Effect of Microstructure on Static and Dynamic Mechanical Properties of Third Generation Advanced High Strength Steels

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ABSTRACT

The recent studies on steels have largely focused on the development of new advanced high strength sheet steels (AHSS), particularly for automotive applications. “First generation AHSS” are steels that primarily possess ferrite-based microstructures with tensile strength (in the as-rolled condition) in the range of 450 to 700 MPa, and “second generation AHSS” are austenitic steels with high manganese content in the range of 900 MPa to 1100 MPa tensile strength. Recently, there has been interest in the development of a “third generation” of AHSS, which are steels with strength-ductility combinations better than the first generation AHSS with a range of 20 000 MPa x %ε but at a cost significantly less than that of the second generation AHSS as a result of reducing expensive alloying elements. Therefore, the current approach to the development of third generation AHSS is to combine all the aspects of first and second generation steels in unique alloy/microstructure combinations to achieve the desired properties. Thus, the third generation of AHSS microstructures consists of a high strength phase (e.g., martensite or bainite) and a significant amount of ductility and work hardening from an austenite that exhibits deformation induced plasticity through transformation or twinning.

In this thesis, four different steel compositions, centered on Mn as the main alloying element, are designated as candidates for third generation AHSS grades. The design of these steels is based on controlling the deformation behavior of the retained austenite. Thus, heat treatment process parameters are determined in order to obtain different amounts and morphologies of retained austenite. The evolution of the microstructure, during processing as well as deformation, is characterized by using optical and electron microscopy techniques and mechanical tests. The effect of alloy composition and processing parameters on the deformation mechanisms of these steels is discussed.
RÉSUMÉ

Les études récentes sur les aciers se sont surtout concentrées sur le développement avancé de nouvelles feuilles d’acier à haute résistance (AHSS, advanced high strength sheet steels, en anglais), particulièrement pour les applications automobiles. Les “AHSS de première génération” sont des aciers qui possèdent principalement des microstructures à base de ferrite ayant une résistance à la traction (à l’état brut de laminage) de l’ordre de 450 à 700 MPa tandis que les “AHSS de seconde génération” sont des aciers austénitiques à haute teneur en manganèse ayant une résistance à la traction de l’ordre de 900 à 1100 MPa. Récemment, un intérêt s’est manifesté pour le développement “d’AHSS de troisième génération” qui sont des aciers ayant une résistance et une ductilité combinées supérieures aux AHSS de Première Génération de l’ordre de 20 000 MPa x ε%, mais à un coût nettement moindre que celui requis pour les AHSS de seconde génération, réduisant ainsi le recours à des éléments d’alliage coûteux. Conséquemment, l’approche actuelle pour le développement d’AHSS de Troisième Génération est d’unir tous les aspects de la première et de la seconde génération d’aciers en des combinaisons uniques d’alliages et de microstructures qui permettront d’atteindre les propriétés désirées. Ainsi, les microstructures d’AHSS de troisième génération sont constituées d’une phase à haute résistance (e.g. Martensite ou bainite) et d’austénite dont la ductilité et l’écrouissage sont importants et qui possède une plasticité induite par déformation suite à une transformation ou un maclage.

Dans cette thèse, quatre compositions d'aciers différents, centrés sur le Mn comme principal élément d'alliage, sont désignés comme candidats pour les grades d’AHSS de Troisième Génération. La conception de ces aciers est basée sur le contrôle du comportement à la déformation de l'austénite résiduelle. Par conséquent, les paramètres du procédé de traitement thermique sont déterminés de façon à obtenir différentes quantités et morphologies d'austénite résiduelle. L'évolution de la microstructure, au cours du traitement et de la déformation, est
caractérisée par microscopie optique et électronique et des tests mécaniques. L'effet de la composition de l'alliage et des paramètres de traitement sur les mécanismes de déformation des aciers est discuté.
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Chapter 1: General Introduction

The automotive industry has an on-going focus on decreasing fuel consumption and gas emissions. Light-weight automobiles have a great advantage in regards to reducing fuel consumption [1]. For this purpose, the steel industry presents several solutions for the automotive industry because steel is one of the most important materials that has the ability to adapt to changing requirements [2]. Additionally, steels are the material with the highest recycling rates [3]. So a wide variety of steel alloys create the opportunity for new and interesting applications, economically as well as technically.

In general, first generation (high strength low alloy (HSLA), dual phase (DP) and transformation-induced plasticity (TRIP) steels, etc.) and second generation (twinning-induced plasticity (TWIP)) advanced high strength steel (AHSS) demonstrate weight reduction plus significant improvements in strength, stiffness, and other structural performance characteristics [4, 5]. However, competition with lightweight materials makes it necessary to improve new generation AHSS to reach over 800 MPa with more than 20% elongation, at the same time as reducing the costs (i.e., amount) of alloying additions.

Hence, third generation AHSS grades are currently one of the materials being increasingly developed to harness the promise of second generation steels without high alloy costs and difficult processing issues. Microstructurally, it is generally accepted that this new generation of AHSS will have a significant amount of metastable (or retained) austenite to attain mechanical properties between TRIP and TWIP steels [5, 6]. Testing and evaluating this is the goal of this thesis.

Firstly, the alloy design of third generation AHSS with medium manganese (Mn) content was investigated with an alloy synthesis based on attaining a level of FCC stacking fault energy (SFE) between that of TRIP and TWIP steels. Thermodynamic modeling approaches were used for the calculation of SFE for
various possible compositions. The effect of alloying elements on the microstructure was also investigated. Then, four different steel compositions centered on Mn as the main alloying element were produced as candidates for third generation AHSS grades.

The design of the microstructure concept is based on controlling the deformation behavior of retained austenite. Therefore, “intercritical annealing” was used to obtain significant amounts of retained austenite. The target microstructure is ferrite on which retained austenite and certain amounts of high strength martensite (thermally-induced $\alpha^1$-martensite) formed during the rapid cooling of metastable austenite. However, the key constituent is retained austenite, which would be expected to increase ductility and strain hardening by forming deformation twins and strain induced martensite ($\varepsilon$-martensite) during room temperature deformation.

Steels were cast, homogenized, and hot rolled, and the effect of the annealing parameters on the microstructure and consequent cold rolling properties of medium manganese AHSS were studied. Cold rolling studies were performed to assess their potential as sheet products.

Static and dynamic mechanical studies were performed on selected microstructures as a key study of this project. Shear punch testing was used since it has the advantage of easy preparation of specimens compared to tensile testing. In the case of high strain rate mechanical testing, Hopkinson bar testing was used to understand the high speed forming and crashworthiness properties of the steel samples by means of dynamic strength and absorbed energy. Finally, microstructural and mechanical properties of all the steel samples were discussed and compared to determine the most appropriate candidate for third generation AHSS.
Chapter 2: Literature Review

This chapter reviews the background theory and literature pertaining to this thesis.

2.1. Third Generation Advanced High Strength Steels for Automotive Applications

Over the last decades, one of the expectations of automotive materials is reduced weight with improved safety and crashworthiness performance. At this point, AHSS steels have demonstrated their ability to meet these demands and have been the fastest growing sheet material in auto body structures [7, 8]. The evolution of this family in recent years has sustained new grades that have a combination of high strength and formability. In particular, TRIP steels and twinning-induced plasticity (TWIP) steels are, in principal, very good options for weight reduction and improved safety performance. As can be seen from Fig. 2.1, these have extraordinary ductility - strength combinations for automotive applications.

![Figure 2.1. The evolution of the mechanical properties of AHSS [8].](image)

The high strength of TRIP steel is obtained by applying special heat treatments and adding elements such as Si, Mn, Al, Cu, etc. This type of steel has a
microstructure of ferrite, bainite, and residual metastable austenite [9]. Then, during room temperature deformation, austenite starts to transform \(\varepsilon\)-martensite (free energy), which enhances the ductility and strength [10]. As a result, TRIP steels demonstrate an advantageous balance of strength and ductility but are limited to a tensile strength of about 800 MPa.

TWIP steels are austenitic steels that are able to accommodate plastic strains by deformation twinning. This is a very favorable mechanism because it exhibits a very high strain hardening, which, in turn, leads to a good combination of strength, ductility (having up to 50% elongation at 1000 MPa tensile strength), and damage tolerance [5, 11]. However, the “second generation” of AHSS have not found commercial application yet, since they are highly alloyed (up to \(~17-22\) wt.% Mn) and present processing problems such as casting, rolling, welding, etc.

As a result, the third generation AHSS, proposed originally by Matlock [5], should be produced via a precise process control to obtain the desired microstructure. These complex microstructures may contain multi phase constituents added to increase strength (e.g., martensite) and enhance strain hardening (e.g., austenite) [2, 12]. Also they should be less alloyed than TWIP steels; having an intermediate combination of 800-1100 MPa strength and 20-30% elongation with supposedly consisting of martensite + austenite structure (Fig. 2.1). Furthermore, there is also the potential for the use of controlled strain hardening that may be obtainable by thermo-mechanical treatments to produce the required increase in yield strength [5].

### 2.2. Mechanism of Plastic Deformation

In general, plastic deformation in metals is due to dislocation motion. Dislocation interactions cause strain hardening of materials, i.e., increases its strength during deformation. However, especially in the case of austenite, whether it is retained austenite as a microstructural minor component or a fully austenitic
microstructure, phase transformations and mechanical twinning can make a major
collection to the deformation process [13-15]. How the austenite reacts to
stresses depends largely on the stacking faults.

2.2.1. Stacking Fault

Stacking faults (SF) are imperfections in the stacking sequence of atomic planes;
in general, they occur in a number of crystal structures, but the common example
is in close-packed structures [16, 17]. If a plane of atoms is missing from a
sequence such as in ABCAB|ABC, an intrinsic stacking fault results. In contrast
to this, if an additional plane is inserted into this sequence as in ABCA|C|BCABC (extra C plane in the sequence), an extrinsic stacking fault results (Fig. 2.2). In
austenite, both intrinsic and extrinsic faults are possible and they are the basic
units for the formation of ε-martensite or mechanical twins, respectively [16-19].

These faults generate pairs of partial dislocations, and the region between this pair
of partials is described as a narrow close-packed (cp) zone. These partial
dislocations have parallel components in their Burgers vectors. The parallel
components of the same sign repel each other, whereas the stacking fault energy
(SFE) involved tends to attract the partials as can be seen from Fig. 2.3. As the
energy of the dislocation is proportional to the square of the Burgers vector, it is
clear that the dissociation has a lower energy compared to a normal dislocation
and becomes favorable as long as the fault energy is appropriate. In this way, the
equilibrium width of the stacking fault increases as its energy decreases.
The deformation mechanisms and mechanical properties of austenitic steels, which have a FCC (face centered cubic) crystal structure, are strongly related to their SFE [10]. Basically, SFE modifies the ability of a dislocation in a crystal to glide onto an intersecting slip plane and determines the amount by which dislocations dissociate into partials [10, 11, 21, 22]. Therefore, to fully exploit the twinning and/or phase transformation mechanism in these steels, the magnitude of SFE has to be properly defined.

Gréass et al. [10] stated that the magnitude of SFE controls the ease of cross-slip of screw dislocations. A low SFE leads to a wide stacking ribbon and difficulties in cross-slip, which promote stress relief by phase transformation or twinning. Figure 2.4 shows the generation of dislocation movement from planar glide to stacking fault and twin formation at near zone axis [23-25].
Figure 2.4. Schematic evolution of deformation twin: (a) an extended dislocation moving towards the cross line; (b) the first leading Shockley partial (L1) transferring to the first conjugate slip plane by stair-rod cross-slip; (c) the first following Shockley partial (F1) reacting with the stair-rod dislocation to form a Frank partial; (d) the second leading Shockley partial (L2) arriving to the cross line of the second conjugate slip plane; (e) the L2 transferring to the second conjugate slip plane by stair-rod cross-slip; (f) the second following Shockley partial (F2) reacting with the new-formed stair-rod dislocation to form another Frank partial; and (g) the deformation twin formed after several twinning partials moving at adjacent plane [25].
In this case, dislocation movement starts at a planar slip plane and then splits into partial dislocations to form stacking faults. An array of SFs generates a twin. Then, repetition of the processes would generate a series of twinning Shockley partials moving at adjacent planes, leading to the formation of deformation twins on the slip plane. Finally, if the deformation continues the twin acts as a fracture source with further shear strain on the plane.

It is known that this movement of dislocation and twin generation is mostly affected by the weight percent of Mn contents via SFE change. In particular, reduction of Mn below 10 wt.% decreases the stacking fault energy to such an extent that stacking fault array does not form, which leads to epsilon phase formation [23, 26]. As shown in Fig. 2.5, it is essential for the occurrence of the strain induced transformation that the SFE be within a relatively narrow range to observe either phase transformation or mechanical twin formation [27]. If SFE is very low (≤ 20 mJ/m²), martensite-induced plasticity is favored in the austenitic matrix. The higher magnitudes of SFE (≈ 25-45 mJ/m²) suppress the \( \gamma \) (FCC) \( \Rightarrow \) \( \varepsilon \) (HCP) phase transformation and favor mechanical twinning [26, 28, 29]. The twinning stress increases with increasing SFE [24, 30]. However, much higher SFE values (≥ 60 mJ/m²) makes the generation of partial dislocations difficult, and therefore the glide of perfect dislocations becomes the dominant deformation mechanism [10, 22, 31].

![Figure 2.5](image)

**Figure 2.5.** Schematic showing the relation between SFE and the operating deformation mechanism [27].
2.2.2. Transformation-Induced Plasticity

Basically, transformation-induced plasticity (TRIP) is the property obtained when a martensite reaction occurs during plastic deformation. In particular, a displacive transformation (e.g., martensite or bainite) results not only in a change of crystal structure and density but also postpones the onset of strain instability (i.e., necking) [2, 22].

Martensitic phase transformations can occur during cooling below the martensite start (M_s) temperature. Above M_s, the critical stress to undergo martensitic phase transformation increases linearly with temperature up to the $M_s^\theta$ temperature defined as the maximum temperature at which martensitic transformation occurs by “elastic” stress. In this temperature range, martensitic transformation is defined as stress-assisted (Fig. 2.6) [32-34]. At temperatures above M_s, significant plastic flow precedes the transformation and an additional contribution to transformation arises from the production of new nucleation sites by plastic deformation. Hence, the phase change is defined as plastic strain induced transformation [33, 35]. The temperature dependence of the critical stress for martensitic transformation is schematically represented in Fig. 2.6 where the M_d temperature is introduced to reflect the stability of the austenitic phase.

![Figure 2.6](image)

**Figure 2.6.** Schematic representation of critical stress for martensitic transformation as a function of temperature [33].
In general, the driving force for the austenite to martensite transformation can be written as a sum of chemical and mechanical contribution. The chemical driving force for the martensitic transformation is a function of the chemical composition of the austenite (C, Mn, Si, Al...) [2, 12, 33]. When the steel receives the appropriate thermomechanical treatments, the metastable austenite is retained and cold deformation above a critical level begins the transformation of the austenite to martensite, and this enhances the ductility and increases the strength [22]. In “classic” TRIP steels, which have relatively low Mn levels (e.g., 1.5%), there is no mention of the influence of SFE on strain induced transformation. However, for medium Mn levels (approx. 10 to 18%) the influence of SFE seems to be apparent.

2.2.3. Twinning-Induced Plasticity

Twinning-induced plasticity refers to the occurrence of mechanical twinning as a plastic deformation mechanism during straining. It is largely believed that this is a main mechanism responsible for the large work-hardening rate of high Mn steels since they play the role of planar obstacles to the dislocation glide [19, 36]. However, despite several decades of research, the precise origins of the large work-hardening rate exhibited by high Mn steels remain unclear.

One explanation for the high work hardening rate of these steels is a typical dynamic strain ageing (DSA) mechanism. According to Dastur and Leslie [37], due to a strong interaction of solute atom dipoles (C–Mn) with the strain field of dislocations, these dislocations are pinned and locked, increasing the dislocation density and thus the work-hardening rate. Macroscopic measurements, such as the rate of dislocation accumulation recently carried out by Hutchinson and Ridley [38] support this kind of influence on the work-hardening rate.

Bouaziz and Guelton [19] claimed that mechanical twinning is accompanied by shear strain and also a strain hardening effect by subdividing the untwined austenite into finer regions, i.e., a “dynamic” (i.e., it occurs during deformation) Hall-Patch effect. As shown in Fig. 2.7 (a) and (b), fine twins are formed in
grains, which involves the creation of new crystal orientations (Fig. 2.7 (d)); in addition, the twins progressively reduce the effective mean free path of dislocations (Fig. 2.7 (e)), increase the flow stress, resulting in a high strain hardening behavior [27, 36]. The huge level of back-stress (up to half of the total flow stress) that appears with strain in these steels is explained from the dislocation pile-ups at the twin boundaries [39].

![Figure 2.7](image)

**Figure 2.7.** Schematic illustration of deformation twins in different scales of observations that gradually reduce the effective glide distance of dislocations: adapted from (a) twins in the grain [36] (b) scale of twins [40] (c) twins are obstacle to dislocation motion (d) sequence of crystal orientation [19].

Recently, Idrissi et al. [36, 41] stated that mechanical twins contain a high level of sessile dislocations resulting from both the twin formation mechanism and dislocation reactions between Shockley partials and forest dislocations. These sessile dislocations lead to a large increase in the critical stress required to induce some plastic deformation within these thin twin-related crystals, therefore improving the strengthening effect [36, 42-44].

Similar to these ideas, Bhadesia et al. [2] believe that twinning-induced plasticity is somewhat analogous to transformation-induced plasticity (TRIP), in which
phase transformation to martensite enhances plasticity. In the case of TWIP, the
symmetry of the lattice is preserved by the twinning operation, and the shape
deformation due to twinning is a shear strain $s = 1/\sqrt{2}$, which is much larger than
the deformation associated with martensitic transformation [2, 45]. Indeed,
mechanical twinning is a simple shear on the twinning plane that remains
undistorted and unrotated. Consider the block of material illustrated in Fig. 2.8
which twins on plane $K_1$. The plane denoted $K_2$ is undistorted but is rotated. In
FCC, if $K_1 (111)$ then $K_2$ is $(11 \bar{1})$ plane [45, 46].

![Figure 2.8. Schematic representation of mechanical twinning of a block material; the interplanar distance for type a plane is lengthened, for c it is shortened, and for b it is kept the same [46].](image)

So the shear strain defined in Eq. 2.1:

$$s = \frac{d}{h} = \frac{|a_{16} <112>|}{d_{111}} = 1/\sqrt{2}$$ (2.1)

where $d_{111}$ is the interplanar spacing of the $(111)$ planes. The alloys are austenitic
and remain so throughout plastic deformation, with plastic strain being
accommodated both via mechanical twinning and slip. However, some of the
studies demonstrate that the impressive elongations characteristic of TWIP steels
have a rather small contribution from the mechanical strain of twinning,
strengthening the case that the increased work hardening attributed to the
partitioning of the austenite grains is the major contributing factor to overall elongation. As noted above, the work hardening rate is believed to be enhanced by subdividing the original austenite into twinned and untwined regions [45, 46]. It is interesting that a similar conclusion has been reached in the context of TRIP steels where the martensitic transformation strain makes only a minor direct contribution to the observed plasticity [45].

2.3. Microstructure Design and Control

All AHSS steels are controlled by many factors, including phase composition and distribution in the overall microstructure, volume fraction, size and morphology of phase constituents, as well as stability of metastable constituents during heat treatment and deformation [6, 47]. However, it is accepted that these complex microstructures contain multi phase constituents added to increase strength (e.g., martensite) and enhance strain hardening (e.g., austenite). To control the microstructure, alloying and heat treatment concepts need to be considered as the main factors.

2.3.1. Thermodynamic Modeling of SFE

Over the last few decades, different approaches to determine the SFE of metals have been presented in the literature. Calculations of the SFE in FCC metals were initially based on the electron theory in metals [48]. Later, Cotterell et al. [49] calculated the SFE of copper by a variation of the method using the central-force approximation, which is an “atomistic” approach. Finally, Olson and Cohen [50] proposed a thermodynamic modeling approach to calculate the SFE in FeMnAlSi TRIP/TWIP steels, which was later modified by various authors. In all of the above approaches, stacking fault energy defines the deformation type in the phase, and that has been calculated for high manganese alloys [51].

According to the Olson-Cohen thermodynamic model, the stacking fault can be formed by the motion of a single Shockley partial dislocation on a close-packed
plane, whereas the motion of such a dislocation on every second plane produces a bulk HCP crystal [52]. Thus, a stacking fault is an HCP embryo that is two planes in thickness. As in classical nucleation theory, the free energy of such embryos relative to the perfect lattice may be expressed in terms of the chemical free energy difference between parent and product phases, a strain energy and a true surface energy [10, 50]. The magnitude of stacking fault energy for FCC alloys is expressed by the following empirical Eq. 2.2:

$$\gamma_{fcc} = 2p\Delta G ^{\gamma \rightarrow \varepsilon} + 2\sigma _{\gamma / \varepsilon}$$  \hspace{1cm} (2.2)

where $p$ refers to the molar planar density of atoms in the close-packed plane of \{111\} ($2.86 \times 10^{-5}$ mol/m$^2$, close to the theoretical density of \{111\} plane), $\Delta G ^{\gamma \rightarrow \varepsilon}$ the molar free energy of the $\gamma \rightarrow \varepsilon$ phase transformation, and $\sigma _{\gamma / \varepsilon}$ the interface (or interfacial) energy between $\gamma$ (FCC) and $\varepsilon$ (HCP) phases (10 mJ/m$^2$ for transition metals) [10, 28, 53-57].

For such a calculation, the situation is complicated in the case of quaternary system. First of all, the stacking fault energy is primarily dictated by the chemical free energy between phases and varies with chemical composition and deformation temperature. Therefore, the tendency to form $\varepsilon$-martensite or twinning boundaries during room temperature deformation depends on the stacking fault energy level of FCC matrix/phase composition [21, 58]. For this purpose, thermodynamic models are developed for the calculation of the SFE of both TRIP and TWIP steels.

Basically, the thermodynamic modeling approach accounts for the change in the Gibbs free energy $\Delta G ^{\gamma \rightarrow \varepsilon}$ of each element upon the $\gamma_{fcc} \rightarrow \varepsilon_{hcp}$ phase transformation, as well as the interaction between the elements by introducing the excess free energy term $\Omega ^{\gamma \rightarrow \varepsilon}$ (interaction parameter) as seen from the flow chart in Fig. 2.9 [14].
According to the chart, based on the regular solution model, the free energy $\Delta G^{\gamma \rightarrow \varepsilon}$ for the $\gamma \rightarrow \varepsilon$ phase transformation is calculated as a function of temperature as seen in Eq. (2.3):

$$\Delta G^{\gamma \rightarrow \varepsilon} = X_{Fe} \Delta G_{Fe}^{\gamma \rightarrow \varepsilon} + X_{Mn} \Delta G_{Mn}^{\gamma \rightarrow \varepsilon} + X_{Al} \Delta G_{Al}^{\gamma \rightarrow \varepsilon} + X_{Si} \Delta G_{Si}^{\gamma \rightarrow \varepsilon} +$$

$$X_{Fe}X_{Mn}(X_{Fe} + X_{Mn})^{-1} \Delta \Omega_{FeMn}^{\gamma \rightarrow \varepsilon} + X_{Fe}X_{Al}(X_{Fe} + X_{Al})^{-1} \Delta \Omega_{FeAl}^{\gamma \rightarrow \varepsilon} +$$

$$X_{Fe}X_{Si}(X_{Fe} + X_{Si})^{-1} \Delta \Omega_{FeSi}^{\gamma \rightarrow \varepsilon} \ldots$$

(2.3)

where $X$ is the mole fraction of the compound. Thermodynamic data and the interaction parameters ($\Delta \Omega_{FeMn}^{\gamma \rightarrow \varepsilon}$, $\Delta \Omega_{FeAl}^{\gamma \rightarrow \varepsilon}$, $\Delta \Omega_{FeSi}^{\gamma \rightarrow \varepsilon}$) can be found in the related publications [28, 53-57].
2.3.2. Effect of Allying Elements

Both TRIP and TWIP steels have nominal compositions based on the contents of their major alloying elements, which are carbon, manganese, silicon, aluminium, and niobium. Indeed, these alloying elements have a considerable effect on the microstructural and mechanical properties, and variations in these compositions cause changes in both the microstructure and mechanical properties [10, 22]. In particular, it can be seen from Fig. 2.10 that modifying the amounts (in wt.%) of major alloying elements (Mn, Si, and Al) influences the stress and elongation ratios of TWIP steels [28, 55, 59].

Clearly, Fig. 2.10 shows that the lower the manganese content, the higher the ultimate tensile strength and the lower the elongation. Furthermore, for a constant Mn concentration, increasing Al and decreasing Si causes a decrease in the tensile strength and an increase in the elongation [22, 59].

![Figure 2.10. Effect of alloying elements on the mechanical properties of FeMnSiAl TWIP steels: (a) yield stress (black bars) and ultimate tensile stress (grey bars) and (b) uniform elongation (black bars) and total elongation (grey bars) [22].](image)

Moreover, these variations affect the deformation mechanisms as well as the mechanical properties. Indeed, depending on the amount of Mn, Si, and Al, twinning becomes dominant and γ (FCC) austenite $\rightarrow\varepsilon$ (HCP) martensite phase transformation can be controlled. As a consequence, a good understanding of the
effect of alloying elements on AHSS makes alloy development much easier for the third generation.

As summarized previously, the metastability of austenite is a function of both intercritical annealing temperature and time. Considering the phase diagram (isopleth) of Fe-C 4.5 wt.% Mn steel as illustrated in Fig. 2.11 (a), it can be predicted that the carbon content of the austenite will increase with increasing annealing temperature. Consequently, austenite is stabilized by increased carbon content and is retained upon cooling rapidly [60, 61].

However, by increasing manganese content, the austenite phase field increases, and the carbon content of the austenite can then decrease with increasing annealing temperature, following the $\alpha + \gamma/\gamma$ phase boundary, as illustrated in Fig. 2.11 (b) [61]. Indeed, Mn raises the A$_4$ and lowers the A$_3$ transformation temperatures, increasing, in effect, the extent of the gamma ($\gamma$) field in the iron-carbon phase diagram.

![Figure 2.11](image.png)

**Figure 2.11.** Fe-C phase diagrams for (a) 4.5 wt.% Mn and (b) 13 wt.% Mn levels [61].

In addition to this, Mn can be considered as a major alloying element in third generation AHSS since it affects the SFE level of the austenite phase [47]. Studies show that higher Mn content first decreases the SFE to a minimum value and then increases it as seen in Fig. 2.12 [62]. Hence, it has been claimed that with increasing Mn content, the deformation mode changes from TRIP to TWIP in austenitic regions [42].
Figure 2.12. Variation of SFE as a function of Mn content in Fe-C-Mn alloys [62].

The addition of Al increases SFE and therefore stabilizes the austenite against the strain induced $\gamma$ (FCC) $\rightarrow$ $\varepsilon$ (HCP) $\rightarrow$ $\alpha'$ (BCT) transformation during deformation [28, 63, 64]. Otherwise, a decrease of the SFE causes strain induced transformation in the microstructure (TRIP) and increases the austenite to martensite transformation ($\gamma \rightarrow \alpha'$). Furthermore, Al also strengthens the austenite by solid solution hardening [65].

In contrast to Al, Si decreases the amount of FCC phase and sustains the $\gamma \rightarrow \varepsilon$ transformation during cooling and deformation [65, 66]. Takaki et al. [51] stated that the addition of Si lowered the SFE of austenite, resulting in an increase in the number of stacking faults.

Carbon is considered an effective austenite stabilizer and is added in modified TWIP steels up to 0.6 wt.% [1]. It is well known that the solubility of carbon is high in austenite, so that carbon alloying can be used to stabilize the austenite and also to strengthen the matrix by solid solution hardening [57].

It is well known that the addition of Mo is important, as it is the additional alloying element that increases the corrosion resistance of steels in various media.
Furthermore, it enhances the formation of ferritic phase \[55\] and increases the \(M_s\) temperature. The addition of Cr to the Fe-Mn-based alloy system raises SFE \[63\].

2.3.3. **Effect of Heat Treatment**

2.3.3.1. **Intercritical Annealing**

Intercritical annealing heat treatments are used to produce dual phase steels to obtain good combinations of strength and toughness by controlling the martensitic microstructure. It is also used to produce TRIP steels by affecting the C concentration in austenite which controls the stability of the austenite.

Fig. 2.13 shows the general schematic of the intercritical annealing process and cooling. In particular, for dual phase steels, annealing between the two phase region \((\alpha + \gamma)\) produces a mixture of ferrite and austenite in low-alloy C steel. The austenite, upon quenching, transforms to islands of martensite, producing a ferrite + martensite dual-phase structure in the steel. Therefore, an increase in intercritical annealing temperature results in an increase in austenite (martensite) volume fraction but also a decrease in martensite carbon content.

![Figure 2.13. Schematic diagram of intercritical annealing and cooling.](image)

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\[\text{Figure 2.13. Schematic diagram of intercritical annealing and cooling.}\]
Higher annealing temperatures and longer annealing times also increase the diffusion of alloy species. This may lead to a “clean” ferrite via the segregation of solute atoms to the austenite phase with possible enhancement of ductility and with little or no loss in tensile strength [67]. Hence, by controlling the annealing temperature and cooling rate, it is possible to control the shape, size, amount, and distribution of the martensite in the duplex microstructure, and the desired combination of strength and ductility can be obtained.

2.3.3.2. Intercritical Annealing and Isothermal Holding

Basically, this is a two-stage heat treatment consisting of an intercritical anneal in the austenite plus ferrite two phase region, followed by an isothermal hold at a temperature in the bainite transformation region, as can be seen in Fig. 2.14. The bainite that is formed will stabilize the remaining austenite by enriching with C from the transforming carbide-free bainite, which is then retained as metastable austenite upon cooling to room temperature [68].

![Graph](image)

**Figure 2.14.** Schematic diagram of intercritical annealing and bainite hold.

In this process, isothermal holding temperature and time control the bainite transformation and support the rejection of carbon into the remaining austenite. Thus, $M_s$ temperature decreases sufficiently and allows austenite to be retained at room temperature [69].
2.3.3.3. **Quenching and Partitioning**

Quenching and *tempering* of martensite is a very well accepted and mature technique. Recently, this process route has been modified and the subsequent process is described as “quenching and partitioning” (Q&P) and is shown schematically in Fig. 2.15 [5]. Basically, Q&P differs from quenching and tempering in that the goal of Q&P is to stabilize austenite in a martensitic microstructure, which apparently leads to increased strain hardening.

The heat treatment sequence involves three steps to obtain final microstructure. Firstly, depending on the initial annealing temperature, the starting microstructure is either fully austenitic or a mixture of ferrite and austenite. Secondly, the materials are rapidly cooled to a specific quench temperature (QT) between the martensite start (Ms) and martensite finish (Mf) temperature to create a controlled fraction of martensite and austenite. Finally, this is followed by a thermal heat treatment at the partitioning temperature (PT), which can either be equal to the quenching temperature (QT) (one-step process), or higher than QT (two-step process) [70].

![Figure 2.15. Schematic illustration of Q&P process](image)

*Figure 2.15. Schematic illustration of Q&P process (Cᵢ, Cγ, and Cm represent the carbon contents of the initial alloy, austenite, and martensite, respectively, and QT and PT are the quenching and partitioning temperatures, respectively) [5].*
During the partitioning stage, in the martensite, the C in solid solution is reduced by migrating from the martensite to the austenite and increasing the austenite stability, which results in carbon-enriched retained austenite at room temperature [70].

### 2.3.3.4. Transformation of Intercritical Austenite

This section includes austenite to martensite transformation due to quenching, as well as the transformation of metastable austenite during plastic deformation. In conventional AHSS steel compositions (either DP or TRIP), martensite forms on quenching from the intercritical region and the austenite transforms to BCT (body-centered tetragonal) martensite [71]. The relationship between the FCC austenite and the BCT martensite can be seen in Fig. 2.16. This figure shows that carbon atoms in the (1/2, 0, 0) type of interstitial sites in the FCC cell can be trapped during the transformation to the body-centered structure, causing the tetragonal structure to be formed in the austenite grain.

![Diagram] Figure 2.16. The martensite transformation of austenite during quenching [71].

During this process, the steel must be quenched, or rapidly cooled, from the stable austenite region to prevent the formation of pearlite, bainite, or primary microstructural constituents. The martensite reaction begins when austenite cools below the $M_s$ temperature (Fig. 2.13 and 2.14). The amount of martensite increases as the temperature decreases. When the temperature passes below the $M_f$, the austenite should have transformed completely to martensite.
Figure 2.17 [72] shows an island of retained austenite in a ferrite-based matrix. In both DP and TRIP steels, strain induced martensite may be formed within the austenitic island as a result of plastic deformation, as shown in Fig. 2.17 (a) and (b). Initially, these martensitic regions often emerge as plates with specific orientations, as illustrated in Fig. 2.17 (b). Higher magnification of a martensitic plate reveals a layered structure of two crystallographic variants of martensite, BCT and HCP, as shown in Fig. 2.17 (c) and (d). To form HCP martensite, the lattice deformation itself must be an invariant-plane strain, i.e., the motion of Shockley partials on the alternate close-packed planes [73]. Simply, the phase transformation may be explained geometrically by a shift of alternate layers in the FCC stacking sequence into the stacking fault sequence, as originally proposed by Burgers [74]. This is equivalent to passing a Shockley partial dislocation (a/6 <112>) over every alternate close-packed plane in the cubic phase. It is believed that the transforming Shockley partial dislocations should be regenerated as almost straight lines such that stacking fault expansion might occur spontaneously with certain SFE [50].

![Schematic illustration of strain induced martensitic transformation within the microstructure](image)

**Figure 2.17.** Schematic illustration of strain induced martensitic transformation within the microstructure [72].
2.4. Dynamic Mechanical Behavior

The dynamic behavior of materials can be defined as the mechanical response of a material that is subjected to impact or impulse loading [75]. It is an area of study that is covered by a number of scientific disciplines, such as materials science, mechanics, chemistry, and applied mathematics and modeling [75, 76]. It is well known that the processes that occur when structures are subjected to rapidly changing loads can differ significantly from those occurring under static or quasi-static loadings [76]. Therefore, the scientific understanding of the deformation and failure of materials under dynamic loading is essential in the design of structures, and it requires a special study of the inertia and inner kinetics of materials.

In metals, the strain rate and temperature dependent mechanical behavior of AHSS is mainly controlled by dislocation dynamics. Hence, both temperature and strain rate affect the formation of dislocation glides, twinning, and phase transformations. Therefore, in order to study the dynamic mechanical behavior and crashworthiness properties of third generation AHSS, a comprehensive description is necessary of the plastic deformation mechanism of these steels.

2.4.1. Dislocation Dynamics

It is rather difficult to define an all-encompassing theory that describes the relationship of strain rate and stress in the plastic deformation area [75]. However, Isaac and Granato et al. [77] stated that the dynamic behavior of a material in the plastic deformation area can be divided into three strain rate regimes. For many metallic materials, the stress is found to increase linearly till the end of the intermediate strain rate. Then the stress increases rapidly in a given applied strain rate as seen in Fig. 2.18 [78].
First of all, at low (quasi-static) and intermediate strain rates, dislocation motion is controlled by thermally activated processes. For most metals, the flow stress increases relatively with increasing strain rate in this regime. Secondly, by increasing the strain rate above between $\dot{\varepsilon} = 10^3$ and $10^5 \text{ s}^{-1}$ levels, dislocation drag controlled deformation mechanisms take place and limits the dislocation velocity, which causes an upturn in the stress values. Finally, at even higher strain rates the velocity approaches the velocity of transverse sound waves and relativistic effects come into play [77, 79, 80].

**Thermally-Activated Dislocation-Motion:** At temperatures below the melting point, the plasticity of a metal is determined by the mobility of its dislocations. Basically, obstacles getting in the way of moving dislocations disturb the mobility and thereby increase the strength of the material [77].

**Dislocation Drag:** According to Gilman [81], the theory of thermally activated dislocation motion is valid only up to a certain critical strain rate. At higher strain rates, the velocity of dislocations is proportional to the applied stress. It is well
known that the energy stored in the material after deformation (as defects) is only a small portion of the energy spent to deform it [75]. The residual energy is usually between 5% and 20% of the total energy and the rest is dissipated by forces opposing the applied stresses [75, 76]. For crystalline materials, this effect is generally attributed to dislocation drag mechanisms, which start to act in addition to the thermal obstacles and finally dominate the deformation behavior at very high strain rates [82, 83].

**Relativistic effects on dislocation motion:** According to Eshelby [84], the third stage of dislocation motion is the one at which the velocity approaches the shear wave velocity, although supersonic dislocations have not yet been observed clearly. Although a lot of assumptions and theories can be found in the literature, an analytical rationale is not possible [75].

An explanation of the strain rate dependent deformation behavior of metals can be given using the theory of thermally-activated dislocation motions. As the strain rate increases, there is less time to overcome the barrier and the thermal energy will be less effective [75]. The contribution of thermal energy to overcoming the barrier eventually becomes zero. In practice, however, there are obstacles, which are classified as either short-range (thermally activated) and long-range (non-thermally activated), as seen in Fig. 2.19 [75, 79]. Hence, taking into account the temperature and strain rate dependence of thermally activated dislocation motion, the flow stress of a material can be expressed in Eq. 2.4:

\[
\sigma = \sigma_G(\text{structure}) + \sigma^*(T, \dot{\epsilon}, \text{structure})
\]  

(2.4)

where the term \(\sigma_G\) is due to the athermal (long-range) barriers determined by the structure of the material, and the term \(\sigma^*\) is due to the thermally activated barrier, which can be overcome by thermal energy. Actually, the principle short-range barrier is the Peierls-Nabarro stress, however, for FCC and HCP metals, dislocation forests are the primary short-range barriers at low temperatures [75].
2.4.2. Strain Rate Dependent Deformation Behavior

The above analysis does not necessarily hold true for TRIP and TWIP steels, since the deformation behavior involves phase transformation or twinning. In fact, there is limited information regarding the effect of strain rate on such steels.

It was reported that both yield strength and ultimate tensile strength increase with increasing strain rate for AHSS [12]. However, these values vary with strain rate levels for different steel grades. Figure 2.20 shows the comparison of yield stress and ultimate tensile strength as functions of strain rate in high Mn content TRIP and TWIP steel grades [22].

According to Fig. 2.20, it is clear that with increasing strain rate the tensile and yield stresses generally increase, although for TRIP steel the tensile strength appears to first decrease in the quasi-static regime. At quasi-static strain rates, there is a difference in strength between the two steels; however, the stress values converge in the intermediate strain rate region.

In general, both materials show similar high strain hardening behavior combined with a high ability to deform without necking up to very high strain rates. As a result, it is expected for TRIP and TWIP steels to exhibit high ductility values. In Fig. 2.21 [22], studies show that TRIP steels do exhibit high ductility over a broad
range of strain rates; however, they also exhibit a slight decrease in ductility as the strain rate increases from the static to the intermediate regime.

**Figure 2.20.** Comparison of yield stress and ultimate tensile strength as a function of strain rate [22].

**Figure 2.21.** Deformability of high Mn TRIP steel under tensile loading with strain rate values [22].
2.4.3. **Strain Rate Sensitivity Factor**

Several commonly used definitions for strain rate sensitivity coefficient can be found in the literature. Basically, the definition is obtained from the relationship between flow stress and strain rate in empirical constitutive equations.

For example, in the Holloman equation:

\[ \sigma = k \times \varepsilon^n \times \dot{\varepsilon}^m \]  \hspace{1cm} (2.5)

\( m \) is the strain rate sensitivity coefficient, \( \sigma \) and \( \varepsilon \) are stress and strain, \( \dot{\varepsilon} \) is strain rate, and \( k \) and \( n \) are constants.

In the Johnson-Cook equation [85]:

\[ \sigma = (A + B\varepsilon^n)(1 + C\ln\dot{\varepsilon})\times(1 - T^*m) \]  \hspace{1cm} (2.6)

\( C \) is the strain rate sensitivity coefficient; \( A, B, n, \) and \( m \) are constants; and \( T^* \) is defined as **Eq. 2.7**:

\[ T^* = \frac{T - T_r}{T_m - T_r} \]  \hspace{1cm} (2.7)

where \( T \) is the instantaneous temperature of the material, \( T_m \) is the melting temperature of the material, and \( T_r \) is the room temperature.

2.4.4. **Crashworthiness**

During the past decade, many investigations have been carried out concerning the material and structural aspects of crashworthiness. Crashworthiness is basically the ability of a structure to protect its occupants during an impact. Depending on the nature of the impact and the vehicle involved, different criteria are used to determine the crashworthiness of the structure. Several different tests are used to assess crashworthiness, such as high-strain rate deformation testing, high-speed crush testing and accelerated impact, in conjunction with computer models or dynamic finite element methods [86]. The injury probability is defined by using
certain parameters (e.g., force, acceleration, deformation) that correlate with injury risk [82, 83].

In terms of materials properties, high-speed deformation testing is a key assessment of crashworthiness, which is quantified by parameters such as dynamic strength and absorbed energy. In the laboratory, high-speed deformation testing is usually examined by Hopkinson bar testing [87] or drop-weight crush-testing [14, 88]. Many investigations have been conducted on the crashworthiness of various high strength steel structures. However, most of them have concentrated on the effect of static strength level and the component design factors [7, 86]. A more thorough understanding of dynamic deformation behavior in terms of the effect of microstructure is required.

### 2.5. Experimental Techniques Used for Static and Dynamic Mechanical Characterization

The basic principle common to almost all mechanical testing techniques is that a load is applied to a material and the material’s resistance to deformation is measured simultaneously. The most common testing techniques corresponding to strain rate ranges and prevailing test conditions are presented in Fig. 2.22 [89]. This figure simply shows the method of loading, strain rate regime, and the important dynamic events that need to be considered for testing in the specified regime.

The details in the testing techniques, however, can differ fundamentally depending on the objective of the test. Performing mechanical tests at varying strain rates requires special considerations due to changing testing conditions and becomes usually more challenging with increasing strain rates.
2.5.1. **Shear Punch**

The choice of the suitable test depends on several factors, such as the material geometry, the available volume of the material to be tested, or the type of application for which the material is used. Therefore, the strength of a material at room temperature can be determined by the shear punch test (SPT) method, which is used when the small size of the available material makes it difficult to make tensile test specimens. In shear punch testing, a cylindrical punch with a flat end is forced to punch a hole in a clamped small disc specimen. The load-displacement plot is obtained during the punch. The test is similar to that of a conventional tensile test and properties obtained by analyzing the test curve are correlated with the corresponding conventional tensile properties. Thus, the tensile and yield strength of the samples can be determined from the load vs. displacement curves.

Figure 2.23 schematically illustrates a shear punch testing machine. A load cell is located in the lower testing bed, and the displacement can be measured by one of the laser displacement meter and strain gauges. The most important characteristic of this test is that the absorbed energy, testing speed, and strain rate can be determined concomitantly [90].

---

**Figure 2.22.** Common mechanical materials testing techniques, their strain rate range, and their test conditions [89].
2.5.2. **Hopkinson Bar**

The dynamic behavior of sheet metals is indeed a key factor to investigating the impact characteristics of the structure in order to calculate the impact energy absorption ratio. Since the dynamic behavior of a material is different from the static behavior because of the high strain rates and the propagation of stress waves, an adequate experimental technique had to be developed for the corresponding strain rate level [91]. For this purpose, the split Hopkinson pressure bar apparatus was developed to measure the stress and strain of a test specimen from the stress waves generated in the output bar after passing through the specimen and from the stress waves of the input bar after being impacted by a striker (Fig. 2.24) [86]. Furthermore, the stress-strain curves for the high strain rates (ranging from 1000 s\(^{-1}\) to 10000 s\(^{-1}\)) can be acquired from the stress waves propagating through the incident and the transmitted bar in the apparatus. Generally, the Hopkinson bar compression specimens are cylindrical, while the tension specimens may be cylindrical or flat [92].

![Figure 2.23. Schematic of the shear punch testing die [91].](image-url)
Chapter 2: Literature Review

Figure 2.24. Schematic illustration of (a) Hopkinson shear punch and (b) split-Hopkinson bar used to conduct the dynamic compression tests.

The Hopkinson bar experiment was developed to its current form by Kolsky in 1949 [75, 93], hence alternative references to it in the literature are as the Kolsky bar method. Several layouts of the Hopkinson bar setup have been used in order to vary the type of tests performed. However, they all share a common configuration consisting of two main cylindrical rods with a sample sandwiched or attached in between. An elastic pressure wave is generated at the end of one bar and eventually reaches and deforms the sample with the stress vs. strain results.
2.6. SFE Calculation via TEM

Based on the above given considerations in section 2.2.1, the SFE can be approximately calculated with Eq. 2.8 [94, 95]:

\[ \gamma = \frac{\mu b^2}{4\pi d} \]  \hspace{1cm} (2.8)

where \( \gamma \) is the SFE, \( \mu \) is the shear modulus, \( b \) is the burgers vector, and \( d \) is the interplanar spacing. The equilibrium separation \( d \) (Fig. 2.3; distance between two partial dislocations) of dislocation partials can be measured experimentally by TEM, and then using the simple relation on Eq. 2.8, SFE can be determined.

The derivations of this method have been widely used with modern image-capture technologies in conjunction with weak-beam dark-field (WBDF) imaging techniques, which enables the precise positioning of dislocations.

It is already known that perfect dislocations present in the matrix split into two Shockley partial dislocations bounding narrow stacking faults, as commonly observed in FCC materials with low SFE [95, 96]. The micrograph of Fig. 2.25 shows an example of this dissociation in WBDF mode with the reflecting plane \( g = 20\bar{2} \) [96]. Two dissociated Shockley partial dislocations can be clearly observed in this image. The burgers vector of the perfect dislocation is parallel to the direction of the lattice vector of the operating reflection.

Therefore, the partial dislocation separation and the angle between the burgers vector of the perfect dislocation and the dislocation line can be readily measured on TEM micrographs. Using these measurements, SFE can be calculated with the following equation (Eq. 2.9):

\[ d = \frac{G|b_p|^2}{8\pi \gamma} \chi \frac{2-v}{1-v} \left( 1 - \frac{2v \cos 2\theta}{2-v} \right) \] \hspace{1cm} (2.9)

where \( d \) is the separation, \( G \) is the shear modulus, \( b_p \) is the magnitude of the burgers vector of the partial dislocations, and \( v \) is the Poisson’s ratio [95].
Figure 2.25. (a) Bright field (BF) and (b) WBDF image of a dissociated dislocation in a sample deformed to the yield point [41].

In addition to these, Fig. 2.25 (a) and (b) shows the difference between the BF and WBDF image of a dissociated dislocation of high manganese steel on its glide plane. From this point of view, alongside the partial dislocation it is much more possible to take several measurements for SFE calculations from the WBDF image.

Finally, it should be stated that the stacking fault images obtained under weak beam diffraction conditions were always found to be symmetrical with respect to the center of the fault [97]. Dislocations can be visualized as sharp bright lines, whereas the perfect parts of the specimen are not visible and appear dark. Therefore, the sample and the incident beam must satisfy specific tilt conditions, which are accepted as the main challenge in this method.
2.7. Research Objectives

Based on the literature review, it seems that the research on medium Mn steels is very limited. However, these steels are regarded as being a very promising candidate for the so-called third generation AHSS, which has properties in between TRIP and TWIP alloys. Therefore, the major objective is to understand how medium Mn steels affect structure and properties and to, therefore, maximize the potential of medium Mn-based steels. Since these steels are targeted for automotive applications, the present study also focuses on crashworthiness properties (i.e., dynamic strength and ductility). In order to fulfill this, the following approach is adopted:

- Producing innovative multiphase microstructures by rational alloy design and thermomechanical treatments. The main goal is to deploy retained austenite.

- Relating microstructural characteristics to the mechanical properties of strength and energy absorption under both static and dynamic conditions.

- Interrelating static and dynamic properties in order to understand deformation mechanisms and to develop engineering design metrics.

- Proposing microstructures and the corresponding processing schedules that provide optimized mechanical properties.
Chapter 3: Experimental Procedure

The background of each of the experimentation stages that a specimen goes through from production to result is presented. These various steps span the metallurgical and mechanical disciplines and are consecutively detailed in the following sections and subsections. The metallurgical aspect of the experimental contribution involves tasks such as the determination of the material chemical composition, heat treatment, metallographic observations of the material microstructure at the different stages of the process flow, and the determination of the volume fraction of retained austenite contained in the produced samples. The mechanical aspect of this work is concerned with the design of the specimens to be mechanically tested and, subsequently, the experimentation on these coupons under various loading rates and conditions.

This chapter consists of three main sections. The first deals with the production of the various third generation AHSS alloys that were processed by casting, hot and cold rolling, and heat treating. The second section deals with the characterization of the microstructure properties and determination of the content of retained austenite. The last section of this chapter deals with the characterization of the mechanical properties under static and dynamic conditions.

3.1. Materials

The steel ingots used throughout this work were supplied by CANMET-MTL (Hamilton, Ontario, Canada). Castings were done in an open induction furnace. The compositions (in wt.%) of the examined steels are shown in Table 3.1. The rationale for these compositions was based on attaining certain levels of stacking fault energy in metastable austenite to promote twinning as opposed to transformation to martensite. Details of the alloy design will be explained in the results section.
Chapter 3: Experimental Procedure

3.2. Processing Stages

The alloys undergo several processing stages, starting with alloy synthesis. Then, homogenizing and hot rolling was conducted to produce steel plates. Several annealing experiments were applied to the hot rolled material to obtain the desired microstructural and mechanical properties before cold rolling studies. After cold rolling with different deformation ratios, microstructural and mechanical characterization was done. The process flow of these steps is depicted on Fig. 3.1.

3.2.1. Hot Rolling

Hot rolling was performed at CANMET-MTL (Hamilton, Ontario, Canada) and TUBITAK-MRC (Gebze, Kocaeli, Turkey). Prior to hot rolling, homogenizing was performed for all steel castings to obtain a more homogeneous distribution of the alloying elements, in particular C and Mn. The segregation of Mn is often seen in TRIP and TWIP steels [98-100].

Two different homogenization times were applied for these compositions. The castings having a thickness of 30 mm were homogenized for 3 hours and 6 hours at 1200°C. The first homogenization was used for hot rolling in CANMET-MTL (Hamilton, Ontario, Canada) and the second one was applied in TUBITAK-MRC (Gebze, Kocaeli, Turkey).

Table 3.1. The chemical composition of steel samples.

<table>
<thead>
<tr>
<th>Sample Nr.</th>
<th>C (%)</th>
<th>Mn (%)</th>
<th>Si (%)</th>
<th>Al (%)</th>
<th>Mo (%)</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>S-1</td>
<td>0.12</td>
<td>4.98</td>
<td>3.11</td>
<td>3.05</td>
<td>0.05</td>
<td>bal.</td>
</tr>
<tr>
<td>S-2</td>
<td>0.19</td>
<td>4.96</td>
<td>3.09</td>
<td>2.99</td>
<td>0.03</td>
<td>bal.</td>
</tr>
<tr>
<td>S-3</td>
<td>0.22</td>
<td>7.15</td>
<td>3.11</td>
<td>3.21</td>
<td>0.05</td>
<td>bal.</td>
</tr>
<tr>
<td>S-4</td>
<td>0.20</td>
<td>10.02</td>
<td>3.17</td>
<td>3.19</td>
<td>0.06</td>
<td>bal.</td>
</tr>
</tbody>
</table>

Figure 3.1. Schematic illustration of process flow.

Some samples were hot rolled in several passes from 30 mm to 6 mm between 1100 to 750°C at CANMET-MTL. Other samples were hot rolled from 30 mm to 5.2 mm between 1100 to 900°C in TUBITAK-MRC. Reheating between passes was not applied in either hot rolling schedule. After hot rolling, some acid pickling (10% HCl + 90% H₂NO₃) was tried but did not remove the oxide layer from the surface and also made some large pitting areas on the surface. Sand blasting was also tried but produced a rough and inhomogeneous surface. As a result, all samples were machined to 4 mm thickness to remove the thick oxidation layer on their surface and to bring all materials to the same thickness with a smooth surface.

3.2.2. Heat Treatment

Since the main objective of the heat treatment was to maximize the retained austenite, all heat treatment sequences involved two steps to obtain the final microstructure. Firstly, a ferrite + austenite (BCC + FCC) structure was generated by an intercritical anneal. Secondly, the steels were quenched in preparation for
Q&PT treatments to generate austenite, which would be retained at room temperature. However, quenching was found to retain considerable levels of austenite, as will be explained in the results section. Therefore, the only heat treatment after intercritical annealing was continuous cooling at three different rates. The intercritical annealing temperatures ($T_{an}$) were selected using FactSage calculations and diagrams. Holding time was kept constant at 15 minutes and different types of cooling media were applied to the steel samples as shown in Fig. 3.2.

![Heat treatment schedule](image)

**Figure 3.2.** Heat treatment schedule.

All heat treatments were conducted in a radiation type furnace, which heats a sample by focusing infrared rays from four lamps. The radiant furnace was programmed to give a constant heating and cooling rate and holding time by a programmable controller, which was actuated by a chromel-alumel thermocouple attached to the sample surface. The temperature of the sample surface was concurrently monitored by a digital read-out installed in the controller. In order to prevent oxidization, all the specimens were protected by argon during annealing.
After intercritical annealing, the specimens were water quenched and air and furnace cooled. Cooling rates were measured by welding a type K (chromel: 90% nickel and 10% chromium) thermocouple on the center of the samples. The average cooling rate between 1200 and 800°C for air cooling was 25°C/sec and between 800°C to room temperature was 13°C/sec. The average cooling rate between 1200 and 800°C for furnace cooling was 5°C/sec, and for 800°C to room temperature it was 1.4°C/sec. In case of iced water quenched samples, the cooling rate could not be measured precisely with the available equipment since the thermocouple was removed from the surface during quenching. However, it was calculated approximately as 85°C/sec by using portable digital thermocouple without welding but placed in contact with the external surface of the specimen during cooling.

3.2.3. Cold Rolling

Annealed plates were cold rolled at different deformation ratios with various reheating processes. Details of the rolling procedure and deformation process in each step before reheating are given in the results section of this thesis. The rolling direction was the same as for the hot rolling without reversing and a lubricant was applied on the rolls. The rolling speed was approximately 35 revolutions per minute (RPM). An annealing schedule was done for the steel samples to maximize reduction before crack formation.

3.3. Sample Preparation

Specimens for optical and electron microscopy (SEM - EDX/EPMA, SEM-EBSD) and x-ray analysis were cut form the cross-section of the rolling direction (from both edges and the center of the sheet) and then hot mounted with Buehler Thermduo 3000 powder to preserve conductivity. Grinding was done with silicon carbide abrasive papers in the order of 240, 400, 600, 800, and 1200 grit. Polishing was done with 3 and 1 μm diamond suspension in an oil based diamond
lubricant. The final step of the metallographic preparation was polishing with 0.5 µm colloidal silica Struers OP-S suspension. For optical metallography, OP-S was applied for 1 to 2 min. For electron microscopy and electron back scattered diffraction (EBSD) samples OP-S was applied for 4-6 hours using a vibrometer.

In the case of EBSD, to index the phases for identification or mapping, as-polished samples were used. On the other hand, for optical microscopy, SEM-EDX/EPMA, and x-ray analysis samples, the following etchants were used to reveal the microstructure and boundaries more clearly.

- 2% Nital

- 2% Nital followed by 10% aqueous sodium metabisulfite (Na$_2$S$_2$O$_5$)

- LePera’s Etchant (equal portions of 1% aqueous Na$_2$S$_2$O$_5$ and 4% picral)

Etching by sodium metabisulfite makes ferrite gray, bainite or martensite black, and retained austenite white [101]. With LePera’s Etchant, martensite or retained austenite appears white, bainite appears black, ferrite appears grey or yellow (due to carbon concentration of surface), and, in most cases, although grain boundaries are not strongly etched, phases were clearly observed [101, 102].

### 3.4. Optical Microscopy

A Nikon Epiphot 200 (25X-1000X) optical microscope with automatic stage was used for optical microscopy. The microscope is linked to Clemex Captive Image acquisition and analysis software, which is also used for image analysis studies.

### 3.5. Electron Microscopy

A Philips XL-30 FE-SEM field emission gun scanning electron microscope operating at 5 to 30kV was used in this study. The microscope is equipped with an
in-lens secondary electron (SE) detector, a back scattered electron (BSE) detector, a TLC electron back scattered (EBSD) detector, and an EDS detector for chemical micro-analysis. High-resolution imaging of the specimens, EBSD phase mapping for identification of their phases, and finally EDS analysis for elemental distribution in their microstructure were analyzed using this microscope. In addition to these, a JEOL 8900 electron probe micro-analyzer (EPMA) was used for the determination of carbon partitioning in retained austenite and elemental mapping.

3.6. X-Ray Diffraction

A Bruker D8 Discovery X-Ray Diffractometer with Co-Kα radiation was used for x-ray diffraction (XRD) analyses. A laser-video alignment system, GADDS software, and HI-STAR area detector enables simple precision alignment of sample features for instant analysis such as phase identification and quantification. The detector position (2θ) was 30º and the apparatus was operated at 35kV accelerating voltage and 45mA beam current.

3.7. Calculation of Retained Austenite Volume Fraction

The retained austenite fractions of specimens were measured by using optical microscopy image analysis techniques and an X-ray diffractometer phase analysis system.

EVA - Diffrac Suite XRD analysis software was used for the analysis of the samples. This enables a qualitative analysis of the system, which is easier to perform for multiphase systems composed of different species. It consists of integrated and/or correlated x-ray diffraction intensity of a single peak, which is dependent upon numerous physical properties of the diffracting crystals and other properties that are related to the interactions and geometry of the x-ray beam impinging on the crystal.
The XRD Intensity Correlation or Direct Comparison method has been widely used to measure the amount of retained austenite in quenched steels. In this method, the retained austenite content was calculated from the integrated intensities of ferrite peaks (110), (200), and (211) and austenite peaks (111), (220), and (311) [103]. Since austenite has a different crystal structure (FCC) than martensite (BCT) and the other forms of steel (ferrite, bainite, and pearlite), the resulting diffraction pattern will also be different. Thus, the amount of retained austenite can be estimated by comparing the intensities of the diffraction peaks arising from each of the phases. In the absence of significant undissolved carbides and preferred orientation, there is a good correlation between the intensity ratio and the volume fraction of the retained austenite. Two standards (ASTM E975 and SAE SP-453) for austenite measurements are in common use. Both assume that the material has a nearly random orientation and has no carbides. The method is illustrated in Fig. 3.3, which compares the \((\alpha^1, \alpha)_{200}\) peak with the \((\gamma)_{220}\).

Figure 3.3. Sample diffraction pattern of S4 after homogenizing.
Finally, correction can be applied and the volume percent retained austenite can be computed with Eq. 3.2 [103]:

\[
V_\gamma = \left[ \frac{I_\gamma}{R_\gamma} \left( \frac{I_\alpha + \alpha^1}{R_{\alpha + \alpha^1}} \right) + \left( \frac{I_\gamma}{R_\gamma} \right) \right]
\] (3.2)

In case of image analyzing, the Clemex Captiva image acquisition and analysis software was used to capture a number of images to make measurements, cumulate data, and generate statistics, with results cumulated instantly. As can be seen from Fig. 3.4, the secondary phase formation is clearly differentiated from that of the matrix phase. Then the data taken from live and stored images are automatically displayed as histograms, scatter plots, and in the browser after each measurement. It is also important to state that there is an option in the software for using the ASTM standards.

![Figure 3.4](image)

**Figure 3.4.** Sample image analysis results of (a), (b) S3 and (c), (d) S4.

Finally, the results were compared with the XRD results, and the ferrite, retained austenite, and martensite fractions of specimens were measured accurately.
3.8. TEM

Thin foil specimens were prepared and examined using a JEOL 2010 transmission electron microscope (TEM) operating at 200kV. The specimen preparation involved mechanical thinning and jet polishing at 50V in a solution containing 340ml butanol, 600ml methanol, and 60ml perchloric acid at -20°C. In order to better observe the dislocations and twins, as well as the morphological variation of phases, a Philips CM-200 transmission electron microscope with a Philips EDS detector was employed.

Firstly, specimens, taken both from tensile testing and cold rolling, were thinned mechanically to a thickness below 80 μm by grinding paper up to 600 grit. Secondly, specimens were further thinned by using a Struers Tenupol-3 jet polisher in a solution of 90 vol pct methanol and 10 vol pct perchloric acid.

The cold rolled samples were taken from a cross-section of the samples 200 μm below the surface. However, it is important to state that only S4 composition was selected for TEM studies since it was the only material in which twinning was observed in initial microstructural characterization.

3.9. Shear Modulus and Poisson’s Ratio

For shear modulus and Poisson’s ratio calculations, ASTM E-132-04 and ASTM E-1876 were used in order.

ASTM E-132-04 is a test method that covers the determination of Poisson’s ratio from tension tests of structural materials at room temperature. This is a test method used for sheet metals or rectangular specimens. Poisson’s ratio calculations of this study were measured using a Zwick Z250 tensile machine with a 100 kN load cell in TUBITAK-MRC in Turkey. Tests were performed with a strain rate of 10 mm/min. In addition to this, both longitudinal and cross-section
strain gauges were adapted to test equipment to measure the negative of the ratio of transverse strain to the corresponding axial strain.

ASTM E-1876 is a standard test method for the calculation of dynamic shear modulus by impulse excitation of vibration. This test method covers the determination of the dynamic elastic properties of elastic materials at ambient temperatures. Specimens of these materials possess specific mechanical resonant frequencies that are determined by the elastic modulus, mass, and geometry of the test specimen. The dynamic elastic properties of a material can therefore be computed if the geometry, mass, and mechanical resonant frequencies of a suitable (rectangular or cylindrical geometry) test specimen of that material can be measured.

Young’s modulus is determined using the resonant frequency in either the flexural or longitudinal mode of vibration. The shear modulus is found using torsional resonant vibrations. The steps for direct contact transducer measurements are given below:

- The transducer is placed in contact with the test specimen to pick up the desired vibration. If the transducer is placed at an antinode (location of maximum displacement), it may mass load the specimen and modify the natural vibration.

- The transducer is placed only as far from the nodal points as is necessary to obtain a reading (see Fig. 3.5). This location will minimize the damping effect from the contacting transducer. The transducer contact force should be consistent, with good response and minimal interference with the free vibration of the specimen.

After the room temperature measurements of the resonance frequency of materials, the young modulus (E) is calculated using Eq. 3.3:

\[
E = 0.9465 \left( \frac{mf_j^2}{b} \right) \left( \frac{L^3}{t^3} \right) T_1
\]  

(3.3)
where, \( m \) is the mass of the plate, \( b \) is the width, \( L \) is the length, \( t \) is the thickness, \( f_f \) is the fundamental resonant frequency of bar in flexure (Hz) and \( T_1 \) is the correction factor for fundamental flexural mode to account for finite thickness of plate and poison ratio. \( T_1 \) is simply calculated following Eq. 3.4 (if \( L/t \geq 20 \)):

\[
T_1 = 1 + 6.585 \left( \frac{t}{L} \right)^2 \tag{3.3}
\]

If the Poisson’s ratio is measured experimentally, Eq. 3.4 can be used to calculate shear modulus (\( G \)) directly.

\[
\mu = \frac{E}{2G} - 1 \tag{3.4}
\]

**Figure 3.5.** Resonant frequency method contact and impulse points for rectangular specimen.

### 3.10. Mechanical Tests

The mechanical tests were done by means of static and dynamic test equipment. Static tests include shear punch and tensile tests. Yet, only dynamic shear punch testing was done. All static mechanical tests were performed using an MTS
Chapter 3: Experimental Procedure

hydraulic pressure machine, which is adaptable for both tensile and shear punch mode using anvils and pressure bars. The machine is shown schematically in Fig. 3.6.

Figure 3.6. Schematic illustration of MTS hydraulic pressure with tensile grips and shear punch-die.

3.10.1. Specimen Dimensions

Static shear punch tests were performed on a hydraulic MTS machine with a special punch-die fixture in which the specimen was sandwiched between the punch and the die (Fig. 3.7). The diameters of the punch and the die are 9.4 and 9.55 mm, respectively, and the punching speed was 0.1 mm/sec. Small square specimens, 30 mm by 30 mm, were cut out from the sheets for heat treatment, microstructural examination, and static and dynamic shear punch testing.

Tensile test samples were machined according to ASTM E-8 sub-size standard with a strain rate of 0.1 mm/sec. Jaw-type grips was used for tensile tests on the
MTS hydraulic testing machine. Figure 3.8 is a schematic illustration of the specimen for tensile testing, with the longitudinal direction along rolling direction. As can be seen, the specimen has a gauge region of 25 mm in length and 6.25 mm in width.

**Figure 3.7.** Shear punch-die fixture used in the experiments.

**Figure 3.8.** Tensile test sub-size specimen dimensions.
3.11. Dynamic Shear Punch

Figure 3.9 shows a schematic illustration of the Hopkinson bar system for dynamic shear punch testing. The strike bar, driven by compressed air, hits the incident bar at a very high speed and the incident bar deforms the specimen placed between it and the transmitted bar. The load and displacement are measured by means of strain gauges attached to the incident bar and transmitted bar. The signals from the strain gages are monitored and acquired by an oscilloscope. The incident bar, which is the punch, and the transmitted bar, which is the die, have the same diameters of 9.4 mm and 9.55 mm as that used in the static shear punch test, and the punching speed was 25 m/sec (90 km/h).

Figure 3.9. Illustration of the Hopkinson shear punch system.

3.12. Thermodynamic Calculation using FactSage

The FactSage software was used to predict the phase diagrams and transformation temperatures of austenite. FactSage, as a fully integrated computing system, has access to the alloys solutions developed by the international SGTE Group, steels databases, Spence Group database, GTT Technologies, CRCT, light metals, and other well-known systems. Figure 3.10 shows the main menu of the software.
Figure 3.10. Illustration of FactSage thermodynamic software.

In this thesis, the calculations were performed in the “Equilib” and “Phase Diagram” module. The results and the graphs were organized using the “Figure” module. A detailed explanation of diagrams and calculation will be given in the results section.
Chapter 4: Results

The first section of this chapter starts with the alloy design using thermodynamic analysis and databases to determine the equilibrium transformation behavior and SFE level of the candidate steel compositions. The following sub-sections describe the determination of heat treatment conditions after casting and hot rolling to emphasize the transformation behavior of austenite.

On the metallurgical side, the results of interest are the volume fraction of retained austenite in the heat treated samples at the various conditions of temperature, time, and cooling rate. The tensile flow curves generate the strength and ductility values of the steel compositions. Five samples were tested to determine the mechanical properties. Subsequently, two of the samples were used to measure the volume fraction of retained austenite using image analysis and XRD techniques as explained in detail in the previous section.

4.1. Alloy and Microstructure Design

4.1.1. Alloy Selection

Two alloying elements, Mn and C, were chosen as the main alloying elements for this study because they are the basic constituents in both TRIP and TWIP steels. Silicon and Al were also considered as a means to minimize Mn levels. Table 4.1 shows the effect of selected elements on the microstructure.

Since these alloys were intended as medium Mn steel candidate compositions, three important factors were considered in the calculations:

- The SFE level of austenite assuming the steel is 100% austenite or assuming the steel is multiphase and the austenite is retained.
- The volume fraction of the retained austenite.
The non-equilibrium C and Mn concentration of the secondary phase (retained austenite/martensite).

Table 4.1. Effect of alloying elements on the microstructure.

<table>
<thead>
<tr>
<th>Element</th>
<th>Austenite (γ) Stabiliser</th>
<th>SFE</th>
<th>Martensite (ε_{hcp}) Refinement</th>
</tr>
</thead>
<tbody>
<tr>
<td>C(HCP)</td>
<td>✓</td>
<td>↑</td>
<td>-</td>
</tr>
<tr>
<td>Mn(BCC)</td>
<td>✓</td>
<td>↓↑</td>
<td>✓</td>
</tr>
<tr>
<td>Al(FCC)</td>
<td>-</td>
<td>↑</td>
<td>-</td>
</tr>
<tr>
<td>Si(FCC)</td>
<td>-</td>
<td>↓</td>
<td>✓</td>
</tr>
</tbody>
</table>

✓: yes, -: no, ↓↑: decrease / increase

4.1.1.1. SFE Consideration

The stacking fault energy $\gamma_{SFE}$ of the Fe-C-Mn-Al-Si system was calculated for the possible composition range over the temperature range $0^\circ C \leq T \leq 400^\circ C$ based on Eq. 2.2. The thermodynamic datasets needed for the calculations were acquired from the literature, mainly from the tables published by the SGTE.

The molar surface density $\rho$ was taken according to Fig. 2.9 close to the close-packed plane of {111} ($2.86 \times 10^{-5}$ mol/m$^2$). The change in the molar Gibbs energy $\Delta G_{\gamma \rightarrow \varepsilon}$ upon the $\gamma_{FCC} \rightarrow \varepsilon_{HCP}$ phase transformation was calculated according to Eq. 2.3.

Figure 4.1 shows the effect of the alloying element on the SFE ($\gamma_{SFE}$) level plotted as a function of temperature. These calculations were initially done to obtain a SFE level of 20 mJ/m$^2$ (see in Fig. 2.5). The aim was to determine an optimum Mn concentration that provides strain induced transformation (either TRIP or TWIP) with this SFE level during deformation.
Figure 4.1. Effect of alloying element (a) Fe-Mn, (b) Fe-Mn-Al, (c) Fe-Mn-Si, and (d) Fe-Mn-C on the SFE level with temperature range between 0 and 400°C.
As can be seen in Fig. 4.1, Al and C are the elements that most effectively increase the SFE level with increasing temperature. The higher the Al and C content, the higher the SFE level, regardless of Mn and Si content. Therefore, C and Al were controlled to obtain the desired SFE level (Fig. 4.1 (d)). Indeed, it is crucial that as long as austenite is formed, the Mn content does not have a large effect on the SFE level below 10 wt.% (Fig. 4.1 (a)) but does influence the stability of the austenite.

Although Si wt.% has no effect on SFE at room temperature, it is important to also add Si to prevent carbon precipitation since C must be in solid solution to influence SFE. Therefore, the minimum negative SFE level was selected with 3 wt.% Si concentration. As noted above, Al is a very important alloying element to control SFE level; it gradually increases the SFE of austenite above 3 wt.% (Fig. 4.1 (b)). However, at relatively high Al levels, there can be processing disadvantages (e.g., weldability, galvanizing, and hot rolling) [104-106]. Therefore, the Al concentration was selected to be a maximum level of 3 wt.% and was kept constant with varying Mn, and C concentrations to be able to compare the steels. In Table 4.2, the room temperature SFE values are presented for the chosen compositions, which are listed in Table 3.1 and are repeated below, while in Fig. 4.2 the SFE of the alloys are plotted at room temperature with modeling variations due to different databases. The relative magnitudes of the SFE of the four steel compositions are $\gamma_{S2} > \gamma_{S1} > \gamma_{S3} > \gamma_{S4}$, but the SFE levels of all steels fall in the targeted value of 20 mJ/m$^2$.

**Table 4.2.** The stacking fault energies ($\gamma_{SFE}$) of steel compositions at room temperature.

<table>
<thead>
<tr>
<th>Sample Nr.</th>
<th>C (%)</th>
<th>Mn (%)</th>
<th>Si (%)</th>
<th>Al (%)</th>
<th>Mo (%)</th>
<th>Fe</th>
<th>SFE (mJ/m$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S-1</td>
<td>0.12</td>
<td>4.98</td>
<td>3.11</td>
<td>3.05</td>
<td>0.05</td>
<td>bal.</td>
<td>19.49</td>
</tr>
<tr>
<td>S-2</td>
<td>0.19</td>
<td>4.96</td>
<td>3.09</td>
<td>2.99</td>
<td>0.03</td>
<td>bal.</td>
<td>21.53</td>
</tr>
<tr>
<td>S-3</td>
<td>0.22</td>
<td>7.15</td>
<td>3.11</td>
<td>3.21</td>
<td>0.05</td>
<td>bal.</td>
<td>18.71</td>
</tr>
<tr>
<td>S-4</td>
<td>0.20</td>
<td>10.02</td>
<td>3.17</td>
<td>3.19</td>
<td>0.06</td>
<td>bal.</td>
<td>16.46</td>
</tr>
</tbody>
</table>
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Figure 4.2. The SFE of the alloys at room temperature with modeling deviations.

The alloys designed in this thesis have much lower Mn levels than the TWIP steels that were used to develop the thermodynamic model used here and, furthermore, are not 100% austenite. Unfortunately, there is no model in the literature that has been created for medium Mn steels. Therefore, we expect that the SFE calculations will not yield correct absolute values but might be appropriate to generate alloys with the desired SFE trends. Finally, it is also important to state that $\sigma^{\gamma/\epsilon}$, “the interfacial energy between $\gamma$ (FCC) and $\epsilon$ (HCP) phases,” was taken as 10 mJ/m² based on the studies of Olson and Cohen concerning high manganese steels [8]. However, recent studies show that this value varies between 8 and 13 mJ/m² [10, 13, 18], which may produce a ±10% modeling error in the calculations.

4.1.1.2. Retained Austenite Considerations

The input values used in the calculations are the composition of the system, constant pressure or volume condition, and temperature range. Five components,
Fe, Mn, C, Al, and Si, and three of the phases, liquid, ferrite, and austenite (immiscibility phase separation), were included in all calculations under 1 atm pressure.

The equilibrium volume fraction of phases is related directly to that of retained austenite. Hence, to estimate non-equilibrium phase formation during solidification, the Scheil cooling approach was used, which assumes a local equilibrium of the advancing solidification path of the two phase region. The wt.% of austenite were plotted for S1 to S4 with respect to Scheil cooling conditions, as can be seen from Fig. 4.3.

**Figure 4.3.** Formation of FCC (mass %) during solidification conditions.

These diagrams give a very important piece of information about the solidification path of steel compositions and in general are compatible with as-cast microstructures. It was predicted that the Scheil cooling diagrams do not represent the phase formation of alloys at room temperature conditions. However, it was expected to approximately estimate the total wt.% of the second phase formation
at room temperature (i.e., retained austenite plus the martensite formed during cooling).

Predicted values of the amount of the austenite wt.% at room temperature are given in Table 4.3. Referring to the compositions of the alloys (Table 3.1), it can be concluded that the amount of austenite of S1 is small, since there is smaller content of C and Mn. S2, which has the same Mn content but with a little more C, has a slightly higher austenite content than S1. Much higher austenite levels were obtained from S3 and S4 with higher C and Mn concentrations. The most interesting result is that in S4, it seems to be the austenite becomes the primary phase and the ferrite is the second phase.

**Table 4.3.** Calculated equilibrium wt.% of ferrite and austenite in each alloy.

<table>
<thead>
<tr>
<th>Sample Nr.</th>
<th>% Ferrite (α)</th>
<th>% Austenite (γ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>77.27</td>
<td>22.73</td>
</tr>
<tr>
<td>S2</td>
<td>75.18</td>
<td>24.82</td>
</tr>
<tr>
<td>S3</td>
<td>62.52</td>
<td>37.48</td>
</tr>
<tr>
<td>S4</td>
<td>42.87</td>
<td>57.13</td>
</tr>
</tbody>
</table>

Using FactSage thermodynamic software, the isopleth iron-carbon (Fe-C) phase diagrams of steel compositions, in particular BCC - FCC phase boundaries in equilibrium conditions, were also generated. The diagrams are shown in the temperature range of interest from 0°C to 1600°C in Figs. 4.4-4.7.

These medium Mn, low C steels lead to austenite existing only in a two phase region (α + γ). Manganese raises the solvus, lowers the two phase transition transformation temperatures, and enlarges the two phase (α + γ) field, which reflects the Scheil cooling predictions.
Figure 4.4. The calculated isopleth phase diagram for S1 composition with the wt.% of C being fixed at 0.1%.

Figure 4.5. The calculated isopleth phase diagram for S2 composition with the wt.% of C being fixed at 0.2%.
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Figure 4.6. The calculated isopleth phase diagram for S3 composition with the wt. % of C being fixed at 0.2%.

Figure 4.7. The calculated isopleth phase diagram for S4 composition with the wt.% of C being fixed at 0.2%.
4.1.2. **Heat Treatment**

The main objective of the heat treatment was to maximize the amount of martensite in preparation for a Q&PT treatment, which would possibly optimize strength and ductility. Therefore, all heat treatment sequences involved two steps to obtain the final microstructure. Firstly, a ferrite + austenite (BCC + FCC) structure was generated by an intercritical anneal (Figs. 4.4-4.7). Secondly, the steels were quenched to prepare for the Q and PT treatment.

Annealing temperatures of $T_{\text{an}-1}$ were determined from Fig. 4.8 as 950°C for S-1, 1020°C for S-2, 1155°C for S-3, and 1240°C for S-4 with their maximum FCC % content. This diagram was obtained from the equilibrium calculations of FactSage. It was generally observed that with increasing Mn content, the desired annealing temperature increased. **Table 4.4** gives the annealing temperatures as well as the corresponding maximum FCC wt.% of the steels. Note that in contrast to the Scheil cooling results, the FCC wt.% of S2 is double that of S1, despite the fact that the Mn levels are the same.

![% FCC vs T(C)](image)

**Figure 4.8.** The change of FCC vol. % as a function of temperature (°C).
The stability of the austenite formed at $T_{\text{an}-1}$ in resisting non-equilibrium transformation on cooling is a function of the intercritical annealing temperature via the C content of the $T_{\text{an}-1}$ austenite; the higher the carbon level at $T_{\text{an}-1}$, the more metastable austenite will be retained at room temperature for a given cooling rate. However, in this work, it was decided to choose the $T_{\text{an}-1}$ that gave the highest level of intercritical FCC.

**Table 4.4.** FCC wt.% content of steel compositions at elevated temperatures.

<table>
<thead>
<tr>
<th>Sample Nr.</th>
<th>Temperature (°C)</th>
<th>FCC (wt.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>950</td>
<td>22</td>
</tr>
<tr>
<td>S2</td>
<td>1020</td>
<td>25</td>
</tr>
<tr>
<td>S3</td>
<td>1150</td>
<td>42</td>
</tr>
<tr>
<td>S4</td>
<td>1200</td>
<td>62</td>
</tr>
</tbody>
</table>

A second annealing temperature ($T_{\text{an}-2}$) was chosen as 900°C for all the steel compositions to maximize the C concentration in the retained austenite. The C concentration in austenite increases with decreasing temperature (Fig. 4.9), but below 900°C, FactSage predicts no FCC. As well, the lower annealing temperature is expected to generate finer polygonal ferrite.

![C % vs T (C)](image)

**Figure 4.9.** C % of austenite as a function of T (°C) in all steel compositions.
4.2. Microstructure Evolution

As a result of the above heat treatments, a variety of microstructures were created, as overviewed in Table 4.5. In general, the microstructure constituents change according to the both annealing temperature and cooling rate. As explained above section, the aim was increasing the retained austenite formation and limiting the transformation of austenite by products (i.e. martensite and/or bainite).

**Table 4.5.** Detailed parameters used in various annealing processes.

<table>
<thead>
<tr>
<th>Sample Nr.</th>
<th>Ann. Nr</th>
<th>T&lt;sub&gt;an&lt;/sub&gt; (°C)</th>
<th>t&lt;sub&gt;an&lt;/sub&gt; (min)</th>
<th>Cooling</th>
<th>Microstructure</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>T&lt;sub&gt;an&lt;/sub&gt;-1</td>
<td>950</td>
<td>10</td>
<td>WQ</td>
<td>F + M + RA</td>
</tr>
<tr>
<td>S1</td>
<td>T&lt;sub&gt;an&lt;/sub&gt;-2</td>
<td>900</td>
<td>15</td>
<td>air</td>
<td>F + M/B + RA</td>
</tr>
<tr>
<td>S1</td>
<td>T&lt;sub&gt;an&lt;/sub&gt;-3</td>
<td>1100</td>
<td>30</td>
<td>FC</td>
<td>F + P/ B + RA</td>
</tr>
<tr>
<td>S2</td>
<td>T&lt;sub&gt;an&lt;/sub&gt;-1</td>
<td>1020</td>
<td>15</td>
<td>WQ</td>
<td>F + M + RA</td>
</tr>
<tr>
<td>S2</td>
<td>T&lt;sub&gt;an&lt;/sub&gt;-2</td>
<td>900</td>
<td>15</td>
<td>air</td>
<td>F + M/B + RA</td>
</tr>
<tr>
<td>S2</td>
<td>T&lt;sub&gt;an&lt;/sub&gt;-3</td>
<td>1100</td>
<td>30</td>
<td>FC</td>
<td>F + P/ B + RA</td>
</tr>
<tr>
<td>S3</td>
<td>T&lt;sub&gt;an&lt;/sub&gt;-1</td>
<td>1150</td>
<td>10</td>
<td>WQ</td>
<td>F + M + RA</td>
</tr>
<tr>
<td>S3</td>
<td>T&lt;sub&gt;an&lt;/sub&gt;-2</td>
<td>900</td>
<td>15</td>
<td>air</td>
<td>F + M/B + RA</td>
</tr>
<tr>
<td>S3</td>
<td>T&lt;sub&gt;an&lt;/sub&gt;-3</td>
<td>1100</td>
<td>30</td>
<td>FC</td>
<td>F + P/ B + RA</td>
</tr>
<tr>
<td>S4</td>
<td>T&lt;sub&gt;an&lt;/sub&gt;-1</td>
<td>1200</td>
<td>10</td>
<td>WQ</td>
<td>F + M + RA</td>
</tr>
<tr>
<td>S4</td>
<td>T&lt;sub&gt;an&lt;/sub&gt;-2</td>
<td>900</td>
<td>15</td>
<td>air</td>
<td>F + M/B + RA</td>
</tr>
<tr>
<td>S4</td>
<td>T&lt;sub&gt;an&lt;/sub&gt;-3</td>
<td>1100</td>
<td>30</td>
<td>FC</td>
<td>F + P/ B + RA</td>
</tr>
</tbody>
</table>

T<sub>an</sub>: annealing temperature; t<sub>an</sub>: annealing time; WQ: water quenching; FC: furnace cooling;
F: ferrite, M: martensite, B: bainite, R. A.: retained austenite
4.2.1. **Steel 1**

The as-cast, homogenized, and hot rolled microstructures and various constituents of S1 are shown in **Fig. 4.10**. The XRD patterns of the given microstructures presented in **Fig. 4.11** and the quantified results of phase fractions, including those obtained by microscopy, are listed in **Table 4.6**.

![Figure 4.10](image)

**Figure 4.10.** The microstructure of S1 (a) as-cast, (b) homogenized, and (c) hot rolled.

The matrix was observed to be ferrite, with retained austenite in both as-cast and homogenized structures. There were dark blue needle-like bands in the austenite after etching with 2% Nital followed by 10% aqueous sodium metabisulfite (Na₂S₂O₅). These structures are martensites nucleated within the austenite islands. In the case of the hot rolled microstructures, most of the retained austenite was transformed. This is due to the thermo-mechanical transformation of austenite during hot rolling.
Figure 4.11. XRD results of as-cast, homogenized, and hot rolled S1.

Table 4.6. The phase volume fraction of S1.

<table>
<thead>
<tr>
<th>S1</th>
<th>Vol. % Ferrite</th>
<th>Vol. % Austenite</th>
<th>Vol. % Martensite</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-cast</td>
<td>79.8</td>
<td>11</td>
<td>9.2</td>
</tr>
<tr>
<td>Homogenized</td>
<td>82</td>
<td>7.4</td>
<td>10.6</td>
</tr>
<tr>
<td>Hot Rolled</td>
<td>86</td>
<td>3.2</td>
<td>10.8</td>
</tr>
</tbody>
</table>

The microstructures of the steels after $T_{an-1}$, $T_{an-2}$, and $T_{an-3}$ annealing are given in Fig. 4.12. XRD patterns of the given microstructures presented in Fig. 4.13 and the quantified results of phase fractions, including those obtained by microscopy, are listed in Table 4.7 (M/B: Martensite and/or Bainite).

As can be seen in Fig. 4.13, the annealed microstructures consist of ferrite (lighter), martensite and pearlite (darker-blackish) as expected, and retained austenite (white). For the detailed characterization of the retained austenite, the volume percentage of the retained austenite was calculated after each process steps by using both image analysis techniques and the XRD intensity correlation method as explained in section 3.7.
Figure 4.12. The microstructures of annealed S1 (a) $T_{an}$-1, (b) $T_{an}$-2, and (c) $T_{an}$-3.

Figure 4.13. The XRD results of annealed S1 (a) $T_{an}$-1, (b) $T_{an}$-2, and (c) $T_{an}$-3.
After annealing with $T_{an-1}$ and $T_{an-2}$, the grain size is finer as a result of the lower annealing temperatures. Mainly as a result of the fast cooling rates, $T_{an-1}$ has much higher martensite than $T_{an-2}$, which is also nucleated inside the austenite grains. In both annealing treatments, the amount of retained austenite is similar to the levels found in TRIP steels.

**Table 4.7.** The phase volume fraction of S1 after the annealing process.

<table>
<thead>
<tr>
<th>S1</th>
<th>Vol. % Ferrite</th>
<th>Vol. % Austenite</th>
<th>Vol. % M/B</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T_{an-1}$</td>
<td>74</td>
<td>15.2</td>
<td>10.8</td>
</tr>
<tr>
<td>$T_{an-2}$</td>
<td>76</td>
<td>17</td>
<td>4</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Vol. % Ferrite</th>
<th>Vol. % Austenite</th>
<th>Vol. % Pearlite</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T_{an-3}$</td>
<td>84</td>
<td>-</td>
</tr>
</tbody>
</table>

Quantitatively, there is not much difference between these two heat treatments, but there is a slightly higher retained austenite for $T_{an-2}$ (Table 4.7). In the case of $T_{an-3}$, the slow cooling rate has transformed all the austenite, generating pearlite as opposed to martensite, and the ferrite volume fractions are higher.

Finally, the microstructures of the cold rolled samples are given in Fig. 4.14, the XRD patterns of the given microstructures are presented in Fig. 4.15, and the quantitative values are listed in Table 4.8.

In this cold rolling process, the important feature is the behavior of the retained austenite. The retained austenite transformed to martensite (either $\alpha^1$ or $\varepsilon$) due to cold rolling. For S1, after cold rolling there is significantly more retained austenite transformed in the $T_{an-2}$ condition compared to the $T_{an-1}$ condition. On the other hand, some retained austenite has been detected in the $T_{an-3}$ specimen, but this is a negligible amount considering the other two annealing processes.
Figure 4.14. The microstructures of cold rolled S1 after (a) $T_{an}$-1, (b) $T_{an}$-2, and (c) $T_{an}$-3.

Figure 4.15. The XRD results of cold rolled S1 after (a) $T_{an}$-1, (b) $T_{an}$-2, and (c) $T_{an}$-3.
Table 4.8. The austenite volume fraction for S1 before and after cold rolling.

<table>
<thead>
<tr>
<th>S1</th>
<th>Before Cold Rolling</th>
<th>After Cold Rolling</th>
<th>$\Delta_{\text{transformed}}\ %$ $(\gamma \rightarrow \varepsilon / \alpha^1)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T_{\text{an}-1}$</td>
<td>15.2</td>
<td>7.2</td>
<td>8</td>
</tr>
<tr>
<td>$T_{\text{an}-2}$</td>
<td>17</td>
<td>4.3</td>
<td>12.7</td>
</tr>
<tr>
<td>$T_{\text{an}-3}$</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

4.2.2. **Steel 2**

The microstructure evolution of S2 (as-cast, homogenized, and hot rolled) is shown in Fig. 4.16. The XRD patterns and the quantified results for phase fractions of the given microstructures are presented in Fig. 4.17 and Table 4.9.

Figure 4.16. The microstructure of S2 (a) as-cast, (b) homogenized, and (c) hot rolled.
Optical micrographs showed that, similar to S1, S2 also has a multiphase microstructure in as-cast and homogenized samples, including ferrite, retained austenite, and martensite. In these structures, needle-like martensite was observed nucleated within the austenite islands. In the case of hot rolled samples, there was a dual phase structure including ferrite (light yellow) and martensite (dark blue) grains after etching with 2% Nital. Compared to S1, less austenite was observed due to a higher C concentration.

![XRD spectra of S2](image)

**Figure 4.17.** The XRD results of as-cast, homogenized, and hot rolled S2.

<table>
<thead>
<tr>
<th>S2</th>
<th>Vol. % Ferrite</th>
<th>Vol. % Austenite</th>
<th>Vol. % Martensite</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-cast</td>
<td>64.2</td>
<td>8</td>
<td>27.8</td>
</tr>
<tr>
<td>Homogenized</td>
<td>72.6</td>
<td>7.4</td>
<td>20</td>
</tr>
<tr>
<td>Hot Rolled</td>
<td>64.5</td>
<td>2.1</td>
<td>33.4</td>
</tr>
</tbody>
</table>

**Table 4.9.** The phase volume fraction of S2.

The microstructures and X-ray diffraction patterns of S2 after T\(_{an}\)-1, T\(_{an}\)-2, and T\(_{an}\)-3 intercritical annealing are given in **Fig. 4.18** and **Fig. 4.19**, respectively. The quantified results of phase fractions are listed in **Table 4.10**, with martensite values generated by optical metallography.
**Figure 4.18.** The microstructures of annealed S2 (a) $T_{an}$-1, (b) $T_{an}$-2, and (c) $T_{an}$-3.

**Figure 4.19.** The XRD results of annealed S2 (a) $T_{an}$-1, (b) $T_{an}$-2, and (c) $T_{an}$-3.
Table 4.10. The phase volume fraction of S2 after the annealing process.

<table>
<thead>
<tr>
<th></th>
<th>Vol. % Ferrite</th>
<th>Vol. % Austenite</th>
<th>Vol. % M/B</th>
</tr>
</thead>
<tbody>
<tr>
<td>T\textsubscript{an}-1</td>
<td>63</td>
<td>9.1</td>
<td>27.9</td>
</tr>
<tr>
<td>T\textsubscript{an}-2</td>
<td>60</td>
<td>14</td>
<td>24</td>
</tr>
<tr>
<td>T\textsubscript{an}-3</td>
<td>86</td>
<td>-</td>
<td>14</td>
</tr>
</tbody>
</table>

The T\textsubscript{an}-1 heat treatment produced dual phase structures, which were clearly observed from optical metallography and quantitative analysis (Figs. 4.18 and 4.19 and Table 4.10). This consists of a ferrite matrix with martensite and a small quantity of retained austenite.

In T\textsubscript{an}-2 annealing, the areas displayed as grey are ferrite, the bright white ones are retained austenite, and the rest are martensite and bainite. One of the interesting results of T\textsubscript{an}-2 annealing is that the retained austenite in T\textsubscript{an}-2 is much higher than in T\textsubscript{an}-1.

In the case of T\textsubscript{an}-3, as for steel S2, the high temperature and very slow cooling rate led to microstructure consisting of ferrite and pearlite with no retained austenite and martensite formation (Fig. 4.19 and Table 4.10).

The morphologies of cold rolled S2 were similar to S1 and representative micrographs are shown in Fig. 4.20. X-ray diffraction patterns of the examined steels and their quantified results of phase fractions are given in Fig. 4.21 and Table 4.11, respectively.

In the case of S2, any retained austenite largely transformed to martensite (either thermally transformed $\alpha'$ or strain induced $\varepsilon$) due to the cold rolling and re-annealing procedures. Similar to S1, there is significantly more retained austenite transformed in the T\textsubscript{an}-2 condition compared to the T\textsubscript{an}-1 condition. No retained austenite has been detected in the T\textsubscript{an}-3.
Figure 4.20. The microstructures of cold rolled S2 after (a) T_{an}-1, (b) T_{an}-2, and (c) T_{an}-3.

Figure 4.21. The XRD results of cold rolled S2 after (a) T_{an}-1, (b) T_{an}-2, and (c) T_{an}-3.
Table 4.11. The austenite volume fraction for S2 before and after cold rolling.

<table>
<thead>
<tr>
<th>S2</th>
<th>Before Cold Rolling</th>
<th>After Cold Rolling</th>
<th>$\Delta_{\text{transformed}}$ % $(\gamma \rightarrow \varepsilon, \alpha')$</th>
</tr>
</thead>
<tbody>
<tr>
<td>T$_{\text{an}}$-1</td>
<td>9.1</td>
<td>4.8</td>
<td>4.3</td>
</tr>
<tr>
<td>T$_{\text{an}}$-2</td>
<td>14</td>
<td>2.6</td>
<td>11.4</td>
</tr>
<tr>
<td>T$_{\text{an}}$-3</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

4.2.3. **Steel 3**

The microstructure of S3 (as-cast, homogenized, and hot rolled) is shown in Fig. 4.22 and the XRD patterns with the quantified results for phase fractions is given in Table 4.12. The microstructures are presented in Fig. 4.23.

The microstructures of S3 were similar to the S1 and S2. Representative micrographs of as-cast, homogenized, and hot rolled samples are presented in Fig. 4.22; brown and bluish regions due to etching are evidence of martensite formation in the (white) retained austenite islands and grey regions represent the ferrite phases in the matrix.

In particular, martensite and retained austenite was observed in both as-cast and homogenized samples, but no retained austenite was observed in hot rolled samples Figs. 4.22-23 and Table 4.12. As suggested for S1 and S2, it was thought that the remaining retained austenite after homogenizing was transformed to ferrite and martensite due to thermomechanical processes. Note that, both hot rolling processes (in CANMET and TUBITAK) for steel samples was tried to perform at relatively high temperatures and then directly quenched as mentioned in the section 3.2.1.
Figure 4.22. The microstructure of S3 (a) as-cast, (b) homogenized, and (c) hot rolled.

Figure 4.23. The XRD results of as-cast, homogenized, and hot rolled S3.
Table 4.12. The phase volume fraction of S3.

<table>
<thead>
<tr>
<th>S3</th>
<th>Vol. % Ferrite</th>
<th>Vol. % Austenite</th>
<th>Vol. % Martensite</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-cast</td>
<td>64.2</td>
<td>8</td>
<td>27.8</td>
</tr>
<tr>
<td>Homogenized</td>
<td>72.6</td>
<td>7.4</td>
<td>20</td>
</tr>
<tr>
<td>Hot Rolled</td>
<td>58</td>
<td>6.6</td>
<td>35.4</td>
</tr>
</tbody>
</table>

The annealed S3 microstructures are shown in Fig. 4.24 and the XRD patterns with the quantified results for phase fractions are given in Fig. 4.25 and Table 4.13. The microstructure of the annealed samples observed using optical microscopy, similar to S1 and S2, again showed that the microstructure consists of three components: ferrite, martensite, and retained austenite in T_{an-1}. After the T_{an-2} treatment, the microstructure contains some bainite with ferrite, martensite, and retained austenite as a result of slow cooling. In the case of T_{an-3}, there were only two components: ferrite and pearlite.

![Figure 4.24. The microstructures of annealed S3 (a) T_{an-1}, (b) T_{an-2}, and (c) T_{an-3}.](image-url)
Figure 4.25. The XRD results of annealed S3 after (a) $T_{an}^1$, (b) $T_{an}^2$, and (c) $T_{an}^3$.

<table>
<thead>
<tr>
<th>S3</th>
<th>Vol. % Ferrite</th>
<th>Vol. % Austenite</th>
<th>Vol. % M/B</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T_{an}^1$</td>
<td>51</td>
<td>17.1</td>
<td>31.9</td>
</tr>
<tr>
<td>$T_{an}^2$</td>
<td>57</td>
<td>20.5</td>
<td>22.5</td>
</tr>
<tr>
<td>$T_{an}^3$</td>
<td>87</td>
<td>1.7</td>
<td>11.3</td>
</tr>
</tbody>
</table>

It can be seen from Figs. 4.24-25 and Table 4.13 that with the decrease of the cooling rate, the fraction of martensite decreases and the microstructural features became coarser. It is also notable that $T_{an}^2$ has slightly more retained austenite compared to $T_{an}^1$, probably because of the lower annealing temperature and slower cooling rate. In the case of $T_{an}^3$, the amount of retained austenite is negligible because of the very slow cooling rate. Furthermore, compared to S1 and S2, the total volume percentage of secondary phase formation (retained austenite plus martensite/pearlite) was increased with increases in the Mn content.
of the alloy. However, Table 4.13 clearly showed that this secondary phase formation concluded with more martensite transformation, instead of stabilizing retained austenite.

The microstructure of cold rolled S3 after annealing with T_{an}-1, T_{an}-2, and T_{an}-3 is shown in Fig. 4.26 and the XRD patterns with the quantified results for phase fractions is given microstructures are presented in Fig. 4.27 and Table 4.14.

Figure 4.26. The microstructures of cold rolled S3 after (a) T_{an}-1, (b) T_{an}-2, and (c) T_{an}-3.

The retained austenite totally transformed to martensite (either thermally transformed α¹ or strain induced ε) due to the cold rolling and re-annealing procedures. Although the existence of a small amount of retained austenite has been revealed by XRD, it could not be observed by using optical microscopy and, instead, martensite was seen. Compared with the result of S1 and S2, higher Mn content of the composition seems to increase austenite stability, resulting in higher amounts of retained austenite stabilized during cold rolling. However, it
was also observed that martensite formation was also increased and, once again, after cold rolling there is significantly more retained austenite transformed in the T_{an}-2 condition compared to the T_{an}-1 condition.

![XRD spectrum](image)

**Figure 4.27.** The XRD results of cold rolled S3 after (a) T_{an}-1, (b) T_{an}-2, and (c) T_{an}-3.

**Table 4.14.** The austenite volume fraction for S3 before and after cold rolling.

<table>
<thead>
<tr>
<th></th>
<th>Before Cold Rolling</th>
<th>After Cold Rolling</th>
<th>Δ_{transformed} (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S3</td>
<td>Austenite Volume</td>
<td>Austenite Volume</td>
<td>(γ → ε / α')</td>
</tr>
<tr>
<td></td>
<td>Fraction (%)</td>
<td>Fraction (%)</td>
<td></td>
</tr>
<tr>
<td>T_{an}-1</td>
<td>17.1</td>
<td>13.7</td>
<td>3.4</td>
</tr>
<tr>
<td>T_{an}-2</td>
<td>20.5</td>
<td>5.7</td>
<td>14.8</td>
</tr>
<tr>
<td>T_{an}-3</td>
<td>1.7</td>
<td>-</td>
<td>1.7</td>
</tr>
</tbody>
</table>

4.2.4. **Steel 4**

As-cast, homogenized, and hot rolled optical micrographs and quantitative XRD results of S4 are given in **Fig. 4.28, Fig. 4.29, and Table 4.15.** As can be seen in **Fig. 4.28,** the microstructure of as-cast and homogenized samples consists of almost half ferrite (grey) and half retained austenite (yellow). After hot rolling,
second phase bands were observed parallel to the rolling direction. These bands are possibly martensite, although they could not be clearly etched.

Figure 4.28. The microstructure of S4 (a) as-cast, (b) homogenized, and (c) hot rolled.

Figure 4.29. The XRD results of as-cast, homogenized, and hot rolled S4.
Table 4.15. The phase volume fraction of S4.

<table>
<thead>
<tr>
<th>S4</th>
<th>Vol. % Ferrite</th>
<th>Vol. % Austenite</th>
<th>Vol. % Martensite</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-cast</td>
<td>44.2</td>
<td>55.8</td>
<td>-</td>
</tr>
<tr>
<td>Homogenized</td>
<td>49</td>
<td>51</td>
<td>-</td>
</tr>
<tr>
<td>Hot Rolled</td>
<td>68</td>
<td>4.3</td>
<td>27.7</td>
</tr>
</tbody>
</table>

The microstructures of S4 after $T_{an-1}$, $T_{an-2}$, and $T_{an-3}$ annealing are given in Fig. 4.30. The volume percentage of the phases and the quantified results of phase fractions were presented with XRD patterns in Fig. 4.31 and listed in Table 4.16 for the given microstructures.

The first noticeable point in the microstructures of the annealed samples showed more or less equiaxed austenitic grains with a certain amount of annealing twins (Fig. 4.30). Limited amounts of martensite and pearlite were observed both in micrographs and XRD analysis.

![Figure 4.30](image)

Figure 4.30. The microstructures of annealed S4 (a) $T_{an-1}$, (b) $T_{an-2}$, and (c) $T_{an-3}$.
Figure 4.31. The XRD results of cold rolled S4 after (a) $T_{an}$-1, (b) $T_{an}$-2, and (c) $T_{an}$-3.

Table 4.16. The phase volume fraction of S4 after the annealing process.

<table>
<thead>
<tr>
<th>S4</th>
<th>Vol. % Ferrite</th>
<th>Vol. % Austenite</th>
<th>Vol. % Martensite</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T_{an}$-1</td>
<td>43</td>
<td>53</td>
<td>4</td>
</tr>
<tr>
<td>$T_{an}$-2</td>
<td>48</td>
<td>47</td>
<td>5</td>
</tr>
<tr>
<td>$T_{an}$-3</td>
<td>47</td>
<td>47.5</td>
<td>5.5</td>
</tr>
</tbody>
</table>

In particular, S4 is quite different to the other alloys in that $T_{an}$-1 leads to a duplex microstructure, which consists only of ferrite and austenite. After $T_{an}$-2, the grain size is more or less same in size, and the duplex microstructure persists but apparently with more annealing twins. The $T_{an}$-3 treatment seems to be no different from the $T_{an}$-2 treatment, which is also a significant departure from the other three steels. As will be discussed later, this seems to indicate that the austenite is much more stable in S4, which could be due to the C and Mn partitioning level. Quantitatively, $T_{an}$-1 yields the highest volume fraction of retained austenite. On the other hand, even with the very different annealing treatments, $T_{an}$-2 and $T_{an}$-3 had similar amounts of ferrite and retained austenite.

The microstructures of the cold rolled samples are given in Fig. 4.32, the XRD patterns of the given microstructures are presented in Fig. 4.33, and the quantitative values are listed in Table 4.17.
Figure 4.32. The microstructures of cold rolled S4 after (a) T_{an}-1, (b) T_{an}-2, and (c) T_{an}-3.

Figure 4.33. The XRD results of cold rolled S4 after (a) T_{an}-1, (b) T_{an}-2, and (c) T_{an}-3.
For S4, cold rolling also transformed a significant fraction of the retained austenite to martensite, as shown in Fig. 4.33 and Table 4.17, with all heat treatments showing similar amounts of transformed austenite. Moreover, deformation twins in the austenite were seen in all cold rolled samples, with twins and martensites occasionally seeming to co-exist in the same austenite grain.

Table 4.17. The austenite volume fraction for S4 before and after cold rolling.

<table>
<thead>
<tr>
<th>S4</th>
<th>Before Cold Rolling</th>
<th>After Cold Rolling</th>
<th>$\Delta_{\text{transformed}}$% $(\gamma \rightarrow \varepsilon / \alpha)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T_{\text{an}-1}$</td>
<td>53</td>
<td>32.7</td>
<td>20.3</td>
</tr>
<tr>
<td>$T_{\text{an}-2}$</td>
<td>47</td>
<td>23.2</td>
<td>23.8</td>
</tr>
<tr>
<td>$T_{\text{an}-3}$</td>
<td>47.5</td>
<td>28.4</td>
<td>19.1</td>
</tr>
</tbody>
</table>

4.3. Typical Tensile Properties

The room temperature mechanical properties of steel alloys were measured by the tensile tests at static strain rate. Engineering stress-strain diagrams were plotted with the standard deviations, from which the values of ultimate tensile strength ($\sigma_{UTS}$) and total elongation (% $\varepsilon_{\text{total}}$) values were generated.

In general, it was observed that increasing annealing time and temperature decreased the strength of these materials significantly but only slightly increased the ductility. After annealing at $T_{\text{an}-1}$ and $T_{\text{an}-2}$, a tensile strength of 800-900 MPa and total elongation of about 10 to 30% was obtained. Tensile strength decreased strongly after $T_{\text{an}-3}$ for all steel compositions to 500-700 MPa, but the total elongation remained largely unchanged (15-25%). As a result, the highest strength was obtained by S2 at $T_{\text{an}-2}$, and the highest ductility was obtained from S4 at $T_{\text{an}-1}$ annealing condition.
4.3.1. **Steel 1**

Figure 4.34 and **Table 4.18** shows typical engineering stress-strain diagrams and mechanical properties of S1 after annealing the hot rolled samples.

![Engineering Stress vs. Strain Diagram](image)

**Figure 4.34.** Engineering stress vs. strain diagram of S1 at static strain rate.

<table>
<thead>
<tr>
<th>S1</th>
<th>UTS (MPa)</th>
<th>% $\varepsilon_{\text{total}}$</th>
<th>Rp0.2 (MPa)</th>
<th>StDv (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T_{\text{an}-1}$ + Tensile</td>
<td>799</td>
<td>17</td>
<td>691</td>
<td>2.43</td>
</tr>
<tr>
<td>$T_{\text{an}-2}$ + Tensile</td>
<td>858</td>
<td>15</td>
<td>766</td>
<td>4.11</td>
</tr>
<tr>
<td>$T_{\text{an}-3}$ + Tensile</td>
<td>674</td>
<td>21</td>
<td>557</td>
<td>2.1</td>
</tr>
</tbody>
</table>

**Table 4.18.** The mechanical properties of S1 after tensile tests.

The results showed that $T_{\text{an}-2}$ has the highest UTS values within the annealed samples that also had the highest martensite volume fraction. On the other hand, there was a large decrease in the UTS values from 855 MPa to 674 MPa after annealing with $T_{\text{an}-3}$, and the highest ductility was obtained with this annealing condition.
4.3.2. Steel 2

Typical engineering stress-strain diagrams and the room temperature mechanical properties of S2 are given in Fig. 4.35 and Table 4.19.

![Engineering stress vs. strain diagram of S2 at static strain rate.](image)

**Figure 4.35.** Engineering stress vs. strain diagram of S2 at static strain rate.

**Table 4.19.** The mechanical properties of S2 after tensile tests.

<table>
<thead>
<tr>
<th>S2</th>
<th>UTS (MPa)</th>
<th>(%\ v_{total})</th>
<th>Rp0.2 (MPa)</th>
<th>StDv (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T\textsubscript{an}-1 + Tensile</td>
<td>828</td>
<td>17</td>
<td>716</td>
<td>2.53</td>
</tr>
<tr>
<td>T\textsubscript{an}-2 + Tensile</td>
<td>941</td>
<td>11</td>
<td>784</td>
<td>3.01</td>
</tr>
<tr>
<td>T\textsubscript{an}-3 + Tensile</td>
<td>561</td>
<td>16</td>
<td>484</td>
<td>1.8</td>
</tr>
</tbody>
</table>

One of the most important points about the mechanical properties of S2 was that the highest UTS value was obtained from T\textsubscript{an}-2 annealing within all the steel compositions. However, total elongation of S2 after all annealing treatments was relatively less than the other steel compositions.
4.3.3. **Steel 3**

The engineering stress vs. strain curve for the tension test, at quasi-static strain rate, is shown in **Fig. 4.36**, and the mechanical properties of S3 with different intercritical annealing conditions are presented in **Table 4.18**. At first sight, the results show a huge variety in terms of UTS between T\textsubscript{an}-1 and T\textsubscript{an}-2 compared to the T\textsubscript{an}-3 but more or less similar ductility range between each other. The most interesting mechanical behavior was obtained with T\textsubscript{an}-3 which had the lowest UTS values for all the steel compositions.

**Figure 4.36.** Engineering stress vs. strain diagram of S3 at static strain rate.

<table>
<thead>
<tr>
<th>S3</th>
<th>UTS (MPa)</th>
<th>% $\varepsilon_{\text{total}}$</th>
<th>Rp0.2 (MPa)</th>
<th>StDv (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T\textsubscript{an}-1 + Tensile</td>
<td>868</td>
<td>20</td>
<td>727</td>
<td>6.35</td>
</tr>
<tr>
<td>T\textsubscript{an}-2 + Tensile</td>
<td>928</td>
<td>15</td>
<td>761</td>
<td>1.61</td>
</tr>
<tr>
<td>T\textsubscript{an}-3 + Tensile</td>
<td>497</td>
<td>18</td>
<td>443</td>
<td>1.40</td>
</tr>
</tbody>
</table>
In the case of T\textsubscript{an}-1, it is clear that the UTS value of the steel compositions gradually increased with increasing Mn composition. But this effect was not observed with T\textsubscript{an}-2 samples. Indeed, in steel samples (S1, S2, and S3) annealed with T\textsubscript{an}-2, the UTS values were, in general, considerably higher than the samples annealed with T\textsubscript{an}-1, but no linear relationship was observed between the compositions.

### 4.3.4. Steel 4

Engineering tensile test curve are displayed in Fig. 4.37 for the three annealed samples, and Table 4.18 summarizes their mechanical properties.

**Figure 4.37.** Engineering stress vs. strain diagram of S4 at static strain rate.

Compared to the other three steels, S4 has significantly different tensile properties, as can be seen clearly from Table 4.18 after annealing treatments. Firstly, the T\textsubscript{an}-1 condition leads to a very high total elongation of 30\%, which is the highest of all the steel compositions, and it was balanced with a relatively high
UTS value of 868 MPa. Annealing with \( T_{an}^-2 \) led to slightly higher UTS but lower ductility. Finally, in the case of \( T_{an}^-3 \), the total elongation was compatible with \( T_{an}^-2 \) but at a much lower strength.

**Table 4.21.** The mechanical properties of S4 after tensile tests.

<table>
<thead>
<tr>
<th>S4</th>
<th>UTS (MPa)</th>
<th>( % \epsilon_{total} )</th>
<th>Rp0.2 (MPa)</th>
<th>StDv (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>( T_{an}^-1 + ) Tensile</td>
<td>868</td>
<td>31</td>
<td>637</td>
<td>7.72</td>
</tr>
<tr>
<td>( T_{an}^-2 + ) Tensile</td>
<td>873</td>
<td>23</td>
<td>694</td>
<td>2.45</td>
</tr>
<tr>
<td>( T_{an}^-3 + ) Tensile</td>
<td>685</td>
<td>25</td>
<td>567</td>
<td>1.62</td>
</tr>
</tbody>
</table>

4.4. **Static and Dynamic Property Relationship**

In this thesis, the static mechanical properties were performed using MTS hydraulic machine in shear punch mode with a static speed of 0.1 mm/sec and the dynamic mechanical properties were performed using a Hopkinson shear punch test with a dynamic speed of 25 m/sec at room temperature conditions. Selected annealing conditions (\( T_{an}^-1 \) and \( T_{an}^-2 \)) were tested and compared with tensile properties.

4.4.1. **Typical Static Shear Punch Curves**

Figures 4.38 to 4.41 show the force-displacement curves of static shear punch test curves of steel compositions. The static speed was 0.1 mm/sec and the tests were performed only for \( T_{an}^-1 \) and \( T_{an}^-2 \) annealed samples. The force-displacement data obtained from shear punch tests was used to compute the compliance of the results with the tensile and dynamic shear punch test results in the following sections. Thus, the mechanical properties shown hereafter, unless indicated, were all measured by shear punch tests at these two different heat treatment conditions.
Figure 4.38. Force-displacement curves of static shear punch test for S1.

Figure 4.39. Force-displacement curves of static shear punch test for S2.
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Figure 4.40. Force-displacement curves of static shear punch test for S3.

Figure 4.41. Force-displacement curves of static shear punch test for S4.
In this study, three samples were tested with a thickness of ~ 2 mm for each heat treatment condition of the steel alloys, and the average values are plotted. During the tests, a 100 N preload was applied to all steel samples that had the same preload values used in the static tensile tests.

In general, the force-displacements curves were similar to each other for both heat treatment conditions. Samples had slightly different thicknesses (± 0.1 mm), but these differences were not enough to introduce significant effects on the flow behavior. There was not much effect of the different heat treatments, but the $T_{an-2}$ annealed samples had slightly higher forces and lower displacements.

After annealing at $T_{an-1}$ and $T_{an-2}$, a maximum force of 42 to 49 kN and displacement range of 1.2 to 1.5 mm was obtained. The maximum force was obtained from S2 at $T_{an-2}$ annealing and the maximum displacement was obtained from S4 at $T_{an-1}$ annealing.

The effect of alloy composition on the force-displacement curves is shown in Fig. 4.42. For $T_{an-1}$ annealing, at low displacements there was little effect of alloy composition (Fig.4.42 (a)). Total displacement ($D_{total}$), i.e., displacement at fracture, increased from steels S1 to S4 in order. However, the trend was changed for the maximum applied force ($F_{max}$), with S2 being the highest.

The $T_{an-2}$ annealing results were somewhat different from $T_{an-1}$ at low displacements, exhibiting a slightly concave curve for all steels (Fig. 4.42 (b)). Again there was a trend in $D_{total}$ increasing from S1 to S4. Similar to the $T_{an-1}$ annealing, S2 had the highest maximum force at $T_{an-2}$ annealing, after which came S3, S4, and S1.
Figure 4.42. Comparison of force-displacement curves of steel samples.
4.4.2. **Comparison of Shear Punch and Tensile Properties**

In general, the force-displacement curve obtained during the punching operation is very similar to that obtained in a conventional uniaxial tensile test (e.g., in Fig. 4.43) preloading (a-b), elastic loading to the yield point (b-c), plastic deformation (c-d), and the ultimate stress at failure (or necking d-e).

However, the shear punch testing generates a non-uniform deformation zone and a complex stress state in which shearing, bending, tension, and compression stresses may occur as the material deforms [86]. It is, therefore, impossible to convert the whole shear punch force-displacement curve into a true stress-true strain curve. However, the properties obtained by analyzing the shear punch test curve can be correlated to the corresponding conventional tensile properties.

![Sample shear punch test curve for S4 at T\textsubscript{an}^-1](image)

**Figure 4.43.** Sample shear punch test curve for S4 at T\textsubscript{an}^-1.

By assuming that a pure shear stress occurs during shear punch deformation, the average shear stress $\tau$ can be related to the force $F$ using the **Eq. 4.1**:

\[
\tau = \frac{F}{W}
\]
\[ \tau = \frac{F}{2\pi rt} \]  \hfill (4.1)

where \( t \) is the specimen thickness and \( r \) is the average of the punch and die radii.

Some researchers \([7, 107, 108]\) studied the static mechanical properties of several materials, including some carbon and alloy steels, by both tensile and shear punch testing. Their results demonstrated that the tensile properties and shear punch properties can be correlated approximately by the following linear relations:

\[ \sigma_t = C \cdot \tau = C \cdot \left[ \frac{F_{\text{max}}}{(2\pi rt)} \right] \]  \hfill (4.2)

\[ RA = \frac{D_f}{t} \]  \hfill (4.3)

where \( \sigma_t, \sigma_y \) is yield or tensile strength, \( F_{\text{yield,maximum}} \) is the yielding or maximum shear punch force, \( RA \) is the tensile reduction in area, \( D_f \) is the failure shear punch displacement, and \( C \) is a regression factor.

The punch diameters used in the studies were diameter of die \( (d_d) \) 9.4 mm and diameter of hole \( (d_h) \) 9.5 mm; the specimen thickness was \( \sim 2 \) mm. The value of \( C \) for the UTS was found to be in the range of 1.05 to 1.13, which is not close to the Von Mises criterion factor \( (\sqrt{3}) \). This seems to indicate that it is questionable to assume that the shear punch deformation is sheared dominant. Furthermore, the \( C \) value for the yield strength was found to be relatively scattered due to variations in measuring yielding point. Some researchers used 1\% offset in the normalized displacement (displacement divided by punch-die clearance \([107, 109]\) or by specimen thickness \([108]\)) as yielding point instead of the deviation from linearity on the curve.

The shear stress-reduction in area (R.A.) curves for the steel alloys studied are plotted in Fig. 4.44. The shear maximum strength is computed from the peak points of the plots. The standard deviations for the measured shear maximum and reduction in area were \( \pm 3\% \) and \( \pm 6\% \) of the respective average values.
Figure 4.44. Shear stress (MPa) vs. reduction in area (R.A.) curves of steel samples (a) $T_{\text{an}-1}$ and (b) $T_{\text{an}-2}$ annealing condition.
A tensile-shear punch property correlation is based on comparing UTS with max shear strength and tensile ductility with maximum shear ductility [86]; the corresponding values from the shear tests and how they are determined is shown in Fig. 4.45 as an example.

![Diagram](image)

**Figure 4.45.** Evaluation of important factors on shear punch curves.

Figure 4.46 (a) shows the comparison of shear punch and tensile mechanical properties, the tensile strength UTS vs. the maximum shear strength (\(\tau_{\text{max}}\), and the total tensile elongation \(\varepsilon_{\text{total}}\) % vs. the reduction in area (R.A.), **Fig. 4.46 (b)**. A linear relationship between shear punch and tensile properties can be derived from both static and dynamic results, and is plotted in this figure.
Figure 4.46. Comparison of shear punch and mechanical properties (a) tensile vs. shear strengths and (b) total elongation vs. R.A.
In this study, parameters taken from shear punch curves for comparing different materials are shown in Fig. 4.46. The UTS and corresponding shear maximum strength ($\sigma_t$) of steel alloys at different annealing conditions is plotted in Fig. 4.46 (a). There appears to be two populations corresponding to the two heat treatments, Tan-1 and Tan-2, as delineated by the two regression lines. These regression lines also reveal two populations of alloy composition, in that S1, S2, and S3 all occupy the two trend lines, but S4 does not. However, for the plot of tensile (% $\varepsilon_{\text{Total}}$) vs. shear ductility (R.A. %; reduction in area) in Fig. 4.46 (b), one trend line seems to fit all the data regardless of composition and heat treatment.

According to the Fig. 4.46, the Tan-1 and Tan-2 annealing schedule, regression equations for the relation between shear punch, and tensile properties can be derived as follows:

$$\text{UTS (Tan-1)} = 0.641 x \tau + 331 \quad (4.4)$$

$$\text{UTS (Tan-2)} = 1.129 x \tau + 4.47 \quad (4.5)$$

$$\varepsilon_{\text{total}} \text{ (both)} = 1.55 x \text{ (R.A.)} - 79.66 \quad (4.6)$$

These results indicate that the shear punch maximum force and uniform displacement give trends that are similar to those measured by tensile testing and can be used to convert to tensile values.

4.4.3. Comparison of Static and Dynamic Shear Punch Tests

An analysis of the propagation of the elastic waves in Hopkinson shear punch test produce three signals recorded during a test that allows the calculation of the shear displacement and the shear maximum force imposed on the specimen (Fig. 4.47). If the split Hopkinson shear punch system is subjected to impact by a striker of the same material and cross-sectional area travelling with velocity “$v$,” then $v$ is associated with the maximum force in the pressure bar. The force and displacement are measured by means of strain gauges attached to the incident bar and transmitted bar.
Chapter 4: Results

The displacement, \( u_1 \), of the face of the incident bar is the result of both the incident and reflected travelling pulses.

\[
    u_1 = c_0 \int_0^t \varepsilon_i \, dt + (-c_0) \int_0^t \varepsilon_R \, dt = c_0 \int_0^t (\varepsilon_i - \varepsilon_R) \, dt \tag{4.7}
\]

where \( t = 0 \) corresponds to the first arrival of the incident wave at the interface. Similarly, the displacement \( u_2 \) of the face of the transmitter bar in contact with the specimen is:

\[
    u_2 = c_0 \int_0^t \varepsilon_T \, dt \tag{4.8}
\]

The velocities of the face of each of the bars in contact with the specimen are:

\[
    v_1 = c_0(\varepsilon_i - \varepsilon_R) \tag{4.9}
\]

\[
    v_2 = c_0 \varepsilon_T \tag{4.10}
\]

The applied loads \( P_1 \) and \( P_2 \) on each face of the specimen are:

\[
    P_1 = EA(\varepsilon_i + \varepsilon_R) \tag{4.11}
\]

\[
    P_2 = EA \varepsilon_T \tag{4.12}
\]

Figure 4.47. A typical pulse record of a Hopkinson shear punch experiment.
The mechanical properties shown in this section, unless indicated, are only from shear punch tests. Dynamic shear punch tests were also performed only for $T_{an-1}$ and $T_{an-2}$, and these were compared to the static shear punch results. The dynamic speed was 25 mm/sec for all steel samples.

Figure 4.48 shows the comparison of a typical static and dynamic shear punch curve. The oscillation of dynamic force-displacement curves is a characteristic consequence of the ringing effect due to the reflection of elastic stress waves in the sample and the machine. As well, the dynamic curve does not reveal peak strength, and, finally, the dynamic curve appears to undergo an increase in work hardening rate as fracture is approached, which is the opposite of the static curve.

**Figure 4.48.** Sample force-displacement curves of static and dynamic shear punch tests for S2.

As may be expected, in general the dynamic force is higher than the static force for a given microstructure (i.e., same composition and heat treatment), and the dynamic fracture displacement (or reduction in area) is smaller than the static
fracture displacement for all the specimens. This is a result of the plastic strain, which is mainly achieved by the thermally activated motion of dislocations. At high strain rates, the thermally activated process becomes more difficult due to shorter times. Therefore, the flow stress increases and ductility is lower at high strain rates compared to the static conditions.

Another typical difference between dynamic and static curves is the absence of a peak force in the dynamic test. Finally, the effect of heat treatment leads to a significant difference in the dynamic results, whereas there is little effect in the static properties.

The effect of strain rate on maximum “strength” is shown in Fig 4.49 by plotting the dynamic maximum force vs. the static maximum force. These curves reveal that there is an effect of heat treatment (i.e., microstructure) on the strain rate “sensitivity,” with the quenched structures illustrating higher strain rate sensitivity. However, as can be seen in Fig. 4.50, the respective maxima do not occur at the same strain. Moreover, the maximum strength of the static test is a peak value, suggesting that it is due to flow instability (as per the UTS in a tensile test), whereas the dynamic maximum strength coincides with fracture.

Since the strain at which the maximum dynamic strength occurs is always lower than the strain at which the peak static strength occurs, plotting the maximum dynamic strength vs. the static strength at the same strain at which the maximum dynamic strength occurs (designated $F_{\text{co-strain}}$) may be more relevant. This plot is shown in Fig. 4.51 and again the effect of heat treatment is qualitatively the same as in Fig. 4.49 but is quantitatively different.

The effect of strain rate on total “strain” (i.e., displacement at fracture) is shown in Fig 4.52. The ductility results generally show an effect of heat treatment, with the higher strain rate decreasing the ductility of the quenched specimens far more than for the air cooled ones.
Figure 4.49. The effect of strain rate sensitivity on maximum shear force.

Figure 4.50. Sample calculation curve for the determination of Static $F_{co-strain}$. 
Figure 4.51. The dynamic shear $F_{\text{max}}$ vs. static $F_{\text{co-strain}}$ curve.

Figure 4.52. The effect of strain rate sensitivity on total displacement.
Since dynamic forces are usually more difficult to obtain experimentally, it is therefore useful to determine an empirical relationship between the dynamic data and the corresponding static test results. Note that the co-strain equations cannot be used to determine the dynamic maximum fracture from static co-strain values since the strain at which dynamic fracture needs to be known before applying these equations. The regression equations for the relation between static and dynamic shear punch tests can be derived as follows.

In this case:

\[ \tau_{\text{max}} (T_{\text{an}-1}) = 14.745 \times F_{\text{max}} + 23.44 \]  \hspace{1cm} (4.7)

\[ \tau_{\text{max}} (T_{\text{an}-2}) = 39.129 \times F_{\text{max}} + 10.51 \]  \hspace{1cm} (4.8)

\[ \tau_{\text{max}} (T_{\text{an}-1}) = 0.253 \times F_{\text{co-strain}} + 46.804 \]  \hspace{1cm} (4.9)

\[ \tau_{\text{max}} (T_{\text{an}-2}) = 0.0201 \times F_{\text{co-strain}} + 54.507 \]  \hspace{1cm} (4.10)

\[ \varepsilon_{\text{total}} (T_{\text{an}-1}) = 84.234 \times (D_{\text{Total}}) - 81.048 \]  \hspace{1cm} (4.11)

\[ \varepsilon_{\text{total}} (T_{\text{an}-2}) = 63.254 \times (D_{\text{Total}}) - 41.048 \]  \hspace{1cm} (4.12)

where the static shear punch speed is 0.1 mm/sec, and the dynamic shear punch speed is 25 mm/sec.
Chapter 5: Discussion

5.1. Formation of Novel Microstructures

The compositional differences appeared to have generated two unique classes of microstructure: a multiphase structure that comprises ferrite, martensite/bainite, and retained austenite (FMNA/FBNA) (S1, S2, and S3) and a duplex retained austenite plus ferrite microstructure (FADP) (S4). Figure 5.1 shows the sample high resolution SEM images of the two novel microstructures.

In general, the FMNA structures are formed with steels having Mn levels between 5 and 7 wt.%, by quenching from intercritical temperatures at which the austenite varied between about 25 and 45 vol.%. The FADP structure was generated from steel with an Mn addition of about 10 wt.%, intercritically annealed at a temperature at which the austenite was about 50 vol.%

In fact, such microstructures have not been reported in the literature. The closest comparison to FMNA structures are dual phase steels; however, such steels have insignificant levels of retained austenite (3 to 7 vol.%) [2]. The so-called Q&PT structures aim at a similar concept, but the RA levels are still much lower than the FMNA structures reported (13 to 16 vol.%) [110, 111]. Moreover, retained austenite fractions obtained from the Q&PT process require prolonged holding times at intercritical temperatures for manganese partitioning.

In this study, the FBNA structures are similar to TRIP structures but again the RA levels in the FBNAs are much higher, and no austempering is required to obtain these structures. Duplex structures at room temperature of retained austenite and equiaxed ferrite have not been reported in the literature.
Figure 5.1. The high resolution SEM images of the two different structures: (a) S3 (also representative of S1, S2, and S3) ferrite and martensite/bainite nucleated in the retained austenite (FMNA) and (b) S4 ferrite plus retained austenite (FADP).
Chapter 5: Discussion

5.1.1. Martensite Start ($M_s$) and Finish ($M_f$) Temperatures

The basic concept of all of the steels in this thesis seems to be focused on the martensite start temperature being close to room temperature. For the FMNA compositions, it can be argued that the $M_s$ temperature appears to be above room temperature, whereas the $M_f$ temperature is below room temperature. For the S4 composition, the $M_f$ appears to be below room temperature. There are many empirical equations to determine the $M_s$ temperatures from the composition of the austenite (Table 5.1).

Table 5.1. The empirical equations for the calculation of $M_s$.

<table>
<thead>
<tr>
<th>Name</th>
<th>$M_s$ Formula</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Andrews (Low alloy heat</td>
<td>$M_s = 539 - 423C - 30.4Mn - 17.7Ni - 12.1Cr - 11Si - 7Mo$</td>
<td>[112]</td>
</tr>
<tr>
<td>treatable steels)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Capdevilla (HSLA steels)</td>
<td>$M_s = 764.2 - 302.6C - 30.6Mn - 16.6Ni - 8.9Cr + 2.4Mo - 11.3Cu + 8.58Co + 7.4W - 14.5Si</td>
<td>[112]</td>
</tr>
<tr>
<td>Dai (Cryogenic austenitic</td>
<td>$M_s = A_{e3} - 199.8(C+1.4N) - 17.9Ni - 21.7Mn - 6.8Cr - 45Si - 55.9Mo - 1.9(C+1.4N)(Mo+Cr+Mn) - 14.4[((Ni+Mn)(Cr+Mo+Al+Si))^{1/2} - 410</td>
<td>[113]</td>
</tr>
<tr>
<td>steels)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mintz (TRIP aided)</td>
<td>$M_s = 539 - 423C - 30.4Mn - 17.7Ni - 12.1Cr - 7.5Mo - 7.5Si + 30Al$</td>
<td>[114]</td>
</tr>
<tr>
<td>Mahieu (TRIP steels)</td>
<td>$M_s = 539 - 423C - 30.4Mn - 7.5Si + 30Al$</td>
<td>[115]</td>
</tr>
</tbody>
</table>

$M_s$: Martensite start temperature; $A_{e3}$: $\gamma\rightarrow\alpha$ phase transformation temperature; HSLA: High strength low alloy

The retained austenite compositions for all the steels in the $T_{an}$-1 condition (i.e., as-quenched condition), measured by EPMA-WDS, are tabulated in Table 5.2. For the $M_s$ analysis, only $T_{an}$-1 need be considered, since martensite is definitely formed after quenching. These values are used to determine the $M_s$ temperatures according to the equations in Table 5.1.
Table 5.2. The retained austenite compositions measured by EPMA.

<table>
<thead>
<tr>
<th>Sample Nr.</th>
<th>C %</th>
<th>Mn %</th>
<th>Si %</th>
<th>Al %</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>1.04</td>
<td>6.32</td>
<td>2.75</td>
<td>2.06</td>
</tr>
<tr>
<td>S2</td>
<td>1.27</td>
<td>6.90</td>
<td>2.82</td>
<td>2.25</td>
</tr>
<tr>
<td>S3</td>
<td>1.09</td>
<td>8.48</td>
<td>2.89</td>
<td>2.79</td>
</tr>
<tr>
<td>S4</td>
<td>1.10</td>
<td>11.2</td>
<td>2.95</td>
<td>2.97</td>
</tr>
</tbody>
</table>

The results of the $M_s$ temperature calculations for the given compositions are depicted in Fig. 5.2. Unfortunately, there is no empirical equation in the literature that has been created for medium Mn steels. Moreover, all these empirical equations have been developed by experiments that begin with 100% austenite, whereas the transformation that is of interest here is from the two phase ferrite plus austenite condition. Therefore, it is expected that the $M_s$ calculations will not yield correct absolute values but might be appropriate to generate a trend that could explain the transition of the quenched microstructure as the Mn content increases from 7 to 10wt.%, namely from the FMNA to the FADP structures, respectively.

Figure 5.2. $M_s$ temperature calculation according to the equation in Table 5.1 for a given composition in Table 5.2.
Generally, all the equations predict the same trends and, apart from the Capdevilla equation, the numerical values are similar. (The Capdevilla equation differs from the other equations mainly in the first term of the equation.) All equations predict that the $M_s$ for S4 is the lowest of the four alloys. Comparing S3 to S4, the C level is lower in S4, therefore the decrease in $M_s$ from S3 to S4 is only due to Mn. Thus, these equations confirm the importance of Mn, but the predicted $M_s$ values do not correspond with the actual results on quenching since the predicted Ms temperatures are either all above or all below the ambient (room) temperature.

5.1.2. Effect of Cooling Rate

The microstructures for S1, S2, and S3 have been formed in a similar manner to TRIP steels, except that after the intercritical anneal, there has been a water quench (as opposed to a bainite hold). In a classic TRIP steel composition, the water quench would lead to a dual phase steel microstructure, but in these steels, the composition is such that the austenite has not been able to fully transform to martensite, retaining a significant level of austenite at room temperature. Thus, the microstructure resembles a TRIP steel but with martensite substituting for the bainite (Fig. 5.3). As can be seen from Fig. 5.3, thermally transformed $\alpha'$ martensite nucleates inside the austenite islands formed during the intercritical annealing of S3.

On the other hand, for S1, S2, and S3, there are indications that air cooling generates a bainitic structure as opposed to a martensitic structure. This can be surmised from the C mapping of S2 for the RA and the phase nucleating inside the RA islands in both $T_{an}$-1 and $T_{an}$-2 annealing, as shown in Fig. 5.4. There is a much greater inhomogeneity of C within the second phase aggregate of RA and the phase nucleated in the RA. In other words, there is a greater difference in C levels between the RA and the nucleated phase within the austenite in the case of $T_{an}$-2. This indicates that C has had time to diffuse during the transformation, suggesting that the phase formed in the austenite due to $T_{an}$-2 (i.e., after air cooling as opposed to quenching) is bainite rather than martensite (Fig. 5.5).
Figure 5.3. The EBSD map of S3: Inverse pole figure and phase distribution (ferrite, austenite, and martensite).

Figure 5.4. The BSE images of S2 after (a) $T_{\text{an}}$-1 and (c) $T_{\text{an}}$-2 annealing and EPMA-WDS elemental mapping of carbon (b)$T_{\text{an}}$-1 and (d)$T_{\text{an}}$-2.
Indeed, differential scanning calorimetry (DSC) analysis at air cooling rates also showed that there seems to be a phase transformation during cooling of steel samples. The effect of the $T_{\text{an-1}}$ and $T_{\text{an-2}}$ heat treatments on the RA of S1, S2, and S3 is negligible. The result of the much slower rate of furnace cooling is the transformation of almost all of the intercritically formed austenite to pearlite, which is expected.

**Figure 5.5.** The differential scanning calorimetry (DSC) analysis of steel samples with transformation during heating and cooling.

By contrast, at all cooling rates used, S4 has a duplex microstructure, which consists only of ferrite and retained austenite. Dilatometer studies with various cooling rates also confirmed that S4 exhibits no sign of any transformation (**Fig. 5.6**). It can be concluded that the C and Mn levels in this composition drastically slow down diffusional transformations and lowers the $M_s$ temperature to below room temperature (**Fig. 5.2**), thus retaining the intercritically formed austenite.
5.2. Mechanical Property and Structure Relationship

It was stated in Chapter 4 that the results of the mechanical tests conducted on FMNA microstructures are strongly related to their martensite/bainite content, which is illustrated in Fig. 5.7, whereas, in general, strength is not systematically affected by retained austenite (Fig. 5.8). However, in general, there is a weak relationship between the two whereby the higher the retained austenite contents the higher ductility (Fig. 5.8).

When $T_{an}$-1 and $T_{an}$-2 are compared to each other for a given martensite/bainite volume fraction in the FMNA structures, the air cooled specimens generally have slightly higher strength than the quenched ones (Fig. 5.7). Given that the air cooled specimens should generate bainite rather than martensite, it seems contradictory that the air-cooled specimens exhibit slightly higher strength levels. An alternative possibility is strain induced transformation of the retained austenite

![Figure 5.6. Dilatometer analysis of S4 composition in various cooling rates.](image)
of the air cooled specimens because the bainite may not provide the same level of stress shielding that is afforded by the martensite. The possibility of strain induced transformation is indicated in the cold rolling results, as discussed later.

Figure 5.7. Effect of martensite fraction on strain and stress.
In regards to other possibilities, the difference between the two heat treatments might be due to a difference in the carbon levels of the martensites and/or bainite (i.e., there is a higher carbon level in the air cooled samples) (Fig. 5.3), which could lead to a higher strength. Although there are few contradictory findings [116-118], several investigators observed that the strengths varied in a non-linear manner with the volume fraction of martensite, with the strengths being dependent on the carbon content of the martensite in both DP and TRIP steels [69, 119, 120].

For the FADP structure, the higher retained austenite leads to a higher ductility and appears to follow the general effect of RA vol.% revealed in the FMNA structures (Fig. 5.8). This may be due to FCC austenite being generally more ductile than BCC ferrite or BCT martensite.

It is also interesting to note that the ductility of the as-quenched specimens is slightly higher than the air cooled ones for a given RA vol.%. Therefore, an explanation is possible for higher ductility of $T_{\text{crit}}$ samples since the intercritically formed austenite is formed at higher temperatures, which have lower carbon content [121], as confirmed by the EPMA results (Fig. 5.4). This lower C retained austenite might make it easier to transform to martensite leading to an enhanced TRIP effect which relaxes the stress concentration thereby delaying the necking and increasing the elongation. However, regions of austenite can be observed to elongate with the majority phase, which is ferrite. With decreasing C levels, this mechanically stable RA is easier to deform.
Figure 5.8. Effect of retained austenite fraction on strain and stress.
5.2.1. Twinning and Strain Induced Transformation (SIT) during Cold Rolling

This study was performed in order to indicate how amenable these grades were to sheet production and to indicate any changes in the austenite due to deformation. All the steels exhibited significant reduction in retained austenite after cold rolling, and it can be assumed that martensite was formed. However, in the case of S4 (FADP), cold rolling not only transformed approximately half of the retained austenite to strain induced martensitic, but twinning was also clearly observed in the optical micrographs. It is possible that twinning may be confused with martensite in optical micrographs, but TEM examination confirms the presence of twins (Fig. 5.9).

To the knowledge of the author, retained austenite that twins as well as undergoes strain induced transformation has never been observed previously. In fact, obtaining retained austenite that twinned was one of the main objectives of this thesis, since the hypothesis was that it would greatly contribute to the ductility of medium Mn steels. However, there was no expectation that twinning and SIT would occur in the same microstructure.

It is clearly of interest to understand exactly why the retained austenite in the FADP steel can undergo both twinning and SIT during deformation, whereas the retained austenite in TRIP steels undergoes only strain induced transformation and the retained austenite in TWIP steels undergoes only twinning. However, in all previous studies, the microstructures that undergo twinning are 100% austenite which is energetically stable, e.g., TWIP and Hadfield steels [40, 57, 98, 122], so there is no chance for strain induced transformation to martensite. Therefore the relevant comparison of FADP steels is with TRIP steels rather than TWIP steels. In this regard, the retained austenite grains in FADP are much larger and the level of retained austenite is much higher. Moreover, there is only one morphological type of retained austenite in FADP, i.e. equiaxed, whereas there are at least three types of retained austenites in TRIP steels ((i) equiaxed small grains, (ii) interlath layers in the bainite and (iii) layers between bainite packets and equiaxed ferrite
Chapter 5: Discussion

grains) [2, 123]. It is well known that decreasing the retained austenite size in TRIP steels increases the resistance against SIT. It may be that size is also a factor in twinning.

Figure 5.9. Deformation twins in S4 (a) bright field image, (b) weak-beam dark field TEM image with SAED pattern oriented along [011] zone axis (FCC), and (c) simulated indexed pattern corresponding to (b).

Equally important is to determine when twinning and SIT take place during deformation, i.e., the evolution of the microstructure with strain. In fact, there are almost certainly three deformation mechanisms operating (i) slip, (ii) SIT, and (iii) twinning. In terms of modeling the events, if these mechanisms are treated purely as ways to relieve the applied stress, then the obvious approach is to use
the concept of critical resolved shear stress via the Schmidt factor. This approach is well known for slip and twinning; the concept of critical stress is also used in SIT [118, 119].

Another way to consider the problem is by a classic Gibbs energy approach; this is commonly used to explain TRIP [2, 124] but not for twinning. With this approach, twinned retained austenite and strain induced martensite could be considered as being two phases in equilibrium. Figure 5.10 shows a schematic diagram of the effect of composition on the free energies of these two phases.

If the diffusion of solutes is allowed, the two phase assemblage is delineated by the common tangent between these curves, which leads to the two co-existing phases having different compositions; this does not occur in this case because both SIT and twinning are diffusionless ‘transformations’, and the compositions of twinned and SIT phases are therefore the same. Therefore, in order to satisfy the co-existence of twinned austenite ($\gamma_{\text{twin}}$) and SIT phase ($\alpha^1$), the Gibbs energies of the two phases should be the same or, in practice, very similar for the existing composition of the retained austenite.

This scenario maybe feasible as twinning and tripping are mechanism at high (about 20%) and low (2%) Mn concentrations, respectively. EPMA analysis [125] has shown that the Mn in the retained austenite of FADP is 11.2%, which is more or less exactly between the high and low Mn levels. However, it would be very fortuitous to have attained the exact compositional ‘fit’ in this experiment where the free energies of strain induced martensite and tinned retained austenite exactly coincide. Therefore, this ‘classical’ $\Delta G$ curve approach does not satisfactorily explain the coexistence of twinning and tripping. A ‘hybrid’ of the CRSS and free energy approaches may be required to satisfactorily analyze this simultaneous twinning and SITing phenomenon. Since, no other approach has emerged in the literature, it is certainly a candidate for future work.
Figure 5.10. Free energy-composition diagram for the austenite and strain induced martensite phases at a given temperature.

Since it is accepted that there is a strong effect of Mn and C on the SFE and transformation kinetics during deformation, one other reason why both SIT and twinning are seen in the retained austenite may be due to an inhomogeneous carbon distribution within the retained austenite. This could lead an incomplete transformation of retained austenite and these regions of austenite tend to transform to martensite or twinning at lower strains [126]. However, this approach is unable to define clearly the relationship between alloys and strain induced transformation.

For the case of strain induced nucleation and/or transformation, it is possible to focus on specific intersecting-shear systems when the austenite stacking fault energy is low, either $\alpha'_{BCT}$ and $\varepsilon_{HCP}$ martensite or mechanical twins can form as a part of the shear displacements. As can be seen from Fig. 5.11, in this case both of them formed in the same retained austenite grain during deformation.
Figure 5.11. Deformation twins in S4: (a) bright field image, (b) weak-beam dark field TEM image with SAED pattern oriented along [011] zone axis (FCC) and along [001] zone axis (BCT), (c) simulated indexed pattern corresponding to BCT structure at (b), and (d) simulated indexed pattern corresponding to FCC structure at (b).

Indeed, these different lattice defects strongly influence the stress-strain response and the evolution of the texture and strain hardening ability of these structures during deformation. But it is questionable how it will affect the evolution of the structure when both of them occur in the same grain. The mechanical twins contain a high level of sessile dislocations, resulting from both the formation mechanism and some potential supplementary dislocation reaction between the twins Shockley partials and the forest dislocations [36]. Therefore, it is believed
that these partial dislocations, which are located between thin twin layers (as can be seen from Fig. 5.12), lead to a large increase in the critical stress required to induce some plastic deformation within these thin twin-related crystals, improving the composite strengthening effect. So dislocations created within the initial austenite crystals, through unavoidable interactions with these twins, present a reduced mean free path that prevents dislocation motion.

**Figure 5.12.** Shockley partial dislocation in the twinning plane observed with: (a) and (c) bright field TEM image and (b) and (d) weak-beam dark field TEM image with SAED pattern oriented along [011] zone axis (FCC).

As a result, twinning and SIT play an important role in the particular dislocation reactions in medium Mn steels, although how to control the transformation of metastable phases during deformation is questionable.
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5.3. Static and Dynamic Property Relationship

In general, strain rate sensitivity decreases as static strength increases, which is the expected behavior [7, 127]. With regard to the relationship between the dynamic and static mechanical properties, there appears to be two different populations of specimens, as was shown in Figs. 4.49 and 4.51. These relationships are similar to those revealed in the comparison of static tensile and static shear values in the sense that there was a noticeable influence of heat treatment on the relationship between these data.

One of the most interesting results of the dynamic testing is that the maximum force of all the alloys for all heat treatments was exhibited by S4 at T_{an}-1 annealing conditions; as well, the maximum displacement of all the alloys was again obtained from S4 but this time at T_{an}-2 annealing conditions. This is not what was observed in the static results, where the maximum force was found in S2-Tan-1 (corresponding to the highest martensite volume fraction) and the maximum ductility was S4- T_{an}-1 (corresponding to the highest retained austenite amount). With the possible increase of transformation at high strain rates, the strength of S4 increases at T_{an}-1 conditions [7] and more strain may be distributed into the retained austenite at T_{an}-2 conditions [127, 128].

5.3.1. Strain Rate Sensitivity

As noted in the literature review, the strain rate sensitivity coefficient can be derived by fitting experimental data to constitutive models, e.g., the extended Hollomon equation (the m-value in Eq. 2.5). Unfortunately, the m-value could not be calculated in this study because of the oscillation of dynamic curves, although the trend was depicted as a function of maximum applied force and strain rate.

The relationship between maximum applied force and strain rate is given in Fig. 5.13. All steels show positive strain rate sensitivity over the measured strain rate range. The FADP structure is slightly more sensitive to strain rate. For both structures an increase in the strain rate sensitivity is observed at lower strength levels.
Figure 5.13. The maximum applied force as a function of strain rate (a) S1, S2, and S3; (FMNA) structure; and (b) S4 (FADP) structure.
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The reason why the maximum force shows somewhat higher strain rate sensitivity in FADP than that of the FMNA structure is not clear. In fact, it is accepted that static strength is the main influence, with generally no consideration that microstructure influences strain rate sensitivity. However, in this case a microstructural effect may be explained by the softening effects of deformation induced heating being more pronounced in the martensite bearing structures compared to FADP.

5.3.2. Dynamic Factor

This section attempts to explain the observed variation of dynamic factor with strength and strain and the effect of microstructure on dynamic factor. Figure 5.14 shows the variation of the dynamic factor of force with the static shear punch force at different displacements (i.e., 0.15, 0.35, and 0.5 mm, as denoted by D$_{0.15}$, D$_{0.35}$, and D$_{0.5}$, respectively). As can be seen, the dynamic factor shows some scatter due to the oscillation behavior of the dynamic forces. The overall trend is that the dynamic factor of force decreases rapidly with increasing static strength to a nearly constant rate. This is generally observed in all other metals.

The variation of dynamic factor with strength and strain and the effect of microstructure on dynamic factor can be explained by the thermal activation theory of plastic deformation [7, 14]. Flow stress can be written as the sum of two components, a thermal component, $\sigma_{th}$, due to short-range obstacles and an athermal component, $\sigma_{ath}$, due to long-range obstacles, as:

$$\sigma = \sigma_{th} + \sigma_{ath}$$  \hspace{1cm} (5.1)

$\sigma_{th}$ is believed to be associated with overcoming the Peierls-Nabarro stress (the stress needed to drive a dislocation through a crystal continuously) and is dependent on strain rate and temperature [109, 129]. $\sigma_{ath}$ is associated with the long-range forces arising (e.g., from long-range stress fields of dislocation pileups) and is dependent on material structure.
It was generally observed [130, 131] that, in the static strain rate range (e.g., $10^{-4}$ to $10^{-1}$ s$^{-1}$) and at room temperature, the thermal stress contributes very little to the overall stress. The dynamic factor of stress, $R$, can then be expressed by:

$$R = 1 + \frac{\sigma_{th}}{\sigma_{ath}}$$ (5.2)

It can be seen from the above equation that at a given strain rate, the dynamic factor decreases with the increase of $\sigma_{ath}$ by increasing static strength. Basically, the physical reason why increasing static strength reduces the dynamic factor, in this simple analysis, is because the athermal stress does not directly affect the thermal stress, and this comes about from the definitions of these two stresses.

Assuming the athermal stress $\sigma_{ath}$ is independent of temperature, the thermal stress $\sigma_{th}$ can be expressed as [130]:

$$\sigma_{th} = \sigma_{th(0)} x \left[1 - \frac{k T}{\Delta G_0} \ln \left(\frac{\dot{\varepsilon}_0}{\dot{\varepsilon}}\right)\right]^2$$ (5.3)

where $\sigma_{th(0)}$ is the thermal stress at 0$^\circ$K, $\Delta G_0$ the activation free energy required for overcoming the local obstacle when the applied stress is zero; $\dot{\varepsilon}$ is the strain rate and $\dot{\varepsilon}_0$ a constant; and $k$ and $T$ are the Boltzmann’s constant and the absolute temperature, respectively. It is considered that the effect of microstructure on the thermal stress probably results from the change in the three factors in the above equation, namely $\sigma_{th(0)}$, $\Delta G_0$, and $\dot{\varepsilon}_0$.

As stated at the beginning of this chapter, there seems to be an effect of heat treatment on the relationship between static and dynamic properties. In general, according to the variation of dynamic factor with static force (Fig. 5.14), samples annealed with $T_{an-1}$ have higher dynamic factor values compared to $T_{an-2}$ annealing. This is possibly a result of quenching the samples, which forms higher martensite content with higher density of mobile dislocations. These mobile dislocations may act as thermal barriers and thereby increase the thermal stress, which might explain why $T_{an-1}$ has higher dynamic factor.
Figure 5.14. The variation of dynamic factors of force vs. static force at different displacements.
Chapter 5: Discussion

The short-range obstacles should be similar, although due to the content of the secondary phase (either martensite in quenching or bainite in air cooling), there may be different short-range obstacle configurations. One possible change is the increase in the average distance dislocations as they move to overcome obstacles because of the larger lattice parameter of martensite compared to austenite and bainite, which leads to a lower thermal stress and dynamic factor. However, the higher carbon (and probably other solutes) content in the martensite may offset this effect somewhat, since solute atoms may also be treated as short-range obstacles [132].

None of the above can explain the effect of heat treatment on the dynamic factor of S4 (FADP) structure, even though it is as much affected by the different annealing treatments as the other alloys. Since no significant effect of heat treatment on the structure was observed, it is difficult to explain the effect of heat treatment on the dynamic factor in this alloy.

5.3.3. Crashworthiness (Absorbed Energy)

Absorbed energy is essentially a combination of strength (yield strength and tensile strength), ductility, and work hardening rate of the flow curve. In the case of high strain rate shear punch testing, the “absorbed energy” area under the curve is also an important factor to evaluate the dynamic mechanical properties as a function of static mechanical properties. It can be readily understood that higher dynamic flow stresses can absorb more impact energy over a given strain. Figure 5.15 shows the variations of the dynamic absorbed energy (Fig. 5.15(a)) and the dynamic factor of absorbed energy (the ratio of dynamic to static absorbed energies, Fig. 5.15(b)) with the static absorbed energy at different displacements for all the microstructures (D_{0.15}, D_{0.35}, and D_{0.5} represent dynamic factors of absorbed energy at displacements of 0.15, 0.35, and 0.5mm, respectively). With increasing static energy, the dynamic energy increases, but again the dynamic factor of absorbed energy decreases. However, the rate of decrease in the dynamic factor of absorbed energy increases with decreasing displacement.
Figure 5.15. Variation of the dynamic absorbed energy with static absorbed energy.

Furthermore, it is also interesting that there is again an effect of heat treatment on dynamic absorbed energy for FMNA structures, but it is different from the effect
of heat treatment on dynamic factor. In fact, T$_{an}$-2 has higher absorbed energy values at static maximum force levels. For the FMNA structures, this may be a reflection of the tougher bainite structures developed after air cooling. There is no string effect of heat treatment for the FADP structures.

The FMNA microstructures in general appear to absorb more dynamic energy than the FADP microstructures. The air cooled S4 microstructure, which has the lowest static strength level, absorbs the lowest dynamic energy. The reason why the absorbed energy continues to increase in the case of the static tests is the much higher increase in force that is experienced with higher secondary phase (martensite/bainite or retained austenite) levels.

5.4. Comparison to Industrial Grades

Static and dynamic shear punch mechanical properties are compared to other steels including AHSS [6, 12, 27, 86] in Fig. 5.16 (a) and Fig. 5.16 (b). Compared to these other grades, it is found that some of the heat treated microstructures obtained in this study have slightly better mechanical and crashworthiness properties than DP and TRIP steels but not as good as TWIP steels, which was the expected behavior for these medium Mn steels.

In particular, when compared to other AHSS steels, the dynamic mechanical properties of medium Mn steels seem closer to TWIP steels than the static mechanical properties. It is believed that, considering the current status of medium Mn steel researches, the alloys used in this study continue to offer many opportunities to explore available property combinations and optimize retained austenite fractions. Therefore, it is clear that the initial results have been very encouraging in regards to understanding the processing mechanisms and exploring industrial processing capabilities. This explains why medium Mn steels is currently a topic of great practical interest and fundamental importance both for steel and automotive industries.
Figure 5.16. The mechanical results of the thesis compared with industry: (a) static and (b) dynamic [3, 6, 12, 86, 133-135].
5.5. Accuracy of FactSage as a Process Design Tool

FactSage was essentially used to select the intercritical annealing temperature, the aim being to maximize the retained austenite by maximizing the austenite at the intercritical temperature. Although no experiments were performed to determine whether the selected temperature generated the most austenite intercritically, it is interesting to compare the FactSage prediction with the measured result. The most appropriate microstructure is after quenching because it can be assumed that the martensite (where present) was austenite at the intercritical temperature. Therefore, the summation of retained austenite and any martensite should be the austenite formed at the intercritical temperature. The comparison is shown in Table 5.3. In general, the predictions are good, apart from the relatively low prediction of S2.

Table 5.3. The comparison of phase formation for $T_{\text{an}-1}$, FactSage predictions vs. measured results.

<table>
<thead>
<tr>
<th>Steel Nr</th>
<th>FactSage wt.%</th>
<th>Martensite vol.%</th>
<th>Retained austenite vol.%</th>
<th>Total vol.%</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>22</td>
<td>10.8</td>
<td>15.2</td>
<td>26</td>
</tr>
<tr>
<td>S2</td>
<td>25</td>
<td>27.9</td>
<td>9.1</td>
<td>37</td>
</tr>
<tr>
<td>S3</td>
<td>42</td>
<td>31.9</td>
<td>17.1</td>
<td>49</td>
</tr>
<tr>
<td>S4</td>
<td>62</td>
<td>4</td>
<td>53</td>
<td>57</td>
</tr>
</tbody>
</table>

Another metric that can be used to judge the accuracy of FactSage is the composition of the retained austenite, again in the quenched condition since the assumption is that the composition at the intercritical temperature will be largely preserved in the retained austenite on quenching. The results for C, Si, Al, and Mn are shown in Table 5.4.

In general, there is a good match between predicted and measured Al and Mn levels, but the predicted C level is much lower than measured and the predicted Si level is much higher than measured.
Table 5.4. The FactSage prediction of composition for retained austenite at $T_{\text{an}-1}$ conditions against EPMA results after $T_{\text{an}-1}$ annealing.

<table>
<thead>
<tr>
<th>Steel Nr.</th>
<th>FactSage wt.%</th>
<th>EPMA wt.%</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>C</td>
<td>Mn</td>
</tr>
<tr>
<td>S1</td>
<td>0.49</td>
<td>7.25</td>
</tr>
<tr>
<td>S2</td>
<td>0.49</td>
<td>6.2</td>
</tr>
<tr>
<td>S3</td>
<td>0.32</td>
<td>8.01</td>
</tr>
<tr>
<td>S4</td>
<td>0.20</td>
<td>11.86</td>
</tr>
</tbody>
</table>

Discrepancies between predictions and measured values can be due to data base issues—the alloy compositions under investigation are quite novel and some discrepancy is to be expected from a data base point of view. Another source of discrepancy is that FactSage is designed for equilibrium conditions. The comparisons in this section concern the structure generated during intercritical annealing, which is performed at a relatively high temperature for a relatively long time. Hence there is an expectation that equilibrium is approached.

In the case of the discrepancy of the austenite prediction for S2, there is no obvious kinetic issue that could explain this discrepancy. However, for the compositional issues of C and Si, there may be a kinetic issue to do with incomplete dissolution of precipitates during intercritical annealing. Unfortunately, this would lead to lower measured values than predicted, which is the opposite of what is observed.

To summarize, FactSage is an excellent program for thermodynamic calculations of steel alloys, but it requires comprehensive databases relevant to the specific compositions being examined. However, it should be stated that it requires some developments to be able to be used efficiently for the alloy design purposes in the sense of non-equilibrium conditions.
Chapter 6: Summary

Steel compositions centered on manganese (Mn) as the main alloying element were designed based on attaining a certain SFE level to control the deformation behavior of retained austenite. A thermodynamic approach, using the FactSage database, was used to calculate the stacking fault energy ($\gamma_{\text{SFE}}$). The data base was also used to select thermal processing temperatures. The objective was to generate microstructures with strength and ductility properties between that of TWIP and TRIP steels.

The main results are as follows:

(1) Two new types of steel microstructure were produced:

i. S1, S2, and S3 microstructures consisting of ferrite matrix and martensite phases nucleated inside austenite islands (FMNA microstructures). These basically resembled TRIP steels, with martensite “substituting” for bainite.

ii. S4 was a dual phase structure of ferrite and retained austenite (FADP microstructures).

(2) For these compositions, 10 wt.% Mn appears to be a critical value transitioning the microstructures (on quenching) from FMNA to FADP, as well as increasing the retained austenite from about 20 vol.% to about 50 vol.%. 

(3) The alloys with <10 wt.% Mn, are sensitive to the cooling rate after intercritical annealing, with the capacity to exist with martensite or bainite or pearlite, whereas S4 (10 wt.% Mn) existed as the FADP structure regardless of the cooling rate.

(4) Cold rolling led to strain induced transformation of austenite in all steels except S1; it is likely that the fine austenite grain size of S1 inhibited strain
induced transformation. S4 exhibited both strain induced transformation as well as twinning in the retained austenite.

(5) Despite the difference between these two microstructures, the mechanical properties of all these steels are similar and overlap the TRIP envelope. The results show that the ductility of these medium Mn steels increases with the retained austenite fraction of the microstructure, and the strength increases with martensite volume fraction.
Chapter 7: Recommendations for Future Work

The following observations can be considered for recommendations for future work:

(1) Considering the novel microstructure obtained throughout this study, it can be clearly said that FMNA structures are heat treatable and can be converted into a tempered martensite (or partitioned structure)–FTMNA, or a TRIP type structure, replacing the martensite with bainite–FBNA structure. Therefore, FMNA, FTMNA, FBNA, and FADP structures are suggested as candidates for future studies. However, it is a fact that more processing and mechanical characterizations are required for the potential of FMNA and FADP structures to emphasize which structure would be more ideal for third generation AHSS concept.

(2) The effect of strain induced transformation and mechanical twinning formation of retained austenite has been proposed as a significant deformation mechanism in this study. However, there is no direct explanation of this occurring at the same time in retained austenite and this is certainly a candidate for future work (i.e., neutron diffraction experiments may be adapted to explore this possible mechanism during deformation).

(3) A more comprehensive study is required for the SFE calculations in medium Mn steels, including a new approach or modeling.

(4) Applying the findings to industrial conditions is necessary, particularly to properly design a heat treatment and rolling schedule that incorporates rolling plant.
Chapter 8: Statement of Original Contributions

(1) For the first time a thermodynamic approach was used to design medium Mn steels on the basis of SFE.

(2) Two novel microstructures were produced, depending on the level of Mn:

(i) ferrite plus martensite nucleated in austenite microstructure (FMNA structures) (alloys S1, S2, and S3) for Mn between 5 to 7%;

(ii) ferrite plus retained austenite duplex structure (FADP steels) (alloy S4) for 10% Mn.

(3) For the first time, retained austenite was observed to undergo both strain induced transformation (SIT) to martensite as well as twinning due to the cold rolling. This occurred in the FADP microstructure.

(4) The effect of generated microstructures on the static and dynamic mechanical properties of medium Mn steels was studied for the first time. Indeed, a strong effect of heat treatment on the relationship between static and dynamic properties was confirmed.

(5) From the above observation, a different response of the absorbed energies at different strains and strain rates with respect to the quenching media was observed.
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