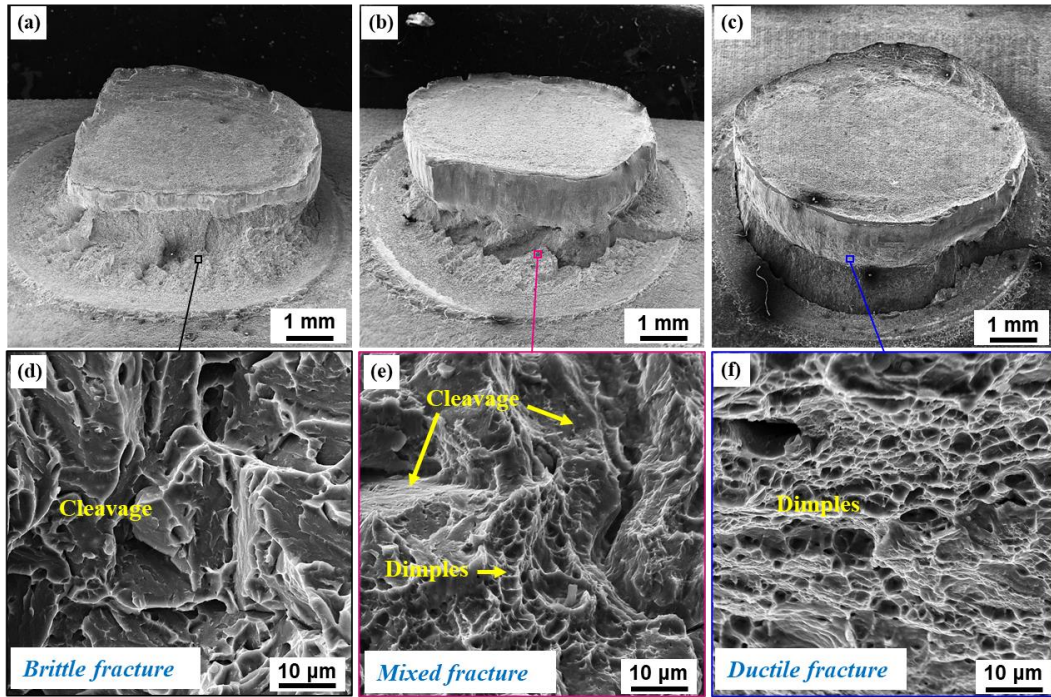


Fig. 6.8: SEM images of failed welds for (a) as-welded (b) DF (c) DF with PWHT; SEM images of failure mode for (d) as-welded (e) DF (f) DF with PWHT; fractography for (g) as-welded (h) DF (i) DF with PWHT schedules.

A strong (101) and (001) texture was observed for the HAG weld, where new equiaxed grains were formed. However, the deformation texture (after mechanical testing) only shows the domination of (101) texture as shown in Fig. 6.7e. It is presumed that there would be a competition between cleavage along the {001} and slip along the {101} plane. However, it is seen in Fig. 6.7f that the activation of main slip systems due to the dominance of (101) texture promoted dislocation generation to accommodate deformation, thereby leading to crack blunting. Based on the failure surface of the welds shown in Fig. 6.8c and f, only dimple features were observed, suggesting that plastic deformation occurred ahead of the crack tip. Tyson et al., [196] reported that plastic deformation at the crack tip is more likely along the {110} plane compared to the {001} plane. This is evident from the IPF map in Fig. 6.5f confirming that crack blunting occurred as the crack navigates through the (101) plane. Thus, the texture along the {101} after the mechanical test in Fig. 6.7e and f indicate that the slip occurred along the crack path for HNG and HAG welds compared to the BWS welds. This analysis underscores the significant impact of crystallographic texture on the resulting mechanical properties and crack propagation behaviour of welds, which has mostly been unexplored in the literature.

6.4.3 Summary of metallurgical principles, mechanical properties and processing time

The previous chapters focused on the pathways to achieve microstructural modifications by controlling different in-situ post-weld parameters (cool time, post-weld current and post-weld time). It was observed that effectively controlling these parameters is key in enhancing the mechanical properties of the weld. Table 6.2 summarizes the major metallurgical principle in relation to optimal post-weld parameters needed to maximize the joint properties.

Table 6.2: Summary of optimal post-weld conditions to trigger microstructural changes in the weld.

Technique		Metallurgical principle and in-situ parameters
1.	Tempering	• Cool time is sufficient to quench below M_s temperature. • Post-weld current must re-heat the weld below the Ac_1 temperature. • Spike tempering with shorter post-weld time and higher post-weld current is more effective for industrial applicability over conventional with longer post-weld time and lower current.
2.	Recrystallization	• Cool time must be short to cool the edge of the FZ to the austenitic region ($Ar_3 < T < T_m$). • Post-weld current has to heat the weld above the Ac_3 temperature but below the melting point. • Maintain the heat above the Ac_3 temperature but below the melting point during the post-weld time. Also, avoid extending the time to prevent excessive grain growth.
3.	Dual Force (DF)	• Keep cool time short to cool the edge of the FZ to the austenitic region ($Ar_3 < T < T_m$). • Ensure dual force is high enough to induce sufficient strain in the weld (a higher force limit is recommended). • Ensure dual force time is sufficient to allow continuous deformation until the end of the cooling cycle (below M_s).
4.	DF with PWHT	• Combines DF with recrystallization principles.

As much as it is important to control the process parameters, the overall time needed to actualize each in-situ microstructural modification is also crucial, particularly from a manufacturing perspective. The overall process time (from squeeze to the end of the hold time)

needed for each technique with the corresponding mechanical properties is shown in Fig. 6.9a and b, while the optimized condition that maximized joint mechanical properties is summarized in Table 6.3. It is seen that the optimal joint properties are achieved with the DF with PWHT schedule with an increase of 61% in peak load and 94% in energy absorption capability to failure compared to the BWS. This is followed by the recrystallization pulse schedule (referred to as PWHT pulse in chapter 4). Even though these two techniques show remarkable improvement in mechanical properties, the recrystallization pulse schedule is more industrially friendly considering the overall process time required and ease of incorporating the weld schedule in the production line as the dual force can be excluded. The recrystallization pulse schedule adds 267 ms (37% increase in process time) to the BWS but results in a 54% increase in peak and 89% in energy absorption capability to failure. Meanwhile, the DF with PWHT pulse adds 384 ms (which is a 54% increase in process time) compared to the BWS. While the DF and tempering improve the joint performance as shown in Table 6.3, the properties are inferior to the recrystallization pulse technique. Besides, the optimal DF schedule adds about 217 ms (i.e., 30%), while the tempering schedule adds 1067 ms (i.e., 148%) to the overall process compared to the BWS. Based on the mechanical property and process time comparison, the recrystallization pulse schedule is taken as the optimal technique to improve weld toughness and was selected to be studied for the paint baking schedule.

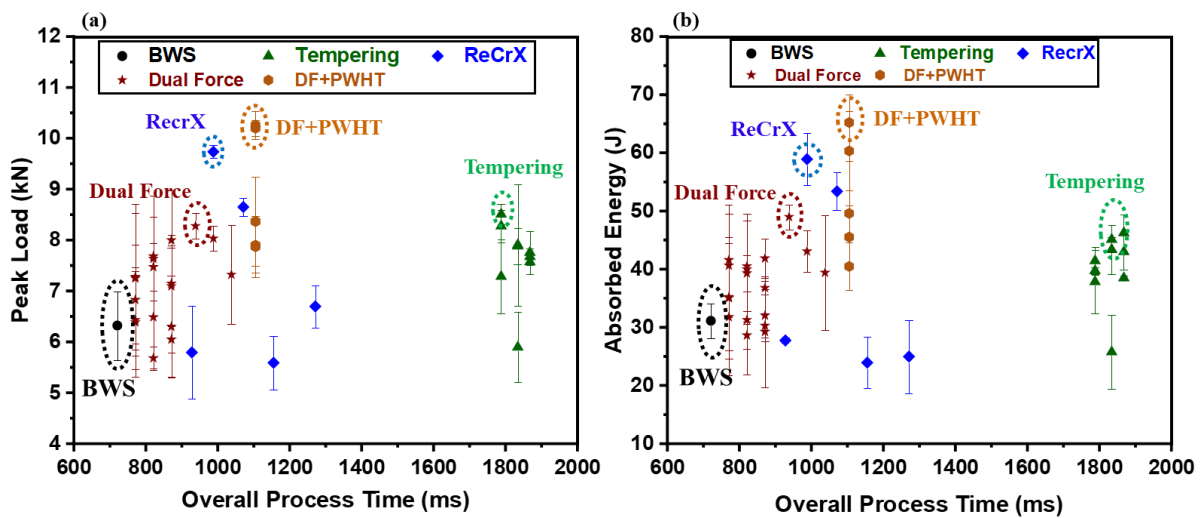


Fig. 6.9: Overall process time for each technique showing (a) peak load (b) energy absorption capability

Table 6.3: Summarized weld technique, process parameters and optimized joint properties.

	Weld schedule	Process time (ms)	Peak load (kN)	% increase in Peak load	Energy absorption (J)	% increase in energy absorbed
1.	Baseline	721	6.32 ± 0.67	-	31.11 ± 2.95	-
2.	Tempering	1788	8.51 ± 0.20	36%	41.43 ± 2.46	33%
3.	Recrystallization	988	9.74 ± 0.13	54%	58.96 ± 4.43	89%
4.	Dual Force	938	8.28 ± 0.25	31%	48.98 ± 2.12	57%
5.	DF+PWHT	1105	10.20 ± 0.16	61%	65.23 ± 4.84	94%

6.4.4 Role of Paint Baking (PB) after PWHT schedule

6.4.4.1 Mechanical properties after PB

As established in section 6.4.3, the recrystallization pulse (RecX) was identified as the optimal technique for improving weld toughness. This technique was further subjected to paint baking. The base metal average hardness before PB was measured to be 490 ± 4 HV, which was reduced to 457 ± 3 HV after the PB schedule as shown in Fig. 6.10. The nugget average hardness was 495 ± 5 HV in the BWS weld and remained at 495 ± 4 HV after applying the PWHT pulse from Chapter 4. It should be recalled that there was no significant difference in hardness after applying the PWHT schedule, as both structures contained martensite which was formed after rapid cooling. After subjecting these welds to PB, the average FZ hardness decreased to 484 ± 6 HV for the BWS+PB welds and 474 ± 4 HV for the RecX+PB welds. This reduction in hardness is believed to be related to microstructural changes induced by the PB schedule, which can lead to different joint responses during tensile testing.

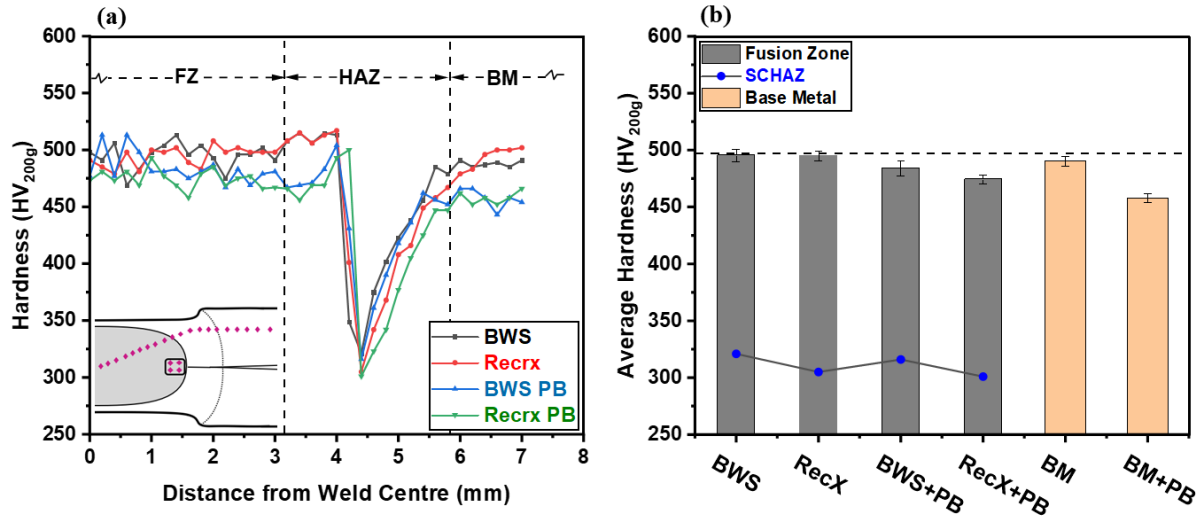


Fig. 6.10: (a) Microhardness of BWS and PWHT pulse before and after PB (b) average hardness of FZ and BM before and after PB.

The average peak load and energy absorption capability of the BWS increased by 16% after PB, with peak load values of 7.35 ± 0.88 kN and energy absorption capability of 36.14 ± 5.93 J compared to the BWS values of 6.32 ± 0.49 kN, and 31.12 ± 2.95 J, as shown in Fig. 6.11. However, it was observed that this increase was not statistically significant. For the welds subjected to the recrystallization pulse (RecX), the peak load increased by 54% to 9.74 ± 0.13 kN. When PB was added to the RecX schedule, the peak load increased by 49% (9.43 ± 0.51 kN) compared to the BWS. Similarly, the energy absorption capability to failure significantly increased by 89% with the RecX pulse and 92% with the addition of PB. These results indicate that paint baking had a less significant impact on the mechanical properties of PHS welds compared to the contributions from the PWHT pulse that resulted in grain refinement and change in weld texture. This finding contrasts with previous studies on 3G-AHSS like quenching and partitioning (Q&P) steel, where a significant increase in peak load and energy absorption capability was observed after PB [86]. The changes in properties for 3G-AHSS were attributed to the decomposition of martensite, formation of ϵ -carbides, and the redistribution of segregated alloying elements from the grain boundary, consistent with the findings of EftekhariMilani et al., [130] and Ramachandran et al., [86]. Although a minor reduction in weld hardness was observed after PB in this work, the hardness reduction alone cannot explain the minor change in CT strength, particularly for the BWS schedule. Hence, it is believed that the failure mode influences the changes in properties, as will be discussed in section 6.4.4.2.

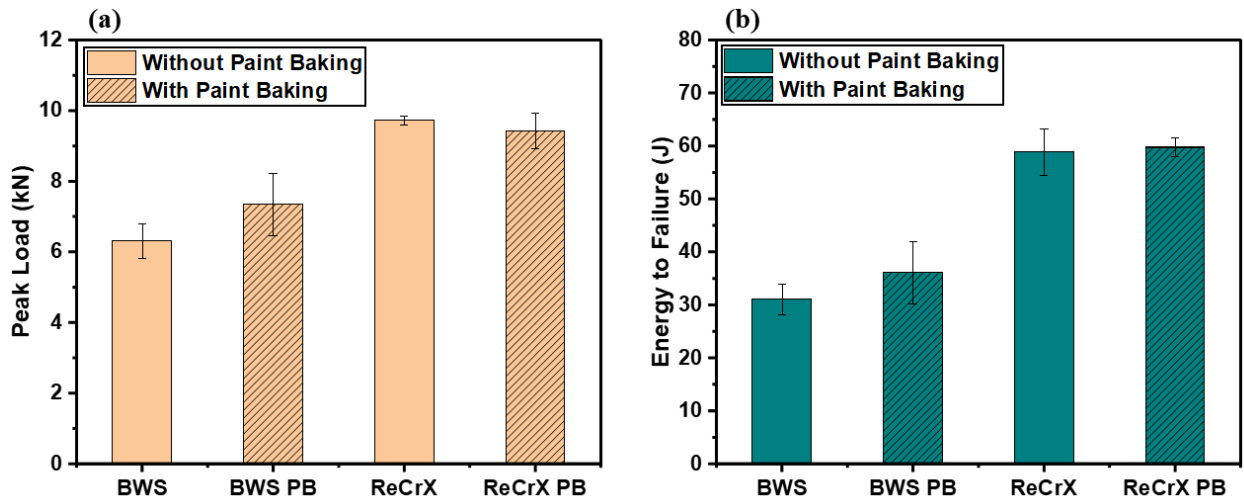


Fig. 6.11: (a) Average peak load for BWS and Recrx pulse before and after PB (b) Average energy absorbed to failure for BWS and Recrx pulse before and after PB.

6.4.4.2 Failure mode and fractography

The failure mode and microstructure for the BWS and PWHT pulse, both before and after PB, are shown in Fig. 6.12. The crack propagation path for the BWS and PWHT has been discussed extensively in previous chapter 4. After PB, the crack propagation path for the BWS remains similar to that before PB, with the crack propagating into the FZ. However, after PB, the crack deviates at the edge of the FZ, resulting in a higher plug ratio compared to the weld without PB. Similarly, a higher plug ratio was observed for the RecX with PB, as the crack deviated along the UCHAZ close to the FZ boundary. In this case, the refined grain for the Recrx pulse schedule inhibits the crack propagation. After PB, it is shown that the martensite disintegrated, indicating that tempering occurred. EftekhariMilani et al., [130] reported the presence of epsilon carbides in the martensitic matrix after paint baking at 180 °C for 20 mins and attributed the presence of the carbide precipitation to the improved mechanical response. It is worth noting that changes in residual stress have also been reported to improve the weld properties after PB [130], however, this was not considered within the scope of this thesis as precise measurement will require the use of synchrotron XRD technique. Hence, it is believed that the effect the grain refinement is more prominent than the tempering effect from the paint-baking cycle.

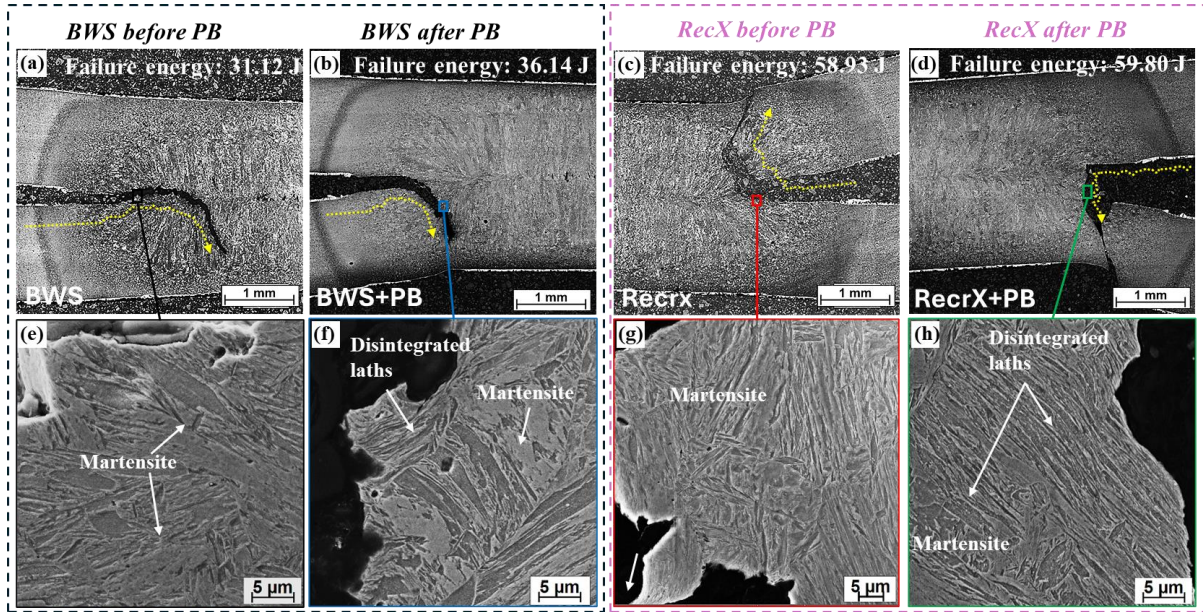


Fig. 6.12: Cross-section of interrupted cross-tension tested welds for (a) BWS (b) BWS+PB; (c) RecX and (d) RecX +PB; SEM image showing microstructure along crack path for (e) BWS (f) BWS+PB; (g) RecX and (h) RecX +PB.

The fracture surface of the welds after CT test is depicted in Fig. 6.13. Before PB, the fracture surface of the BWS shows cleavage features, which are indicative of brittle failure. In contrast, after PB, the BWS weld exhibits a mixed-mode failure, with both cleavage and dimple features present as shown in Fig. 6.13c and g. This suggests a combination of brittle and ductile failure modes. On the other hand, the fracture surface of the RecrX pulse before and after PB shows completely ductile failure, characterized by dimple features as shown in Fig. 6.13f and h.

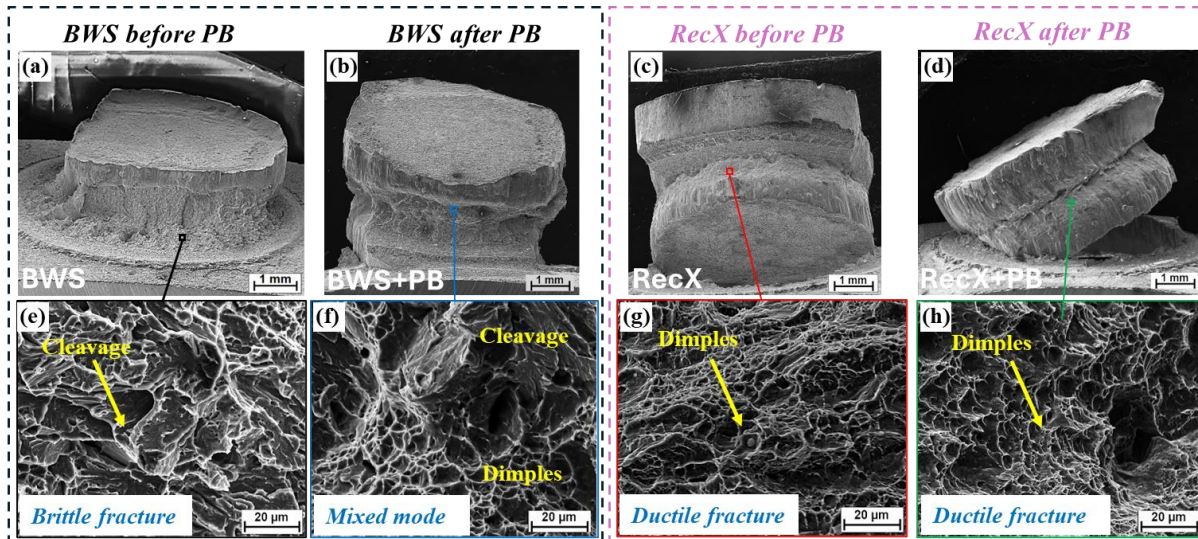


Fig. 6.13: Failure mode for (a) BWS (b) BWS+PB; (c) RecX and (d) RecX+PB; SEM fractography images for (e) BWS (f) BWS+PB; (g) RecX and (h) RecX+PB.

6.5 Summary

This chapter extensively discussed how different microstructural factors contribute to the mechanical properties of PHS welds. It was found that refining the grain structure leads to optimal mechanical properties, which is achieved at a relatively shorter time compared to the other PWHT schedules employed in this study. Contrary to previous literature where P segregation was observed in steel with P content, there was no correlation between P homogenization at the weld of the FZ with the resulting mechanical properties. Instead, a change in texture from the cleavage (001) planes for the BWS to the (101) planes were found to enhance plastic deformation ahead of the crack tip after the PWHT schedule. Furthermore, paint baking of PHS welds did not yield notable improvement in mechanical properties as per this study.

CHAPTER

7

7. Conclusion and Future Recommendations

7.1 Conclusion

The martensitic microstructure along with the columnar structure in the FZ of PHS welds is problematic, as it results in brittle failure showing relatively low energy absorption capability. This brittle failure is undesirable for vehicle crashworthiness and needs to be improved for better reliability in case of crash events. This research sought to understand the various pathways to enhance the mechanical properties and failure behaviour of PHS welds. All the thermo-mechanical treatments studied in this research resulted in improved joint properties, with in-situ grain refinement proving to be the most effective technique. In this work, an understanding of the correlation between post-weld parameters and the physical metallurgical transformation occurring during the non-equilibrium process is presented. These transformations were also monitored from the changes in dynamic resistance during the post-weld process, which has not been explored before now. In addition, this study delineated the contributions of various metallurgical factors that occur concurrently during post-weld treatment to the changes in the weld properties and found the dominant factors to be the changes in the PAG structure and weld texture. This research also offers insights into how weld properties can be maximized and recommends principles for microstructural modifications, highlighting their applicability in an industrial environment. The following conclusions can be drawn from the present study:

1. In-situ grain refinement achieved via austenite recrystallization was found to be the most effective technique that yields the optimal enhancement of mechanical properties. The changes in dynamic resistance and thermal history during the in-situ PWHT were used to describe the metallurgical transformation. It was found that when the edge of the FZ is austenitic and below the melting point, recrystallization is promoted by reheating that region, which refines the grain structure. Conversely, when the PWHT current is applied after the edge of the FZ has transformed to martensite, austenite is formed by martensite reversion on reheating and the original epitaxial structure is retained after cooling. This insight extends beyond PHS welds and can be transferable to other AHSS and potentially to dissimilar stack-ups.
2. The addition of strain energy after the edge of the FZ is solidified refines the substructure of martensite and increases dislocation density within that region compared to the baseline welds. The microstructural changes due to the induced strain energy were found to increase the yield strength of martensite by 13% compared to the

baseline welds, thereby improving the overall strength of the weld. On the other hand, combining strain energy and thermal energy provides sufficient driving force for both PAG and substructure refinement, with the majority of grains showing the 24 variants. These microstructural modifications effectively change the crack path during tensile loading, thereby improving the energy absorption capability of PHS welds.

3. Variation in hardness at the weld center and edge after in-situ tempering was attributed to the presence of voids in the FZ, which affects the temperature distribution in the nugget during post-weld tempering. This variation drastically increased the scatter in mechanical property measurements. However, a well-designed pre-pulse schedule effectively reduced voids in the FZ, resulting in effective tempering of the nugget as a more homogenous temperature was attained during post-weld tempering. Welds tempered with reduced voids exhibited a reduced standard deviation in the mean peak load and energy absorption capability. Thus, it was concluded that reducing voids in welds is essential for effective post-weld heat treatment.
4. Energy absorption capability improved by 33% after tempering, 89% after in-situ grain refinement, 57% after dual force application, and 94% by combining dual force with PWHT current compared to the baseline weld schedule (BWS). For each schedule, crack propagation was retarded at the edge of the FZ where the microstructural modification occurred, unlike in the BWS welds where the cracks propagate directly into the FZ, leading to a partial interfacial failure mode. Additionally, a transition from cleavage features (indicating brittle failure for the BWS) to dimple features (indicating ductile failure) occurred after subjecting the welds to the different microstructural changes.
5. Among the factors contributing to improved joint performance, in-situ grain refinement and change in texture were found to be the most significant. Contrary to reports in the literature, no correlation was found between phosphorus segregation and mechanical properties in this study after applying the PWHT schedule. Instead, changes in crystallographic texture, particularly from the cleavage (001) plane in welds made with the BWS to the (101) planes after the PWHT schedule, allowed plastic deformation ahead of the crack tip and contributed to the overall improved toughness in the PWHT schedule welds. Although paint baking is known to induce additional microstructural

changes that can improve joint performance, its effect on PHS welds did not show significant improvement in this work.

7.2 Areas of Future Research

Although this work has predicted the metallurgical transformations occurring with each PWHT studied in this research, particularly for the in-situ grain refinement which has not been well examined in the literature, the proposed sequence was based on thermal history from simulation and final microstructural observations from experiment. Future studies can be done to observe the intermediary transformation in real-time using advanced microscopy techniques such as confocal laser microscopy (CLM). By replicating the same thermal history from the post-weld schedule in the CLM, the transformations can be observed in real time to validate the proposed sequences from this work. This understanding can add more insights into the solid-state transformations occurring during the non-equilibrium in-situ RSW process.

Metallurgical factors that dictate the joint properties were mostly considered in Chapter 6 of this work, but the effect of the post-weld schedules on residual stress, particularly at the edge of the FZ, was not investigated. It is hypothesized that the post-weld schedules can reduce residual stress at the front of the notch, decreasing the stress field around the crack and retarding rapid crack propagation. Accurate measurement of the residual stress will require synchrotron x-ray diffraction. Further studies can explore this area to quantify the changes in residual stress after the microstructural modification techniques explored in this work.

This work has provided insight mainly focusing on a high-strength material with low ductility to improve the weld toughness. The understanding gained can be extended to other grades of AHSS, and more interestingly, to stack-ups of dissimilar metal combinations. These studies can expand the validity of the microstructural modifications explored in this work and establish its influence on improving the mechanical performance of multiple sheet stack-ups. Moreover, more work is still needed to test these PWHT schedules under dynamic loading and to understand the role of the microstructure change in energy absorption capability under high strain rates. A mathematical model can also be developed after experimentation (for both quasi-static and dynamic loading) to predict the energy absorbed under dynamic conditions.

Microstructural modification from martensite to tempered martensite was shown to be effective in improving joint mechanical properties in this study. However, it is unknown how the formation of other steel microstructural phase such as bainite can be achieved in the weld FZ, and how it will govern the resulting joint properties. As shown in the literature review, another

promising pathway termed “slow cooling” can prevent martensitic transformation and promote the formation of softer phases in the weld nugget. This can be metallurgically achieved by attaining a cooling rate below the critical cooling rate of the material and can be made possible during in-situ RSW PWHT by applying a lower current pulse to slowly cool the weld from the austenitic region. It is hypothesized that an optimized post-weld current pulse will prevent a rapid temperature drop and allow short-range diffusion of alloying elements such as carbon which could favour the formation of softer microstructural phases such as bainite. Preliminary investigations and findings from this study propose a sloped-down current pulse, which is a possibility achievable on numerous car manufacturers' welding equipment. This can further enhance the possibility to control the RSW process to achieving different microstructural modifications.

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