Understanding Microstructure Evolution in Rapid Thermal Processing of AISI 8620 Steel

A Thesis

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ABSTRACT

A new steel heat treatment process has been developed to achieve strength comparable to Advanced High Strength class of steels. This process is unique for its very short treatment time (<10s) and is hence termed Flash Process. The strength of the steel obtained from this process (ultimate tensile strength (UTS): 1694 MPa, elongation: 7.1%) showed at least 7% higher UTS and 30% greater elongation than published results of martensitic advanced high strength steel (UTS: 1585 MPa, elongation: 5.1%). Arc welding of flash processed steel resulted in softening in the heat affected zone, which could be a potential site for crack initiation. In order to devise better welding procedures to join this steel, the microstructure evolution during flash processing has to be understood.

The main goal of this thesis is to explain the formation of the flash process microstructure, which governs the strength of the steel. In order to achieve this goal the task was divided into two parts. In the first part, the thermal profile of the flash process is quantified. The second part focuses on the characterization of the microstructure in the final steel. Based on the results obtained from the two analyses, the microstructure evolution is
explained. This work forms a basis for future research on the weldability of the steel produced from this process.

Initial confirmatory tests were carried out to verify the properties of the flash processed steels. The steels showed high strength (UTS > 1500 MPa) and good ductility (elongation > 8.7%). Thermocouples were attached to the steel plates to measure the thermal profile of the process. The total time of the process was recorded to be around 6s with a peak temperature of over 1000°C. Analysis of the heating curve indicated complete austenitization with Ac1 and Ac3 temperatures lying in the ranges 925-930°C and 1045-1052°C respectively and a very short dwell time (< 2s) in the austenite region. Analysis of the thermal profile provides an insight into the microstructure evolution during the flash process.

The initial microstructure of the steel consisted of ferrite with carbides. Optical images of the flash processed steel showed very fine features that could not be clearly identified. Scanning electron microscopy of the final steel also indicated the presence of these carbides. This pointed to incomplete carbide dissolution during the process. On further analysis, transmission electron microscope images showed the presence of bainite in the steel, along with martensite.

The presence of carbides shown by microstructural characterization and the short austenite dwell time from thermal analysis, indicate a non
homogenous carbon distribution in the austenite phase. This inhomogeneity is related to the formation of a mixed microstructure of bainite and martensite.

Finally, the high strength observed in the flash process steel is rationalized by modifying published theoretical models on the strength of steel. It is shown that a mixed microstructure of bainite and martensite is much more stronger than completely martensitic steel.
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FIELDS OF STUDY


Studies in Heat Treatment and Weldability of Steels
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CHAPTER 1

INTRODUCTION AND MOTIVATION

In the face of the growing energy crisis, the automotive industry is faced with the challenge of complying with passenger safety norms while maintaining good fuel efficiency and minimizing emissions. One of the most promising routes to achieve these objectives is to reduce the overall weight of vehicles. With this goal, Ultra Light Steel Automotive Body-Advanced Vehicle Concept (ULSAB-AVC) consortium developed a new class of steels that can be used to make a range of body components while reducing the net weight [1].

This class of steels has been named Advanced High Strength Steels (AHSS) for their superior mechanical properties over conventional steels. Their properties are governed by the underlying microstructure achieved by controlled thermal cycles. However, many challenges arise when such steels are welded [1]. Welding imposes localized thermal cycles that modify the carefully designed microstructure of the steel, thereby rendering it unsuitable for its intended application. The type of welding process and other welding parameters (i.e. the sample geometry, pre-heat, filler metal, weld heat input,
the cooling practice and post weld heat treatment etc.) are important parameters for a weld procedure development. However, for developing welding practices that retain the properties of the steel, a thorough knowledge of the underlying microstructure and the original thermomechanical processing is necessary.

A relatively new heat treatment steel processing technique has been developed, that has been termed ‘Flash Process’ for its very short treatment time (~10s). Flash Process is believed to create AHSS type microstructures in low alloy steels plates. This process was essentially developed as an energy efficient alternative to the traditional heat treatment practices that use continuous annealing lines. This process has been claimed to produce steels with AHSS properties reaching ultimate tensile strengths up to 1600 MPa and elongations of well over 8%. However, no prior knowledge of microstructure evolution during the flash process, that causes the observed strength, was available. As a preliminary test to access weldability, two flash processed steels were welded.

1.1 Preliminary Welding of Flash Processed Steel

A 75 mm wide and 6.3 mm thick steel sheet of the grade AISI 4130 was flash processed. This steel was reported to have strength (UTS) greater than 1800 MPa and a total elongation of 11%. Furthermore, the hardness of the steel was measured to be 408 HV. Information about the microstructure prior to flash processing was not known. However, after flash processing the
microstructure of the steel appeared to be very fine and consisted of martensitic type lath structures. Welding of this flash processed steel showed softening in a narrow region of the far heat affected zone (HAZ). Welding was performed with no preheat. For the weld, 1/8” diameter ER70S – 6 filler rod was used and seven weld passes were made. No other welding parameters were recorded. Hardness measurements were carried out over the welded region.

1.2 Weld Hardness Measurements

Over 6000 micro-hardness indents were made over a region comprising of the weld metal, the HAZ and the base metal. Fig.1.2 shows an image map of the hardness measurements. The weld metal (region 1) clearly shows a region of lower hardness than the HAZ (regions 2) and the base metal (regions 3). Within the HAZ, the region closer to the weld metal is harder (~300 HV) than the region farther away from the HAZ (~210 HV). However, the whole weld zone is softer than the base metal by ~100 HV. With the knowledge of the initial microstructure, weldability of the steel can then be evaluated by using available predictive tools [2].

In a recent study, weldability of DP and TRIP type AHSS steels was evaluated. Two steels, DP780 and TRIP780, were welded using two different filler wires, ER70S-6 and ER100S-G. The study showed that the cooling rate of the welds played a major role in the distribution of hardness.
For example, a predominantly high hardness martensite region (~375HV) was reported in the HAZ close to the weld in DP 780, for high weld cooling rate. However, the peak hardness observed for the same steel for, from a low cooling rate weld was ~260HV. The hardness distribution over the HAZ was also found to depend on the microstructure and alloying additions in the base metal. The DP 780 grade steel was predominantly ferritic along with islands of martensite. Comparatively, TRIP 780, which was alloyed with aluminum, remained mostly ferritic during welding. This resulted in a microstructure predominantly of ferrite with some martensite near the fusion boundary, leading to a lower hardness than DP 780 steels. Tensile tests conducted on these welded joints resulted in failure predominantly along the softened HAZ for both grades of steels [3].

Following the observations of the above study, in the welded flash processed steel, two possible regions of weld failure initiation are
hypothesized. One being at the fusion zone (region 1) due to under matched weld metal strength when compared to the base metal (the difference in hardness being ~200 HV between them) and the other being at the softened region of the HAZ as was observed in the study.
2.1 Advanced High Strength Steels: Properties and Processing

In recent times, one of the goals of the automotive industries has been the weight reduction of vehicles in order to achieve improved fuel efficiency while not compromising on the safety of the passengers. The focus has been on a new class of steels first developed by ULSAB-AVC [4,5] called the Advanced High Strength (AHS) Steels. This family of steels shows good formability while maintaining very high strength values (yield strength >300 MPa, ultimate tensile strength (UTS)>700 MPa [6]). Compared to high strength steels (HSS), where the strengthening was achieved by precipitation strengthening, solid solution strengthening and grain refinement, the strengthening in AHSS is attained through phase transformation and precise microstructure control. Based on the microstructure and the properties exhibited, the AHS steels are classified into the following grades: dual phase (DP), transformation induce plasticity (TRIP), complex phase (CP) and martensitic (MART) steels. All theses steels have multi-phase
microstructures that essentially determine their properties that include improved formability and crash worthiness. Due to the superior properties exhibited by these steels, their integration in vehicle design is believed to result in essential weight savings. However, the processing of these steels currently involves complex thermo-mechanical processing steps followed by controlled annealing and cooling cycles that requires considerable time and energy [6]. In the next section, a brief comparison is made between the mechanical properties, the governing microstructure and the thermo mechanical processing cycles for the four different grades of steel mentioned above.

2.1.1  *DP Steels*

**Microstructure and Mechanical Properties**

The term “Dual Phase” refers to a microstructure consisting of ferrite and martensite. However, dual phase steels often contain small volume fractions of other microstructures consisting of bainite, retained austenite and even pearlite due to non-ideal thermo-mechanical processing cycles. The main reason for the development of this steel was to achieve an improved formability when compared to commercial high strength low alloy (HSLA) grade of steel, while maintaining similar strength values [7]. Commercially the volume fraction of martensite contained in dual phase steel is in the range of 10-25%. Further increase in the amount of martensite causes a reduction in toughness (88 J at 0.2 volume fraction to 40 J at ~0.5 volume
fraction) and general formability of the steel [8]. The cause for this is believed to be due to formation of coarse martensite microstructures.

When considering the strength of dual phase steel, the amount of carbon in martensite is expected to play a governing role as martensite with higher carbon is expected to have higher strength. This means martensite formed by quenching from higher temperature is expected to have lower strength (lower carbon) than that formed by quenching from lower temperature (higher carbon). Furthermore, the volume of martensite formed also depends on the amount of carbon and the quenching temperature. For example, consider a steel with ‘C’ wt% of carbon as shown in a section of the binary Fe- C phase diagram (Fig. 2.1a). If this steel is heated to two different temperatures in the intercritical region (T_1 and T_2), the carbon present in the austenite at higher temperature (T_2) is C_2 (obtained by drawing a tie-line to austenite phase). Similarly, the carbon present in austenite for the lower temperature (T_1) is C_1. Clearly from the figure, C_2 < C_1. Hence the martensite formed by quenching the higher temperature T_2 would have lower strength (lower carbon) than that formed by quenching from T_1.

However, in dual phase steels, the strength of the steel was found to be dependent only on the volume fraction of martensite and not on the carbon content within the martensite [9].
Fig. 2.1a: Schematic of a binary Fe-C diagram showing an alloy with ‘C’ wt% of carbon heated to two different temperatures $T_1$ and $T_2$ leading to a carbon concentration of $C_1$ and $C_2$ in their respective austenite phases.

Fig. 2.1b: 0.2% flow stress and tensile strength as a function of percent martensite for the Fe-Mn-C alloys. (Reference: [9])
Fig.2.1b shows the strength of a Fe-Mn-C alloy that was quenched from different temperatures, as a function of the volume fraction of martensite plotted for a constant flow stress of 0.2\% [9]. The above graph indicates that the strength of dual phase steel increases linearly with the volume fraction martensite irrespective of the quenching temperature (or carbon in martensite). For example, steel quenched from 740\(^{0}\)C has a carbon content of 0.57 wt% in martensite and that quenched from 800\(^{0}\)C has 0.38 wt% C in martensite. Hence the former steel is expected to have higher strength than the later. However, this rule is not followed and the strength is found to vary linearly solely with respect to the volume fraction of martensite. When the graph is extrapolated to 100% martensite, the strength from the above trend is lower than the observed strength. The cause for the observed phenomenon is not very clear but it is believed that the strength of martensite formed when surrounded by a ferrite matrix is lower than that formed from 100% austenite matrix [9]. This is believed to be because the surrounding ferrite phase would be softer than the corresponding austenite phase due to lower amount of carbon present in it.

Hence, depending on the volume fraction of martensite, DP steels with different levels of strength can be produced. These steels typically have low yield strength, high tensile strength and good uniform elongation. Steels containing 10-20% martensite show ultimate tensile strengths close to 600
MPa with a yield strength ranging from 300 to 400 MPa and ductility of 25-30% [4,5].

**Processing of DP steels**

There are three processing routes for obtaining DP steels. 1.) Continuous annealing, 2.) As rolled and 3.) Batch annealing. Among these, the batch annealing processing is involves long cooling cycles (days) and is typically not employed. Continuous annealing and as rolled processing methods would be discussed below.

The continuous annealing process can be divided into three processing steps. The initial condition of the steel subjected to the processing cycle is typically a ferrite + pearlite rolled microstructure with some carbides. In the first step, this steel is rapidly heated to a temperature above the $Ac_1$ temperature. On reaching above the $Ac_1$ temperature nucleation of austenite starts along the ferrite grain boundaries and the carbides present in the steel begin to dissolve. The second stage of the processing is considered critical for in order to generate an ideal ferrite and austenite mixture. In this stage the pearlite and carbides microstructures should be completely dissolved. This stage is called *intercritical annealing*. Speich et al [10] explained the formation of austenite during this stage and considered it to be a three-step mechanism. In the beginning of the annealing stage, the dissolution of pearlite and growth of austenite into pearlite is the predominant process. The growth of austenite is governed by the diffusion of carbon in the austenite
with the diffusion path lying along the austenite-pearlite interface, where the
diffusion distance is governed by the interlamellar spacing (~0.2 \( \mu \text{m} \)) [10]. As
the diffusion distances for carbon are very short, depending on the annealing
temperature, the growth of austenite in this stage can be very rapid. However, for lower annealing temperature, substitutional alloying element
diffusion becomes the rate determining step. It has been reported that
heating from an annealing temperature of \(730^\circ\text{C}\) to \(780^\circ\text{C}\), the
transformation shifts from a substitutional element controlled to an
interstitial diffusion controlled growth.

After the dissolution of pearlite, the growth of austenite in ferrite
becomes the governing step. At this stage, carbon partitions between
austenite and ferrite to attain equilibrium carbon concentration given by the
lever rule in the intercritical region. In most cases, the substitutional alloys
do not partition and the rate is governed by carbon diffusion alone. This mode
of transformation is called \textit{paraequilibrium mode} (discussed later). After this
step, the later part of the annealing stage is governed by the diffusion of
substitutional elements (usually manganese). In the final processing stage,
the steel is cooled form the annealing temperature to the room temperature.
The cooling rate involved depends on the alloys present in the steel. For
steels with low hardenability, the cooling rate used is higher than that used
for steels microalloyed steels. Usually slower cooling rates are preferred as
higher strength and ductility can be achieved by slow cooling.
In the as-rolled processing, the steel is chosen to attain 80-90% ferrite in the final rolling pass. The formation of martensite from the remaining 10-20% austenite occurs during the final slow cooling process. However, alloy composition of the steel chosen should have an elongated ferrite C curve i.e. the ability to form ferrite over a reasonable wide range of cooling rates. Such a condition would allow the formation of ferrite in a short annealing time as well as suppress the pearlite formation during the cooling operation.

![Fig.2.1c: Schematic of the cooling schedule in the thermo-mechanical processing of DP steels (Reference: [6])](image)

Fig.2.1c shows the schematic of the cooling schedule used for producing DP steel microstructures in the as-rolled processing. A rolled, completely austenitic steel is held at the intercritical temperature to obtain a 80-90% ferritic microstructure and then cooled to the $M_S$ temperature to transform the austenite to martensite.
3.1.2 TRIP Steels

Microstructure and Mechanical Properties

Transformation induced plasticity or TRIP refers to a phenomenon observed in the similarly named grade of steel, in which a significant increase in plasticity is observed due to phase transformation. In these steels, application of an external stress is the driving force for the phase change $\gamma \rightarrow \alpha$. Typically, these grades of steels have a microstructure consisting of inter-critical ferrite matrix with a dispersion of bainite and retained austenite in the final processed condition. The underlying cause for the high plasticity is due to the formation of mechanically induced martensite from the metastable retained austenite. Studies have shown that this effect causes an improvement in the strength and ductility by maintaining a high work hardening rate during straining [12]. This increase in mechanical properties is believed to be due to two mechanisms; 1.) Stress assisted nucleation of favorably oriented variants of martensite with respect to the applied external load, called the orientation or Magee effect [13] and 2.) Plastic strain generated in austenite due to the volume and shape change caused by martensite formation which is called the accommodation or Greenwood-Johnson effect [13]. For these steels, the governing condition for the observed transformation is that the martensite start ($M_s$) temperature lies just below the room temperature. This allows the formation of retained austenite required for the stress-induced transformation. As the martensite
transformation is a diffusionless shear transformation, the application of external stress would assist in the transformation. On cooling, martensite phase transformation would occur at $M_s$ without any external applied stresses. The critical stress required to initiate martensite transformation above $M_s$ increases linearly till the $M''_s$ temperature. $M''_s$ is the highest temperature where the martensite transformation occurs by elastic stress and the transformation is termed as ‘stress assisted transformation’. Above the $M''_s$ temperature, plastic flow steel occurs before the transformation to martensite. In this temperature regime, the transformation is defined as ‘plastic strain induced transformation’ [14]. Fig.2.1d shows the temperature dependence of the critical stress required to cause the TRIP effect schematically, where $M_d$ represents the temperature above which there is no martensite formation (also known as the $T_o$ temperature). Using phenomenological models [15], the TRIP effect in the steels can be evaluated by taking in consideration both the orientation effect and accommodation effect.

The strength achieved by this steel is < 1000 MPa and typical lies in the range of 600- 800 MPa. It shows high elongation and high work hardening ratio with good formability [4,5]. TRIP steels have high energy absorption potential and can be used for dynamic loading applications.
Alloying in TRIP Steels

In recent times, low alloy C-Mn-Si steels are used to achieve the multi-phase TRIP steel microstructure. The total alloying element content is around 3.5 wt%. The choice of alloying elements is governed by multiple criteria which include attaining the appropriate $M_s^0$ temperature typically 15–25°C lower than the room temperature, minimizing the cost of alloying and the time spent on the continuous ageing lines in steel mills. Such an $M_s^0$ temperature range ensures that the strain induced transformation influences upon the deformation behavior just above room temperature. Based on the

Fig.2.1d: Schematic representation of critical stress required for martensitic transformation as function of temperature (Reference: [14])
concept by Matsumura, the original range of alloying elements contained in TRIP steels is 0.12-0.55 wt% C, 0.2-2.5 wt% Mn and 0.4-1.8 wt% Si [16].

In this steels, the role of Mn is twofold. Mn is an austenite stabilizer and hence is required to achieve good hardenability and it lowers the cementite precipitation temperature. A higher Mn content (> 2.5 wt%) causes excessive austenite stabilization, which is undesirable. Si, on the other hand, retards the formation of cementite. For example, a Fe-1.2C-1.5Mn steel, with 1.5 wt% of Si, forms no cementite as opposed to the same steel with 1 wt% of silicon, which forms 0.1 volume fraction of cementite, at 400°C [17]. Si is mainly added as it increases the carbon activity in ferrite and austenite and it reduces the solubility of carbon in ferrite. From the industrial point of view, the addition of Si reduces the kinetics for the formation of bainite. This implies that the C-Mn-Si type TRIP steel requires a long aging time. In addition, addition of Si causes the formation of a surface oxide film over the steel, which prevents it from being galvanized [18]. Despite these disadvantages, a small amount of Si is still preferred (0.3-0.8 wt%) as Si is an effective suppresser of cementite during the austempering stage. Si has a very low solubility in cementite. But the displacive (paraequilibrium) nature of carbide growth traps Si in the cementite lattice. This causes the free energy change due to precipitation to reduce and hence retards cementite formation.
C-Mn-Al alloy composition has been proposed to make TRIP steels in order to replace Si. Like Si, Al is also insoluble in cementite and causes a high C content in the retained austenite. Most importantly Al accelerates the formation of bainite [19]. The disadvantages of using Al are lowering of solid solution hardening and the increase in the $M_s$ temperature, typically above the room temperature. Currently, TRIP steels are made using C-Mn-Si-Al low alloy steels with small addition of Phosphorous. Replacing Si with Al makes the steel galvanizable and accelerates bainite formation. Small additions of P (0.05-0.1 wt%) increase the solid solution strengthening and suppress cementite formation and increases carbon in retained austenite [19].

**Processing of TRIP steels**

The processing cycle for TRIP steels can be divided into five stages [20]. The initial microstructure of the steel used for the TRIP steel processing consists of a cold rolled ferrite plus cementite microstructure. The initial microstructure of the steel is very important and governs the kinetics of the first stage. During the first stage, the rolled steel is rapidly heated above the $Ac_1$ temperature during which recrystallization of ferrite, dissolution of cementite takes place. In this heating stage, when the temperature reaches above $Ac_1$ austenite formation starts. The rate of heating is believed to be rapid and hence the dissolution of cementite in this stage depends on its morphology (blocky or pearlitic). In the second stage, the steel is held at a
temperature in between the $Ac1$ and $Ac3$ temperatures and the stage is called *intercritical annealing*. In this stage it is essential to dissolve the cementite completely in order to achieve good properties in the final steel. The essential phenomenon occurring in this stage is the formation of a mixed ferritic and austenitic microstructure. The early stage of intercritical annealing is governed by carbon diffusion between supersaturated austenite and ferrite. In the later stages substitutional alloys (Mn and Si) redistribute between the two phases. This stage of processing lasts for 2-4 mins.

In the third stage, the intercritically annealed steel is rapid cooled to the austempering temperature. This stage involves formation of new ferrite which grows on the existing ferrite and into the austenite. However, formation of new ferrite in this stage is not desirable. For this reason, the cooling rate employed in this stage is high (10 to 50°C/s). The fourth stage in the processing is called the *Austempering* stage. The ideal mixed microstructure of ferrite and austenite changes with the formation of bainite. This stage is considered to be a critical stage as it determines the volume fraction of austenite, the size of austenite grains and the carbon content in retained austenite. Formation of carbide free bainite causes the carbon to diffuse from bainite into the surrounding austenite. This carbon enrichment of austenite causes the $M_s$ temperature of the local austenite region to be suppressed below the room temperature. The austempering stage lasts for 4-8 mins. The final stage of processing is a relatively slow cooling stage (~6°C/s).
leading to the formation of an ideally austenitic and bainitic microstructure with intercritically annealed ferrite.

Fig.2.1e: Schematic of the cooling schedule in the thermo-mechanical processing of TRIP steels (Reference: [20])

Fig.2.1e shows a schematic of the process cycle for the formation of TRIP steels. The TRIP steels would require a lot more processing time and a better control over the cooling cycle than that require for DP steels.
2.1.3 CP and MART Steels

Microstructure and Mechanical Properties

Complex-phase or CP steels are characterized by a microstructure consisting of fine ferrite, bainite and martensite along with fine precipitates achieved by microalloying and some retained austenite. These fine precipitates are nanoscale carbides or carbonitrides obtained by the addition of Nb, Ti and V. These along with substitutional elements like Mn, Cr, Mo etc., determine the austenite decomposition kinetics to produce the multiphase microstructure. The final steel is characterized by the volume fraction of the formed phases, the grain size of ferrite and strengthening solutes and precipitates [21]. In comparison to DP steels, the presence of higher fraction of bainite (~0.25) in CP steels provides higher strength to it. Comparing DP and CP steels with same ultimate tensile strength, CP steels have a lower yield strength and show increased work hardening. Geiger et al compared the yield strength of a DP and a CP grade of steel having the same ultimate tensile strength [22]. They showed that the 0.2% proof stress of CP steel (624.47 MPa) was higher than that of the DP steel (513.7 MPa). It was also shown that the strain hardening exponent of CP (0.095) was smaller than of DP800 (0.135) indicating a higher strain hardening in the CP steel.

Mesplont et al [23] used different coiling temperatures to attain a different multiphase microstructures and compared their strength and work hardening behavior. They found that steels containing a mixture of bainite
with small fraction of martensite or martesite-austenite (MA) constituents provided a high true tensile strength value of 900 MPa. Based on their results, they predicted the optimum volume fraction of different microstructure constituents to achieve a good compromise between high strength and good elongation. According to them CP steels with about 63% ferrite, 25% bainite and 12% martensite/austenite constituents would give the best set of properties. Fig.2.1f shows the effect of the different microstructure content on the tensile strength of CP steels.

![Diagram showing the effects of constituent microstructure on tensile strength](image)

Fig.2.1f: Effects of constituent microstructure on the tensile strength (TS) of CP steels (Reference: [22])

MART steels, on the other hand, are produced by fast quenching of austenite to obtain lath martensite. These have a microstructure that is
completely martensitic. The mechanical properties of this AHSS steel is typically between (UTS) 900 and 1,500 MPa and typical elongations of ~5%. This grade of steel is considered to be the least formable and is typical applied for parts that act as anti-intrusion components of the car body.

**Processing of CP and MART steels**

In order to obtain multiple phases in the final processed steel, it is critical to have a precise control over the thermo-mechanical processing. The addition of alloying elements can help control the phase transformation kinetics. Hot rolling followed by controlled cooling is used make CP steels having a mixture of ferrite, banite and martensite. The initial microstructure of the steel for the hot rolling operation should be austenitic [21]. During the hot rolling operation in the austenitic regime, pancake shaped austenite grains are obtained. Such a microstructure is ideal to obtain fine grains of multi phase microstructure. Softening of austenite occurs during the hot rolling process and often recrystallization of $\gamma$ grains is expected alone with grain elongation. Addition of alloying elements like Nb, Mo, Ti and Mn retard the softening and recrystallization by solute drag effect and by forming precipitates [24]. This allows the elongated structure of austenite to be preserved. This creates a high grain boundary area per unit volume for the nucleation of ferrite. The ferrite nucleation rate is further enhanced by using rapid cooling. However, addition of alloying elements retards the growth of ferrite and changes the ferrite morphology. For example, depending on the
alloying addition and the cooling rate employed, the ferrite morphology changes from grain boundary ferrite to acicular or could lead to the formation of banite and martensite [21]. Hence the presence of alloying elements plays a significant role in the formation of CP steel microstructure.

MART steel grades can be made directly by quenching after annealing in the continuous annealing lines or by postforming heat treatment operations. Because of its limited elongation, martensite steels are usually roll-formed. More complex shapes of the components can however be fabricated by hot forming and quenching of lower carbon grades of steel. Fig.2.1g depicts the strength versus the elongation of various commercial high strength steels.

Fig.2.1g: Overview of published tensile strength and ductility that can be achieved for a wide range of advanced high strength steels  (Reference: [25])
To address the challenge of producing low-cost and advanced steel, steel researchers throughout the world have considered different approaches. For example, researchers from Colorado school of mines have developed steels that rely on the formation of martensite and partitioning of carbon to retained austenite to produce high-strength and high toughness microstructures [26]. Similarly, University of Cambridge researchers have developed high-strength high-toughness microstructures by low-temperature aus-tempering process by which a high-carbon austenite transforms to nano-scale thick bainitic microstructure [27] leading to strengths reaching over 2.5GPa (UTS). In addition, researchers from Japan have focused on achieving the fine ferrite grain structure by coupled deformation and phase transformation effects [28]. Other researchers have also focused on maximizing the packet and sub-unit size in low-carbon martensite to achieve advanced high strength steels [29]. In addition to the production of these steels, there exists a critical need to develop reliable and robust process for forming [30] and welding [31] these steels too.

Although, the above examples are not a comprehensive representation of the published literature on advanced high strength steels, it is sufficient to summarize that there is a renewed interest to develop low-cost steel processing methods to obtain steels with high-strength and toughness while minimizing expensive alloying additions and process time.
One of such innovative processing techniques that produces high strength steels using minimum processing steps and processing time is the ‘Flash Process’. To understand the cause for the high strength of the final steel obtained from the Flash Process, it is critical to identify the underlying microstructure governing the strength. Furthermore, a clear knowledge of the microstructure evolution due to the imposed process conditions, is essential to explain the formation of the final microstructure. For this purpose, knowledge of the transformation behavior of the high temperature phase (austenite) is required.

2.2 Austenite to Ferrite Phase Transformation Characteristics

In this section, possible on-cooling phase transformations of the parent austenite phase are reviewed and the formation of various final microstructures from these transformations are explained.

Austenite phase transformations can be broadly classified into two categories based on the motion of the atoms of the parent material and the alloying elements, the shape change experienced by the transforming component. These modes of transformations are termed as reconstructive transformations and displacive transformations [32].

2.2.1 Reconstructive Transformations

In reconstructive transformations, a rebuilding of the crystallographic and chemical components of the parent phase takes place. Such a
transformation involves a relative motion of solute atoms relative to its
surrounding neighbors by diffusion. In this mode, the product phase retains
the shape of the parent phase with a possible change in the volume. This
transformation is characterized by redistribution of solutes between the
parent and the product phase. (See Fig.2.2a)

2.2.2 Displacive Transformations

![Diagram of Displacive Transformation]

Fig.2.2a: Schematic illustration of the transformation mechanism in reconstructive
and displacive transformations. (Reference: [33])

Displacive transformation occurs by a cooperative motion of a large
number of atoms simultaneously, during the transformation of the parent
phase into the product phase. During such a transformation, the motion of an atom with respect to its neighboring atoms is less than an inter-atomic distance i.e. the relative relationship with the neighbors is maintained. Hence, no diffusion of either solute of solvent atoms is involved in this mode. An invariant plane strain with a large shear component is involved causing the shape of the parent phase to change during the transformation.

Fig. 2.2a shows a schematic illustration of the mechanism of the reconstructive and displacive transformations using solute and solvent atoms, which are represented by different shapes and numbers. In the reconstructive transformation, the numbered atoms are distributed randomly representing a transformation governed by diffusion. In the displacive transformation however, the numbered atoms maintain their relative positions and a shape change in the parent phase is attained.

The free energy curves for the austenite and ferrite phases are schematically depicted for a Fe-C binary system in Fig. 2.2b, at a particular temperature T. The points of intersection of the curves to their common tangent, correspond to the points on $Ae_1$ and $Ae_3$ lines of the equilibrium phase diagram as shown. The point of intersection of the two free energy curves (pt. c) is represented by a point $c'$ on the phase diagram. The locus of $c'$ plotted from the free energies at different temperatures, is a line that lies between the $Ae_1$ and the $Ae_3$ lines as shown and is represented by $T_0$. At a fixed temperature, for systems containing carbon lower than the $T_0$
concentration at that temperature, a diffusionless transformation of austenite to ferrite is possible. Moreover, for a system with a higher carbon concentration than the $T_0$ concentration line, displacive transformation is thermodynamically not feasible.

Fig.2.2b: Illustration of $T_0$ concentration in a binary Fe-C system from the free energy curves of austenite ($\gamma$) and ferrite ($\alpha$) phases at a temperature $T$. (Adopted from: [44])

The final structure of the transformation product accompanying the any of the above two modes of transformation could however differ. Based on the composition of the steel and the cooling rate of the austenite present at high temperatures, different types of ferrite morphologies can be obtained.
The different microstructures of ferrite that can be formed from the austenite are as a variation of the above two transformation modes are classified as:

- Allotriomorphic ferrite
- Idiomorphic ferrite
- Widmanstätten ferrite
- Acicular ferrite
- Bainitic ferrite
- Martensite

2.2.3 *Allotriomorphic ferrite*

Allotriomorphic ferrite is characterized by its formation along the grain boundary between two adjacent austenite grains (\(\gamma/\gamma\) grain boundary). For this reason, this type of ferrite growth is also called grain boundary ferrite or polygonal ferrite. The \(\gamma/\gamma\) grain boundary acts as good heterogeneous nucleation site for the initiation of the transformation. Hence the growth of ferrite occurs preferentially along the grain boundary as compared to the direction that is perpendicular to the grain boundary. The formation of this microstructure is purely by reconstructive mechanism.

2.2.4 *Idiomorphic ferrite*

Idiomorphic ferrite differs from Allotriomorphic ferrite in that it forms primarily inside an austenite grain and not along the \(\gamma/\gamma\) grain boundary. Non- metallic inclusions or precipitates present inside an austenite grain act
as sites for heterogeneous nucleation leading to the formation of ferrite within the grain. Crystallographically idiomorphic ferrite appears facetted unlike allotriomorphic ferrite, which follows the contour of the austenite grain boundary. Fig.2.2c shows a schematic of the growth morphology of allotriomorphic and idiomorphic ferrite.

![Figure 2.2c: Schematic of the growth of allotriomorphic and idiomorphic ferrite](image)

Both allotriomorphic and idiomorphic ferrite have a reconstructive mode of transformation and hence involves the partitioning of alloying elements between ferrite and austenite. Extensive studies on the growth of allotriomorphic ferrite have been carried out in recent time [34], [35], [36]. Bhadeshia, in a review [37] on the diffusional growth of ferrite explained the mechanism and the governing factors for the formation of allotriomorphic ferrite. In the Fe-C steel system, the growth of allotriomorphic ferrite is
governed by the partitioning of carbon in the austenite ahead of the interface. However, in the presence of substitutional alloying elements, the diffusional controlled growth is controlled by the diffusion of both the interstitial carbon and the substitutional element. This situation is complicated, as the local equilibrium at the interface has to be maintained [37] by the diffusion of alloying elements with a substantial difference in their diffusion coefficients. Under local equilibrium at the interface, the composition of the alloying elements in the two phases is determined by tie lines drawn to the phases in the equilibrium phase diagram for the system. In order to achieve a unique interface velocity by matching the diffusional fluxes of the two diffusing elements, the only route possible is to vary the concentration gradient of the two alloys ahead of the interface (this is assuming that the substitutional element also partitions into austenite). Fig.2.2d shows the schematic of a concentration profile developed ahead of the moving $\alpha/\gamma$ interface. The concentration of C and the alloying element X on either side of the interface is determined by the tie-line in the Fe-C-X ternary phase diagram at that temperature. Using the condition of conservation of mass at the interface moving with a velocity “$v$”, for both the diffusing elements, the equations obtained are:

\[
(x_c^\alpha - x_c^\gamma)v = D_c \nabla x_c
\]  
(1)

\[
(x_X^\alpha - x_X^\gamma)v = D_X \nabla x_X
\]  
(2)
where, $x_c$ and $x_x$ represent the concentrations of C and X, $D_c$ and $D_x$ are the diffusion coefficients of C and alloying element X at that temperature, and

$\nabla x_c$ and $\nabla x_x$ are the gradients in the concentration of C and X in the austenite respectively. The term $x^{\alpha}$ represents the concentration of an alloying element in $\gamma$ when in equilibrium with the $\alpha$ phase and similarly, $x^{\gamma}$ is the concentration of an alloying element in $\alpha$ when in equilibrium with the $\gamma$ phase.

Fig.2.2d: Schematic of the concentration profile ahead of a moving $\alpha/\gamma$ boundary with velocity ‘$v$’

For the common alloying elements in steel, $D_c >> D_x$ and hence the above two equations cannot usually be satisfied simultaneously. However, under two specific cases, such a growth is achievable:
1.) By selecting a tie line which satisfies the condition \( x_C^{\nu} = \bar{x}_C \), where \( \bar{x}_C \) is the bulk concentration of carbon in the transforming austenite. This condition would reduce the carbon diffusion flux to be consistent with the diffusion of X. Fig.2.2e illustrates the concentration profile generated for such a growth condition. This type of ferrite growth is called “Partition Local Equilibrium” (PLE) mechanism [37] with long range diffusion of alloying element X in austenite.

![Low Supersaturation Condition PLE](image)

Fig.2.2e: Schematic illustration of the ‘partition local equilibrium’ mode of growth (Reference: [37])

2.) Alternatively, increasing the gradient of X ahead of the growth interface would lead to a larger diffusion flux of the element X. Such a condition can be attained by a tie-line satisfying \( x_X^{\nu} = \bar{x}_X \), with \( \bar{x}_X \) being the bulk concentration
of X in austenite. Growth of this mechanism is called “Negligible Partitioning Local Equilibrium” (NPLE) [37]. Fig.2.2f shows a schematic of the concentration profile generated from such a type of growth.

![High Supersaturation Condition (NPLE)](image)

Fig.2.2f: Schematic illustration of the ‘negligible partition local equilibrium’ mode of growth (Reference [37])

However, as the alloying addition in the system is increased, simultaneously maintaining the diffusion of all the alloying elements at the interface to achieve an equilibrium growth becomes increasing difficult. In most of the practical situations, redistribution of all the elements is not possible. The diffusion of some of the elements could be very slow due to large differences in the diffusion coefficients of the alloying additions. In such a
situation, the growth of ferrite is by a constrained equilibrium where carbon maintains equilibrium at the interface by diffusion but the ratio of iron to other substitutional elements at both side of the interface is the same. Such a growth mechanism is called a “Paraequilibrium” growth mechanism. In situations where high cooling rates are involved, the formation of ferrite can be assumed to follow paraequilibrium mechanism where the growth is controlled by the diffusion of carbon.

Babu et al, studied the crystallography of allotriomorphic ferrite and compared it to that of the two adjacent austenite grains (γ₁, γ₂) [38]. It was found that during nucleation, allotriophic ferrite would have a preferred orientation with respect to one of the two austenite grains (γ₁). The orientation relationship usually followed is given by Kurdjumov-Sachs (KS) where the close packed planes of austenite and ferrite are parallel and the close packed directions of austenite and ferrite are parallel to each other i.e.

\[
\{111\}_γ \parallel \{110\}_α \text{ and } \langle 1\overline{1}0 \rangle_γ \parallel \langle 1\overline{1}1 \rangle_α
\]

Sometimes, a slight deviation from the close packed direction is observed, which is given by Nishiyama-Wasserman (NW) orientation relationship:

\[
\{111\}_γ \parallel \{110\}_α \text{ and } \langle 1\overline{1}2 \rangle_γ \parallel \langle 1\overline{1}0 \rangle_α
\]

The presence of such orientation relationships causes the formation of semicoherent phase boundary between austenite and ferrite, which causes
reduction in the surface energy for the nucleation of ferrite. It was also shown that the ferrite growth in the adjacent austenite grain ($\gamma_2$) did not follow any fixed orientation relation and the growth is random. The interface at this random orientation growth has a disordered boundary and the growth into this austenite is rapid [38]. Using in situ transmission electron microscopy, this difference in growth rate was shown by Purdy [39]. The semicoherent boundary present in $\gamma_1$ plays an important role in the formation of secondary Widmanstätten ferrite.

2.2.5 **Widmanstätten ferrite**

Widmanstätten ferrite is a ferrite phase formation that has a plate or lath morphology. Based on the site of its nucleation Widmanstätten can be divided into two types. Primary Widmanstätten ferrite is characterized by the formation of thin wedge shaped plates, which nucleate from the austenite grain boundary. In contrast, the secondary Widmanstätten ferrite nucleates at the semicoherent interface boundary between austenite and ferrite [40]. Fig.2.2g shows a schematic of the growth of secondary Widmanstätten ferrite that shows a sawtooth morphology. Widmanstätten ferrite is believed to grow by a displacive type growth mechanism due to the invariant plane strain shape change observed [41].
Fig. 2.2g: A schematic of sawtooth like secondary Widmanstätten ferrite forming over grain boundary or allotriomorphic ferrite.

It typically forms at temperatures below that of allotriomorphic ferrite formation, hence the growth follows a paraequilibrium mode of transformation and growth rate is controlled by the diffusion of carbon in austenite. The thickening of the Widmanstätten ferrite plates is suppressed by the diffusion fields of adjacent plates. This phenomenon is referred as ‘soft impingement’.

2.2.6 Bainitic Ferrite

Bainite usually forms as a cluster of thin platelets, which are termed as bainitic subunits or sheaves. For the formation of bainite, two different theories on the modes of growth are proposed. One of the theories proposes a reconstructive mode of growth where diffusion of carbon is the governing factor for the transformation [42]. In this mechanism, a competitive growth of
cementite and ferrite occurs during eutectoid decomposition. The second theory on the growth mechanism of bainite advocates a displacive mode of transformation where in no diffusion of alloying elements takes place during its formation. According to this theory, a sub-unit of bainite forms with a supersaturation of carbon via a displacive mode of transformation involving an invariant shape change deformation. With the help of atom probe characterization techniques, it was shown that substitutional alloying elements do not partition during the formation of bainite [43]. This observation supports both the above theories as according to both the theories, distribution of substitutional elements is not expected. The nucleation for the formation of the first subunit takes place at the austenite grain boundary and subsequent subunits nucleate at the tip of the previous subunit [44]. The diffusion of carbon follows after the formation of such individual subunits i.e. after the transformation. The formation of bainite takes place below the $T_0$ temperature. This supports the theory of displacive transformation, as below this temperature is it thermodynamically possible to achieve a diffusionless transformation. The growth of bainite stops when the carbon concentration in the austenite surrounding the sheaves exceeds that given by the $T_0$ curve [44].

Two types of bainitic ferrite morphologies are observed in steels. Upper bainite refers to the bainite formation where carbides are formed in between the bainite subunits. Such growth morphology occurs when the time required
for the carbon to diffuse out of the bainite subunits is lower than the carbide formation time. Conversely, during the formation of lower bainite, the time required for the carbon to diffuse out of bainite higher than the time needed for the formation of carbides. Hence, in lower bainite, the carbides are formed within the bainitic subunits. The growth of bainite follows a fixed KS-NW orientation relationship with parent austenite [44]. Due to the carbon enrichment in the surrounding austenite, the austenite can transform into martensite or decompose into carbides during the cooling of the steel.

2.2.7 Acicular Ferrite

Acicular ferrite in steels appears typically like an interlocked growth of thin fine grains and is typically referred to as a ‘basket weave’ structure. This type of ferrite growth is typically observed in welds where such a microstructure is tailored in ordered to achieve high strength and toughness which is due to its interlocking nature. Yang and Bhadeshia [45] explained the thermodynamics for the formation of acicular ferrite steel welds. The presence of intragranular inclusions is a necessary prerequisite for the formation of such a microstructure. In the absence of such inclusions, the formation of bainitic microstructure is observed. Both bainitic ferrite and acicular ferrite show similar transformation characteristics and the two microstructures competitive to grow in the austenite. For example, an increase in the austenite grain boundary area has been shown to promote the formation of bainite over acicular ferrite [45]. Similar to bainite, the growth
of acicular ferrite does not involve partitioning of substitutional solutes [46] and both growth mechanisms seize when the surrounding austenite has a higher carbon composition than that represented by the $T_0$ curve. Acicular ferrite is also characterized by invariant plane strain as observed in bainite. The growth of acicular ferrite also follows KS-NW orientation relationships with respect to the parent austenite [47].

2.2.8 Martenite

The formation of martensite from austenite occurs by a displacive transformation. Martensite transformation is achieved by cooling the steel below the martensite start temperature ($M_s$). This transformation is defined as an athermal transformation for the extent of martensite formation depends on the undercooling experienced below $M_s$ and does not depend on the holding time below $M_s$ [48]. Complete transformation of austenite to martensite occurs when the steel is cooled below the martensite finish ($M_f$) temperature. Presence of austenite at room temperature is possible if the $M_f$ temperature lies below it. The formation of martensite is achieved by invariant plane strain displacement. The crystal structure exhibited by martensite in carbon steels is body center tetragonal (bct) ($a = b \neq c$) and differs from the structure of ferrite (bcc). This tetragonal deformation of bcc crystal is due to the presence of carbon along four edges of the bcc crystal elongating it into one direction. An increase in carbon addition in the steel increases the tetragonal distortion [40] and also the hardness of martensite.
The morphology exhibited by martensite could be lath, lenticular or plate like. The growth of martensite also follows KS-NW orientation relations.

During the growth of lath martensite, near parallel plates of martensite grow along the habit planes following the KS orientation relation, within a prior austenite grain. Impingement of such parallel laths from different habit planes leads to the formation of martensite ‘packets’. Within these packets, following the KS orientation, six different variants of martensite laths are formed. These variants grow from the same habit plane but would have different growth directions. Among these, the variants that have low misorientation angles between them are group together as ‘blocks’ [49]. Fig.2.2h shows the different sub-features of martensite formed inside an austenite grain when transforming into martensite, leading to the appearance of a lath structure.

![Fig.2.2h: A schematic of martensitic sub-microstructural features inside a prior austenite indicating the formation of martensite packets and blocks. (Source: 31)](image)

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The complex morphology of martensitic microstructures affects the deformation characteristics. As shown in the figure, the blocks formed within a packet are further made of parallel laths [50]. Due to the growth from a single habit plane, the blocks within a packet appear to be parallel. In addition, the packets and the blocks, within a prior austenite grain are separated by high angle boundaries whereas the sub-blocks and laths are delineated by low angle boundaries as indicated in the figure [50]. Due of the presence of high angle boundaries between both the packets and blocks, both are expected to contribute as dislocation barriers during the deformation of the lath martensite. The size of the prior austenite grain would also play an important role by influencing the size of the packets and blocks.

Fig. 2.2i: Schematic of a TTT diagram showing the microstructures formed in the reconstructive and displacive modes and the temperature of transition between these modes.
Fig. 2.2i shows the regions of formation the above microstructures on a TTT diagram. The formation of the above microstructures is strongly dependent on the alloy composition of the steel and the kind of thermal processing it is subjected to. In order to correlate the formation of any microstructure to the processing conditions of the steel, analysis of the thermal cycle of the process is essential. Such analysis is usually carried out to investigate the occurrence of thermal events that could be corresponded to transformations taking place within a material. In the following section a few techniques available for thermal profile analysis would be discussed.

### 2.3 Thermal Cycle Analysis Techniques

Fluctuations in a thermal profile of a sample that is in a well-defined system with constant heat extraction or heat input conditions, is representative of endothermic or exothermic events occurring within the sample. Thermal analysis techniques like differential thermal analysis (DTA) or differential scanning calorimetry (DSC) are capable of accurately recording such thermal events. This information can then be used to identify phase transformation occurring in a material or to estimate phase fraction of solid in a liquid to solid solidification etc. However, due to small sample sizes and need of expensive equipment, alternative techniques have been used for such purposes. Techniques like thermodynamic modeling can be used for systems with known thermodynamic data. For example, commercial softwares like ThermoCalc® can be used to calculate phase transformation characteristics.
for common alloy systems with known thermodynamic database. These techniques follow equilibrium phase diagram and lever rule principles to estimate phase fractions for given external conditions of temperature and pressure. The main drawback of this technique is that most calculations ignore the kinetics effects on the transformation and equilibrium conditions are assumed at each stage of calculation. In practical applications however, systems are far from equilibrium and often the kinetics aspect of a process cannot be ignored.

For the above reasons, thermal profile analysis is considered as a good alternate for understanding transformations occurring within a system. Two such thermal profile analysis techniques, with investigative purposes, are discussed below.

2.3.1 Single Sensor Differential Thermal Analysis

Single Sensor Differential Thermal Analysis (SSDTA) is an in-situ thermal measurement technique that does not require any reference sample unlike other commonly used techniques like DTA, DSC for analysis of a thermal profile. Developed by Alexandrov and Lippold [51], SSDTA is a software-based technique that is capable of detecting transformation temperatures from the measured thermal cycles by using well-described heat transfer conditions given by Newtonian, Gaussian or Rosenthal equations. The transformation temperatures are detected by looking for the start of the deviation of a measured heating (or cooling) curve from a generated reference
heating (or cooling) curve, due to endothermic (or exothermic) effects.

Fig. 2.3 shows a schematic illustration of the working of the SSDTA technique. The reference curve is defined for a given cooling condition. The point of deviation of the measured cooling curve is the transformation start temperature. By direct comparison with dilatometric techniques, the sensitivity of SSDTA in recording various transformations has been evaluated and documented [52].

![Diagram](image)

**Fig. 2.3:** A schematic showing the working of the SSDTA thermal analysis technique

With the help of a high-speed data acquisition system, the thermal cycles can be recorded easily, thereby enabling the technique to accurately
determine transformation temperatures. The drawback of this technique however is that the fitting of a reference curve does not follow any statistical fitting criteria like goodness of fit and the choice of selecting a region of the original curve to create a fit is mostly visual.

2.3.2 Cooling Curve Analysis (CCA)

Another objective of a thermal curve analysis is to estimate the solid phase fraction in a liquid to solid phase transformation. As the name suggests, CCA is a method limited to cooling curve analysis and uses the derivative of the cooling curve for comparison with a reference called the ‘zero’ or ‘base’ curve. This zero curve is defined as the derivative of a cooling curve, which has not thermal energy released or absorbed due to thermal events like phase transformation or any chemical reaction. The difference between this zero curve and the derivative of the cooling curve represents heat evolved due to phase transformation. Furthermore, the cumulative area between these two curves normalized over the entire area over the solidification range, represents the solid fraction of that alloy.

Based on the method to calculate the zero curve, CCA is divided into Newtonian and Fourier methods [53]. However due to difference in the heat capacities of the liquid and solid phases, the zero curve rarely fits the derivative of the cooling curve in the liquid region too [53]. To avoid this problem, John Gibbs and Patricio Mendez [54] developed an equation based Newtonian (EBN) method that does not consider a zero curve solid fraction.
This method is developed from heat transfer analysis and considers Newtonian cooling and uses differential equations for solid fraction calculations. For a solidification process, a simple heat balance equation is considered where, the heat released by the solid and liquid as the sample cools, along with the latent heat of fusion, is equated to the heat loss to the surroundings with a known heat transfer coefficient for the container.

\[ mf_s c_p s \, dT + mf_l c_p l \, dT - mLdf_s = -KA(T - T_0)\, dt \]  

(3)

where:

\begin{itemize}
  \item A = area of the sampling cup (m²), \( C_{ps} \) = heat capacity of solid (J/kg K)
  \item \( C_{pl} \) = heat capacity of liquid (J/kg K), \( f_s \) = mass fraction solid (kg/kg), \( f_l \) = mass fraction liquid (kg/kg), \( K \) = heat transfer coefficient (W/m² K), \( L \) = isothermal latent heat of solid to liquid transformation (J/kg), \( m \) = mass (kg) \( T \) = temperature (°C), \( T_0 \) = ambient temperature (°C) and \( t \) = time (s)
\end{itemize}

By the modification of the above equation [54], the mass fraction of product and parent phase can be estimated. The shortcoming of this method however is that all the reactions have to be a transformation involving only two phases. In this current form, this method cannot be used for multiple phase fractions though, with the elimination of the dependence on a zero curve, this method could be expanded for multiple phase system and work is being done in this regard [55].
In order to explain the cause for the observed strength in FP steels based on its microstructure, existing models on the strength of steels have to be understood. In the following section, published theoretical models for the strength of steel would be reviewed.

2.4 Models to predict the strength of steels

2.4.1 Models for the strength of martensitic/ bainitic steels

Most theoretical models that particularly deal with the strength of individual martensitic/ bainitic microstructures involve a combination of several strengthening mechanisms. The strength of the martensitic steel is given by

\[ \sigma_{\text{Total}} = \sigma_f + \sigma_{\text{Dis}} + \sigma_{\text{SS}} + \sigma_C + \sigma_{\text{Ppt}} + \sigma_{\text{Grain}} \]  

(4)

where;

\( \sigma_f \) (MPa) is the Peierls force required to move the dislocation is a perfect matrix.

\( \sigma_{\text{Dis}} \) (MPa) is the contribution to the strength due to the dislocation density.

\( \sigma_{\text{SS}} \) (MPa) considers the contribution of substitutional alloying elements to the total strength.

\( \sigma_C \) (MPa) is the carbon interstitial strengthening term

\( \sigma_{\text{Ppt}} \) (MPa) strength due to precipitates and

\( \sigma_{\text{Grain}} \) (MPa) considers the effect of the features of the grains on the strength
The strengthening from these mechanisms are evaluated based on data collected from earlier published test results and also from calculations involving microstructural and compositional factors. For example, Naylor [56] estimated the strength of Mn-Ni low carbon martensitic steels by estimating the individual strengthening factors given above. A similar method was proposed by Bhadeshia [57,58] to estimate the strength of martensitic/bainitic steels. For both the models, the strengthening factors are the same, however, the method of quantifying some of these factors and their contribution to the total strength differed. In the following section, the strengthening terms in these two methods would be introduced and compared. A superscript ‘N’ would be used to denote strengthening terms of Naylor [56] and ‘Y’ for the strengthening terms of Young and Bhadeshia [59].

*Peierls-Nabarro force* ($\sigma_f$): For the pure BCC iron matrix, the Peirels force is the force required to move a dislocation when iron is in the fully annealed condition. This force is dependent on the temperature at which the tests are carried out [60,61] and decreases as the temperature increases. An average value of 40 MPa is considered as the strengthening contribution of this term at room temperature, for the BCC iron lattice.

*Substitutional solid solution strengthening* ($\sigma_{ss}$): Substitutional alloying elements cause strengthening of the parent matrix due to solute size misfit. However, their contribution to the strength is generally much smaller when compared to interstitial or dislocation strengthening. For the contribution of
strength, in the analysis of the Cr-Mn-Ni low carbon steel [56], only the strengthening from Mn was considered in the among the solid solution alloying elements as strengthening from Ni, Cr and other alloy additions were found to be negligible [62]. The contribution of Manganese to the strength was found to vary from 10 to 30 MPa per wt% of Mn [62]. For low alloy steels, this strengthening is negligibly small.

**Interstitial solid solution strengthening ($\sigma_c$):** Carbon is the main interstitial solid solution strengthener in steels. Following the work by Honeycombe and Pickering [63], Naylor assumed a linear variation of martensitic strength with the carbon content in the steel given by 4500 MPa per wt% of carbon. However, Honeycombe and Pickering do not explicitly talk about the effect of interstitial carbon on the strength of martensite microstructure.

On the other hand, Young and Bhadeshia [59] consider the strengthening from carbon in two separate cases based on lower and higher carbon content. Fleischer, [64] in his view on the strength of martensite from interstitial carbon, predicts a linear relationship of strength with the square root of carbon concentration in wt. percent ($c^{1/2}$). This model was consistent with data from many experimental results [65, 66, 67]. An interstitial strength value, governed by:

$$\sigma_c^{\gamma} = 1722.5 \cdot C^{1/2} \text{ MPa} \tag{5}$$
was considered by Bhadeshia [59]. However, for higher carbon content (>0.4 wt%), the increase in stress appears to saturate and the above relation for interstitial strength is no longer followed [68]. Hence, for strength analysis of microstructure with higher carbon content, the relation found by Winchell and Cohen is followed [69, 70] where the interstitial strength was reported to vary linearly with cube root of carbon concentration in wt%. \((C^{1/3})\). For such a microstructure the strength was taken to vary as:

\[
\sigma_c^Y = 1171.3 \cdot C^{1/3} \text{ MPa}
\]  

(6)

**Dislocation Strengthening** \((\sigma_{\text{Dis}})\): The plastic accommodation due to the phase transformation in martensite and bainite, causes accumulation of dislocations. In the as quenched condition, the contribution of this term to strengthening is considered to be significant. Naylor calculated the dislocation strengthening by the method followed by Cox [71] where the dislocation density was approximated to the inverse of the square of the subcell diameter \((L_d)\)(i.e. \(\rho = (1/L_d^2)\)); where \(\rho\) is the dislocation density. The contribution of this term to the strength then was given to be

\[
\sigma_{\text{Dis}}^N = K_{\text{Dis}}^N \rho^{1/2} = \alpha_i \mu b \rho^{1/2} \text{ MPa}
\]  

(7)

where \(\alpha_i\) is a constant, \(\mu\) is the shear modulus and \(b\) is the Burger’s vector. The value of \(\alpha_i\) was taken to be \(5.2 \times 10^{-9}\) as reported by Cox and a Burger’s vector value of 2.48 Å was taken for the iron matrix. The shear modulus for iron is \(8.25 \times 10^{10} N/m^2\).
According to Takahashi and Bhadeshia [72], the phenomena of plastic accommodation and recovery are governed purely by the transformation temperature. For low alloy steels, they proposed a formulation to calculate the dislocation density of both bainite and martensite microstructures.

\[
\log(\rho_{\text{dis}}) = 9.284 + \frac{6880.73}{T} - \frac{1780360}{T^2}
\]  

(8)

where \( \rho_{\text{dis}} \) is the dislocation density with units in m\(^{-2}\) and \( T \) is the transformation temperature in Kelvin. This equation is based on data obtained from experimental results from 570 to 920K. The relation for the strength due to the dislocation density was obtained as from experiments [73].

\[
\sigma'_{\text{dis}} = 0.38 \mu b \rho_{\text{dis}}^{1/2} = 7.34 \times 10^{-6} \rho_{\text{dis}}^{1/2} \text{ MPa}
\]

(9)

where \( \mu \) and \( b \) are the same as in Naylor’s equation (6).

**Precipitation Strengthening (\( \sigma_{\text{ppt}} \))**: Both Naylor’s and Young and Bhadeshia’s models account for the strengthening due to precipitates, representing it in the Orowan form, \( P \cdot \Delta^{-1} \), where \( P \) is a constant and \( \Delta \) is the effective distance between the precipitates.

**Strength from the microstructure (\( \sigma_{\text{Grain}} \))**: It is well known that grain refinement would improve the strength and toughness of steels [74, 75, 76]. However, the definition of the effective grain is not very clear when dealing with martensitic or bainitic steels due to the complex structural features.
formed within a prior austenite grain. Lath martensite forms in low and medium carbon steels and typically has parallel arrangement of lath shaped crystals.

Many investigations have shown a Hall- Petch kind relationship to exist between the strength of the steel and the prior austenite grain size [77,78,79]. A few others however believe that the strength depends on packet size and not the prior austenite grain size alone [80,81,82,83], though it can be argued that the packet size would be dependent on the prior austenite size. Naylor [56], in his analysis on the effect of morphology on the yield strength in Fe-Mn and FE-Mn-C steels, considered both the block width and the length of the block to be of importance and considered the length of block to be equivalent to the packet size. He defined an average slip plane length parameter that included both the block width and length in Hall – Petch type strength dependent calculations. Furthermore, in their work on characterizing martensite microstructure, Wang and co-workers [84] reported a Hall – Petch type dependence of the strength to the block width when working with commercial Cr-Ni-Mo steels. Thus, the smallest unit that would govern the strength properties in martensitic steels is still under deliberation but both the block width and packet size are now considered essential factors controlling the strength of martensitic steels.

Naylor [56, 85], in his strength model, defined an equivalent slip plane length for a block as follows.
\[ M = \frac{\varepsilon_l}{\cos \theta} \quad \text{for } \theta \leq \arccos \left( \frac{\varepsilon_l}{d_p} \right) \quad \mu \text{m} \quad (10) \]

Where \( M \) is the equivalent slip plane length for a block, taking into account both the block width and block length, \( \varepsilon_l \) is the block width and \( d_p \) is the packet diameter, which represents the block length. Hence, an average slip plane length term was defined as a function of block width and packet diameter. This is given as:

\[
\overline{M} = \frac{1}{\pi} \left[ \int_0^{\arccos(\varepsilon_l/d_p)} \frac{\varepsilon_l}{\cos \theta} d\theta + \int_{\arccos(\varepsilon_l/d_p)}^{\pi/2} d\theta + \int dp \right]
\]

\[
\overline{M} = \frac{2}{\pi} \{ \varepsilon_l \ln \left[ \tan \left( \frac{\arccos(\varepsilon_l/d_p)}{2} + \frac{\pi}{4} \right) + \frac{\pi}{2} d_p - d_p \arccos(\varepsilon_l/d_p) \} \}
\quad (11)
\]

where \( \overline{M} \) is the average slip plane length in \( \mu \text{m} \). Using the calculated value of \( \overline{M} \) from measured sizes of the microstructural features, Naylor estimated the strength from the martensitic microstructure via a Hall Pitch type strength equation given by:

\[
\sigma_{\text{Grain}}^N = k_{\text{Grain}} \cdot \overline{M}^{-1/2} \quad \text{MPa} \quad (12)
\]

where, \( \sigma_{\text{Grain}}^N \) is the Naylor’s term for strengthening by the microstructure and \( k_{\text{Grain}} \) is a constant. The value of \( k_{\text{Grain}} \) was found to be \( 3.63 \times 10^{-2} \text{N/m}^{3/2} \) from regression analysis.
However, Bhadeshia [57] approximated the average the slip plane diameter to be dependent on the thickness of the blocks ($t$) that are present within a packet and defined a term $L=\tau$, where $L$ is a measure of the mean intercept on the blocks. The strength from this factor is given by:

$$
\sigma_{\text{Grain}}^{\gamma} = K_{\text{Grain}}^{\gamma} \frac{1}{(L)^{-1}} = 115(L)^{-1} \text{ MPa}
$$

(13)

$\sigma_{\text{Grain}}^{\gamma}$ is the strength from the microstructure from Bhadeshia’s model and $L$ is in $\mu$m.

It is essential to note that the above two models, which were based on martensitic microstructure, are also applicable for bainitic microstructure. The difference in the application of the above two models, for the two microstructures, would be in the interstitial strengthening factor. This is because, the amount of carbon in bainite would be much lower (~0.03 wt% C) than in martensite, due to its partitioning into the surrounding austenitic. The difference would also arise in the dislocation strengthening term where the amount of dislocation in bainite would be lower than that in martensite due to the higher transformation temperature of bainite formation.

The above models can also be extended to evaluate the strength of systems containing a mixed microstructure of martensite and bainite. For this, the strength from individual microstructures can first be evaluated using the above models. Then, by using theoretical models for mixed microstructure steels, the strength of the steel can be evaluated. This strength would be a function of the strength and volume fraction of
constituent microstructures. These mixed microstructure models, for a system with bainite and martensite, are explained in the following section.

2.4.2 Models for the strength of mixed microstructure steels

Tomita and Okabayashi [86,87], worked on steels having martensite and bainite mixed microstructure. In their work, tensile tests of steels with varying volume fractions of constituent microstructures showed unexpected results. Fig.2.4a shows a schematic of the trend in strength observed by Tomita and Okabayashi as the volume fraction of bainite is varied in the steels.

![Graph showing the trend in strength of (martensite+bainite) mixed microstructure steels compared to the strength from the rule of mixtures.](image)

**Fig.2.4a:** Observed trend in the strength of (martensite+bainite) mixed microstructure steels compared to the strength from the rule of mixtures.
They observed that for small volume fractions of bainite, the strength of the steel attained was higher than that observed for 100% martensitic steels. This trend (Curve B) deviates from the expected trend (Curve A) strength given by the simple rule of mixtures (Eq. 14). In an attempt to explain their findings, Tomita and Okabayashi developed a model for the strength of bainitic and martensitic mixed microstructure steel [86,87,88,89]. This model was a modification of the common model used to explain the strength of a composite as given by Eq. (14). (where, \( M \): martensite and \( B \): Bainite)

\[
\sigma^\text{Mix}_{0.2} = \sigma^M_{0.2} \cdot (1 - V_B) + \sigma^B_{0.2} \cdot V_B \quad \text{MPa}
\]  

(14)

According to them, two factors are essentially the cause for the observed increase in the strength of the mixed microstructure a.) Refinement in martensite substructure due to the formation of bainite and b.) Strengthening of bainite by the plastic restraining effect created during martensite growth. In their view, the effective austenite grain size for the martensitic transformation is reduced due to the formation of bainitic sheaves, giving rise to refined martensite. This refinement hence increases the strength of martensite (\( \sigma^M_{0.2} \)) above the expected value via a ‘Hall Petch’ type-strengthening route. This is indeed consistent with the reduction of austenite grain size leading to reduction of martensite blocks and laths. Furthermore, following the research on strengthening observed in brazed alloys, they concluded that a similar phenomenon of plastic restraining to
also be a contributing factor for the strengthening. A modified empirical law of mixtures was proposed which included the above two factors.

\[ \sigma_{0.2}^{\text{Mix}} = (\sigma_i + k_{\text{Hall}} S^{-1/2}) \cdot (1 - V_B) + \sigma_{0.2}^B \cdot V_B \quad \text{MPa} \]  

(15)

Where \( \sigma_i \) is a combination of all the remaining strengthening factors, \( k \) is a constant and \( S \) is the packet size. The \((\sigma_i + kS^{-1/2})\) term is a Hall-Petch kind relation to account for the strengthening due to refined packet size in martensite. To explain the peak strength from restraining, it was assumed that \( \sigma_{0.2}^B \) reaches \( \sigma_{0.2}^M \) due to the high plastic restraining of bainite during plastic deformation caused by martensite formation, when the volume fraction of bainite is low \( (V_B < 0.3) \). Hence the modified equation of strength in the final developed form was:

\[ \sigma_{0.2}^{\text{Mix}} = \sigma_i + k_{\text{Hall}} S^{-1/2} \quad \text{MPa} \]  

(16)

Eq. (16) showed good agreement with observed results for \( V_B < 0.25 \) but deviated considerably for higher values of bainite. Young and Bhadeshia [59] however explained this strengthening in a slightly different manner.

They developed a quantitative method to accurately predict the trend and the strength of mixed bainitic and martensitic microstructure, as a function as a function of \( V_B \). Their method retained the theory of restraining effects experienced by bainite with the growth of martensite proposed by Tomita and Okabayashi. The increase in the strength of martensite is explained by carbon partitioning between bainite and martensite. First, the strength from
the individual, martensitic and bainitic microstructures ($\sigma_{0.2}^B$ and $\sigma_{0.2}^M$) are estimated using the models mentioned earlier [57, 58]. The combined effect on the strength due to the mixing these microstructures is then calculated by assuming a ‘rule of mixtures’ type (Eq. (14)) combined final strength as followed by Tomita and Okabayashi. However, Young and Bhadeshia explained the strengthening in the martensite formed to be mainly due to carbon partitioning and not by the refinement of the prior austenite grain as assumed by Tomita and Okabayashi. The formation of bainite within a prior austenite grain leads to an increase in the local carbon concentration in austenite regions adjacent to the formed bainite. The bainite formed was assumed to have a carbon solubility of 0.03 wt.% and the remaining carbon partitioned in the austenite. This carbon-enriched austenite then transforms into a stronger martensite with higher strength and causes a plastic constraining effect on the softer bainite.

The plastic restraining effect that Tomita and Okabayashi used to explain the additional strengthening was also considered by Young and Bhadeshia. Moreover, this strengthening was quantized under the lines of strength estimation from brazed layer thickness. It is known that the normalized stress in brazed joint varies exponentially with the normalized thickness of the brazed layer. Fig.2.4b depicts the observed exponential variation in the normalized strength of a braze joint as a function of the normalized thickness of the braze layer. Volume fraction of bainite was assumed be analogous to
the thickness in braze alloys as long as the volume fraction of bainite was low.

![Graph](image)

Fig.2.4b: Schematic of variation of normalized strength of brazed joint versus the normalized thickness of the brazing material. ($\sigma_0$: Strength of base material; $t_j$: total joint thickness; $V_b$: Volume fraction of bainite). (Reference: [59])

An exponential expression was derived, by fitting of curve to the data points of Tomita and Okabayashi, to estimate the strength of constraint bainite and it was given as:

$$
\sigma_{0.2}^{\beta} = \sigma_{0.2}^{\beta}(0.65 \exp\{-3.3V_b\} + 0.98) \leq \sigma_{0.2}^{M} \text{ MPa}
$$

(17)

where $\sigma_{0.2}^{\beta}$ and $\sigma_{0.2}^{\beta}$ represent strength of constraint and unconstraint bainite respectively and $\sigma_{0.2}^{M}$ is the strength of martensite. From the equation, for low
volume fractions of bainite, the strength of constraint bainite would reach the strength of martensite.
CHAPTER 3

OBJECTIVE

In order to develop good welding procedures to weld flash processed steels, the underlying microstructure of the steel has to be clearly understood. With the knowledge of the microstructure, the weldability of the steel can be improved. Hence, the aim of this study is to clearly understand the flash process technique, characterize the resulting microstructure from it and finally to explain the mechanism of the evolution of this microstructure during the rapid thermal treatment of flash process, from an initial microstructure. In addition, using microstructure characterization and available strengthening models, the final microstructure is to be co-related to the good mechanical properties observed in the flash processed steels. With this objective in mind, AISI 8620 grade of steels are taken as candidate steels for understanding the flash processed and subjected to the heat treatment for subsequent characterization and testing.
CHAPTER 4

FLASH PROCESS AND INITIAL TESTING

4.1 Flash Process

Fig. 4.1: A schematic illustration of experimental set-up in flash process technique

Fig. 4.1: A schematic illustration of experimental set-up in flash process technique
This work pertains to a novel heat treatment procedure that shows high potential as an efficient alternative for the production of AHSS steels [90]. This process has been termed as ‘Flash Processing’ for the incredibly short time (< 10 seconds) of heating and cooling of the steel sheets that has been reported.

A schematic of the flash process setup is shown in Fig.4.1. The assembly consists of a pair of rollers that transfers the steel sheets through a heating and cooling stage. At a critical distance from the top pair of rollers, the heating assembly is placed. The heating stage can be based either on oxy-propane flame or with electrical induction heating. For the current analysis, oxygen-propane based heating port was used. The heating consists of 17 flame nozzles spaced evenly to spread the flame uniformly over the steel. Immediately below the heating port, a cooling trough is situated. Any cooling medium can be used in this trough and for the present work, water is used as a cooling medium. The water in the trough is continuously agitated and is maintained at a constant temperature by a water chiller circulation system. To avoid any in-efficiency in heating due to the upward flow of steam, a graphite separator film is used to separate the heating and cooling stages. The cooling trough has a slit in the middle of its base to allow the steel to pass through. A rubber lining around the slit acts as a seal and prevents water from flowing down through the slit along with the steel. It is important to point out that these seals do not deteriorate due to any heating,
since the steel surface reaches the ambient water temperature as the steel exits the water trough.

The assembly is also fitted with three infrared pyrometers for spot temperature check along the width of the steel sheets. These infrared heat sensors are used as a control to drive the feed rollers and for positioning of the heating and cooling units. With these controls and sensors, the steel heating/cooling rate and the maximum temperature can be controlled and this provides flexibility to control the process cycles. For experiments, the steel sheets were fed through the heating and cooling system at a rate of 28 cm/min.

In this study, steel sheets of the grade AISI 8620, with an average width of 76.77 mm and an average thickness of 1.524mm, were taken as the candidate grade for testing and were subjected to the Flash Process. The composition of the AISI 8620 steel used is given in Table 1. For the remainder of this report, an acronym ‘FP’ would be used to denote this process and to refer to the steels that are processed using this method. In order to compare the flash processed samples with another reference sample with traditional heat treatment, the AISI 8620 un-processed steel samples were subjected to a quench and tempering treatment. These steels samples were austenitized at 1000°C for two minutes and quenched into a water to form martensite. These quenched samples were then immediately tempered at 220°C for four minutes.


<table>
<thead>
<tr>
<th>Element</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Ni</th>
<th>Co</th>
<th>Mo</th>
<th>S</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition</td>
<td>0.21</td>
<td>0.27</td>
<td>0.73</td>
<td>0.48</td>
<td>0.48</td>
<td>0.007</td>
<td>0.156</td>
<td>0.002</td>
<td>0.009</td>
</tr>
</tbody>
</table>

Table 1: Composition of AISI8620 steel used for the present study

4.2 Reconfirmation of Properties

In an earlier study, the strength of FP steels was evaluated [90]. It is reported that flash-processing of plain carbon-steels (> 0.15 C) lead to high yield strength (>1200 MPa), tensile strength (>1500 MPa) and ductility (> 7%) and that these processed steels could be classified under the category of AHSS. In a preliminary confirmatory study, the FP AISI8620 steel samples were tested for strength and compared with the reported strength of FP steels. Tensile tests were also carried on the quenched and tempered (QT) AISI8620 steel samples. In addition, to understand the governing cause for the said strengthening from the flash processing treatment, the microstructure of FP AISI8620 samples were observed under an optical microscope.

4.2.1 Sample Preparation and Testing

Tensile tests were carried out on the FP and QT samples to compare the strength and to extract the stain hardening exponents in the plastic zone. Three samples were extracted from the edge (2) and from the middle of the sheets. The tests were performed along the longitudinal direction. The gauge length of the test samples was set at 25 mm. The width of the samples at the
gauge length was 6.3 mm. The tests were performed at room temperature with a crosshead displacement rate of 1.27 mm/min following ASTM E8 mechanical testing specification for sub-size specimen. All the tensile testing were performed using testing machines available at Edison Welding Institute. Similar tests were repeated for the QT samples.

Optical microscopy was used to observe the resulting microstructure from the FP heat treatment. The sectioned FP samples were mechanically polished down to less than 1 µm surface finish. The polished samples were etched with 2% Nital etchant for 5 seconds. The underlying microstructure in both the FP and QT samples were recorded.

4.2.2 CCT and TTT Diagrams

Using standard predictive methodologies [91], the continuous cooling transformation (CCT) and time-temperature transformation (TTT) diagrams for the AISI 8620 steels were calculated. The above predictive techniques have been developed and calibrated by using a wide range of steels and their time-temperature transformation (TTT) data [92]. The conversion of these TTT data into CCT diagrams is carried out by assuming additive law [93], which has shown good predictive accuracy [94] and are considered to be fairly accurate for low allow steels including AISI 8620.

Fig.4.2 shows the calculated TTT and CCT diagrams for the initiation of paraequilibrium transformation of austenite to reconstructive
transformation and displacive transformation. The paraequilibrium mode of transformation is controlled by carbon partitioning during which the ratio of substitutional to iron atoms are configurationally frozen in place on either side of the austenite to ferrite transformation interface. Since FP involves rapid cooling process, the assumption of paraequilibrium transformation is considered valid.

Fig. 4.2: TTT and CCT diagrams showing reconstructive and displacive transformation regions for AISI 8620 steels
4.3 Preliminary Results

4.3.1 Optical Microstructure

The microstructure of the FP AISI8620 steel sheets is presented in Fig.4.3a. Optical microscopy from the FP samples shows very fine mottled and featureless lath microstructure, which appears to be martensitic upon cursory visual analysis. Such a structure was observed throughout the width of the sample indicating a uniform transformation product formation from the process. From the optically observed images, a co-relation is sought between the observed properties and the underlying microstructure.

Fig.4.3a: Optical Micrograph of AISI8620 FP sample showing very fine indistinguishable features
4.3.2 Tensile Test Results

The measured engineering tensile properties for both FP and QT samples (3 each) are tabulated below in Table 2. From the obtain results of tensile tests, the variation of the true stress ($\sigma_{Tr}$) of the samples with respect to the true strain ($\varepsilon_{Tr}$), were plotted as shown in Fig.4.3b In order to evaluate the formability of FP steels, the ratio of YS and UTS and true stress-true strain characteristics were calculated.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Ultimate Tensile Strength (MPa)</th>
<th>0.2% Yield Strength (YS) (MPa)</th>
<th>Reduction of Area (%)</th>
<th>Elongation (%)</th>
<th>Ratio of YS to UTS</th>
</tr>
</thead>
<tbody>
<tr>
<td>FP#1</td>
<td>1664.1</td>
<td>1442.8</td>
<td>39.4</td>
<td>8.8</td>
<td>0.867</td>
</tr>
<tr>
<td>FP#2</td>
<td>1619.3</td>
<td>1386.9</td>
<td>38.0</td>
<td>9.9</td>
<td>0.856</td>
</tr>
<tr>
<td>FP#3</td>
<td>1520.7</td>
<td>1300.0</td>
<td>38.3</td>
<td>9.9</td>
<td>0.855</td>
</tr>
<tr>
<td>QT#1</td>
<td>1607.6</td>
<td>1333.8</td>
<td>37.4</td>
<td>6.8</td>
<td>0.829</td>
</tr>
<tr>
<td>QT#2</td>
<td>1657.9</td>
<td>1464.1</td>
<td>49.8</td>
<td>10.0</td>
<td>0.883</td>
</tr>
<tr>
<td>QT#3</td>
<td>1642.8</td>
<td>1487.6</td>
<td>14.8</td>
<td>4.3</td>
<td>0.905</td>
</tr>
</tbody>
</table>

Table 2: Measured engineering tensile properties of FP and QT samples

The strain hardening exponent (n) and pre-exponent strength coefficient ($K_H$) for uniform plastic strain was estimated by fitting the Hollomon’s power law relationship for strain hardening (Eq. 18), to the plastic regime of the true stress-strain curve for both the FP and QT samples and the results of the fitting analysis are presented in table 3.

$$\sigma_{Tr} = K_H \cdot \varepsilon_{Tr}^n$$  \hspace{1cm} (18)
Table 3: Calculated pre-exponent ($K_H$) and strain hardening exponent ($n$) derived by fitting the equation (18) to the plastic regime of the true stress-strain curve.

<table>
<thead>
<tr>
<th></th>
<th>FP#1</th>
<th>FP#2</th>
<th>FP#3</th>
<th>QT#1</th>
<th>QT#2</th>
<th>QT#3</th>
</tr>
</thead>
<tbody>
<tr>
<td>$K_H$ (MPa)</td>
<td>569.6</td>
<td>622.1</td>
<td>613.7</td>
<td>1305.6</td>
<td>536.3</td>
<td>543.9</td>
</tr>
<tr>
<td>$n$</td>
<td>0.1542</td>
<td>0.1568</td>
<td>0.1348</td>
<td>0.1950</td>
<td>0.1342</td>
<td>0.1471</td>
</tr>
</tbody>
</table>

Fig.4.3b. Comparison of true stress versus true strain of QT (a) and FP (b) specimen
4.4 Discussions

The evaluation of optical micrographs indicates fine, martensitic type, lath microstructure to be present in FP samples. Due to the rapid heating and rapid cooling thermal cycles involved, this observation is supported by established theories of phase transformation and microstructure evolution in steels [40]. Based on the calculated CCT diagram (Fig.4.2), one can expect complete transformation of austenite to 100% martensite in AISI8620 steels at cooling rates faster than 70 K/s. As the FP is believed to be a very short heat treatment process (<10s), based on the CCT diagram a complete transformation of austenite to 100% martensite in FP AISI 8620 is expected.

The tensile tests reconfirmed that the flash processing of AISI 8620 steels did lead to yield and tensile- strengths greater than 1300 MPa and 1500 MPa, respectively. In addition, the elongation (8.8-9.9%) and reduction in area do show good ductility in these samples. The UTS of the FP tensile specimen are comparable to that of the QT samples however, the FP samples show a consistent and higher elongation values compared to the QT samples.

The calculated K and n values (see Table 3) are similar values to the published grades of AHSS steels for dual phase and complex phase steels [25]. The measured UTS and YS from the tensile tests of FP are overlaid in the map (Fig.3.1g) showing the range of currently available AHSS steels to get Fig.4.4. This comparison shows that the FP steels are indeed a part of AHSS family. Furthermore, both UTS and the elongation of FP steels are
higher than the reported values for AHSS based on a martensitic microstructure.

Fig.4.4: Overview of published tensile strength and ductility that can be achieved for a wide range of advanced high strength steels and measured tensile strength and ductility of flash processed steels

Based on the observed microstructure, the mechanical properties exhibited by the FP samples are unexpected. A rapid heating to austenite phase field and quenching would lead to the formation of martensite, which is expected to be brittle in the untempered condition. During tensile testing, these martensitic steels may not lead to any appreciable plastic deformation.
Hence, with the expected, completely martensitic microstructure, the elongation (8.8-9.9%) exhibited by the FP samples is greater than the levels (4-5%) usually reported for martensitic steels [25]. This ambiguity in the structure property co-relation could not be explained through the preliminary analysis carried out on the FP samples. Hence, a thorough analysis of the process is carried out which focuses on understanding the microstructure evolution during the process through thermal cycle analysis and in-depth microstructure characterization.
CHAPTER 5

THERMAL CYCLE ANALYSIS

5.1 Introduction

The flash process is known to have a very short treatment time. However, the heat treatment information of the process pertaining to the heating/cooling rates, peak temperatures attained, total time for the treatment etc., were not measured. Moreover, the spot pyrometers attached to the FP assembly are fixed and hence are limited to measuring the temperature of steel only at certain fixed regions during the process.

In order to understand the thermal cycles experienced by samples subjected to FP treatment and to quantify the heat treatment parameters, the detailed temporal variations of temperature were measured using contact thermocouples. Furthermore, in order to develop a mechanistic understanding of microstructure evolution during FP, it was important to record the various phase transformations taking place during the process. However, in-situ analysis of phase transformation is difficult as most phase transformation analysis techniques like DTA, DSC are used ex-situ. For this
reason, SSDTA analysis technique was used to identify the phase transformations taking place. The apparatus and the experimental setup required for this study are described below.

As a reference for above phase transformation analysis, the equilibrium phase transformation characteristics for the AISI 8620 steel sample are evaluated using standard computational modeling tools that utilize equilibrium thermodynamics databases for calculations. The transformation temperatures for various phases formed during equilibrium heating and equilibrium cooling of AISI 8620 steels are evaluated and compared with the values measured in FP.

5.2 Computational Modeling

In order to evaluate thermodynamics of phase transformation for AISI 8620 steel, ThermoCalc™ [95] software were used. The TCFE5 database was used to estimate the thermodynamic quantities and equilibrium transformation temperatures using the 8620-grade steel composition.

The equilibrium Ac1 and Ac3 temperatures of this steel were calculated to be 702°C and 806°C, respectively. In addition, the calculated equilibrium volume fractions of carbides that can form in these steels as a function of temperature are also shown in Fig.5.2.
Fig.5.2: Calculated variation of carbide volume fraction as function of temperature obtained from ThermoCalc14 and TCFE5 database

This result shows that as the steel heats above 806°C, it should reach 100% austenite. The bainitic (Bs) and martensitic (Ms) start temperatures were calculated to be 553°C and 426°C, respectively [41,91].

5.3 Thermal Analysis - Procedure

To record the thermal profile experienced during FP, thermocouples had to be attached to the AISI 8620 steel sheet. However, the oxy propane flame heating method does not lend itself for the direct measurement of temperature due to possible deterioration of thermocouples by the high intensity flame. Therefore, the flame heaters were substituted with the induction heating method for the temperature measurement experiments. In order to impose the similar heating conditions as of that of the flame heating, the feed-rates and the temperature gradients were controlled to be identical
using the pyrometers. Type K (Chromel–Alumel) thermocouples were attached to the middle of the steel sheets. The temperature of the steel was measured, as the sheet traversed through the heating and cooling ports. These thermal measurements were recorded using a high-speed data acquisition system capable of recording temperature at a sampling rate of up to 5 KHz. To check the repeatability of the process, multiple heat treatment runs were made and the temperatures from each run were recorded. The obtained temperature profile was analyzed to determine the instantaneous heating and cooling rates throughout the FP. In addition, ferrite ($\alpha$) to austenite ($\gamma$) transformation was evaluated by analyzing the thermal cycle data using the SSDTA technique.

5.4 Thermal Profile of FP

Using the thermal measurements made by the data acquisition system, the thermal profile of the FP is plotted. Fig.5.4a shows the thermal profile of FP from two different runs. From the plot, the repeatability of the process is very evident. In addition, the instantaneous heating and cooling rates at each thermal measurement point is evaluated. The temperature of a point 5 time steps ahead is used as the cooling rate required to evaluate the rate of change of temperature at a point. These calculated instantaneous rates of change of temperature are plotted as a function of time in Fig.5.4b coupled with the thermal profile in FP.
Fig. 5.4a: Thermal profile of two AISI 8620 samples showing repeatable thermal cycles of FP.

The thermal profile of FP can be divided into four regions based on the temperature range. In region I, the steel temperature increases gradually due to heat conduction from the hot stage. In region II, the increase in temperature is rapid due to its proximity to heat source. In this region, maximum heating rate of 410 K/s was observed at 780°C. This temperature is between the calculated A1 and A3 transformation temperatures for AISI8620 steel from equilibrium calculations. Above 780°C, the heating rate starts reducing, which indicates that there is a dynamic equilibrium between
the heat flux, the heat conduction, and endothermic/exothermic effects due to phase transformations [52]. In region III, the heating rate reaches zero when the sample reaches a peak temperature of 1100 °C.

Fig.5.4b: Heating/cooling rate and temperature variation versus time in typical FP
As per the equilibrium thermodynamic calculations, at this temperature, the sample should be 100% austenite. After reaching the peak temperature, the sample starts cooling down gradually. The total dwell time for this slow cooling near 1100°C is measured to be 2s. In region IV, within 1.2s of reaching peak temperature, the sample reaches the water bath and starts cooling rapidly. The maximum instantaneous cooling rate of 3150 K/s is achieved when the sample reaches a temperature of 393°C. This temperature is much below the calculated Bs (553°C) and Ms (426°C) temperatures. [41,91] As per the measured thermal cycles and the calculated CCT diagram (Fig.4.2), the austenite phase from high temperature should transform to 100% martensite during FP. Hence the thermal analysis also appears to support the initial hypothesis after the preliminary tests.

5.5 SSDTA Analysis

Using the SSDTA technique, the heating regime of the FP thermal cycle was analyzed. The sensitivity and accuracy of SSDTA technique, in measuring α to γ transformation during high heating rates (~500 K/s), has been documented earlier [52] by direct comparison with dilatometric techniques. By fitting reference curves over sections of the original heating curve, the points of deviation resulting from endothermic (or exothermic) effects during phase changes, are taken as the transformation start/end temperatures.
Fig.5.5a Single sensor differential thermal analysis of the heating regime of the first run showing two endothermic thermal events.

Fig.5.5b Single sensor differential thermal analysis of heating region of second run showing two endothermic thermal events similar to Fig.5.5a.
Fig.5.5a and Fig.5.5b show the results of the SSDTA analyses of the thermal profiles for the heating cycle of the two FP runs. In these analyses, the reference curves were denoted by polynomial functions that describe well-controlled heat transfer conditions. Both the results show a small endothermic phenomenon around the calculated [95] Curie temperature (~750°C) of bcc ferrite and another large endothermic peak, 900°C. These large endothermic peaks are interpreted as Ac1 (926 and 930°C) and Ac3 (1046 and 1052°C) temperatures by SSDTA software. From the figures it is evident that the results are reproducible.

The reproducibility of these measurements is within the 0.4% accuracy of the type K thermocouple. The accuracy of these analyses is also affected by the electromagnetic noise generated by the induction heating. It is important to point that the technique estimates a higher and non-equilibrium Ac1 and Ac3 temperatures, in comparison to the equilibrium A1 (702°C) and A3 (806°C) temperatures.

In the case of the flat plate steels, the analysis of the cooling regime of the thermal cycle would not be accurate. Due to the assembly of FP, a major part of the cooling occurs within the chilled water. This causes the thermocouple attached to the surface of the steel plate to come in direct contact with the water during the process, which would lead to erroneous phase transformation temperatures. However, while recording the thermal cycle, no bubble formation was observed due to the continuous agitation of
water. In another study, work focusing on the analysis of the cooling regime of FP was carried out (see Appendix 1). In this work, pipe samples were subjected to FP and the thermocouples were attached to the inner surface of the pipe, which prevented them from coming in contact with water. In this work, SSDTA applied to the cooling regime showed two transformations taking place as the steel cooled from austenite to the temperature of water. The higher temperature transformation was concluded to be the bainitic transformation and the lower temperature one pertained to the martensitic formation.

5.6 Summary

From the thermal cycle analysis, the total treatment time in FP was found to be less than 8 s. Peak temperatures of well above 1000 °C was achieved in the process which is higher than the calculated A3 temperature from equilibrium thermodynamics. The process was recorded to reach instantaneous heating rate of 400 K/s and instantaneous cooling rates >3000K/S with an average cooling rate between 800 °C to 500 °C (t8-5) of ~900 K/s.

From SSDTA performed over the heating regime, the non-equilibrium Ac1 and Ac3 were recorded to be between 925-930 °C and 1045-1052 °C respectively. These temperatures are higher than the A1 and A3 temperatures by ~200 °C indicating a delayed transformation with respect to temperature. The analysis also shows that the sample is completely
austenitized during FP.

Analysis of the cooling regime (Appendix A), in a similar work, showed two transformations to be taking place, which were connected to the bainite, and martensite transformations. Hence, the final microstructure from FP is expected to be a mixture of bainite and martensite. This discovery completely contradicts the expected 100% martensitic microstructure from preliminary tests. In order to confirm this finding, exhaustive microstructural characterization was carried out.
CHAPTER 6

MICROSTRUCTURE CHARACTERIZATION

6.1 Introduction

To completely understand the microstructure evolution during FP, an in-dept characterization of the final microstructure was essential. Initial cursory evaluation of the FP microstructure, using optical microscopy showed very fine, lath martensite type microstructure (Fig.4.3a). However, from SSDTA analysis, FP is expected to result in martensitic and bainitic mixed microstructure. Hence, advanced characterization techniques were used to quantify the microstructure of FP steel.

For developing the mechanistic model for phase transformation during the process, the microstructure of the un-processed AISI 8620 steel was also characterized. In the similar way, the microstructure of the reference QT sample was also analyzed.

Micro-hardness measurements were made over the cross section of the FP and QT AISI 8620 samples, to observe and compare the distribution of hardness and evaluate the spread of the microstructure in both cases.
6.2 Characterization Procedure

6.2.1 Instrumentation and Techniques

Standard characterization techniques were used to characterize unprocessed, QT and FP samples. All the samples were observed under an Olympus digital optical microscope and also using Quanta200 scanning electron microscopy (SEM) and Philips XL-30 Environment SEM, equipped with secondary electron, backscattered and X-ray energy dispersive spectroscopy (EDS) detectors. Phase identification testing and phase mapping was carried out using a TSL electron back scattering diffraction (EBSD) analysis system in the XL-30 ESEM. For imaging purposes, the SEM was operated at an accelerating voltage of 25 kV and a spot size of 5. For EBSD pattern generation, the SEM was operated at an accelerating voltage of 20 kV and a spot size of 5, which corresponds to a beam current of 2.41 nA and an aperture of 4.1 nm. For all EBSD patterns, a step size of 0.1 μm was used.

Transmission electron microscopy was used to observe the very fine features of the microstructure in FP samples. The samples were imaged using two types of transmission electron microscopes. General observation of microstructures and associated electron diffraction analyses was performed with a Phillips CM12 and Tecnai F20 transmission electron microscope operating at 120kV with a tungsten filament source. In addition, High angle annular dark field (HAADF) and Bright field scanning transmission electron
microscopy (STEM) techniques were performed using a Tecnai F-20 field emission gun (FEG) TEM at 200kV operating voltage. STEM dark field imaging using the high angle annular detector minimizes contrast due to diffraction and makes it easier to image the lath structure and the inter-lath boundaries that have very low misorientation. The annular detector records only high angle elastically scattered electrons. In this way the contribution of the Bragg reflections is minimized and incoherent thermal diffuse scattering becomes the prevailing contribution to the image intensity. Such incoherent STEM images are almost insensitive to defocus or thickness changes of the sample. EDS was also used to characterize the chemical composition of the precipitates.

Two-dimensional hardness distributions were measured over the cross section of FP and QT samples. For each sample more than 2000 indents were made on QT using an AMH43 automatic hardness testing system with the load of 300g. The measured hardness distributions were analyzed in a map format and also using frequency distribution curves for a qualitative and quantitative measure of mechanical heterogeneity of the samples. This was performed using a technical graphing and data analyzing software, IGOR Pro.

6.2.2 Sample Preparation

For all the above techniques, the samples need to be prepared to attain a certain surface finish. The sectioned samples were mechanically polished
down to less than 1 µm finish. The polished samples were etched in 2% Nital etchant for 5 seconds for optical microscopy and SEM images. The samples were not etched for EBSD analysis and hardness measurements. TEM samples were prepared by electrolytic thinning of 3mm discs of 90 µm, abrasively thinned samples. A chemical solution of 33% Nitric acid, 67% Methanol was used as the electrolyte. The polishing was carried out at 25V at a current of 10-15 mA and the temperature of the electrolyte was maintained at -25°C.

The results from the characterization of all the three samples are described in the following sections.

6.3 Initial Microstructure of the Annealed 8620 steel sheets

The initial microstructure of the 8620 steel sheets is presented in Fig.6.3a. Optical microscopy showed the presence of ferrite grains with carbides. Most of the carbides were smaller than 1 µm and some were larger than 2 µm, suggesting a bimodal nature. These carbides also appear to be aligned suggesting that these could have precipitates in between the pancake shaped ferrite structures during processing.

Since the details of all the treatment given to the initial steel were not known, further analyses were performed to identify the composition of these carbides. Scanning electron microscopy (see Fig.6.3b) with back-scattered imaging showed that most of these carbides were associated with ferrite grain boundaries.
Fig. 6.3a: Optical image microstructure of AISI8620 unprocessed sample showing extensive carbide distribution, especially along grain boundaries, in ferrite matrix.

Fig. 6.3b: SEM backscattered image showing presence of carbides along grain boundaries in microstructure of AISI8620 unprocessed sample.
Energy EDS analysis of the particles shows increase in peak intensities of chromium (Cr) when compared to the EDS of the bulk sample (Fig.6.3c) which indicates that the particles could be Cr rich cementite ($M_3C$) particles.

Fig.6.3c: Comparison of EDS signals obtained from matrix and carbide particle observed in AISI8620 unprocessed sample (signals from particle show higher Cr/Fe ratio indicating that particle could be chromium carbide)

6.4 Microstructure of QT AISI8620 steel sheets

The QT samples were characterized with optical microscopy and scanning electron microscopy. The optical microscopy of QT samples showed (Fig.6.4a) a featureless and mottled fine tempered martensitic lath
microstructure very similar to that seen in FP samples. The scanning electron microscopy (Fig.6.4b) with back-scattered electron imaging revealed the martensitic lath structure with some brightly imaging carbides. By tracing the lath packet boundary, typical prior austenite grain size was estimated to be around 20 to 30 μm.

Fig.6.4a: Optical image QT AISI8620 sample showing fine microstructure consisting of martensitic structures.

Due to the short duration in the austenitizing time (2 min at 1000°C), extensive austenite grain growth had not occurred. The volume fraction and size of these carbides in the QT samples were much smaller than the carbides that are observed in the untreated samples (Fig.6.3b). It is possible for the
microstructure to contain nano-meter sized carbides that might have formed during the tempering (220 °C for 4 minutes) stage. Usually the sizes of these carbides that form during early stages of tempering are below the resolution of scanning electron microscopy [96,97]. However, it is fair to conclude that initial carbides that are present in the un-treated samples have dissolved during austenitizing at 1000 °C for 2 minutes.

Fig.6.4b: SEM backscattered image of AISI8620 QT sample showing martensitic lath structures with prior austenite grain boundary

In order to measure the features formed with a prior austenite grain, EBSD technique with crystallographic orientation analyses was required [98]. Fig.6.4c shows an inverse pole figure image of the AISI 8620 QT sample.
The prior austenite grain boundaries are clearly highlighted following the method explained earlier and the average prior austenite grain size is found to be 7.98 μm.

Fig.6.4c: An inverse pole figure image of the AISI 8620 QT sample in which the lath structures are clearly visible along with the highlighted grain boundaries

6.5 Microstructure of FP AISI8620 steel sheets

As shown in Fig.4.3a, the optical microscopy image from the FP samples showed mottled and featureless lath microstructures, which is very similar to that observed in QT samples. However, scanning electron microscopy with electron backscattered mode of imaging revealed interesting
features. Fig.6.5a shows an SEM image taken from the cross section of the FP sample. Fine lath structures with uniformly distributed brightly imaging particles could be observed. Following the same procedure as for QT sample, by tracing the lath packet boundary, typical prior austenite grain could be delineated.

Fig.6.5a: SEM backscattered image of AISI8620 flash processed sample showing fine lath structures with brightly imaging particles and typical prior austenite grain boundary

A typical trace of the prior austenite grain is shown in Fig.6.5a. This qualitatively shows that the prior austenite grain size in FP samples are much finer than that of the QT samples. This could be attributed to the rapid
heating and cooling above the Ac3 temperature. In the next step, the composition of carbides was also evaluated with EDS analyses. The measured spectrum and calculated composition of the analyzed region are presented in Fig.6.5b. Surprisingly, similar to the untreated samples, these carbides were found to be rich in Chromium.

Fig.6.5b: Comparison of EDS signals obtained from matrix and carbide particle observed in AISI8620 FP sample (signals from particle show higher Cr/Fe ratio indicating that particle could be chromium carbide)

Since the FP process involved rapid heating (> 400°C/s) and cooling (> 3000°C/s) rates, these carbides might be the same that were present in the
original base material. Fig.6.5c shows an EBSD image of the FP 8620 sample surface with the prior austenite grain outlined. Three such EBSD images were obtained from FP samples, which should similar features. Fig.6.5d shows a cropped prior austenite grain from Fig.6.5c. The average grain size measured from the EBSD image analysis is 5.4 μm.

![Fig.6.5c: EBSD image of the FP AISI 8620 sample showing prior austenite grain and 6.5d a cropped prior austenite grain with the packet boundaries outlined](image)

Fig.6.5e shows a typical grayscale image outlining the blocks present in the FP samples. Such images were used to measure the block width for the
two samples and the distribution of the block width for both FP and QT samples were plotted (Fig.6.5f). For the FP sample, the average block width was found to be 0.623 μm with a standard deviation of 0.28 μm. For the QT samples however, the average block width was found to be 0.78 μm with a standard deviation of 0.25 μm.

![EBSD greyscale image showing blocks present in FP samples](image)

**Fig.6.5e: EBSD greyscale image showing blocks present in FP samples**

<table>
<thead>
<tr>
<th></th>
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<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>FP Sample</td>
<td>5.4</td>
<td>2.73</td>
<td>0.623</td>
</tr>
<tr>
<td>QT Sample</td>
<td>7.98</td>
<td>4.62</td>
<td>0.779</td>
</tr>
</tbody>
</table>

**Table 4: The difference in the microstructural features between QT and FP samples**
These values are shown in Table 4. It is interesting to note that the difference in the block widths of the FP and QT samples is not significant as compared to the differences in the prior austenite grain size (5.4µm and 7.98µm respectively) and the average packet size (2.73µm and 4.62µm respectively). This observation is consistent with that made by Maoqiu Wang et al [84] who noticed that the variation in the block width was not as prominent as the variation in the prior austenite grain size for samples austenitized at different temperatures. For example, in their experiments, samples austenitized at different temperatures ranging from 1133K to 1523K showed prior austenite grain size ranging from 337µm to 6µm. However, the block width for the samples ranged from 1-14µm.

Fig. 6.5f: Distribution of block width in FP and QT samples
Although through cursory observation of the optical and scanning electron microscopy, one may conclude that the FP samples are predominantly martensitic, to confirm the final microstructure in FP, detailed transmission electron microscopy was required. A low magnification TEM image [see Fig.6.5g] shows a prior austenite-austenite (\(\gamma/\gamma\)) grain boundary with presence of fine-scale martensitic laths with high dislocation density and coarser bainitic ferrite with reduced dislocation density. The identification of bainitic ferrite was made based on the size of these plates and the presence of sheave like structure with sub-units as shown by Bhadeshia [57].

![TEM image showing a \(\gamma/\gamma\) grain boundary with parallel laths growing out of it](image.png)

Fig.6.5g: Images (TEM) showing a \(\gamma/\gamma\) grain boundary with parallel laths growing out of it
In another region of the sample [see Fig.6.5h], three bainitic (marked as 1, 2, 3 in the images) sheaves were observed. A point to note is that each and every individual sheave is made up of ferrite sub-unit as the electron diffraction analyses failed to identify any retained austenite film in between these sub-units [99]. The analyses of relative orientation relationship between these bainitic sheaves were done and the summary of the results are presented in inset diffraction patterns in Fig.6.5h.

Fig.6.5h Three bainitic sheaves (1, 2, 3) in FP sample with a overlaid diffraction pattern from the same region. Sheaves 1 and 2 belong to the same prior austenite grain and sheave 3 grows in an adjacent prior austenite grain
The sheaves #1 and #2 are indexed to be in the [111] BCC zone axis with relative misorientation of only 10 degrees. Assuming that these bainitic sheaves have KS/NW orientation relationship [100] with the parent austenite (FCC) phase, one can conclude that the bainitic sheaves #1 and #2 could have formed in a single austenite ($\gamma_A$) grain, within the regions that are separated by small angle boundaries. In contrast, the sheave #3 was indexed to be close to the [011] zone axis. Using the axis-angle pair analyses [38, 101], the relative orientation between #3 and #1 or #3 and #2 was found to be of high-angle type. The details of the orientation relationship analyses are provided in Appendix A. Using the same assumption of KS/NW orientation relationship of bainitic sheaves with austenite and symmetry relations [38] one can also conclude that the #3 sheave formed in adjacent austenite grain ($\gamma_B$) and the orientation relationship between $\gamma_A$ and $\gamma_B$ should be of high-angle type. Copious presence of such austenite grains with large angle boundaries will promote bainitic and/or martensitic plates with wide varying orientations and may impact the properties [102]. It is also important to note that the above results are typical and we cannot conclude similar conditions throughout the sample. To evaluate this throughout the sample, we need to do extensive EBSD analyses and correlate the orientation distribution function to the properties.

In another region, HAADF STEM image shows extensive carbide distributions. Some of them were associated with the ferrite sub-unit grain
boundaries (Fig.6.5i). Size of these carbides was about 100nm. Elongated carbides between the lath boundaries are probably cementite. The precipitates are predominantly darker than the matrix indicating that their average atomic number is lower than the iron matrix. However, the precipitate thickness effect is convoluted with the atomic number contrast and further work is required to establish the chemical composition of these precipitates. Some of the large carbides show (marked by arrow) shows no correlation to the ferrite boundaries (Fig.6.5j). The EDS analyses show these carbides were rich in silicon and chromium (Fig.6.5k). The detailed analyses of elemental distribution within these carbides can only be attained by atom probe field ion microscopy [96, 97].

Fig.6.5i HAADF STEM image showing extensive carbides with different size range, Fig.6.5j HAADF STEM image showing fine carbides in between bainitic subunits.
Fig. 6.5k Measured energy dispersive X-ray spectrum obtained from matrix and carbides showing higher Cr/Fe ratio similar to that of carbides from unprocessed samples.

Based on the rapid rate of heating and cooling which will limit the diffusion controlled growth during cooling, these large carbides are tentatively attributed to the un-dissolved carbides from the base materials before processing. From the above TEM analyses one can conclude that the FP microstructures contain bainitic ferrite, martensitic laths, as well as, large and small carbides.

### 6.6 Mechanical Heterogeneity Evaluation

Although, the microstructure of FP sample showed this unique mixture of microstructures consisting of bainite, martensite and carbides, it is necessary to evaluate the heterogeneity of these microstructures within the overall sample. Since it is practically impossible to evaluate the microstructural heterogeneity by a series of optical, SEM or TEM techniques
throughout the sample, hardness mapping was adopted to evaluate the mechanical heterogeneity. In this method, series of hardness indents with equal spacing are made across the cross-section of the samples and the mechanical heterogeneity [103] is estimated by analyzing the hardness distribution. This quantitative mechanical heterogeneity may be used as an indirect measure of microstructural heterogeneity. A point to note is that it is possible for different microstructure distribution to provide the same hardness values. Therefore, the above measure has to be used only as a way to evaluate the possible scatter in final mechanical properties. The mechanical heterogeneity results from QT and FP samples are presented below.

The cross section of both the FP and QT samples were cut into three pieces (edge 1, middle, edge 2) for the ease of experiment. The hardness maps from these three regions of the sample cross-section from QT is shown in Fig.6.6a.

![Hardness contour map of cross-section of QT sample cut into three pieces showing distribution of hardness as measured from over 2000 micro-indent points](image)

Fig.6.6a: Hardness contour map of cross-section of QT sample cut into three pieces showing distribution of hardness as measured from over 2000 micro-indent points
The sample showed a large soft region (blue color) in the edge1 region. In addition, the hardness variations are large in this sample. This is attributed to in-efficient quenching during manual cooling of the austenitized sample. This is also reflected in the results of the tensile tests, where QT samples showed low yield strength [see Fig.6.3a and Table 2] and high strain hardening exponent. The hardness distribution curve from the QT sample is shown in Fig.6.6a. The above results are interpreted by fitting multiple peaks to the distribution. The fits show that the QT sample can be divided into two regions, i.e., a large (v_{QT1} =88%) region 1 with an average hardness of 475±47 HV with a large scatter and a smaller (v_{QT1} =12%) region 2 with an average hardness of 483±5 with a much smaller scatter.

Fig.6.6b: Histogram plotted from hardness values observed in cross-section of QT sample, shows large scatter peak (v_{QT1}=88%) and small scatter peak (v_{QT2}=12%)
In contrast, the mechanical heterogeneity in the flash processed steel was smaller and the FP sample was harder than QT samples. Fig.5.6c and Fig.5.6d show the hardness contour maps of the cross section of FP sample and the frequency histogram of the hardness values respectively. The image Fig.6.6c shows that a slightly harder region (yellow color) is in the middle of the FP steel sheet compared to the edge of the steel sheet.

![Hardness contour map](image_url)

Fig.6.6c: Hardness contour map of cross-section of FP sample cut into three pieces showing distribution of hardness as measured from over 2000 micro-indent points

From the flash process assembly (Fig.4.1), it can be expected that the middle of the steel sheet is exposed to the highest heat density and as a result highest heating rate and high peak temperature may be expected in this region. Similar to QT sample analyses, the calculated hardness distribution was analyzed by fitting multiple peaks to the distribution and the obtained histogram is shown Fig. (6.6d)
Fig. 6.6d: Histogram plotted from hardness values observed in cross-section of FP sample shows three distinct regions of different hardness range; 498±7, 512±11 and 534±9 HV

The fits show that the FP sample can be divided into three distinct regions, i.e., region 1 ($v_{FP1}$ 14%) with an average hardness of 498±7, region 2 ($v_{FP1}$ =74%) with an average hardness of 512±11 and region 3 ($v_{FP1}$=12%) with an average hardness of 534±9 HV. This presence of interspersed small soft regions within a hard region is tentatively attributed to the improved properties of the flash processed steels.
CHAPTER 7

MICROSTRUCTURE EVOLUTION IN FLASH PROCESS

7.1 Introduction

In this section, a mechanism for the microstructure evolution during FP is hypothesized. In this hypothesis, the results from the mechanical testing, phase transformation characteristics and microstructural characterization would be utilized to explain the good mechanical properties obtained from the flash process. Such an understanding is essential for retaining the properties during other thermo-mechanical processing operations including forging or welding, which potentially change the underlying microstructure of the steel.

Based on the results, it can be concluded that FP leads to a complex microstructure that contains martensite, bainite, and carbides. In addition, the above complex microstructure also leads to a preferred mechanical heterogeneity that is better than the reference QT sample. Furthermore, the FP steels can be classified under the category of advanced high strength
steels. Also, FP steels show at least 7% higher UTS and 30% higher elongation than published values for martensitic advanced high strength steels. However, the results have to be discussed in context with the underlying mechanism for the microstructure evolution during rapid heating and cooling.

7.2 Stepwise Mechanism of Microstructure Evolution

7.2.1 On Heating–Carbide Dissolution

As per equilibrium thermodynamic calculations, the 8620 steels should be 100% austenite with no carbides above 800°C. From the measured thermal cycle during the flash process, the sample was heated above 1100°C. Therefore, according to equilibrium thermodynamics, the sample should have completely transformed into austenite at this temperature and all the carbides should have dissolved. However, from the SEM and TEM characterization showed that the precipitates have not dissolved completely. This discrepancy is attributed to a very short dwell time in the austenitizing region and that time is not sufficient to dissolve a large proportion of the precipitates. This is supported by the observation of less precipitates in the QT samples since the samples were austenitized for 2 minutes at 1000°C. It is well known that, extended time in the austenite phase field will promote the complete dissolution of carbides by enhancing the substitutional element diffusion [104].
7.2.2 On Heating–Ferrite to Austenite Transformation

Another possibility is that the rapid heating rates (> 400 °C/s) in flash processing may affect the Ac1 and Ac3 Temperatures. Using published Ac1 and Ac3 temperatures, neural network analysis [105], and Gaussian process modeling [106], it has been shown that the Ac1 and Ac3 temperature of steels may increase above the equilibrium temperatures. The level of superheating to nucleate and growth austenite phase depends on the alloy composition, initial microstructure and the heating rate. This increase in Ac1 temperature also been rationalized with the need to nucleate austenite phase from a ferrite + carbide microstructure. On the other hand, if the initial microstructure contains retained austenite, there is no need for nucleation barrier [107]. Since our initial microstructure contained only ferrite and carbide, the heating rate may also lead to an increase in Ac1. Currently there is no direct method to measure this increase in Ac1 temperature during flash processing to confirm SSDTA results. Similar arguments can be made for an increase in Ac3 temperature due to reduced dwell time for diffusion-controlled growth of austenite into ferrite. It is also important to note that the research by Gavard et al [105] does not contain any data with the heating rates above 100 °C/s. Therefore, the validity of the continued increase in Ac1 and Ac3 far above the equilibrium Ac3 temperature has to be considered.

In a recent work, Elmer et al [108] have developed an overall transformation kinetic model for ferrite to austenite formation in 1005 steel
based on synchrotron diffraction measurements during a heating cycle of a weld. The model was calibrated using the data from low heating rates below 100 °Cs⁻¹. However, the model calculations can be extrapolated to higher heating rates. The calculations indicate a superheating of 100°C or more above equilibrium Ac₃ for the completion of austenite formation at heating rates above 300°C/s. This result supports the notion that the Ac₁ and Ac₃ temperature during flash processing (> 410°C/s) may also increase. This increase in Ac₁ and Ac₃ temperature will also affect the carbide dissolution, since the presence of austenite is necessary for the initiation of dissolution. In addition, the time taken for austenite grain growth also will be reduced. This is supported by the small prior austenite grain size (<10 μm) measured in the flash processed samples.

Another major consequence of the small dwell time above Ac₃ temperature, the carbon diffusion and redistribution in the matrix would not been complete [40]. Hence, the steel that is being cooled from the peak temperature, will have inhomogeneous carbon distribution throughout its matrix. Rapid cooling of this steel could lead to complex decomposition of austenite into different ferrite morphologies and martensite. This mechanism is discussed below.

7.2.3 On Cooling – Austenite Decomposition

Based on the calculated CCT diagram and cooling rate (> 3000 °C/s) during flash processing, the steel should have transformed to 100%
martensite. To evaluate the effect of gradient in carbon concentration within the austenite on the final microstructure, the CCT diagrams for 8620 steels with different carbon concentrations ranging from 0.01 to 0.2 wt.%, with the same substitutional alloying element concentrations, were calculated using the methodology described earlier [103]. These series of CCT diagrams are overlaid with the measured cooling curve in Fig.7.2. The comparison shows that even with very low carbon content the microstructure should be 100% martensite. However, the microstructure contains bainitic ferrite, martensite and carbides.

![Calculated CCT curves for AISI8620 steel austenite with same substitutional concentration (Fe–0.21C–0.27Si–0.002S–0.009P–0.73Mn–0.48Cr–0.48Ni–0.007Co–0.156Mo–0.178Cu in wt-%) but with different carbon concentrations (diagram is overlaid with measured cooling curve during FP)](image)

Fig.7.2: Calculated CCT curves for AISI8620 steel austenite with same substitutional concentration (Fe–0.21C–0.27Si–0.002S–0.009P–0.73Mn–0.48Cr–0.48Ni–0.007Co–0.156Mo–0.178Cu in wt-%) but with different carbon concentrations (diagram is overlaid with measured cooling curve during FP)
This discrepancy is attributed to an inherent inadequacy of the transformation kinetic methodologies. The models for the development of CCT data were based on the TTT data generated by traditional heat-treatment conditions [91,92], which involved slow heating and isothermal hold at austenitizing temperature exceeding 5 minutes. The extension of these models to austenite with inhomogeneous carbon concentration coupled with a condition of rapid heating rates (>400 °C/s) may not be completely reliable. There is hence a need to develop models that consider these unique features of processes like flash processing.

7.3 Conclusion

Based on the above results discussions, the microstructure evolution during flash processing is proposed with the help of Fig.7.3. At room temperature, the steel microstructure contains ferrite and carbides [Fig.7.3a]. On rapid heating, the austenite nucleation is expected to occur at the carbide-ferrite boundaries [109] and the austenite is expected to grow at the expense of ferrite and carbide dissolution. Due to rapid heating rates, the temperature at which the nucleation and growth occurs may be pushed to higher temperatures (> 900°C) [108]. However, before the complete dissolution of austenite, the cooling cycle starts. At this point, the austenite is expected to have inhomogeneous distribution of carbon content. With the onset of rapid cooling, the carbon enriched regions may transform to martensite and carbon-
depleted regions may transform to bainite. This would lead to a final microstructure with complex distribution of bainite, martensite and carbides.

Fig.7.3: Schematic illustration of microstructure evolution (a) unprocessed ferrite with carbides; (b) growth of austenite and start of carbide dissolution; (c) inhomogeneous carbon distribution in austenite with reduction in carbides; (d) martensite with bainitic plates formed from inhomogeneous carbon distribution in austenite

This is supported by the complex microstructure seen with the SEM and a mixed microstructure of bainite and martensite observed under the TEM. This hypothetical mechanism also suggests that the initial microstructure
will have strong effect on the final microstructure and properties. With this proposed model, further work was done to quantify the amount of constituent microstructures present in FP steels.

7.4 Summary

In Summary, a rapid heating (400 K/s) and cooling (3000 K/s) flash process has been shown to produce a microstructure with good combination of yield (1280 MPa), UTS (1600 MPa) and appreciable ductility of up to 9.9%. The above strength and ductility levels are significantly better than martensitic based AHSS (1400–1500 MPa UTS). Based on the above data, the flash processed steels could be classified as part of the advanced high strength steel family. As the overall processing time (<10 s) is very short, this process could be an alternative route for producing AHSS sheets. The underlying microstructures in flash processed samples were characterized with optical and analytical electron microscopy. The characterisation results showed that the steel contains bainite, martensite and carbides. The hardness map analyses showed that the flash processed samples contain a minor fraction of soft microstructure interspersed within a harder microstructure. This unique distribution of soft and hard microstructure is correlated with the improved YS, UTS and ductility. Traditional phase transformation models indicated that, for the cooling rates measured in the flash process, the microstructure should be 100% martensite after processing. This discrepancy is addressed with a theoretical mechanism that involves an
increase in Ac1 and Ac3 temperatures due to rapid heating rate, incomplete
dissolution of carbides and decomposition of austenite with non-uniform
carbon concentration.
CHAPTER 8

MODELING THE STRENGTH OF FLASH PROCESSED STEEL

8.1 Introduction

In view of the challenges that might arise in subjecting the flash processed steel to thermo-mechanical processing, it would first be important to quantify the amount of microstructure constituents that govern the strength of the FP steels. This knowledge would be vital in order to determine the steps required to retain them during such processing. In this section, a theoretical model is developed to estimate the volume fraction of bainite in FP steels.

From the above experiments, FP is now known to cause a mixed microstructure of bainite and martensite. Investigations on the strength of such mixed microstructure steels have shown strengthening [86,87,88,89] due to the presence of a small volume fraction of bainite. Fig. 3.4a shows a schematic of the observed trend in strengthening from the above researchers. With the help of such a model, the volume fraction of bainite can be back
calculated using the experimentally measured strength. Instead of applying the model directly, the existing models

This chapter deals with the steps taken to develop a model for mixed microstructure steels that shows good predictability with experimental results. This model is then used to evaluate the amount of bainite in FP samples, based on their strength.

8.2 Model Development- Strength of Martensite/ Bainite

Existing models that predict the strength of mixed microstructure steels, depend on the strength of individual constituent microstructures (i.e. martensite and bainite). Furthermore, theoretical models have been proposed to evaluate the strength of individual microstructures [56,57,58]. However, when tested, these models deviated from the experimental strength by a considerable amount.

Fig.8.2 Deviations of the predicted strengths from experimental strengths of untempered martensitic steels using a) Naylor's Model and b) Bhadeshia's Model
Fig.8.2a and Fig.8.2b show experimentally observed strengths for martensitic steels [81, 84] and the strengths predicted by Naylor’s and Bhadeshia’s model for the same steels. The deviations from the experimentally observed strengths are evident.

Hence there is a need for a model that can predict the strength of a wide range of martensitic steels as well as the strength of mixed (martensitic+bainitic) microstructure steels. A new model for strength estimation is proposed by curve fitting and by modifying some of the strengthening factors from the above two models. For the most part, the strengthening factors proposed by Bhadeshia are retained and only the proportionality constants are changed. However, the strengthening factor due to grains is adopted from Naylor’s model as explained below.

First, a model is developed to predict the strength of untempered martensite. For this, the same method of evaluating individual strengthening factors is followed. The equations for individual strengthening, factors are taken from Bhadeshia’s model except the term of strength due to grains, which is taken from Naylor’s model. This is because Naylor considers both block width and packet size as a contributing factor to the strength due to their high angle boundaries. Table 5 shows the equations for the new model.
Table 5: The strengthening terms used for new model to predict strength of untempered martensitic steels.

<table>
<thead>
<tr>
<th>Source</th>
<th>Equation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dislocation Density $\sigma_{\text{Dis}}$ Bhadeshia [59]</td>
<td>$K_{\text{Dis}} \mu b \rho^{1/2}$</td>
</tr>
<tr>
<td>Substitutional Strengthening $\sigma_{\text{SS}}$ Naylor [56]</td>
<td></td>
</tr>
<tr>
<td>Interstitial Strengthening $\sigma_{\text{C}}$ Bhadeshia [59]</td>
<td>$\text{Martensite: } K_{\text{Mat}} C^{1/3}$</td>
</tr>
<tr>
<td>Grain size Strengthening $\sigma_{\text{Grain}}$ Naylor [56]</td>
<td>$\text{Bainite: } K_{\text{Bain}} C^{1/2}$</td>
</tr>
<tr>
<td></td>
<td>$K_{\text{Grain}} M^{-1/2}$</td>
</tr>
</tbody>
</table>

Fig.8.2c Experimental strength vs. the predicted strength of three different grades of martensitic steels from the new model
Using the reported strengths of two different grades of steels [81, 84], the constants $K_{\text{Dis}}$, $K_{\text{Mar}}$ and $K_{\text{Grain}}$ were evaluated. The obtained equation was then tested using another set of independent experimental strength results [110]. Fig.8.2c shows the predicted strength from this new model plotted against the experimental values and good predictability is evident from this new model. The values of the new and the old coefficients are given in Table 6.

<table>
<thead>
<tr>
<th>Strengthening Term</th>
<th>Equation</th>
<th>Old Coefficient</th>
<th>New Coefficient</th>
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<td>7.34 x 10^{-6}</td>
<td>7.34 x 10^{-6}</td>
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<td>Substitutional Strengthening $\sigma_{\text{SS}}$</td>
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<td>60</td>
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<tr>
<td>Interstitial Strengthening $\sigma_{\text{C}}$</td>
<td>$\text{Martensite}: K_{\text{Mar}} C^{1/3}$</td>
<td>1722.5 – Martensite</td>
<td>222.539 – Martensite</td>
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<td></td>
<td>$\text{Bainite}: K_{\text{Bain}} C^{1/2}$</td>
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<td>1039.4 – Bainite</td>
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<tr>
<td>Grain size Strengthening $\sigma_{\text{Grain}}$</td>
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<td>11.5 x 10^{-5}</td>
<td>3.35 x 10^{-5}</td>
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</table>

Table 6: Coefficients in the old and new model for untempered martensite strength

8.3 Model Development- Strength of Mixed Microstructure Steel

The presence of a small volume fraction of bainite has been shown to increase the strength of mixed microstructure steel. This has been predicted to be due to two reasons; a) Partitioning of carbon between bainite and
martensite and b) due to the plastic constraining effect experienced by bainite due to martensite.

Extending the above-developed model for martensite/ bainite, the strength of mixed microstructure steel is calculated.

8.3.1 Strength due to carbon partitioning

When considering strengthening by carbon in bainitic microstructure, the amount of carbon dissolved in the interstitial solution is taken to be 0.03wt% [59]. However, for the martensitic microstructure, the strengthening by carbon is important, as the amount of carbon in martensite can be higher than the average bulk carbon concentration. This is primarily due to the carbon rejected from bainite during its formation causing enrichment of the surrounding austenite with carbon. Using mass balance equations, the amount of carbon in austenite when a certain volume fraction of bainite is formed can be calculated as follows:

\[ C_r = \frac{\overline{C} - V_b C_b}{1 - V_b} \]  

(19)

where \( C_r \) is the carbon in the residual austenite before transforming to martensite, \( V_b \) is the volume fraction of bainitic ferrite and \( C_b \) is the amount of carbon in bainite and \( \overline{C} \) is the bulk carbon concentration. This carbon would be present in the final martensite. The strength can then be calculated by the interstitial strengthening term from Table 6.
8.3.2 Strength due to plastic constraining effect

It is known that the normalized stress in brazed joint varies exponentially with the normalized thickness of the brazed layer. Volume fraction of bainite was assumed be analogous to the thickness in braze alloys for volume fraction of bainite. An exponential expression was then derived, by fitting of curve to the data points of Tomita and Okabayashi, to estimate the strength of constraint bainite and the equation is given by:

\[
\sigma'_b = \sigma_b (K_{Pl} \cdot \exp(C_{Pl} \cdot V_b) + 0.98)
\]  

(20)

where \(\sigma'_b\): Strength of constraint bainite; \(\sigma_b\): unconstraint bainite and \(K_{Pl}\) and \(C_{Pl}\) are constants.

Table 7 shows the plastic constraining effect from the new model and from Young and Bhadeshia’s model.

**Tempering Effect:** Tempering causes softening in martensite and it is taken into account by using the Avrami equation for carbon diffusion. The strength of tempered martensite is given by

\[
\sigma_{MT} = \sigma_M + \tilde{\xi}(t)(\sigma_M - \sigma_b)
\]  

(21)

where: \(\sigma_{MT}\): tempered martensite; \(\sigma_M\): untempered martensite; \(\sigma_b\) strength of bainite; \(\tilde{\xi}(t)\): is an Avrami equation function \(\tilde{\xi}(t) = 1 - \exp(-kt^n)\); t being temper time in hours and \(k = k_0 \exp(-Q/RT)\) with \(k_0\) is given by \(K^* \times \bar{X}^{0.635}\); \(\bar{X}\): atom fraction of carbon, \(Q\): activation energy, \(R\): Universal gas constant and \(T\): tempering temperature in Kelvins.
<table>
<thead>
<tr>
<th>Factor</th>
<th>Source</th>
<th>Old Model</th>
<th>New Model</th>
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<td>Plastic Constrain</td>
<td>Young and Bhadeshia</td>
<td>$K_{Pl} = 0.65$</td>
<td>$K_{Pl} = \left[1.2 - 1.86 \times 10^{-3} \cdot \exp(5.493 \times 10^{-3} T)\right]$</td>
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<td>Effect</td>
<td></td>
<td>$C_{Pl} = -3.3$</td>
<td>$C_{Pl} = -3.3$</td>
</tr>
<tr>
<td>Tempering</td>
<td>Young and Bhadeshia</td>
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<td>$n = 1.138$</td>
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<td></td>
<td></td>
<td>$k = 4.07 \times 10^4$</td>
<td>$k = 45.2674$</td>
</tr>
</tbody>
</table>

Table 7: The plastic constraining effects constants from the old and the new model; $T$ and $t$ represent the tempering temperature and time in K and Hrs respectively.

![Graph](image)

Fig.8.3: The variation of strength with the volume fraction of bainite as reported by Okabayashi and Tomita compared to the predicted strength by the model.
Fig. 8.3 shows the strength observed by Okabayashi and Tomita and the strength predicted for the same steel by the newly developed model.

**NOTE:** In the model, the constant in the plastic constraining term 
\[ K_{Pl} = [1.2 - 1.86 \times 10^{-3} \cdot \exp(5.493 \times 10^{-3} T)] \], has been correlated to tempering time and temperature. However, this has been done purely as a fitting parameter and further work is needed to evaluate the accuracy of this relation.

### 8.4 Predicting the strength of FP steel

Based on the block width and packet size measurements, (Table 4) and developed model for martensite strength, the strength of the FP steel is calculated. For this calculation, initially, it is assumed that no bainite is present in the steel. The calculated strength of the bainitieless, completely martensitic FP steel thus obtained is 1330.07 MPa. The contribution of various strengthening factors to this strength is tabulated in Table 8.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Peierls Force (MPa)</th>
<th>Dislocation strengthening (MPa)</th>
<th>Interstitial strengthening (MPa)</th>
<th>Microstructure strengthening (MPa)</th>
<th>Substitutional strengthening (MPa)</th>
<th>Total Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>HP</td>
<td>40</td>
<td>665.218</td>
<td>130.142</td>
<td>452.709</td>
<td>42</td>
<td>1330.07</td>
</tr>
</tbody>
</table>

Table 8: The calculated individual strengthening terms for FP steels assuming that they are completely martensitic.

From tensile test (Table 2) results it can be seen that the strength for FP steel can however be higher than this calculated value. Also, FP was
characterized to have bainite along with martensitic. From the average value of observed strength, the volume fraction of bainite is then back calculated using the model for mixed microstructure. Fig.8.4 shows the variation of strength in AISI 8620 steel with varying volume fraction of bainite, having the same microstructural features as obtained from FP. When the tensile tests results of the FP samples are superimposed on this curve, a band or region (between the dotted lines) can be obtained within which FP steel can lie. In Fig.8.4, the band and the strength curve from the model intersect at two regions ($V_b < 0.2$ and $V_b > 0.5$). However, as FP does not provide enough time for the growth of large amounts of bainite, FP steels cannot lie in the region with $V_b > 0.5$. Hence from the model and tensile test results, the volume fraction of bainite in FP steels is expected to lie close to 0.1.

Fig.8.4: Expected volume fraction of bainite in FP steel ($0 < V_b < 0.1$) from developed model for mixed microstructure.
8.5 Summary

By the modification of existing models for strength of mixed microstructure, a new model has been developed that shows a good correlation with experimental results. Based on this model, the volume fraction of bainite in FP steel is back calculated. From the calculations, the volume fraction of bainite is expected to be close to 0.1 volume fraction.
CHAPTER 9

CONCLUSIONS AND FUTURE WORK

9.1 Mechanical Properties of Flash Process Steels

The aim of this part of the study was to confirm that FP produced steels with strength and ductility that matched the reported values for AHS steels. Tensile tests were conducted on sample taken from FP AISI 8620 steel sheets the conclusions from these tests are:

1. The ultimate tensile strengths of the FP AISI 8620 samples were greater than 1500 MPa and the total elongation of the samples was in the range of 8.8-9.9%. The observed elongation is much greater than the martensitic type AHSS grade of steels (5%). This proved that FP does produce steels with strength matching AHS steels along with good ductility.

2. Initial microstructure characterization under optical microscopy revealed an indiscernible very fine microstructure consisting of fine lath
features and more advanced characterization techniques are needed to reveal the underlying microstructure constituents.

3. CCT diagrams constructed for AISI 8620 grade steels indicated that for the reported short processing time of FP (<10s), the steel is expected to form a completely martensitic structure with very low ductility. This contradicts the observed mechanical properties of FP AISI 8620 steels. Hence a deeper understanding of the process is required to understand the cause for the observed properties.

9.2 FP Thermal Cycle Analysis

This part of the work focused on understanding the thermal profile generated during the flash process. Furthermore, in order to record the phase transformations occurring during the process, a thermal analysis technique (SSDTA) was used and the conclusions from these experiments are as follows.

1. Thermocouples attached to the AISI 8620 steel surface revealed the thermal cycle of FP. The total time for the process was recorded to be close to being around 6s. The peak temperature recorded was near 1100°C. The cooling cycle in the process was just under 2s. The peak temperature reached is higher than the Ae1 and Ae3 temperatures for the steel which were calculated to be 702°C and 806°C.

2. Heating and cooling rate analysis of the thermocouple data showed a maximum instantaneous heating rate of over 400°C/s just before the
heating unit and instantaneous cooling rates of greater than 3000°C/s. This clearly shows that flash process is a rapid heating and rapid cooling process with a very short processing time.

3. SSDTA analysis on the heating part of the thermal cycle indicated a thermal event that is correlated to the $\alpha \rightarrow \gamma$ phase transformation. The transformation start ($\text{Ac}_1$) and finish ($\text{Ac}_2$) temperatures were recorded to be in the ranges 925-930°C and 1045-1052°C. This shows that due to the rapid heating involved in the process, the transformation temperature is pushed higher. The transformation finish temperature also indicates that the steel becomes complete austenitic during the heating cycle.

4. In another related work, cooling curve analyses of the FP for AISI 4130 grade steel indicated two thermal transformation events, one at a higher temperature and the other at a lower temperature. However, detailed microstructure characterization was required to correlate the above thermal events to the microstructure.

9.3 Microstructure Characterization

In this part of the work, the focus was to understand the underlying microstructure generated by FP. Using advanced characterization techniques like SEM, TEM and EBSD pattern analysis, FP samples were analyzed and the conclusions from the observations made are as follows.
1. SEM image of the unprocessed sample shows a rolled microstructure consisting of ferrite and carbides. EDS analysis of the carbides indicated a high Cr content in them. Hence the initial microstructure consisted of ferrite and chromium carbides.

2. Under the SEM, FP samples showed lath type microstructures along with clusters of carbides. EDS analysis of the carbides again indicated an enrichment of chromium. As no tempering was provided for the steels, the presence of carbides shows that complete dissolution of carbides does not take place in the austenite regime during FP. This is concluded to be due to the very short dwell time in the austenite region.

3. TEM image analysis of the samples showed the presence of bainite along with lath martensite. This proved that FP leads to the formation of a mixed microstructure of martensite and bainite.

4. EBSD analysis was primarily used to quantify the size of the packets of and sub blocks of martensite formed within a prior austenite grain. The size of prior austenite grains in FP samples was found to be 5.4 μm with packets and block sizes of 2.73 μm and 0.623 μm respectively. When compared to a normal quenched and tempered sample, these grain sizes were finer. This indicated that the prior austenite grain transforming into bainite and martensite are very fine.
The cause for the fine grains is believed to be due to a very short dwell time above the Ac3 temperature.

9.4 Microstructure evolution

Based on the results from the analysis of the thermal cycles and microstructure characterization, a hypothesis was formed for the microstructure evolution in the FP.

1. The initial microstructure of ferrite and carbide leads to an inhomogeneous carbon distribution at the initial stages of the formation of austenite. This conclusion was supported by the presence of undissolved carbides in the final microstructure.

2. Due to the very short dwell time in the austenite phase field, the process of carbon homogenization is incomplete. Hence the microstructure that enters the cooling cycle is a consists of austenite with inhomogeneous carbon content and undissolved carbides. From such a microstructure, regions containing very low amounts of carbon transforms into bainite and regions with higher carbon transforms to martensite. This hypothesis is supported by SSDTA results and presence of bainite in the final steel.

3. In conclusion, the final microstructure obtained from flash process is highly dependent on the initial microstructure of the unprocessed steel.
and to obtain a mixture of bainite and martensite, the steel should have an initial microstructure of ferrite with a dispersion of bainite.

9.5 Amount of Bainite in FP

As most advanced characterization techniques have a localized field of view and since the features in FP samples could only be discerned in a TEM, it is highly difficult to quantify the volume fraction of bainite in FP. Microhardness maps cannot be completely reliable as the hardness indents (~10μm diagonal) are usually larger than the steel features. Hence the volume fraction of bainite is calculated using models for the strength of steel.

Based on the model, the strength of the steel increases with the inclusion of a small amount of bainite. Using the strength obtained from tensile tests of FP samples, the volume fraction of bainite is back calculated from the model. For the AISI 8620 FP steels, the volume fraction of bainite was calculated to lie between 0- 0.15.

9.6 Future Work

9.6.1 Testing of welded FP steels

For evaluating the weldability of FP steels, welds were made on FP steel samples. This work was performed at the Pennsylvania State University. Seven steels samples were welded and three different welding processes were employed to weld these steels. The welding processes included low heat input laser welding, laser-arc hybrid welding and gas metal arc
welding (GMAW). For the laser and hybrid welds, a 12kW Ytterbium (Yb) fiber laser operating at 4500 W was used. Laser energy was focused through a transmissive fused silica collimation and focus optics having focal lengths of 200 and 500 mm, respectively. The laser beam focal position was placed on the surface of the workpiece, producing a 0.744 mm diameter laser beam. For all the welds, a shielding gas consisting of Ar-10CO₂ was used.

**Laser Welds:** Zero-gap butt welds were made on FP samples using autogenous laser welding (LBW). For these samples Bead on Plate (BOP) autogenous welds were also made at the same conditions as the square butt welds. This was done as some of the joints were seen to open up during the initial butt weld. The BOP welds were made after the plates were allowed to cool to room temperature. Four samples were welded in this fashion and the process parameters for these welds are given in table 9.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Travel Speed (m/min)</th>
<th>Laser power (W)</th>
<th>Heat Input (KJ/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 1</td>
<td>2.54</td>
<td>4500</td>
<td>106.3</td>
</tr>
<tr>
<td>Sample 2</td>
<td>3.56</td>
<td>4500</td>
<td>75.98</td>
</tr>
<tr>
<td>Sample 3</td>
<td>4.06</td>
<td>4500</td>
<td>66.54</td>
</tr>
<tr>
<td>Sample 4</td>
<td>4.57</td>
<td>4500</td>
<td>59.06</td>
</tr>
</tbody>
</table>

Table 9: Process parameters for the laser welded FP samples
Laser-arc hybrid weld: Similar to the laser welds, one zero-gap butt welds was made using laser-arc hybrid welding. The laser power used was the same as was used for laser welds. The arc was generated with the help of a pulsed GMAW process using an 0.045 inch diameter 100S-1 wire filler wire. The nominal arc voltage and current used were 25 V and 165 A respectively. The laser-arc separation in the hybrid weld was maintained at 1 mm.

GMAW weld: One FP sample was welded using the GMAW process. For this joint, both sides of the sample were welded using the same filler and process parameters were used as for the laser hybrid weld. The second side of the sample was welded after the plate was allowed to cool to room temperature. The process parameters used for the laser-arc welds and the GMAW welds are tabulated. (See: Table 10)

<table>
<thead>
<tr>
<th>Weld</th>
<th>Travel Speed (m/min)</th>
<th>Laser Power (W)</th>
<th>Wire Feed Rate (m/min)</th>
<th>Volts (V)</th>
<th>Current (A)</th>
<th>Heat Input (KJ/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hybrid</td>
<td>4.06</td>
<td>4500</td>
<td>7.96</td>
<td>25</td>
<td>165</td>
<td>127.16</td>
</tr>
<tr>
<td>GMAW</td>
<td>4.06</td>
<td>-</td>
<td>7.96</td>
<td>25</td>
<td>165</td>
<td>121.65</td>
</tr>
</tbody>
</table>

Table 10: Welding parameters for the hybrid and GMAW welds on FP samples.
The hybrid and GMAW welds on the FP samples resulted in significant distortion of the welded samples. This is believed to be due to the relatively higher heat input in these two processes.

Microhardness tests were initially made on the Hybrid weld and the on the BOP sample 4. Fig.9.6a shows the color plot generated from hardness measurements on the hybrid weld.

Fig.9.6a: Hardness plot of the hybrid weld on FP 1020 steel.

In the figure, the weld metal has a hardness in range of 450 to 500 HV whereas, the HAZ has a hardness of ~ 300 HV and the base metal has a hardness of 400 HV. The thickness of the HAZ on either side of the weld is around 2 mm. Clearly this type of weld on FP 1020 shows HAZ softening and can be a region of weld failure in service. The higher hardness in the weld metal could be due to martensite formation due to sufficient time spent in
austenitizing temperature and rapid cooling. The softening at the HAZ could be due to the high heat input from the hybrid weld (127 KJ/m). Cursory inspection of the weld under an optical microscope did not show the presence of any cracks. However, microstructure characterization is required to observe the microstructure that is obtained due to welding in the HAZ.

![Hardness plot of the laser weld on FP 1020 steel.](image)

**Fig.9.6b:** Hardness plot of the laser weld on FP 1020 steel.

Hardness measurements were made on a BOP laser weld (sample 4) and the hardness plot obtained is shown in Fig.9.6b. The image shows a welded region of hardness ~475 HV and a base metal hardness of ~325 HV. A very narrow HAZ (~50 μm) (see Fig. 9.6c) is also distinguishable and it has a hardness of ~425 HV. Due to the very low heat input from the laser welds
(59.06 KJ/m) and high cooling rate resulting from it, the HAZ is not large. Furthermore, due to the high cooling rate experienced by the region just adjacent to the weld, there is no HAZ softening unlike that seen in the hybrid welds.

Fig. 9.6c: Optical image of the HAZ in the laser welded FP 1020 steel. The thickness of the HAZ is ~50 µm.

The high hardness in the weld could be due to the formation of martensite resulting from the high cooling rate of the weld. In the laser weld however, the flash processed steel had a softer base metal than that observed in the hybrid weld. The cause for this is not clearly understood and further work is required to understand this observation. No visible crack formations in the laser welded FP samples, when seen under the microscope. Similar hardness testing has to be performed on the remaining welded samples.
From the above observations, laser welding appears to be an appropriate welding technique to weld FP steels. However there a need for mechanical testing which would include tensile and charpy impact testing to confirm it.

9.6.2 Thermal cycle simulation using Gleeble

Using a thermo-mechanical simulator like Gleeble, it is possible to replicate the FP thermal profile. With this capability, it is possible to test the proposed mechanism for microstructure evolution in FP of steels. The results from SSDTA could be confirmed by using a dilatometer. For example, the heating cycle of FP can be used in the Gleeble to confirm whether the steel becomes completely austenitic during FP, using a dilatometer. In a similar way, cooling curve analysis of the flat plate sample can be performed in the Gleeble to look for two transformation events occurring during cooling which would support the formation of mixed microstructure steel.

A number of tests can also be tailored to involve step heating which can be used to observe and study the carbon dissolution characteristics. This could then be used to support or disprove the hypothesis that believes that the presence of undissolved carbon could cause an inhomogeneous distribution of carbon in the austenite just before quenching.
9.6.3 Assembly for Processing Thicker Sheets

The current work on understanding the flash process was carried out on a prototype assembly capable of processing steels with a width of ~7.6 cm and thickness ranging between 1.7-4 mm. However, in order to make this process industrially competent, a larger assembly with a capability of processing thicker sheets has been built. Furthermore, the assembly has been converted to have a horizontal throughput for easy of processing as compared to the vertical processing setup of the prototype. With this assembly, steels with thickness ~12.5 mm and width 600 mm can be processed. One of the major changes in the new assembly is that quenching of steel is carried out by using jets of water impinging on both sides of the hot steel surface in place of an agitated water trough due to the restriction imposed by the horizontal processing assembly.

However, the effect of this change in the quenching condition on the microstructure of the final steel is not completely understood. Microhardness testing over the cross section of the steel plate (Fig.9.6d) shows two distinct bands of hard and soft regions. The above result can be hypothesized by the following observation; during the process, water impinging on the top surface of the steel becomes stagnant and forms puddles over the surface. Whereas, water hitting the bottom surface, just skims the surface of the steel before flowing back down. This difference in the water flow characteristics is believed to create an inhomogeneous quench condition. However, the above
observation has to be further understood in order to modify the assembly for attaining a homogenous quench condition during the process. This can most likely be achieved by using a larger number of nozzles in the water jet assembly.

Fig.9.6d: Hardness map of a horizontally processed AISI4130 FP sample showing a clear variation in the through thickness hardness indicating a clear difference in the quenching conditions at the top and bottom

9.6.4 Impact Toughness

Impact toughness measurements were carried out on both the horizontally and vertically processed AISI4130 FP steels. Charpy samples
were cut from both the transverse and longitudinal directions of the vertically and horizontally processed FP samples and were tested at two different temperatures (233K and 300K). Fig.9.6e shows the results obtained for the above tests. From the results of the charpy impact toughness tests, for samples taken from the same orientation, the vertically processed steels show almost twice the impact toughness compared to the corresponding horizontally processed sample at 300K. Also in general, at 233K, the vertical samples continue to show higher toughness than the horizontal processed samples. As mentioned above, due to non uniform quenching in the horizontal process, the impact toughness of the steel is believed to be sensitive to the region from which the test sample was taken and the location where the charpy ‘V’ notch was created.

Fig.9.6e: Impact toughness measurements on vertically and horizontally processed FP steels for samples taken from both the longitudinal and transverse samples. (H: Horizontal and V: Vertical)
In order to understand the reason for the differences in impact toughness between the horizontal and vertical processes, the difference in the two processing conditions must be realized. For this, the thermal profile of the horizontal process should be recorded and compared with that of the vertical process. Using thermal analysis techniques like SSDTA, the phase transformations occurring in the horizontal process must be recorded. Further work is hence required with a focus on reproducing the vertical flash process properties in the horizontal assembly.
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APPENDIX A

Towards Understanding the Microstructure Development During Flash Heating and Cooling of Steels

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Materials Science and Technology, Maney Publications: online paper.

Abstract

A rapid heating and cooling heat treatment procedure, with cycles times less than 10 seconds, for the production of advanced high strength steel sheets has been developed. According to the steel phase transformation theories and published continuous cooling diagrams, these thermal cycles should have lead to the formation of martensitic microstructure. Contrary to this expected result, many candidate steels (e.g. steel specifications AISI 8620, 4130) showed high yield strength (1350 to 1400 MPa) and tensile strength (1694 to 1900 MPa), while maintaining a good ductility (>7%). The current work summarizes the investigation of microstructure evolution under this process through (a) single sensor differential thermal analysis (SSDTA),
(b) optical and electron microscopy characterization and (c) computational models in a 4130 grade steel.

A.1 Introduction

In the wake of the rising need for weight reduction in automobiles to improve their efficiency, a new class of steels called Advanced High Strength Steels (AHSS) has been developed [4, 5]. Their production typically involves time-consuming complex heat treatment cycles [6]. Different research directions are being explored in order to produce advanced steels [26,27,28,29]. An innovative heat treatment process that aims at improving the strength of steels with extremely short heat treatment cycle times, has been developed by SFP Works. Due to the incredibly fast treatment cycle involved (< 10 seconds), the process has been named Flash Processing (FP). This process focuses on the production of AHSS while eliminating the complex heat treatment cycles involving large dwell times at different temperatures.

FP involves rapid heating of steels to temperatures just over 1000 °C using an oxy-propane flame or electrical induction heating. This is immediately followed by rapid quenching in agitated water maintained at a constant temperature. The surprising aspect of this process is, contrary to well-established theories of phase transformation, rapid quenching doesn’t appear to render the processed steel brittle [40]. Mechanical testing of AISI
8620 flat plates processed by the flash process, showed good mechanical properties (YS: 1300-1440 MPa, TS: 1500-1650 MPa and elongation: 8.8-10%). A scanning electron microscopy (SEM) image of a FP 8620 steel sample (see Fig.6.5a) shows a mixed bainitic and martensitic microstructure with undissolved carbides. In this work, the generality of this microstructure evolution was tested in 4130-grade (Fe-0.5 Mn-0.04 S-0.225 Si-0.95 Cr-0.2 Mo wt.%) steel. The phase transformations characteristics were evaluated with Single Sensor Differential Thermal Analysis (SSDTA) [51, 52], hardness mapping and microstructure analysis. The data was interpreted using well-established structure property correlations.

A.2 Experimental Work

A.2.1 Processing Setup

The schematic of the FP setup is shown in Fig.4.1. This setup consists of rollers placed at the top of the arrangement to guide the steel followed by a heating stage which is made of either oxy-propane flame or electrical induction heating source. A water filled trough placed below the heating heads at a certain distance to quench the hot steel. A rubber lined, watertight slit in the middle of this trough allows the steel to pass through and makes this a continuous feed process. Finally rollers placed at the bottom, guide the processed steel out of the trough. The water in the trough is maintained below the room temperature (~25 °C) through a chiller system. It is also
continuously agitated to avoid the formation of vapor barriers around the steel when it is passing through the water. However it is to be noted that all the experiments mentioned in this paper were performed on the AISI 4130 pipe steel to ensure that the results from the thermal measurements are not influenced by the formation of any vapor barrier or steam bubbles during the quenching in water.

A.2.2 Single Sensor Differential Thermal Analysis (SSDTA)

SSDTA is an innovative method to record thermal fluctuations in a material during continuous heating or cooling process. It has the capability to detect reactions or phase transformations taking place during a process. SSDTA utilizes the change in temperature involved with the exothermic/endothermic nature of any reaction or phase transformation [51]. This process is very similar to the normal Differential Thermal Analysis (DTA). However, unlike DTA, there is no standard reference sample involved for comparison. This is because it essentially utilizes a single sensor to measure the thermal history of the material of interest, which gives it a flexibility of usage in non-laboratory conditions. The sensitivity of the SSDTA technique during high rates of heating and cooling have been documented [51,52] by direct comparison with dilatometric techniques. For the current investigations, thermocouples were attached to the inner surface of these steel pipes during the process. Thermocouples were then connected to a high-
speed data acquisition system to record the temperature profile generated at a sampling rate of up to 5 KHz. The SSDTA data was used to delineate the possible phase transformation temperatures during the rapid cooling.

A.2.3 Hardness and Microstructure

Micro hardness tests were performed with over 2000 indents over the cross section of FP steel. The load used was 300g and the mechanical heterogeneity [103] was estimated by analyzing the hardness distribution. Two-dimensional hardness distribution maps and histograms were drawn based on these hardness data. Optical images were taken after etching the polished steel with 2% Nital etchant. Microstructures were also observed under a scanning electron microscope (SEM) using both Secondary Electron (SE) and Back-scattered Electron (BSE) imaging modes.

A.3 Results and Discussion

A typical plot of the thermal cycle experienced by the 4130-pipe sample is shown in Fig.A.3a. The highest temperature achieved in the process is 1150 °C. The total duration to heat from 400°C to 1150°C and then cool to 400°C was around 10 seconds. Analysis of the SSDTA curves clearly shows the presence of two thermal events during the cooling cycle (see Fig.A.3b). The analyses show a high temperature event (from 650°C to 559.3°C) and a low-temperature event (from 459°C to 359°C). These events are delineated by careful analyses of individual regions (not shown in the graph) due to the
release of enthalpy of transformation. The occurrence of these two events points to the possibility of two types of phase transformations. In the next step, the above-observed phenomena were correlated to underlying microstructure.

Initial analysis using optical microscopy showed indiscernible and complex microstructure. However, SEM images (see Fig.A.3c) from the steel clearly shows bainitic type lath structures dispersed among a martensitic microstructure. The prior austenite grain boundaries are clearly visible along with un-dissolved carbide particles.

Fig. A.3a: Recorded thermal profile of the AISI 4130 pipe sample when subjected to FP
Fig. A.3b: SSDTA results showing two-phase transformation events in 4130-steel during cooling cycle between (a) 650°C to 550°C and (b) from 459°C to 359°C

Energy dispersive spectrum (EDS) analysis of the carbides shows chromium enrichment. Some of the prior austenite grain boundaries are delineated in Fig. A.3c. Based on these analyses, the austenite grain sizes are estimated to be in the order of 10 µm. These small austenite grain sizes indicate that the dwell time in the austenitizing temperature regime is extremely short.

Since it is practically impossible to evaluate the microstructural heterogeneity through the series of microscopy techniques throughout the sample, hardness mapping was adopted as a measure of the microstructural
heterogeneity [93]. In this method, series of hardness indents were made on the samples and the mechanical heterogeneity [110] is estimated by analyzing the hardness distribution.

Fig.A.3c: SEM- Microstructure of 4130 Flash processed sample which is very similar to that of 8620 Flash processed sample.

Microhardness data, measured as a function of spatial coordinates, was transformed into an image format (see Fig.A.3d). The map shows a uniform distribution of lower hardness (500-550 VHN) in a matrix of predominantly higher hardness (600-650 VHN). These regions are referred as LHR and HHR in this paper, from this point forward. A histogram Fig.A.3e of all the hardness data shows two peaks in the hardness distribution, a smaller
peak (~525 VHN) with a relatively lower hardness and a larger peak (~625 VHN) of higher hardness. The above analyses indicate that the LHR and HHR regions lie within a specific range of hardness values. This relatively intimate mixture of hard and soft regions in the microstructure is believed to give the steel improved mechanical properties.

Fig.A.3d: Hardness contour map of a cross section of FP 4130 tube sample, showing the distribution of hardness;
Fig.A.3e: Histogram plotted from the same data shows a large peak (600-650 VHN) and a small peak (500-550 VHN)

It will be of importance to correlate these LHR and HHR regions to underlying microstructure. Due to the difficulty of extracting the steel regions under these indents, a theoretical approach is considered in this paper. In the literature, empirical relations exist to correlate microstructure to hardness. These equations were developed by regression analyses by measuring the hardness of a sample with well-controlled microstructure and hardenability theories [110]. Theses microstructures are considered to form during the cooling of steels from the austenitizing temperature. The following are the formulas developed for evaluating the hardness of the martensitic and bainitic microstructures. These equations are valid when the cooling rate
experienced by the system is below 500 °C/s. For example, hardness of martensitic microstructure (in HV) is given by equation (23)

\[ HV = 127 + 949 \cdot C + 27 \cdot Si + 11 \cdot Mn + 8 \cdot Ni + 16 \cdot Cr + 21 \cdot \log(CR) \] (23)

Similarly, the hardness of the bainitic microstructure (in HV) is given by:

\[ HV = \{89 \cdot +53 \cdot -55 \cdot -22 \cdot -10 \cdot -20 \cdot -33\} \cdot \log(CR) + 185 \cdot C - 323 \\
+ 330 \cdot Si + 153 \cdot Mn + 65 \cdot Ni + 144 \cdot Cr + 191 \cdot Mo \] (24)

In the equations (23) and (24), the elemental symbols represent the concentration (wt.%) of that particular alloying element and CR represents the cooling rate (K/hr). Using these standard hardness equations and by assuming a candidate microstructure distribution, the carbon content of the steel sample can be back calculated. If the candidate microstructure is correct, then the calculated carbon content should match with the nominal concentration of the steel. In this case, the alloy composition (in wt.%) is equal to 4130 grade of steel composition given earlier. The cooling rate involved in FP is 203.8 °C/s. This is estimated by measuring the time taken to quench from the highest temperature of 1150 °C to 200 °C (see Fig.A.3a). For such high cooling rates, paraequilibrium mode of phase transformation is possible. Considering such a mode of transformation, only the carbon would redistribute between bainitic ferrite and austenite, after every sub-unit of bainitic ferrite forms, while other alloying elements are configurationally...
frozen. Using the equations 23 and 24, the carbon concentration from both the martensitic (1) and bainitic microstructures (2) is calculated using the hardness values from LHR and HHR regions, as well as, the measured cooling rate. Table A.1 shows the calculated weight percent of carbon for each type of microstructure based on the measured hardness value.

<table>
<thead>
<tr>
<th>Assumed microstructure</th>
<th>Carbon (wt.%) in HHR.</th>
<th>Carbon (wt.%) in LHR.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Martensite</td>
<td>0.3633</td>
<td>0.258</td>
</tr>
<tr>
<td>Bainite</td>
<td>0.7308</td>
<td>0.5358</td>
</tr>
</tbody>
</table>

Table A.1: Estimated carbon concentration of LHR and HHR with assumed microstructure

With the observed hardness distribution, three possible scenarios could be possible to ascribe the microstructure for these region as following, (1) LHR and HHR contain only martensitic microstructure, (2) both LHR and HHR contain bainitic microstructure, or (3) LHR contains purely bainitic and HHR is purely martensitic. In order to select the appropriate microstructure, the bulk carbon content of 4130-steel was back calculated. From the hardness histogram (see Fig. A.3e), the average hardness of the LHR and HHR regions are taken as 525 HV and 625 HV, respectively. The volume fraction of LHR and HHR was calculated using by the frequency of the hardness data as
shown in Fig.A.3e (frequency in LHR = 190 and frequency in HHR = 900).

Using weighted average of the calculated carbon concentration in Table A.1, the estimated bulk carbon concentration is given in Table A.2 for the three scenarios discussed above.

<table>
<thead>
<tr>
<th>Scenarios</th>
<th>Microstructure in the LHR</th>
<th>Microstructure in the HHR</th>
<th>Wt.% C in HHR</th>
<th>Wt.% C in LHR</th>
<th>Calculated wt.% C in Bulk</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Martensite</td>
<td>Martensite</td>
<td>0.3633</td>
<td>0.258</td>
<td>0.345</td>
</tr>
<tr>
<td>2.</td>
<td>Bainite</td>
<td>Bainite</td>
<td>0.7308</td>
<td>0.5358</td>
<td>0.723</td>
</tr>
<tr>
<td>3.</td>
<td>Bainite</td>
<td>Martensite</td>
<td>0.3633</td>
<td>0.5358</td>
<td>0.393</td>
</tr>
</tbody>
</table>

Table 2: Bulk carbon concentration obtained by the carbon concentration and volume fraction of LHR and the HHR

For AISI 4130 grade of steels, the carbon concentration lies in the window of 0.28 to 0.33 wt.% The calculations suggest that the underlying microstructure should be a mixture of martensite with two different carbon concentrations. However, this conclusion is not consistent with microstructural data and other mechanical property measurements. The other possibility is that the underlying microstructure could a mixture of bainite and martensite (scenario 3). This is qualitatively supported by the above calculations considering the fact that only the peak value of the hardness distribution is considered and not the whole distribution. For lower values of hardness the value of the carbon in that region would be lower. This
would lead to lowered bulk carbon concentration, which would be more consistent with the carbon concentration of 4130-steel. Theoretically, it is possible to get such a microstructure if the regions being quenched have different carbon concentration. Such conditions may arise, if the steel is being heated rapidly and then quenched before sufficient time for complete homogenization of carbon in the austenite phase field. Rapid heating rate may also affect the Ac1 and Ac3 temperature. Neural network analysis [105] and Gaussian processing modeling [106] have shown that the Ac1 temperature increases as the heating rate increases. This increase in Ac1 temperature can also been rationalized based on the need to nucleate austenite phase from a ferrite microstructure. Therefore, the rapid heating rate involved in FP should lead to an increase in Ac1 above the equilibrium value. Similar arguments can be made for an increase in Ac3 temperature due to reduced time for diffusion-controlled growth of austenite into a ferrite matrix. Such an increase in Ac1 and Ac3 temperatures will also affect the carbide dissolution, since the presence of austenite is necessary for the initiation of dissolution. In addition, the time taken for austenite grain growth will also be reduced. This is supported by the small prior austenite grain size (<20 μm) measured in the flash processed samples. Further research with computational thermodynamic and kinetic calculations is necessary to describe the microstructure evolution in the flash processing of steels.
A.4 Conclusions

Flash processing of steels leads to formation of steels with high YS (>1350MPa), UTS (>1600MPa) and elongations (>7%) comparable to that of AHSS type of steels. SSDTA analysis of the cooling curve of the FP treatment shows two transformations taking place. These transformations are interpreted as the formation of bainite (higher temperature thermal event) and martensite (lower temperature thermal event). Optical and SEM images show complex microstructures consisting of bainitic lath structures along with martensite and un-dissolved carbides. Micro hardness tests over the surface of the 4130 pipe shows that the flash processed sample contains a minor fraction (~17%) of soft microstructure interspersed within a harder microstructure. This unique distribution of relatively soft and hard microstructure is correlated to the high strength with good ductility.
APPENDIX B

Development of rapid heating and cooling (flash processing) process to produce advanced high strength steel microstructures

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Materials Science and Technology, - Online Article
Development of rapid heating and cooling (flash processing) process to produce advanced high strength steel microstructures

T. Lolla¹, G. Cola², B. Narayanan³, B. Alexandrov⁴ and S. S. Babu¹

Flash processing of an AISI8620 steel sheet, which involves rapid heating and cooling with an overall process duration of <10 s, produced a steel microstructure with a high tensile strength and good ductility similar to that of advanced high strength steels. Flash processed steel [ultimate tensile strength (UTS): 1694 MPa, elongation: 7-1%], showed at least 7% higher UTS and 30% greater elongation than published results on martensitic advanced high strength steel (UTS: 1585 MPa, elongation: 5-1%). The underlying microstructure was characterised with optical, scanning electron, transmission electron microscopy as well as hardness mapping. A complex distribution of bainitic and martensite microstructures with carbides was observed. A mechanism for the above microstructure evolution is proposed.

Keywords: Flash processing, Advanced high strength steels, Heat treatment, Bainite, Phase transformation, Microstructure characterisation

Introduction

There is a need for weight reduction of automotive structures in order to achieve improved fuel efficiency, while not compromising the safety of passengers. To meet these two demands, a new class of steels known as advanced high strength steels (AHSS) was developed by the ultra light steel automotive body advanced vehicle concept consortium. This family of steels shows good formability while maintaining very high strength values [engineering yield strength (YS)>300 MPa and ultimate tensile strength (UTS)>700 MPa]. These AHSS grades include dual phase, transformation induced plasticity, complex phase and partially martensitic steels. All these steels achieve their mechanical properties by engineering the fractions of ferrite, bainite and martensite microstructure as well as austenite phase. This is achieved either through energy intensive thermomechanical processing steps and/or by expensive alloying additions (e.g. manganese).

The need to focus on the area of advanced steel development is imminent. This is demonstrated by research directions of many researchers throughout the world. In addition to the production of these steels, there exists a critical need to develop reliable and robust processes for forming and welding of these steels too. The above examples are not a comprehensive representation of the published literature. However, it proves that there is an impetus to develop processing techniques to produce AHSS.

The present paper pertains to an innovative heat treatment procedure that shows a potential as an alternative route for the current production of AHSS steels. This process has been termed as ‘flash processing’ for the incredibly short time (<10 s) for heating and cooling of the steel sheets. Throughout the present paper this term or an acronym ‘FP’ will be used to denote steels that are processed using this method. Preliminary work has shown that FP of plain carbon steels (>0.15C) may lead to high YS (>1200 MPa), tensile strength (>1500 MPa) and appreciable ductility (>7%). As a result, FP processed steels could be classified under the category of AHSS.

Curious evaluation of the above claim leads to skepticism, since it contradicts the well established theories of phase transformation and microstructure evolution in steels. Heating of steels to the homogenous austenite phase field and rapid quenching will lead to the formation of martensite. During tensile testing of this martensitic microstructure, no appreciable plastic strain is expected. Therefore, the objective of the present paper is to relate the observed thermal cycle, microstructure and the observed properties by developing a mechanistic understanding of the microstructure evolution.

Experimental

Steel composition and initial condition

In earlier work, Cola has investigated many steels including AISI8620 steels by FP. In the current paper, to limit the effect of the steel compositions on the interpretations of the microstructure and property correlations, only AISI8620 grade steel sheets were
Development of rapid heating and cooling – Flash processing

A schematic of the process set-up is shown in Fig. 1. The assembly consists of a pair of rollers that transfers the steel sheets through a heating and cooling stage. At a controlled distance from the top pair of rollers, the heating stage is placed. The heating stage is based on either oxy propane flame or electrical induction heating. The flame heating consists of 17 flame nozzles spaced evenly to spread the heat over the steel sheets. The above set-up has also been equipped with infrared pyrometers, instrumentation to drive the feed rollers, mechanical fixtures for positioning heating and cooling units, and heating intensity controls. Spot temperature check using an Inca infrared pyrometer showed a small variation of temperature of approximately ±10°C from the middle to the edges of the sample. Immediately below the heating port, a cooling trough is placed. To avoid inefficient heating due to the upward flow of steam, a graphite separator film is used to separate the heating and cooling units, and heating intensity controls.

Thermal cycle measurements and analyses

Cola et al. calculated thermal cycles in different regions using infrared pyrometers and imposed feedrates of steel sheets. However, the detailed temporal variations of temperature were not measured using contact thermocouples. Such measurements are necessary to develop a mechanistic understanding of microstructure evolution during FP. However, the oxy propane flame heating method does not lend itself for the direct measurement of temperature due to possible deterioration of thermocouples by the high intensity flame. Therefore, the induction heating method was used during temperature measurement experiments. In order to impose the similar heating conditions as that of the flame heating, feedrates, temperature gradients and the peak temperature were controlled to be identical. Type K (Chromel–Alumel) thermocouples were attached to the middle of the steel sheets. The temperature of the steel was measured, as the sheet traverses through the heating and cooling ports. These measurements are made with a high speed data acquisition system capable of recording temperature at a sampling rate of up to 5 KHz. The measured thermal cycles were analysed to determine the instantaneous heating and cooling rates throughout the FP. In addition, ferrite (\(\alpha\)) to austenite (\(\gamma\)) transformation was evaluated by analysing thermal cycle data using single sensor differential thermal analysis (SSDTA) technique. The sensitivity and accuracy of SSDTA technique, in measuring \(\alpha\) to \(\gamma\) transformation during high heating rates (\(\sim 500 \text{ K s}^{-1}\)), has been documented earlier by direct comparison with dilatometric techniques.

Mechanical property measurements

In order to evaluate the spatial variations of steel microstructures, two-dimensional hardness distributions were measured in the cross-section of samples. For each sample, >2000 indents were made on QT and FP samples using an AMH43 automatic hardness testing system with a load of 300 g. The measured hardness distributions were analysed in a map format and using frequency distribution curves to provide quantitative measure of mechanical heterogeneity of the samples.

Tensile samples were extracted from the edge regions and from the middle regions. Tensile tests were performed along the longitudinal direction of the coils. The gauge length of the test samples was 25 mm. The width of the samples at the gauge length was 6.3 mm. The tests were performed at room temperature with a crosshead displacement rate of 1.27 mm min\(^{-1}\). All the tensile testing were performed using testing machines available at Edison Welding Institute. Similar tests were repeated in QT samples also.

Microstructure characterisation

Standard optical microscopy was used to characterise microstructure from the FP and QT samples. In the next step, the samples were characterised using a Quanta200 scanning electron microscope (SEM), equipped with secondary electron, backscattered and X-ray energy dispersive spectroscopy (EDS) detectors. The microscope was operated at an accelerating voltage of 25 kV.

Transmission electron microscopy (TEM) samples were prepared by electrolytic thinning of 3 mm discs of 90 μm thick samples. A chemical solution of 33% Nitric acid, 67% Methanol was used as the electrolyte. The polishing was carried out at 25 V at a current of 10–15 mA and the temperature of the electrolyte was maintained at \(-25\) °C. The samples were imaged using two types of transmission electron microscopes. General microstructure and electron diffraction analyses were performed in a Philips CM12 transmission electron microscope operating at 120 kV. In addition, high angle annular dark field (HAADF) and bright field imaging was performed using STEM technique in a Tecnai F-20 FEG/TEM operating at 200 kV.
Computational modelling
ThermoCalc\textsuperscript{14} software with TCFE5 database was used to estimate the thermodynamic quantities and equilibrium phase transformation temperatures. In addition, the time temperature transformation (TTT) diagrams for the initiation of reconstructive and displacive transformations were predicted using the methodology developed by Bhadeshia.\textsuperscript{15,16} The TTT data were then converted into a continuous cooling transformation (CCT) diagram using the methodology developed by Babu and Bhadeshia.\textsuperscript{17} The source code for these calculations can be downloaded from an internet location\textsuperscript{18} and example calculations can be made using an online calculator.\textsuperscript{19}

Results and discussion

Calculated phase transformation characteristics
In order to analyse the results from the FP experiments with reference to expected transformation behaviours, the transformation temperatures were calculated using the methodologies described before\textsuperscript{14–19} for the AISI8620 steel composition. The equilibrium $A_1$ and $A_3$ temperatures of this steel were calculated to be 702 and 806°C respectively. In addition, the calculated equilibrium volume fractions of cementite (M\textsubscript{6}C\textsubscript{3}) and alloy carbides (M\textsubscript{23}C\textsubscript{6} and MC) that can form in these steels as a function of temperature are shown in Fig. 2a. This result shows that as the steel heats above 810°C, it should transform to 100% austenite.

The bainitic and martensitic start temperatures\textsuperscript{15,16} were calculated to be 553 and 426°C respectively. Calculated TTT and CCT diagrams for the initiation of paraequilibrium (transformation controlled by carbon partitioning and the ratio of substitutional to iron atoms are configurationally frozen in place on either side of austenite and ferrite transformation interface) reconstructive mode of austenite to allotriomorphic ferrite transformation and displacive mode of austenite to Widmanstätten and bainitic transformations are shown in Fig. 2b. Since the FP involves rapid cooling process, the assumption of paraequilibrium is indeed valid. Based on the above results, one can expect complete transformation of austenite to 100% martensite in AISI8620 steels at cooling rates faster than 70 K s\textsuperscript{-1}. These predictive methodologies have been developed using a wide range of steels and their TTT data presented in the literature.\textsuperscript{16,20} Therefore, the predictions are expected to be fairly accurate for low alloy steels including AISI8620 grades. For example, these calculations have accurately predicted the transformation start during continuous cooling of low alloy steels.\textsuperscript{21}

Thermal cycles during FP
A typical heating and cooling thermal cycle during FP is shown in Fig. 3. The plot also shows the heating and cooling rates as a function of time. These thermal profiles can be divided into four regions based on the temperature range. In region I, the steel temperature increases gradually due to heat conduction from the hot stage. In region II, the steel temperature increases rapidly due to its proximity to heat source. In this region, maximum heating rate of 410 K s\textsuperscript{-1} was observed at 780°C. This temperature is between the calculated $A_1$ and $A_3$ transformation temperatures for AISI8620 steel. Above 780°C, the heating rate starts reducing indicating that there is a dynamic equilibrium between the heat flux, the heat conduction, and endothermic/exothermic effects due to phase transformations.\textsuperscript{12} In region III, the heating rate reaches zero when the sample reaches a peak temperature of 1100°C. As per the equilibrium thermodynamic calculations, at this temperature, the sample should be 100% austenite. After reaching the peak temperature, the sample starts cooling down gradually. The total dwell time for this slow cooling near 1100°C is measured to be 2 s. In region IV, within 1-2 s of reaching peak temperature, the sample reaches the water bath and starts cooling rapidly. The maximum cooling rate of 3150 K s\textsuperscript{-1} is achieved when the sample reaches a temperature of 393°C. This temperature is much below the calculated $B_6$ (553°C) and $M_6$ (426°C) temperatures.\textsuperscript{13} As per the measured thermal cycles and the calculated CCT diagram (see Fig. 2b), the austenite phase from high temperature should transform to 100% martensite during FP. Figure 4 shows the complete thermal profile of two separate FP treatment runs recorded from thermocouples attached at the centre of the sheet. This shows the thermal cycle repeatability that can be achieved in FP.

Alexandrov and Lippold\textsuperscript{12} have developed the SSDTA software to detect transformation temperatures from measured thermal cycles, in a well described (Newtonian, Gaussian or Rosenthal type) heat transfer

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{diagram.png}
\caption{a) calculated variation of carbide volume fraction as function of temperature obtained using ThermoCalc\textsuperscript{14} and TCFE5 database and b) TTT and CCT diagrams showing reconstructive transformation and displacive transformation regions}
\end{figure}
condition. The transformation temperatures are detected by looking for a departure of measured temperature from a reference heating (or cooling) curve due to endothermic (or exothermic) effects during phase changes. Figure 5 shows the results of the SSDTA analyses of thermal profiles, for the heating regime only. In these analyses, the reference curves were described by a polynomial function that describes a well controlled heat transfer conditions.

Both the results show a small endothermic phenomenon around the calculated Curie temperature ($\approx 750^\circ$C) of bcc ferrite and another large endothermic peak $\approx 900^\circ$C. These large endothermic peaks are interpreted as $AC_1$ (926 and 930°C) and $AC_3$ (1046 and 1052°C) temperatures by SSDTA software. The reproducibility of these measurements is within the 0-4% accuracy of the type K thermocouple. The accuracy of these analyses is also affected by the electromagnetic noise generated by the induction heating. It is noteworthy that SSDTA technique estimates a higher and non-equilibrium $AC_1$ and $AC_3$ temperatures, in comparison to equilibrium $A_1$ (702°C) and $A_3$ (806°C) temperatures. Possible mechanisms for these are discussed later.

**Hardness and tensile properties**

Measured engineering tensile properties are presented in Table 1. Table 1 also shows the tensile properties of the FP samples tested by Cola$^{10}$ and the current work. The data show that the flash processed AISI8620 steels have yield and tensile strengths greater than 1300 and 1500 MPa respectively. In addition, the elongation and reduction in area indicate appreciable ductility in these
The ductility of 8.8-9.9% is indeed greater than the levels reported for martensitic AHS steels. The UTS of the FP tensile specimen is comparable to that of the QT sample; however, the FP samples, consistently, show higher elongation values.

In order to evaluate the formability of FP steels, the ratio of YS and UTS and true stress–true strain characteristics were calculated (see Fig. 6). The strain hardening exponent $n$ and pre-exponent strength coefficient $K$ for uniform plastic strain were estimated by fitting the following equation to the plastic regime of the true stress–strain curve.

$$\sigma = K \cdot e^n$$  \hspace{1cm} (1)

The calculated $K$ and $n$ values (see Table 2) are similar values to the published grades of AHSS steels for dual phase and complex phase steels. The measured UTS and YS from the current work are overlaid in a map of current AHSS steels. This comparison confirms that the flash processed steels (see Fig. 7) are indeed a part of AHSS steel family. It is interesting to note that both UTS and the elongation are higher than the reported values for AHSS based on a martensitic microstructure.

### Table 1 Measured engineering tensile properties of FP (from previous and current work) and QT samples (current work).

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Ultimate tensile strength, MPa</th>
<th>0.2% yield strength, MPa</th>
<th>Reduction of area, %</th>
<th>Elongation, %</th>
<th>Ratio of YS to UTS</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ref-FP1</td>
<td>1665.77</td>
<td>1241.75</td>
<td>...</td>
<td>6.3</td>
<td>0.737</td>
<td>Ref. 10</td>
</tr>
<tr>
<td>Ref-FP2</td>
<td>1694.04</td>
<td>1314.83</td>
<td>...</td>
<td>7.3</td>
<td>0.776</td>
<td>Ref. 10</td>
</tr>
<tr>
<td>Ref-FP3</td>
<td>1676.12</td>
<td>1275.53</td>
<td>...</td>
<td>7.1</td>
<td>0.761</td>
<td>Ref. 10</td>
</tr>
<tr>
<td>Ref-FP4</td>
<td>1669.91</td>
<td>1292.77</td>
<td>...</td>
<td>6.9</td>
<td>0.774</td>
<td>Ref. 10</td>
</tr>
<tr>
<td>FP no. 1</td>
<td>1664.1</td>
<td>1442.8</td>
<td>39.4</td>
<td>8.8</td>
<td>0.867</td>
<td>Present paper</td>
</tr>
<tr>
<td>FP no. 2</td>
<td>1619.3</td>
<td>1386.9</td>
<td>38.0</td>
<td>9.9</td>
<td>0.856</td>
<td>Present paper</td>
</tr>
<tr>
<td>FP no. 3</td>
<td>1620.7</td>
<td>1300.0</td>
<td>38.3</td>
<td>9.9</td>
<td>0.855</td>
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</tr>
<tr>
<td>QT no. 1</td>
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<td>1464.1</td>
<td>49.8</td>
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<td>QT no. 3</td>
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<td>1487.6</td>
<td>48.8</td>
<td>4.3</td>
<td>0.905</td>
<td>Present paper</td>
</tr>
</tbody>
</table>

*Sample dimensions used in Ref. 10: width 13 mm; thickness 1.5 mm. Sample dimensions in the present work: width 6.4 mm; thickness 1.23 mm.

### Table 2 Calculated pre-exponent $K$ and strain hardening exponent $n$ derived by fitting equation (1) to plastic regime of true stress–strain curve

<table>
<thead>
<tr>
<th></th>
<th>FP no. 1</th>
<th>FP no. 2</th>
<th>FP no. 3</th>
<th>QT no. 1</th>
<th>QT no. 2</th>
<th>QT no. 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>$K$, MPa</td>
<td>569.6</td>
<td>622.1</td>
<td>613.7</td>
<td>1305.6</td>
<td>536.3</td>
<td>543.9</td>
</tr>
<tr>
<td>$n$</td>
<td>0.1542</td>
<td>0.1568</td>
<td>0.1348</td>
<td>0.1950</td>
<td>0.1342</td>
<td>0.1471</td>
</tr>
</tbody>
</table>
Microstructure of heat treated samples

In this section, the underlying microstructure in the original steel sheets before processing and after the FP and QT heat treatments are presented.

Initial microstructure of annealed AISI8620 steel sheets

Optical microscopy of original steel sheets showed the presence of ferrite grains with carbides (Fig 8a). Most of the carbides were <1 μm and some of them were >2 μm, suggesting a bimodal nature of carbide distribution. These carbides also appear to be aligned in between pancaked ferrite structure suggesting that these could have precipitated during legacy thermomechanical processing of these sheets. Scanning electron microscopy (see Fig. 8b) with backscattered imaging showed that most of these carbides were associated with ferrite grain boundaries. Energy dispersive X-ray spectroscopy analyses (see Fig. 8c) of these carbides show a higher ratio of Cr/Fe peak intensities compared to that of ferrite.

7 Overview of published tensile strength and ductility that can be achieved for a wide range of advanced high strength steels and measured tensile strength and ductility of flash processed steels

8 a optical image microstructure of AISI8620 unprocessed sample showing extensive carbide distribution, especially along grain boundaries, in ferrite matrix, b SEM backscattered image showing presence of carbides along grain boundaries in microstructure of AISI8620 unprocessed sample and c comparison of EDS signals obtained from matrix and carbide particle observed in AISI8620 unprocessed sample (signals from particle show higher Cr/Fe ratio indicating that particle could be chromium carbide)
matrix. Based on the above analyses and thermodynamic calculations, the carbides are assumed to be Cr rich cementite (M₇C₃).

**Microstructure of QT AISI8620 steel sheets**

Since the QT and FP microstructures showed similar properties, the microstructure of the QT samples was characterised with optical microscopy and scanning electron microscopy. Owing to the fine nature of the microstructure, optical microscopy (see Fig. 9a) could not discern the martensitic and bainitic microstructures. The scanning electron microscopy (see Fig. 9b) with backscattered electron imaging revealed the martensitic lath structure with brightly imaging carbides. By tracing the lath packet boundary, typical prior austenite grain size was estimated to be around 20–30 μm. Owing to the short duration of the austenitising time (2 min at 1000°C), extensive austenite grain growth was not observed. A quantitative distribution of the austenite grain size in the overall sample was not performed due to the difficulty of identifying all the prior austenite grain boundaries by this technique. A backscattered electron diffraction technique with crystallographic orientation analyses is required and is the focus of the ongoing work.

The volume fraction and size of carbides in the QT samples is smaller than that of the untreated samples (see Fig. 8b). It is possible that the microstructure may contain nanometer sized carbides that formed during the tempering (220°C for 4 min) stage. Usually the sizes of these carbides that form during early stages of tempering are below the resolution of scanning electron microscopy. However, it is fair to conclude that initial carbides that are present in the untreated samples have dissolved during austenitising at 1000°C for 2 min.

**Microstructure of flash processed AISI8620 steel sheets**

Since the focus of this paper is related to FP, extensive characterisation was performed on FP samples. Optical microscopy (see Fig. 10a) of the FP samples again showed features that were difficult to distinguish. However, scanning electron microscopy with backscattered electron imaging revealed interesting features (see Fig. 10b). Fine lath structures with brightly imaging particles could be observed. By tracing the lath packet boundary (see Fig. 10b), typical prior austenite grain size was estimated. This qualitatively shows that the prior austenite grain size of FP samples (<10 μm) is much finer than that of the (20–30 μm) QT samples. This could be attributed to the rapid heating and cooling above the Ac₃ temperature. In the next step, the composition of carbides was evaluated with EDS analyses. The measured spectrum and the calculated composition of analysed regions are presented in Fig. 10c. Surprisingly, similar to the original steel samples before processing, these carbides were found to be rich in Chromium. In addition coarser and finer carbides were also observed which is similar to untreated samples. Since the FP process involves rapid heating (>400 K s⁻¹) and cooling (>3000 K s⁻¹) rates, these carbides are interpreted as the undissolved carbides that were present in the original base material.

With cursory observation of optical and scanning electron micrographs, one may conclude that the FP samples are predominantly martensitic. However, to substantiate this conclusion, detailed transmission electron microscopy was performed. A low magnification TEM image (see Fig. 11a) shows a prior austenite/austenite (γ/γ) grain boundary with presence of fine scale martensitic laths with high dislocation density and coarser bainitic ferrite with slightly reduced dislocation density. The identification of bainitic ferrite was made based on the size of these plates and the presence of sheave-like morphology with subunits as shown by Bhadeshia.

In another region of the same TEM sample (see Fig. 11b), three bainitic (marked as 1, 2 and 3 in the images) sheaves were observed. It is interesting to note that each and every individual sheave is made up of many ferrite subunits with similar orientation in space. Electron diffraction analyses failed to identify any retained austenite film between these subunits. Crystallographic orientation relationships between these bainitic sheaves were calculated and the summary of the results is presented in inset diffraction patterns in Fig. 11b. The sheaves nos. 1 and 2 are indexed to be in the [111] bcc zone axis with a relative misorientation...
of only 10 degrees. Assuming that these bainitic sheaves have a Kurdjumov–Sachs/Nishiyama–Wassermann (KS/NW) orientation relationship, with the parent austenite (fcc) phase, one can conclude that the bainitic sheaves nos. 1 and 2 could have formed a single austenite \( \gamma_A \) grain, within the regions that are separated by small angle boundaries. In contrast, the sheave no. 3 was indexed to be close to the \([011]\) zone axis. Using the axis angle pair analyses, the relative orientation between no. 3 and no. 1 or between no. 3 and no. 2 was found to be of high angle type. Using the same assumption of KS/NW orientation relationship of bainitic sheaves with austenite and symmetry relations, one can also conclude that the no. 3 sheave formed in adjacent austenite grain \( \gamma_B \) and the orientation relationship between \( \gamma_A \) and \( \gamma_B \) should be of high angle type. Copious presence of such austenite grains with large angle boundaries will promote bainitic and/or martensitic plates with wide varying orientations and may improve the properties. It is stressed that the above results are typical and one cannot conclude similar crystallographic conditions throughout the FP sample. To evaluate this throughout the sample, we need to do extensive backscattered electron diffraction analyses and correlate the orientation distribution functions to the properties, which is the focus of the ongoing work.

In another FP sample, HAADF STEM image showed extensive carbide distributions. Some of them were associated with the ferrite subunit grain boundaries (see [Image](image.png)).
Size of these carbides was \( \approx 100 \text{ nm} \). Elongated carbides between the lath boundaries are probably cementite. The precipitates are predominantly darker than the matrix suggesting that their average atomic number is lower than the iron matrix. However, the precipitate thickness effect is convoluted with the atomic number contrast and further work is required to establish the chemical composition of these precipitates. Some of the large carbides (marked by arrow) show no correlation to the ferrite boundaries (Fig. 12b). The EDS analyses show that these carbides were rich in silicon and chromium (Fig 12c). The detailed analyses of elemental distribution within these carbides can only be attained by atom probe field ion microscopy.\(^{24,25}\) Owing to the rapid rate of heating and cooling, the diffusion controlled dissolution and/or growth is expected to be sluggish. Therefore, these large carbides are interpreted as the undissolved carbides from the original steel sheets that existed before processing. From the above TEM analyses one can conclude that the FP microstructures contain bainitic ferrite, martensitic laths as well as uniform distribution of large and small carbides.

**Evaluation of mechanical heterogeneity in heat treated samples**

Although, the FP samples showed this unique mixture of bainite, martensite and carbide microstructure, it is necessary to evaluate the heterogeneity of these microstructures within the overall sample. Since it is practically impossible to evaluate the microstructural heterogeneity through the series of optical, SEM and TEM techniques throughout the sample, hardness mapping was adopted as a measure of the heterogeneity.\(^{17}\) With this method, series of hardness indents were made on the samples and the mechanical heterogeneity\(^{11}\) is estimated by analysing the hardness.
distribution. The average size of the indents in the FP sample was 42 μm which is bigger than the prior austenite grain size and also the bainitic and martensitic packet sizes. It is highly possible that the different microstructure distribution may provide similar hardness values. Therefore, the above measure has to be used only as a way to evaluate the possible scatter in final mechanical properties. The mechanical heterogeneity results from QT and FP samples are discussed below (see Figs. 13–16).

The hardness maps from the cross-section of QT (Fig. 13) sample showed a large soft region (blue colour) in the edge 1 region. In addition, the hardness variations are larger in the QT sample. This is attributed to the inefficient quenching during manual insertion of the austenitised sample into the water. This is also reflected in the results of the tensile tests, where QT samples showed low YS (see Fig. 6a and Table 1) and high strain hardening exponent. The hardness distribution curve from the QT sample is shown in Fig. 14. The above data were interpreted by fitting multiple peaks to the distribution. The fitted peaks show that the QT sample can be divided into two regions, i.e. large (ν_{QT1}=88%) region 1 with an average hardness of 475±47 HV with large scatter and a small (ν_{QT2}=12%) region 2 with an average hardness of 483±5 HV with small scatter.

In contrast, the mechanical heterogeneity of the flash processed samples was less than QT samples (see Figs. 15 and 16). The image also shows that a slightly

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**13** Hardness contour map of cross-section of QT sample cut into three pieces showing distribution of hardness as measured from over 2000 micro-indent points over cross-section area.

**14** Histogram plotted from hardness values observed in cross-section of QT sample, shows large scatter peak (ν_{QT1}=88%) and small scatter peak (ν_{QT2}=12%).

**15** Hardness contour map of cross-section of FP sample cut into three pieces showing distribution of hardness as measured from over 2000 micro-indent points over cross-section area.

**16** Histogram plotted from hardness values observed in cross-section of FP sample shows three distinct regions of different hardness range; 498±7, 512±11 and 534±9 HV.
harder region (yellow colour) is in the middle of the FP steel sheet compared to the edge of the steel sheet. The reader may recall the description of the FP process setup (see Fig. 1) in which the middle of the steel sheet gets exposed to the highest heat density, as a result of highest heating rate and high peak temperature. Similar to QT sample analyses, the calculated hardness distribution was analysed by fitting multiple peaks to the distribution. The fits show that the QT sample can be divided into three distinct regions, i.e., region 1 (\(v_{\text{FP1}}\) 14%) with an average hardness of 498 ± 7 HV, region 2 (\(v_{\text{FP2}}\) = 74%) with an average hardness of 512 ± 11 HV and region 3 (\(v_{\text{FP3}}\) = 12%) with an average hardness of 534 ± 9 HV. This presence of distinct and homogeneous mixing of soft and hard regions in the FP sample is tentatively attributed to the improved mechanical properties of the flash processed steels.

**Mechanism of microstructure evolution during FP**

Based on the above results, one can conclude that the FP leads to a complex microstructure that contains martensite, bainite and carbides. In addition the above complex microstructure also leads to a preferred mechanical heterogeneity that is better than the reference QT sample. In addition, the FP steels can be classified under the category of AHSS based on the strength observed. Furthermore, the flash processed steels show at least 7% higher UTS and 30% higher elongation than published martensitic advanced high-strength steels show at least 7% higher UTS and 30% higher strength observed. Furthermore, the flash processed steels should be 100% austenite above 800°C. As per equilibrium thermodynamic calculations, the on heating carbide dissolution

As per equilibrium thermodynamic calculations, the 8620 steels should be 100% austenite above 800°C. From the measured thermal cycle during the flash process, the peak temperature was 1100°C. Therefore, the sample should have completely transformed into austenite at this temperature and all the carbides should have dissolved. However, electron microscopy showed that the carbides have not dissolved completely. This discrepancy is attributed to a very short dwell time in the austenitising region which limits the dissolution of carbides. This hypothesis is supported by the observation of fewer carbides in the QT samples, which were austenitised for 2 min at 1000°C before quenching. It is well known that, extended time in the austenite phase field will promote the complete dissolution of carbides by enhancing the substitutional element diffusion.

**On heating ferrite to austenite transformation**

Another possibility of such unique microstructure evolution can be attributed to an increase in \(A_{\text{C1}}\) and \(A_{\text{C3}}\) temperatures due to rapid heating rates (>400 K s\(^{-1}\)) during FP. This hypothesis is in agreement with neural network analysis and Gaussian process modelling. The level of superheating required to nucleate and grow the austenite phase depends on the alloy composition, initial microstructure and the heating rate. This increase in \(A_{\text{C1}}\) temperature also been rationalised with the need to nucleate austenite phase from a ferrite-carbide microstructure. On the other hand, if the initial microstructure contains retained austenite, there is no need for nucleation barrier. Since the initial microstructure of AISI8620 steel sheets contained only ferrite and carbide, there is a need to nucleate austenite. Therefore, the rapid heating rate should lead to an increase in \(A_{\text{C1}}\) temperature above equilibrium value. Similar arguments can be made for an increase in \(A_{\text{C3}}\) temperature due to reduced dwell time for diffusion controlled growth of austenite into a ferritic matrix. It noteworthy that the published research did not consider very high (>100 K s\(^{-1}\)) heating rates. Therefore, we need to consider the validity of the continued increase in \(A_{\text{C1}}\) and \(A_{\text{C3}}\) temperatures far above the equilibrium \(A_{\text{C3}}\) temperature. Recently, Elmer et al. have developed an overall transformation kinetic model for ferrite to austenite transformation formation in 1005 steel based on synchrotron diffraction measurements during a heating cycle of a weld. The model was calibrated using the data from low heating rates (<100 K s\(^{-1}\)). However, the model calculations were extrapolated to higher heating rates. The calculations suggest a superheating of 100°C temperature or more above the equilibrium \(A_{\text{C3}}\) temperature for the completion of austenite formation at heating rates >300 K s\(^{-1}\). This result supports the notion of non-equilibrium \(A_{\text{C1}}\) and \(A_{\text{C3}}\) temperatures during FP (heating rate >410 K s\(^{-1}\)). Moreover, the SSDTFA analyses of the heating curve (Fig. 5a and b) indicate that the \(A_{\text{C1}}\) temperature is definitely above 900°C, 925-7°C (Fig. 5a) and 929.6°C (Fig. 5b).

**On cooling austenite decomposition**

Based on the calculated CCT diagram and cooling rate (>3000 K s\(^{-1}\)) during FP, the steel should have transformed to 100% martensite. To evaluate the effect of carbon concentration gradients within the austenite matrix, which could trigger complex decomposition of austenite into different ferrite morphologies and martensite.

Based on the calculated CCT diagram and cooling rate (>3000 K s\(^{-1}\)) during FP, the steel should have transformed to 100% martensite. To evaluate the effect of carbon concentration gradients within the austenite matrix, which could trigger complex decomposition of austenite into different ferrite morphologies and martensite. This discrepancy is attributed to the inadequacies of transformation kinetic modelling methodologies. These models were developed using the TTT data generated by
traditional heat treatment conditions involving slow heating and isothermal hold at austenitising temperature exceeding 5 min and homogenous austenite. The extension of these models to austenite with inhomogeneous carbon concentration may not be reliable. These situations are similar to the rapid heating and cooling conditions experienced by heat affected zone of welds.

Based on the above results and discussions, a hypothetical microstructure evolution during FP is proposed (see Fig. 18). At room temperature, the initial microstructure contains ferrite and carbides (Fig. 18a). On rapid heating, the austenite nucleation is expected to occur at the carbide–ferrite boundaries and the austenite is expected to grow at the expense of ferrite and carbide dissolution. Owing to rapid heating rates, the temperature at which the austenite nucleation and growth occurs may be elevated to higher temperatures (>900°C) (Fig. 18b). But before the complete dissolution of cementite, the cooling cycle starts. At this juncture, the austenite is expected to have inhomogeneous distribution of carbon content (Fig. 18c). With the on-set of rapid cooling, the carbon enriched regions may transform to martensite and carbon depleted regions may transform to bainite or low carbon martensite. This leads to a final microstructure with complex distribution of bainite, martensite and carbides (Fig. 18d). This hypothetical mechanism suggests that the initial microstructure will have a strong effect on the final microstructure and properties achieved by FP. Ongoing experiments are focusing on evaluating these effects and methods to track the phase transformations during heating and cooling for a wide range of steels to prove or disprove this hypothesis.

Conclusions

A rapid heating (>400 K s⁻¹) and cooling (>3000 K s⁻¹) flash process has been developed that produces a microstructure with good combination of yield (1280 MPa), UTS (1600 MPa) and appreciable ductility of up to 9-9%. The above strength and ductility levels are significantly better than martensitic based AHSS (1400–1500 MPa UTS). Based on the above data, the flash processed steels can be classified as part of the advanced high strength steel family. As the overall processing time (<10 s) is very short, this process could be an alternative route for producing AHSS sheets.

The underlying microstructures in flash processed samples were characterised with optical and analytical electron microscopy. The characterisation results showed that the steel contains bainite, martensite and...
carbides. The hardness map analyses showed that the flash processed samples contain a minor fraction of soft microstructure interspersed within a harder microstructure. This unique distribution of soft and hard microstructure is correlated with the improved YS, UTS and ductility.

Traditional phase transformation models indicated that, for the cooling rates measured in the flash process, the microstructure should be 100% martensite after tempering to 77°C. This discrepancy is addressed with a theoretical mechanism that involves an increase in Ac1 and Ac3 temperatures due to rapid heating rate, incomplete dissolution of carbides and decomposition of austenite with non-uniform carbon concentration.

References