MICROSTRUCTURE EVOLUTION DURING PROCESSING OF
DUAL PHASE AND TRIP STEELS

by

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ABSTRACT

In this study, a systematic experimental study on the microstructure evolution for a dual phase and a TRIP steel during intercritical annealing and intermediate temperature holding (for the TRIP steel) was conducted. In the reheating stage, recrystallization occurs for the cold-rolled materials, which were characterized by the microhardness and metallography. A mathematical description of the recrystallization kinetics can be made using the Avrami equation. It was found that for dual phase steel, ferrite recrystallization may extend into the austenite formation region thus overlapping with the austenite formation reaction. While for the TRIP steel, which has faster recrystallization rates, the recrystallization and austenite formation processes are sequential for the processing parameters (i.e. heating rates of 1, 10 and 100 °C/s) considered in this study.

In the intercritical annealing stage, the materials were subjected to both continuous and ramp plus isothermal hold heat treatments with various heating rates. The kinetics of austenite formation was determined by dilatometry. Very significant effects of heating rate on both the fraction of austenite and its spatial distribution and morphology were observed. The material behaviour during austenite formation can be understood by considering the effect of heating rate on the nucleation and growth of austenite. The basic trends can be rationalized by the competing mechanisms for nucleation and growth of austenite and how these depend on the starting microstructure (i.e. spatial distribution of pearlite and the degree of ferrite recrystallization). The interaction between ferrite recrystallization and austenite formation is strong and it affects not only the kinetics of austenite formation but also the spatial distribution
and morphology of austenite.

A new approach using neutron Bragg-edge transmission (BET) was applied to studying the austenite decomposition which occurs when the material is cooled to the intermediate hold stage (i.e. during processing of TRIP steel). The neutron BET technique allows for the analysis of the evolution of volume fraction for the participating phases; and also reveals unique, 'in-situ' carbon redistribution information which may be derived from the change in the lattice parameter. The volume fraction results were compared to the results from traditional XRD, dilatometry and optical metallography. Excellent agreement between these results was observed. In addition, carbon enrichment in the austenite phase during bainite transformation is clearly observed from this technique.
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DEDICATION

This thesis is dedicated with love

to my wife Duyun
Chapter 1 Introduction

Facing environmental problems, efforts have been made in the automotive industry to produce lightweight vehicles while maintaining satisfactory functionality and crash safety. Steel is widely used in the automotive industry. In the past, the steel products applied as structural and outer body materials are far from reaching their full potential in terms of mechanical properties, resulting in more mass on the vehicle. In recent years, medium and high strength steels have been displacing low strength steels as automotive materials to reduce vehicle weight and therefore reduce fuel consumption and vehicle emissions. Besides high strength, it is desirable for these steels to possess high formability, in order to produce complicated automotive components. Advances in steel research have made it possible to significantly increase the material's strength without losing the formability; therefore, great weight saving potential can be realized by applying these materials in the automotive industry. Among these steels, dual phase and TRIP (Transformation-Induced Plasticity) steels are two promising materials that can help to achieve the mass reduction goal.

Dual phase and TRIP steels belong to a common family of moderately high-strength steels exhibiting excellent mechanical characteristics. It is of technological interest to exploit the full potential of these materials. This can be achieved by developing a clear understanding of their behaviour along the processing route of reheating, intercritical annealing, cooling and low temperature bainite transformation. Many metallurgical phenomena can be observed for this processing route, such as recrystallization, austenite formation and austenite decomposition. Although each metallurgical phenomenon in the individual stage of the
processing has been studied more or less, little information is available on the material behaviour along the multiple stage heat treatments as an integrated process. For example, the interaction between the recrystallization and austenite formation is not clear so far, nor is the effect of heating rate on the austenite formation kinetics of the material. For the bainite transformation, despite the fact that it has been extensively studied, important information such as the carbon redistribution between austenite and bainite during the transformation is not experimentally available.

This study follows the microstructure evolution of the material along the entire process (i.e. reheating, intercritical annealing, cooling and bainite transformation) using conventional techniques (i.e. metallography, dilatometry) and a novel technique (neutron Bragg-edge transmission), in order to obtain the kinetic information for each individual metallurgical phenomenon involved and examine the possible interactions between them. The materials are subjected to systematically varied processing parameters (e.g., heating rate, temperature), so that their behaviour can be revealed and characterized.

This work will help to achieve better understanding of the microstructure development of the steels throughout the entire integrated process and develop industrially relevant processing routes that will lead to good properties with a minimum of variation. The thesis is organized in the following manner. Chapter 2 is a literature review which provides some background information about the development, processing and the associated microstructure evolution of the dual phase and TRIP steels, as well as the related experimental techniques being used currently. Chapter 3 outlines the objectives of this work. Chapter 4 deals with the recrystallization of the steels. The details of isothermal and non-isothermal recrystallization experiments, results, discussion and modeling of the recrystallization behaviour of steels are
Chapter 5 focuses on the austenite formation behaviour of the cold-rolled materials during the intercritical annealing process, with an emphasis on the interesting effect of the initial microstructure at the onset of austenite formation (where the recrystallization dealt with in the previous Chapter is important) and the effect of heating rate on the austenite formation behaviour. Chapter 6 is devoted to the study of bainite transformation using a novel neutron Bragg-edge transmission technique, to complete the present study on each of the significant microstructure evolution phenomena involved during the processing for these types of steels. Finally, Chapter 7 is a summary of the conclusions that can be drawn and a outlook into future developments that can be extended from the present work.
Chapter 2 Literature Review

2.1 Area of Interest

The history of steel as an alloy of primarily iron and carbon is long, dating back to at least the beginning of the tenth century B.C. in ancient China and India\textsuperscript{[1, 2]} Over the years, a wide variety of steels has been developed, which can be categorized in a number of ways, i.e. according to their chemistry (carbon concentration, alloy concentration), strength level, phase composition, application, etc. The interesting feature for steel, an iron-carbon alloy, is the change of phase composition associated with the carbon concentration change in the material. A small change in the carbon concentration from 0 to 1 wt.% leads to a large change in the phase fractions and composition, thus affecting microstructure and mechanical properties dramatically. Figure 2-1\textsuperscript{[3]} illustrates the Fe-C phase diagram where one can see the effects of temperature and composition on the important phases, i.e. ferrite, austenite and Fe\textsubscript{3}C. The combination of these constituents provides a variety of ways to manipulate the steel's microstructure and mechanical properties (i.e. changing the carbon concentration of the steel or changing the processing parameters). The addition of other alloying elements such as Mn, Ni, Mo, Nb, etc. can move the phase boundaries significantly, adding more freedom in achieving the desired properties, yet making the system more complex.

One of the most important ways in which steels are characterized is in terms of their mechanical properties. It is commonly observed that as strength goes up, ductility (and hence, formability) goes down. Figure 2-2\textsuperscript{[4]} plots the total elongation vs. tensile strength for a wide
variety of steels. The dotted line in Figure 2-2 represents the behaviour for conventional steels.

Figure 2-1 Fe-Fe₃C phase diagram. (Adopted from Massalski[3])
Figure 2-2 Strength-elongation balances for different steels. (After Heimbuch[4]).
It can be observed that a number of recently developed steels such as bake hardenable, dual phase and TRIP (Transformation Induced Plasticity) steels fall to the right of conventional steels indicating superior performance. These materials are of considerable interest, particularly for automotive applications. Recently, in a project set up by the ULSAB (UltraLight Steel Auto Body) consortium consisting of 35 prominent steel producers, advanced high strength steels such as dual phase and TRIP steels were proposed to be used in more than 79% of the body structure, resulting in 36% of weight saving\textsuperscript{[5]}.

In the following sections, properties and processing of these steels will be reviewed.

### 2.2 Overview of Dual Phase Steels

#### 2.2.1 Introduction to Dual Phase Steels

Renewed interest in high strength, low alloy steels for automotive application has been stimulated by the need for increased fuel economy and reduced emissions from automobiles. This goal may be achieved by reducing vehicle weight through the use of higher strength and therefore lighter gauge steels. However, the formability of these steels must be sufficient to meet the demands for fabricating increasingly complex parts. This challenge has led to the development of dual phase steels\textsuperscript{[6,7,8]}.

To illustrate the advantages of dual phase steels, it is useful to compare the stress-strain behaviour of a dual phase steel with two traditional steels (i.e. a plain carbon and an HSLA steel) as shown in Figure 2-3\textsuperscript{[9]}.

Compared to the HSLA steel, the dual phase steel has the following positive attributes:

- high tensile strength
- low Y.S. (Yield Strength)/UTS (Ultimate Tensile Strength) ratio
• high initial work hardening rate
• high uniform elongation
• no yield point elongation

These characteristics are particularly attractive to the automotive industry. Continuous yielding and high uniform elongation allow for the fabrication of complex shaped parts and finally high level of work hardening rate results in high strength in the as-formed parts.

Figure 2-3 Typical stress-strain curves for high-strength automotive steels. (After Morrow\cite{9})

The chemistry found in most dual phase steels falls in the Fe-C-Mn family\cite{10,11,12}, while other chemistries, such as Fe-C-Si with micro alloying additions\cite{13}, also can be used to form dual phase steel. For the Fe-C-Mn system, the chemical composition of the major
alloying elements falls in the following range:

<table>
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<th>Range</th>
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<tr>
<td>Carbon</td>
<td>0.04-0.2 wt.%</td>
</tr>
<tr>
<td>Manganese</td>
<td>0.24-2.3 wt.%</td>
</tr>
</tbody>
</table>

2.2.2 Processing and Microstructure of Dual Phase Steels

In the 1970s, a large amount of research was conducted to demonstrate that various industrial processes could be used to produce duplex ferritic-martensitic microstructures from plain carbon steels usually containing microalloying additions\cite{8, 10, 11, 14}. These processes are summarized as follows,

I. As-hot-rolled -- Cooling from single $\gamma$ phase into the intercritical ($\alpha+\gamma$) region, then quenching.

II. Continuously annealed (intercritical) -- Heating from ($\alpha + \text{Fe}_3\text{C}$) into the intercritical ($\alpha+\gamma$) region, then quenching.

Of these two processes, the current work is more interested in the latter one, since automotive body panel is normally produced from cold-rolled steel products.

The microstructure of the dual phase steel consists of a “clean” ferrite matrix and hard martensite (which is transformed from austenite phase during cooling to room temperature) plus a small amount of retained austenite which is almost always found in dual phase steels\cite{12, 15}. Aside from the volume fraction, $f$, that carries no information on the distribution and morphology of the component phases in the material, Becker, et al.\cite{16} introduced the following parameters to characterize the dual phase microstructure,
\[ \Delta = \frac{C_\gamma}{C_{aa}} \]  
\[ \delta = \frac{C_{\alpha\gamma}}{C_{aa}} \]  
where \( C_{aa} \), \( C_\gamma \) and \( C_{\alpha\gamma} \) are the densities of ferrite-ferrite grain boundaries, austenite-austenite grain boundaries and ferrite-austenite phase boundaries, respectively.

The characterization of dual phase microstructures using \( \Delta \) is illustrated schematically in Figure 2-4\textsuperscript{[16]}. \( \Delta = 0 \) represents a dispersion structure with isolated austenite grains separated by ferrite. As \( \Delta \) increases from 0 to \( \infty \), there is a transition in the distribution of the \( \gamma \) phase from dispersion to a continuous network structure, as the \( \gamma \) phase becomes interconnected and the \( \alpha \) grains separated, i.e. the \( \Delta \) value provides a description of the morphology of the phases in the microstructure. However, the \( \Delta \) parameter does not contain information on the coarseness of a dispersion structure. Instead, this information can be characterized by the \( \delta \) parameter, as illustrated in Figure 2-5. The dispersion becomes finer with the increasing \( \delta \) value for the microstructure.

2.2.3 Mechanical Properties of Dual Phase Steel

The mechanical properties of dual phase steels are related to their microstructure. Referring to Figure 2-3, it is clear that the uniform elongation of the dual phase steel is much higher than for a similar strength HSLA steel. It is known that during tensile loading, necking occurs when the considere criterion is reached, i.e. \( \frac{d\sigma}{d\varepsilon} = \sigma \)\textsuperscript{[17]}. The uniform elongation is thus increased if the work hardening rate can be raised. In dual phase steels, the work hardening behaviour is modified due to having a hard phase (martensite) in a soft matrix.
(ferrite). There have been many attempts to rationalize the mechanical properties of dual phase steels in terms of the micro-mechanics of the two-phase system. The phase averaged stress and strain can be described as[^18]:

![Figure 2-4 Schematic survey of dual phase microstructures. (After Becker, et al.[^16])](image-url)
Figure 2-5 characterization of different types of dispersion structure. (After Becker, et al.\textsuperscript{[16]})
\[ \sigma = (1 - f_M) \sigma_\alpha + f_M \sigma_M \]

and
\[ \varepsilon = (1 - f_M) \varepsilon_\alpha + f_M \varepsilon_M \]  

(2-3)

where \( \sigma \) and \( \varepsilon \) are stress and strain, subscripts \( \alpha \) and \( M \) denote the ferrite and martensite phase, respectively and \( f_M \) is the volume fraction of martensite. The difficulty lies in predicting how the stresses and strains are partitioned between the two phases during deformation. Two extreme possibilities, equality of strain and equality of stress, predict widely differing behaviour and reality lies between these extremes\(^{19}\). Not only are stresses and strains different in the two phases, they are inhomogeneously distributed within each phase, as demonstrated by computer simulation using finite element methods (FEM)\(^{20,21}\).

In summary, by carefully controlling the chemistry and thermal path, material with different microstructures, hence different mechanical properties can be produced. In principle, if the martensite volume fraction, size and distribution are known, the mechanical properties can be predicted. However, a good understanding of the microstructure evolution of the material is required to develop an overall process model to predict mechanical properties.

### 2.3 Overview of TRIP Steels

#### 2.3.1 Introduction to TRIP Steels

The acronym TRIP (Transformation Induced Plasticity) was coined by Zackay et al.\(^{22}\) in 1967, with respect to their seminal study on austenitic stainless steels. The classic TRIP steels used in these studies have a high level of alloy additions, in particular 4.5-12% Ni. More recently, ferrite based steels which exhibit the TRIP effect can be produced by careful control of alloy elements additions and a proper heat treatment, so that substantial amounts of retained austenite are found in the final microstructure. It has been found that the strength-formability
characteristics of these steels are enhanced by the TRIP effect, in this case, the transformation of retained austenite to martensite occurs during deformation. This has lead to the development of TRIP steels\cite{23, 24} which contain ferrite, bainite, martensite and retained austenite in the microstructure. In order to retain austenite in the final microstructure, a two step cooling pattern is introduced into the heat treatment schedule\cite{25}.

The major alloying elements found in ferrite based TRIP steels are carbon, manganese and silicon. The chemical composition of TRIP steel typically falls in the following ranges\cite{26}:

- $C$: 0.15 – 0.40 wt.%
- $Mn$: 1 – 2 wt.%
- $Si$: 1 – 2 wt.%

with perhaps the most widely studied being 0.1-0.2 C, 1.5 Mn and 1.5 Si in wt.%. Al can also be found in some TRIP steels, replacing some of the Si, as the conventional Si-TRIP steels might present casting and galvanizing problems\cite{27, 28}.

The microstructure of the TRIP steels is complex and usually consists of a fine dispersion of metastable retained austenite, martensite, bainite and ferrite. The size, distribution and composition of the various phases are critical in controlling the properties of the material. The TRIP steels cover a tensile strength range from 600-800 MPa and offer a combination of good strength and formability\cite{29} (see Figure 2-2).

2.3.2 Processing Routes

Two principal processing routes for producing TRIP steels have been proposed\cite{29,30,31}, as schematically illustrated in Figure 2-6\cite{32}.
Figure 2-6 Thermomechanical paths for TRIP steels. (Adopted from Eberle[32])
1. Controlled cooling after hot-rolling to obtain hot-rolled TRIP steels.

2. Intercritical annealing of ferrite/pearlite mixture followed by an isothermal tempering stage in the bainite transformation temperature ranges.

During the tempering process, part of the austenite transforms to bainite, whereas a significant amount of residual austenite may become sufficiently stabilized by carbon rejection from bainite so as to not transform to martensite during the final cooling to room temperature[33, 34, 35, 36].

2.3.3 Role of Retained Austenite

As discussed in section 2.2.1, there is a small amount of retained austenite phase present in dual phase steels, which was initially thought to play a minor role. The effect of retained austenite in low carbon steels on the mechanical properties was not fully recognized until 1980’s during which many studies demonstrated that retained austenite improves the mechanical properties of the steels through the TRIP effect, if the stability of retained austenite can be increased[37, 38, 39]. Therefore, the key to understanding the better properties of TRIP steel is to understand the transformation of metastable austenite to martensite during deformation. In the thermodynamic aspect of the problem, the external applied stress makes it possible for the metastable retained austenite to transform to martensite by increasing the thermodynamic driving force[40], as illustrated schematically in Figure 2-7[41], where $T_0$ is the temperature at which the parent austenite phase and daughter martensite phase are in thermodynamic equilibrium. At any temperature below $T_0$ the change in bulk free energy given by $\Delta G^\gamma - a' = G^a' - G^\gamma$ is negative and therefore the reaction is favorable. However, the formation of a martensite plate produces a
substantial amount of elastic strain due to the shape change and the constrain of the surrounding matrix and the formation of a martensite nucleus also creates a new interfacial area. The associated strain energy $\Delta G_e$ and interface energy $\Delta G_I$ are positive and they form the activation barriers to nucleation. Further cooling to a temperature $M_S$ below $T_0$ is essential to give sufficient driving force to begin the martensite transformation. At this temperature, the free energy change $\Delta G_{M_S}^{\gamma' - \alpha'}$ is negative. At a temperature $T_1$ between $T_0$ and $M_S$, retained austenite under external applied stress can also be transformed to martensite, if an added mechanical driving pressure $U'$ to the chemical driving force $\Delta G_{T_1}^{\gamma' - \alpha'}$ meets the requested driving pressure $\Delta G_{M_S}^{\gamma' - \alpha'}$.

Figure 2-7 Mechanical stability of retained austenite. (After Laquerbe\textsuperscript{[41]})
The applied strain can also assist the formation of martensite from retained austenite by creating extra nucleation sites or embryos\cite{40}. The martensite islands which are produced act as strong barriers to slip and therefore increase the macroscopic work hardening rate. In summary, the mechanism of TRIP is believed to be that during plastic deformation some of the austenite in the most severely strained portions of the material will transform to martensite by the action of the applied strain/stress. The martensite produced during straining prevents early failure due to necking, via stabilization of the neck through increases in the strain hardening rate. This leads to an overall increase of both tensile strength and elongation\cite{22}.

2.4 Effect of Alloying Elements in Dual Phase and TRIP Steels

The major alloying elements for dual phase and TRIP steels are carbon, manganese and silicon. In general, it is desirable to minimize the alloying additions due to a number of considerations including cost, coatability and weldability of the product.

Carbon is a very efficient austenite stabilizer which leads to a general retardation of reaction kinetics\cite{42}. Carbon also has a large effect on the range of temperature over which austenite decomposition occurs. The following empirical equation describes the effect of alloying elements on the bainite start temperature $B_S$\cite{43}:

$$B_S (°C) = 830 - 270C - 90Mn - 37Ni - 70Cr - 83Mo \quad (2-4)$$

and martensite start temperature $M_S$ (Andrew’s equation\cite{44}):

$$M_S (°C) = 539 - 423C - 30.4Mn - 17.7Ni - 12.1Cr - 7.5Mo \quad (2-5)$$

where the alloying elements are in weight percent. It can be seen from the above equations that carbon is the most efficient alloying element in decreasing the bainite start temperature and martensite start temperature.
The addition of the alloying elements Mn and Si to a normal plain carbon steel produces several important changes. First, their presence changes the phase boundaries in the Fe-Fe₃C phase diagram (Figure 2-1). Manganese acts as an austenite stabilizer by lowering the two-phase (α+γ) region toward room temperature and raising the two-phase (δ+γ) zone to the melting range, thus opening the γ phase region, i.e. expanding the temperature range for stable austenite. Manganese addition also lowers the $M_s$ temperature, as shown empirically in Andrew's equation (2-5). In fact, Mn has the second most significant effect in lowering the $M_s$ temperature among the other alloying elements (except for carbon).

Silicon is reported to stabilize austenite by inhibiting carbide precipitation due to its extremely low solubility in cementite and therefore promoting enrichment of residual austenite with carbon during bainite transformation. As a result, studies of the TRIP steels usually focus on alloys containing rather high concentrations of silicon (>1 wt.%) [49]. However, Jacques et al. [50] recently proposed that by carefully controlling the bainitic tempering time and temperature, the carbide precipitation can be hindered in a low carbon, low silicon (0.39 wt.%) cold-rolled steel. Consequently, a volume fraction of austenite as large as nearly 10% can be stabilized in the microstructure and excellent strength-elongation combination stemming from the TRIP effect as well as the contributions from other second phases (bainite and martensite), can be obtained.

The alloying elements Mn and Si also change the kinetics of phase transformations in the system. Like most of the familiar alloying elements, Mn and Si displace the TTT curve of pearlite transformation for a plain carbon steel to the right, i.e. towards longer transformation times [51], thus making martensite formation easier, thereby increasing the hardenability of the
2.5 Microstructure Evolution during Industrial Processing

In the heat treatment of cold-rolled dual phase and TRIP steels, the microstructure evolution involves recovery/recrystallization, austenite formation, followed by austenite decomposition. Information about these phenomena is given in the following sections.

2.5.1 Recovery and Recrystallization

The first significant phenomenon that happens to the cold-rolled steel during reheating to the intercritical annealing temperature is recovery and recrystallization. Recovery is defined as the restoration of physical properties of the cold worked metal without any observable change in microstructure\(^{[52]}\). It is driven by the stored energy in the deformed material. Generally, recovery in plastically deformed metals is controlled by the dislocation glide, climb and cross slip. The stored energy of the material from the cold working is lowered through the dislocation annihilation and re-arrangement\(^{[53]}\).

Recrystallization involves the formation of new strain-free grains in certain parts of the specimen and the subsequent growth of these grains to consume the deformed or recovered microstructure\(^{[54]}\). This is accomplished through the reorientation of crystals in the specimen by the passage of high-angle boundaries\(^{[55]}\). As a result, recrystallization produces a drastic change of mechanical properties of the deformed material (such as hardness, yield strength, tensile strength, ductility, etc.) to the level corresponding to those of the annealed condition. The recrystallization process is affected by the prior recovery process in the following ways: 1) recovery plays an important role in nucleating recrystallization; 2) the nature and kinetics of
recrystallization is influenced by a significant amount of prior recovery, as recovery lowers the driving force for recrystallization. On the other hand, the progression of recrystallization will terminate any further recovery since deformation substructure is consumed.

The kinetics of recrystallization can usually be described using the JMAK (Johnson-Mehl-Avrami-Kolmogorov) equation:\(^{56}\):

\[ X_v = 1 - \exp (-bt^n) \]

(2-6)

where \( X \) is the volume fraction recrystallized in time \( t \), \( n \) and \( b \) are adjustable parameters.

Although most of the recrystallization researches have been carried out under isothermal condition, attempts have been made to apply the JMAK equation to continuous heating condition\(^{57}\). Studies show that recrystallization of the deformed ferrite grains in dual phase and TRIP steels may occur during reheating (below \( Ac_1 \) temperature) and/or during the early stages of intercritical annealing process (i.e. at temperatures between \( Ac_1 \) and \( Ac_3 \)), depending on the heat treatment conditions, i.e. heating rate, holding time\(^{25,58}\). However, limited results are available for the intercritical annealing process, and thus, the interaction between the recrystallization of ferrite and the formation of austenite remains unclear. For example, it cannot be ruled out that the recrystallization process may extend into the austenite formation regime. The possibility of overlapping between the recrystallization and austenite formation depends on many factors, but it seems that the alloying elements play a major role. For example, Reis et al.\(^{59}\) have found that in an 95 % cold-rolled IF steel (0.0024C–0.044Ti in wt.%), the recrystallization has completely finished before the onset of the ferrite to austenite formation, even under a heating rate of 4500 °C/s. However, in the work of Yang et al.\(^{60}\) on a 50 % cold-rolled steel containing more alloying elements (0.08C–1.45Mn–0.21Si in wt.%), it
was observed that the recrystallization extended well into austenite formation regime when reheated using a salt bath.

2.5.2 Austenite Formation

Intercritical annealing is a critical processing step required to produce dual phase and TRIP steels from cold-rolled steels. Austenite is formed during the intercritical annealing process. The formation of austenite has been widely investigated over the last century albeit not as extensively as for austenite decomposition. What appears to be the first account that austenite forms as a nucleation and growth process upon heating, can be traced to Arnold and McWilliam\textsuperscript{61}. Roberts and Mehl\textsuperscript{62} confirmed the nucleation and growth nature of the process by discussing in detail the rate of austenite formation from ferrite-cementite aggregates. A substantial body of work was published in the 1980s in conjunction with extensive research on dual phase steels\textsuperscript{63-78}. Studies on the austenite formation from ferrite and pearlite\textsuperscript{64, 78, 79, 80}, ferrite and cementite\textsuperscript{79, 80, 81}, and martensite\textsuperscript{82, 83, 84, 85, 86} suggest that the austenite nucleation is sensitive to initial microstructure. For example, Speich \textit{et al.}\textsuperscript{64} concluded that the first stage of austenite formation in 1.5 wt.% Mn low carbon steels with ferrite-pearlite microstructure occurs by nucleation of austenite at ferrite-pearlite interfaces and the subsequent growth of the austenite into pearlite until pearlite dissolution is complete. The austenite formation features a quite rapid initial rate and is controlled by carbon diffusion, followed by sluggish kinetics in the later stages which are associated with the partitioning and diffusion of substitutional alloying elements\textsuperscript{64, 71}. Although the steel properties depend on the austenite decomposition following austenitization, the initial austenitic condition before intercritical annealing is important for the development of the final microstructure and its mechanical properties.
It was found\textsuperscript{[60]} that in the cold-rolled steel, the distribution of austenite can be affected by the recrystallization process prior to the austenite formation. Austenite first forms on boundaries between elongated, unrecrystallized ferrite grains resulting in bands of austenite parallel to the rolling direction. Moreover, rapid pearlitic cementite spheroidization in the cold-rolled material during reheating was also observed. In another work of Yang et al.\textsuperscript{[87]}, it has been demonstrated that even in normalized ferrite-pearlite microstructures, spheroidization of the pearlitic cementite occurs at short intercritical annealing times. The results suggested that austenite formation is slower with the spheroidized pearlitic cementite microstructure.

Gau and Thomas\textsuperscript{[13]} examined the influences of different transformation paths on the final microstructure of dual phase steel. In their study, three transformation paths were chosen, as depicted in Figure 2-8. In the process I, the material was intercritically annealed. The dominant nucleation sites for the austenite phase during the annealing were ferrite-cementite boundaries and ferrite grain boundaries. The resulting microstructure is fine globular martensite dispersed in a ferrite matrix with a grain size of \(~20 \, \mu m\). In the process II, the material was first austenitized, then cooled into the intercritical region. In this case, ferrite nucleates and grows from the unstable austenite during the annealing, resulting in a coarse two-phase morphology, indicating that this heat treatment scheme has no grain refinement effect. In the process III, the material was first austenitized, followed by quenching to room temperature, then intercritically annealed. It was found that reheating to the two phase annealing temperature from the initial \(~100\%\) martensite involves the nucleation of austenite at prior austenite grain boundaries, martensite lath boundaries and possibly other lattice defects. The final microstructure has a fibrous morphology which indicates that the austenite formed during
the annealing grows in an acicular manner along martensite lath boundaries.

A further example of a study on the intercritical annealing of a cold-rolled (0.11C-1.5Mn-1.5Si) multiphase steel can be found in the work of Samajdar et al. In this work, it was found that the austenite volume fraction and the intercritical austenite grain size increases with an increase in intercritical soaking periods and temperature, but the austenite stability decreases (as $M_s$ temperature increased), as larger austenite grains are expected to be less stable with respect to martensite transformation. On the other hand, both the volume fraction of retained austenite and its carbon content dropped with prolonged soaking. At the very initial stages of soaking, small size and high carbon content of the austenite colonies ascertained their survival at room temperature—indeed no martensite could be formed by room temperature quench. With a longer soaking time and a carbon homogenization in intercritical austenite,
smaller dimensions of the austenite domains were possibly more crucial for their stability. Prolonged soaking also formed larger amounts of martensite surrounding small austenite colonies.

2.5.3 Decomposition of Austenite

For TRIP steel, after intercritical annealing, the temperature is lowered and held at a temperature below the nose of α/pearlite reaction, i.e. in the bainite region. Bainite transformation in steels probably remains the least understood of all the decomposition reactions of the high-temperature γ phase even after extensive studies. Two opposite theories about the transformation mechanism exist. The diffusional reaction mechanism suggests that the growth of bainitic ferrite is considered to take place by the diffusion-controlled movement of ledges and the carbon concentration of growing bainitic ferrite is in equilibrium with respect to austenite. The bainite reaction, in fact, is assumed to be an extension of the proeutectoid ferrite reaction. The bay in transformation-time-temperature (TTT) diagrams at the bainite start temperature and the incomplete transformation phenomenon of bainite are explained in terms of a solute drag effect (SDE) produced by the segregation of certain alloying elements into the mobile austenite/ferrite boundaries. These alloying elements trapped in boundaries depress the carbon activity in austenite in contact with the growing ferrite, thereby reducing the diffusional flux of carbon into austenite and retarding ferrite growth. This may lead to growth stasis or the complete cessation of bainite growth.

On the other hand, the displacive reaction mechanism proposes that bainite transformation is diffusionless, as far as the solvent and substitutional atoms are concerned.
Bainitic ferrite supersaturated with carbon forms displacively with carbon diffusing from ferrite into austenite after the growth event or, alternatively, precipitating as carbide inside the ferrite. Minote et al.\textsuperscript{[93]} studied the bainite transformation behaviour of a TRIP steel (0.19C-1.51Mn-1.45Si), in which the evolution of bainite volume fraction was determined and compared to the model predictions based on diffusional and displacive mechanisms, respectively. This analysis suggested was found that the bainite transformation follows the diffusional mechanism above 350 °C and displacive mechanism below 350 °C during the bainite transformation.

The bainite transformation is also strongly influenced by alloying elements in steel. The bainite transformation start temperature $B_s$ is depressed by many alloying elements but carbon has the greatest effect, as indicated in equation (2-2)

Carbide-free bainite transformation promotes the carbon enrichment in the neighboring austenite, since carbon is rejected into nearby austenite when bainitic ferrite is formed and results in a higher volume fraction of retained austenite. Bai et al.\textsuperscript{[94]} showed that a short holding time (2 min.) during the bainite transformation following the intercritical annealing of a TRIP steel (0.20C-1.5Mn-1.5Si) not only led to some of austenite transforming to martensite but also lowered the carbon content in the retained austenite. On the other hand, holding for too long a time (10 min.) at this temperature also leads to a decrease in the carbon content due to formation of carbides. Clearly, the alloy addition, temperature and time in the bainite transformation region are critical to the formation of retained austenite in optimal condition.

During the final cooling stage following the bainite transformation process, remaining austenite either transforms to martensite or remains as retained austenite depending on composition. The martensite transformation is a diffusionless process, i.e. no change in
composition, accomplished by homogeneous lattice shear\cite{95}.

2.5.4 Retained Austenite

As mentioned before, the retained austenite plays an important role in the mechanical properties of the TRIP steels. In order to utilize the TRIP effect, the stability of the austenite must be increased so that a significant volume fraction of retained austenite can be obtained in the final microstructure. It is proposed\cite{96} that the presence of austenite in the final microstructure be affected by:

*Alloy content.* With the presence of enough alloying elements, $M_S$ and $M_F$ temperatures are lowered to marked degree (see Eq. 2-2), therefore the retained austenite becomes more stable. In addition, alloying elements cause solid solution hardening and hinder the nucleation of martensite\cite{97}.

*Intercritical annealing temperature.* As the austenitization temperature is raised, the amount of retained austenite is increased because of a greater degree of carbon and other elements in the austenite acquired at the higher austenitization temperatures. Thus, overheating tends to take more carbon into solution because of dissolution of more carbides and greater diffusion rates at higher temperature. With increasing carbon content in austenite, the $M_S$ temperature is lowered. This results in the retention of additional austenite at room temperature, when the material is quenched.

*Cooling rate.* The rate of cooling or severity of the quench also affects the amount of retained austenite in steel. If on quenching the rate of cooling in the $M_S - M_F$ range is slowed down, there is a greater amount of retained austenite formed than if the cooling rate is continuous and rapid. During cooling, the partitioning of carbon leads to a time dependent
austenite stabilization. The diffusion of interstitials to dislocations generated in austenite can cause atmospheres and strengthen the austenite\(^{98}\).

*Austenite grain size.* Grain size affects the stability in two ways, (i) the probability of the occurrence of a critical nucleus size is small in small grains, and (ii) the volume of martensite produced per nucleus is small for small grains, due to closely spaced boundaries\(^{99}\).

The work by Jeong *et al.*\(^{33}\) showed that during the plastic deformation of TRIP steel, most of retained austenite with particle size larger than 1 \(\mu\)m has transformed to martensite at a strain of about 5\%, therefore retained austenite with particle size smaller than 1 \(\mu\)m are considered to be stable and can make significant contribution to the ductility of the material.

### 2.6 Experimental Techniques for the Study of Microstructure Evolution

A number of traditional experimental techniques e.g. metallography, dilatometry, etc., have been widely adopted to characterize the microstructure of steel and reveal the phase transformation kinetics. In addition, with the advances in the technology, *in-situ* information such as carbon redistribution during the austenite decomposition process, which was not available experimentally in the past, becomes possible to obtain. In this section, the relevant experimental techniques are reviewed.

#### 2.6.1 Metallography

Metallography is widely used as a traditional microstructure characterization tool. In this technique, normally an appropriate etchant is used to reveal the microstructure of the material. For TRIP steel with multi-component microstructure consisting of ferrite, bainite,
martensite and retained austenite, conventional etchant such as nital and picral used in the optical metallography technique cannot serve the purpose of phase identification well. Tint etching has been developed to better identify the component phases. Van der Voort\cite{100} suggested a sodium metabisulfite (SMB) etchant to reveal isothermally treated steels. It is capable to uniformly reveal ferrite grain boundaries and colorizes ferrite, pearlite, martensite, bainite and retained austenite. LePera\cite{101,102} proposed an etchant made of a mixture of 1% SMB and 4% picral that distinguishes martensite, bainite and ferrite in a high-strength dual phase steel. However, the grain boundaries are not revealed properly with this etchant. Lawson et al.\cite{103} developed a double etching technique using picral-alkaline chromate which is able to differentiate the ‘new’ ferrite (produced from austenite on cooling) and ‘old’ ferrite (proeutectoid ferrite not transformed during he intercritical annealing). Table 2-1 summaries the tint etching techniques adopted in the study of dual phase and TRIP steels.

<table>
<thead>
<tr>
<th>Etchant</th>
<th>Etching Result</th>
<th>Ref.</th>
</tr>
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| 2% nitol, followed by: 10% SMB in distilled water | Ferrite: gray  
Austenite: white  
Martensite: brown  
Bainite: dark gray | \cite{100,104} |
| 1 part (1% SMB) + 1 part (4% picral) | Martensite: white  
Bainite: black  
Ferrite: tan | \cite{101} |
| 4% picral, followed by: (8g CrO₃, 72ml H₂O and 40g NaOH) | “Old” ferrite: gray  
“New” ferrite: white  
Martensite: black | \cite{103} |
2.6.2 Dilatometry

Dilatometry is one of the classic techniques, along with the quantitative analysis of microstructures, most commonly employed to determine the phase transformations in steels. This technique is based on the fact that when a steel undergoes a phase transformation, its lattice structure changes and this is accompanied by a change in the specific volume. For example, when austenite in pure iron is transformed into ferrite phase, a volume expansion of about 1.6 % can be observed\(^{[105]}\). This volume change associated with the phase transformation can be detected by measuring the length change of the sample. The transformation kinetics can be determined when the relationship between the dilation, the phase compositions, the transformed phase volume fractions and temperature\(^{[106]}\).

Dilatometry has been extensively applied to the studies of austenite formation and decomposition kinetics\(^{[105, 107, 108, 109, 110, 111, 112]}\). The work by Quidort\(^{[113]}\) has shown that the dilatometric data analysis based on the lever rule can be applied to the austenite formation process. For the austenite decomposition process where several transformation products are formed, the lever-rule method seems to over-estimate the transformed fraction of the proeutectoid ferrite and pearlite\(^{[114]}\). In this case, several corrections to the data analysis method have been proposed\(^{[105, 106, 114, 115]}\), taking into account the redistribution of carbon between the transformed ferrite and the remaining austenite and their different thermal expansion coefficients.

2.6.3 Neutron BET Technique

Neutron Bragg-Edge Transmission (BET) technique is closely related to the neutron diffraction technique in a way that both share the same operational principle, the Bragg's
law:  

$$\lambda = 2d_{hkl} \sin \theta$$  \hspace{1cm} (2-7)$$

where $\lambda$ is the wavelength of the incident beam, $d_{hkl}$ the lattice spacing for the crystal plane $\{h k l\}$ and $\theta$ the diffraction angle. Therefore, information such as phase compositions, volume fractions and lattice parameters can be obtained for the study of steel phase transformations. Most interestingly, the lattice parameter can be used to derive the carbon concentration information in the austenite and ferrite, as has been demonstrated by various researchers.  

Previously, neutron BET technique has been applied for structural phase transformations\[120, 121, 122, 123, 124\] as well as strain measurement \[125, 126\]. In the work of Meggers et al.\[127\], it has been demonstrated that a time resolution of 20 $\mu$s is achievable in obtaining phase fractions and lattice parameters using a single pulse of neutron. Pioneer work by Vogel\[128\] has been conducted to demonstrate the application of the neutron BET technique in the study of bainite transformation in steel; however, the study suffered from poor temperature control on the sample. In a time-resolved kinetic phase transformation study, a large amount of neutron BET datasets can be generated in a single run, rendering manual data analysis rather low in efficiency. Practical data analysis involves software automation of the data processing procedure\[129\].

2.7 Summary

Dual phase and TRIP steels have a good combination of strength and ductility. During
the processing of dual phase and TRIP steels, complex reactions take place, such as recrystallization, austenite formation and austenite decomposition. The ferrite recrystallization behaviour of the cold-rolled material can be described using the Avrami equation. The kinetics of recrystallization is affected by the alloying additions in the steels. Austenite formation is a classical nucleation and growth process with the initial stage controlled by carbon diffusion and later by the diffusion of substitutional elements such as Mn. For the TRIP steel, the austenite phase can be stabilized by the carbon enrichment during the bainite transformation. As a result, austenite phase can survive during the final quench to room temperature and present in the microstructure as retained austenite, which contributes to the TRIP effect.

While information on the individual reactions is available in the literature, the interaction between those reactions is not clear. In particular, the effect of ferrite recrystallization, when it has the chance to extend into the austenite formation region, on the austenite formation kinetics and austenite distribution and morphology. For the bainite transformation, the 'in-situ' information about the carbon redistribution between the austenite and bainitic ferrite is missing.
Chapter 3 Objectives

The primary goal of this study is to obtain a quantitative understanding of the microstructure evolution for dual phase and TRIP steels along the processing route. This will provide a solid ground for the development of a process model which describes the microstructure evolution of the dual phase and TRIP steel during processing. The mechanical properties of the material can also be predicted if the model links the microstructure and mechanical behaviour. The following specifies the objectives:

1. The isothermal recrystallization behaviour of dual phase and TRIP steels will be characterized experimentally. This behaviour will be modeled using a semi-empirical approach and then applied to non-isothermal conditions.

2. A comprehensive set of experiments will be conducted to characterize the austenite formation kinetics of dual phase and TRIP steels during the intercritical annealing process and to understand the effect of recrystallization on the austenite formation behaviour of the steels.

3. New experimental technique (neutron Bragg-edge Transmission) is to be explored to study the bainite formation behaviour of a TRIP steel and to obtain information about carbon redistribution in the material during the bainite transformation.
Chapter 4 Study of Ferrite Recrystallization

The first metallurgical phenomenon to be examined in this work is the recrystallization of the cold-rolled material as it is heated to the intercritical region. In this chapter, an experimental study is described that has been conducted to characterize the isothermal recrystallization kinetics for Fe-C-Mn-Mo and Fe-C-Mn-Si steels. The kinetics of recrystallization are then described by an Avrami type model which can be generalized to non-isothermal heating conditions.

4.1 Experimental

This study has examined two steel chemistries, i.e. one typical of a dual phase steel and the other typical of a TRIP steel. The chemical compositions of the two steels are given in Table 4-1. In order to examine the possibility of the overlap between the ferrite recrystallization and austenite formation process, the thermodynamic calculations for the ferrite-austenite two phase region were carried out using the Thermo-Calc® software with FE2000 database. Two conditions were considered for the calculation: orthoequilibrium (OE) and paraequilibrium (PE). In the orthoequilibrium condition, the ratio of substitutional alloying elements such as Mn and Si to Fe in the ferrite and austenite were allowed to vary, i.e. the redistribution of the substitutional elements was allowed. In the paraequilibrium condition, such redistribution was not allowed so the ratio of substitutional elements to Fe in the ferrite and austenite were kept constant for the calculation. The $T_{Ae1}$ and $T_{Ae3}$ as well as the austenite volume fraction in the two phase region can be obtained from the calculations under the OE
and PE conditions.

Samples of the Fe-C-Mn-Mo steel were received from STELCO Inc. in the hot-rolled and cold-rolled state. The hot-rolled and cold-rolled material had thicknesses of 3.8 mm and 1.5 mm, respectively. The cold reduction was 55% (i.e. \( \frac{h_0 - h}{h_0} \times 100 = \% \) cold reduction, where \( h_0 \) and \( h \) are the thickness of the material before and after the cold-reduction, respectively).

The Fe-C-Mn-Si steel was cast as a 50 kg laboratory ingot which was subsequently hot-forged to a bar of 50 x 50 x 200 mm. The as-received material was cut into 12.7 mm thick samples and then hot-rolled to 3 mm thickness using a laboratory scale rolling mill. The rolling temperature was 950 °C, with air-cooling at the end of the hot-rolling process. The hot-rolled steel was then given a 50% cold reduction, resulting in 1.5 mm thick sample for the following study.

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Mn</th>
<th>Mo</th>
<th>Si</th>
<th>Ti</th>
<th>Nb</th>
<th>V</th>
<th>Cr</th>
<th>Ni</th>
<th>Cu</th>
<th>P</th>
<th>S</th>
<th>Ca</th>
<th>ASA*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-C-Mn-Mo</td>
<td>.06</td>
<td>1.86</td>
<td>.15</td>
<td>.07</td>
<td>.011</td>
<td>.002</td>
<td>.004</td>
<td>.048</td>
<td>.014</td>
<td>.015</td>
<td>.015</td>
<td>.0041</td>
<td>.0012</td>
<td>.043</td>
</tr>
<tr>
<td>Fe-C-Mn-Si</td>
<td>.178</td>
<td>1.55</td>
<td>.005</td>
<td>1.70</td>
<td>.004</td>
<td>.002</td>
<td>.002</td>
<td>.016</td>
<td>.015</td>
<td>.042</td>
<td>.007</td>
<td>.005</td>
<td>.0013</td>
<td>.026</td>
</tr>
</tbody>
</table>

*ASA -- Acid Soluble Aluminum

The microstructure of the starting material was examined using optical microscopy. Samples parallel to the rolling direction and perpendicular to the rolling plane were mounted and then mechanically grounded and polished, with the final polishing step using 0.05 micron silica solution. The etching procedure used for the metallographic sample was as follows: 3
seconds pre-etching in 2% Nital, rinse in water and final etching for 25 seconds in 10% sodium meta-bisulfite aqueous solution. Grain size was determined using Jeffries method outlined in the ASTM standard E112-96. The volume fraction of pearlite was determined using the Clemex image analysis system, in which the pearlite colonies were traced out and the area fraction of pearlite calculated.

For recrystallization studies, test coupons of 10 x 60 x 1.5 mm were machined from the as cold-rolled materials with the longitudinal direction of the test coupon aligned with the rolling direction of the material. Figure 4-1 illustrates the sample used in this study. The Gleeble 1500 thermal-mechanical simulator was employed in the recrystallization studies due to its unique capability to control the heating rate and sample temperature precisely over a wide range of conditions. Temperature was controlled using a type K thermocouple spot-welded on the centre of the sample and all annealing was conducted under a vacuum of ~26Pa.

![Figure 4-1 Schematic illustration for the dimension of the recrystallization sample.](image)

The temperature homogeneity on the sample has been measured to find out the working zone on the sample. The results showed a maximum temperature variation of 2 °C in a region within 2 mm away from the thermocouple junction, which forms the working zone for the
sample. The subsequent microstructure observations and hardness measurements were confined to this working zone. Figure 4-2 gives an example of the sample temperature-time profile during an annealing experiment conducted on the Gleeble system.

![Temperature-time profile diagram](image)

Figure 4-2 An example of sample temperature-time profile during an annealing experiment conducted on the Gleeble system.

For the isothermal recrystallization study, the samples were heated at a rate of 50 °C/s to the desired annealing temperature. After a predetermined isothermal annealing time, the sample was cooled rapidly to room temperature using helium gas quenching. The average cooling rate between the holding temperature and 250°C was 100 °C/s. The isothermal annealing conditions used in this study are summarized in Table 4-2. After the heat treatments, samples were sectioned longitudinally along the centre of the sample using a diamond saw.
The samples were then mounted, polished and etched using the same procedure described earlier in this section. Optical micrographs were taken in the region underneath the thermocouple junction. Microhardness (Hv) was measured using the MICROMET3 microhardness tester with a load of 25g weight. The indentations were located as much as possible in the ferrite grains.

Table 4-2 Isothermal annealing conditions applied to the recrystallization study.

<table>
<thead>
<tr>
<th>Initial heating rate</th>
<th>Isothermal annealing temperatures, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>50 °C/s</td>
<td>Fe-C-Mn-Mo: 600, 650, 680, 710</td>
</tr>
<tr>
<td></td>
<td>Fe-C-Mn-Si: 600, 650, 680</td>
</tr>
</tbody>
</table>

After the isothermal annealing experiments, the recrystallization behavior of the materials under non-isothermal conditions was examined using continuous heating test. The samples were heated at heating rates of 1 and 10 °C/s and then quenched out at various temperature points, followed by microhardness measurement and quantitative metallography on selected samples. The quantitative metallography for the ferrite recrystallization measurement was done as follows: first the ferrite grains were traced out in the micrograph to separate them from other phases that maybe present in the microstructure (e.g. pearlite, martensite), and their total area $A_{ferrite}$ calculated. Following that, the ferrite grains were analyzed to obtain the aspect ratio of each grain. The criterion for the recrystallized ferrite grain was taken as the aspect ratio less than 3. Then the recrystallized ferrite grain area $A_{Rex}$ can be obtained by summing up all the area of the ferrite grains that conforms to the recrystallization criterion. Finally the ferrite
volume fraction recrystallized was determined by the expression: \( \frac{A_{Re}}{A_{ferrite}} \times 100\% \).

4.2 Results

4.2.1 Starting microstructure

The microstructures of the hot and cold-rolled steels are shown in Figure 4-3. It can be observed that both hot-rolled steels feature a banded microstructure, i.e. pearlite bands in an equiaxed ferrite grain matrix (Figure 4-3a and 4-3c, for Fe-C-Mn-Mo and Fe-C-Mn-Si steel, respectively). The ferrite grain size was measured to be 6 \( \mu \text{m} \) and 11 \( \mu \text{m} \) for the Fe-C-Mn-Mo and Fe-C-Mn-Si steels, respectively. After cold rolling, the ferrite grains deform into an elongated shape consistent with the level of cold reduction. The pearlite colonies also elongate in proportion to the cold reduction consistent with the observations from other studies that ferrite and pearlite co-deform\(^{[60]} \). The spacing of pearlite bands in the hot-rolled and cold-rolled Fe-C-Mn-Mo steel is 31 and 15 \( \mu \text{m} \), respectively. The values for the hot and cold-rolled Fe-C-Mn-Si steel are 49 and 27 \( \mu \text{m} \), respectively. The reduction in pearlite spacing is consistent with the macroscopic deformation level for these two steels. The results from quantitative metallography indicate that the pearlite volume fraction is 11 % for the hot-rolled Fe-C-Mn-Mo steel and 25 % for the hot-rolled Fe-C-Mn-Si steel.

4.2.2 Ferrite recrystallization kinetics

During annealing, deformed ferrite grains are replaced by equiaxed grains, as can be seen in Figure 4-4 and Figure 4-5 for the cold-rolled Fe-C-Mn-Mo and Fe-C-Mn-Si steel, respectively, where the materials were annealed at 650\(^\circ\text{C}\). Closer observation using the scanning electron
Figure 4-3  Microstructures for the a) hot-rolled, b) 55% cold-rolled Fe-C-Mn-Mo steel and c) hot-rolled, d) 50% cold-rolled Fe-C-Mn-Si steel. Pearlite: dark ; ferrite: gray
microscope indicates that spheroidization of pearlite also occurs during annealing, as shown in Figure 4-6, in which the white regions are Fe₃C and the dark regions are the ferrite matrix. In the case of Fe-C-Mn-Mo steel, the recrystallized ferrite grain sizes was observed to be dependent on annealing temperature increasing from 6.3 to 8.9 µm as the annealing temperature increased from 600 to 710 °C (see Table 4-3).

<table>
<thead>
<tr>
<th>Isothermal Annealing Temperature, °C</th>
<th>600</th>
<th>650</th>
<th>680</th>
<th>710</th>
</tr>
</thead>
<tbody>
<tr>
<td>Recrystallized Ferrite Grain Size, µm</td>
<td>6.3</td>
<td>7.2</td>
<td>8.8</td>
<td>8.9</td>
</tr>
</tbody>
</table>

The results for the microhardness measurements of the ferrite phase as a function of annealing time are shown in Figure 4-7. It is clear that higher isothermal annealing temperature leads to faster ferrite softening process. The fraction softening of ferrite was calculated from the hardness measurements, i.e.

$$X_S = \frac{H_0 - H}{H_0 - H_{Rex}}$$ (4-1)

where $H_0$ is the initial microhardness of ferrite in the as cold-rolled steel, $H$ the microhardness after annealing and $H_{Rex}$ the microhardness corresponding to fully recrystallized steel.
Figure 4-4 Microstructure evolution for the cold-rolled Fe-C-Mn-Mo steel during isothermal annealing at 650 °C, with initial heating rate of 50 °C/s. Pearlite: dark; ferrite: gray.
Figure 4-5  Microstructure evolution for the Fe-C-Mn-Si steel during isothermal annealing at 650 °C, with initial heating rate of 50 °C/s. Pearlite: dark; ferrite: gray
Figure 4-6 Spheroidization of pearlite in the Fe-C-Mn-Si steel for annealing at 680 °C.
Figure 4-7 Microhardness measurements for a) Fe-C-Mn-Mo and b) Fe-C-Mn-Si steel annealed at various temperatures.
The fraction softening of ferrite obtained from microhardness measurement was compared to the fraction recrystallization from quantitative metallography, as shown in Table 4-4. It shows that the fraction softening for the ferrite phase measured from the microhardness is consistent with the fraction recrystallized measured from the quantitative metallography. Thus, the fraction softening for the ferrite phase ($X_S$) was used to represent the fraction recrystallized of ferrite in both steels.

Table 4-4 Comparison of the ferrite recrystallization kinetics determined by microhardness measurement and quantitative metallography for the cold-rolled Fe-C-Mn-Mo isothermally annealed at 680 °C.

<table>
<thead>
<tr>
<th>Isothermal Annealing Time s</th>
<th>Fraction Recrystallized</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Results from microhardness measurement</td>
</tr>
<tr>
<td>10</td>
<td>0</td>
</tr>
<tr>
<td>30</td>
<td>0.15</td>
</tr>
<tr>
<td>60</td>
<td>0.48</td>
</tr>
<tr>
<td>180</td>
<td>0.74</td>
</tr>
<tr>
<td>300</td>
<td>0.82</td>
</tr>
<tr>
<td>600</td>
<td>1</td>
</tr>
</tbody>
</table>

4.3 Discussion

The microstructures of the hot-rolled Fe-C-Mn-Mo and Fe-C-Mn-Si steels feature pearlite colonies with a banded morphology surrounded by equiaxed ferrite grains. After cold rolling, the morphology of both phases becomes elongated, i.e. the ferrite grains become
elongated and contain deformation bands and other substructures, while the pearlite is also deformed, as seen in Figure 4-3b and Figure 4-3d. The cold-rolling of the steel has the following effects on the material: 1) there is an increase in the stored energy of the material due to the high dislocation density and this provides the driving pressure for the ferrite recrystallization upon annealing\cite{130}; 2) the total ferrite grain boundary area is increased and 3) the cementite laminar structure in pearlite is broken down. The latter has been shown to promote spheroidization of cementite during subsequent annealing processes\cite{60,131}.

Upon annealing of the cold-rolled material, the stored energy is released primarily by recrystallization. The isothermal recrystallization kinetics for the ferrite phase in Fe-C-Mn-Mo and Fe-C-Mn-Si steels are shown in Figure 4-8. A suitable fit to the experimental results can be obtained using the Avrami equation\cite{132,133}, i.e.

\[ X = 1 - \exp(-bt^n) \]  

(4-2)

where \( X \) is the volume fraction recrystallized in time \( t \), \( n \) and \( b \) are fit parameters with \( b \) being a function of temperature:

\[ b = b_0 \exp\left(-\frac{Q}{RT}\right) \]  

(4-3)

where \( b_0 \) is a constant and \( Q \) is an effective activation energy for the recrystallization process. Overall, a good fit to the data can be found with \( n = 1 \) and \( Q = 350 \text{ kJ/mol} \) for both steels. However, some data scatter can be found for the Fe-C-Mn-Mo steel. The recrystallization rates are higher for the Fe-C-Mn-Si steel, and this is reflected in the value for \( b_0 \), i.e. \( b_0 = 1.6 \times 10^{18} \text{ s}^{-1} \) for the Fe-C-Mn-Si steel compared to \( b_0 = 7.2 \times 10^{16} \text{ s}^{-1} \) for the Fe-C-Mn-Mo steel.
Figure 4-8  Recrystallization kinetics and model predictions for the a) Fe-C-Mn-Mo and b) Fe-C-Mn-Si steel.
The activation energies for ferrite recrystallization in the Fe-C-Mn-Mo and Fe-C-Mn-Si steel are considerably higher than the 251 kJ/mol reported for the self-diffusion of iron in BCC ferrite\textsuperscript{[134]} and 227 kJ/mol reported by Yang and co-workers for a 50 % cold-rolled low carbon manganese steel\textsuperscript{[60]}. This can be understood in terms of the effects of solute additions such as Mn\textsuperscript{[135, 136]} and Mo\textsuperscript{[137]} which have been shown to delay the growth of newly recrystallized grains due to solute drag on migrating grain boundaries. The difference in the rate of recrystallization for the Fe-C-Mn-Mo and the Fe-C-Mn-Si steel is summarized in Figure 4-9 which shows the time for 50% recrystallization vs. temperature. It can be observed that the time to 50 % recrystallization is shorter for the Fe-C-Mn-Si steel than for the Fe-C-Mn-Mo steel. The slower recrystallization in the Fe-C-Mn-Mo steel can presumably be attributed to a particularly strong solute drag effect of Mo which has been well documented for austenite recrystallization\textsuperscript{[138]} and the austenite-to-ferrite transformation\textsuperscript{[139]}.

To examine recrystallization under non-isothermal conditions, experiments were conducted at constant heating rates to a set temperature at which point the sample was quenched out. The microstructure evolution for the cold-rolled Fe-C-Mn-Mo and Fe-C-Mn-Si steel during the continuous heating condition is shown in Figure 4-10, Figure 4-11 and Figure 4-12. It can be seen that at higher temperatures, the deformed ferrite grains are replaced by recrystallized ferrite grains. Figure 4-13 shows the results for fraction recrystallized. The solid lines are the predictions from applying equation (4-2) in differential form (taking $n=1$):

$$\frac{dX}{dt} = nb^n \left( \ln \left( \frac{1}{1-X} \right) \right)^{n-1} (1 - X)$$

(4-4)
Figure 4-9 Time for 50% recrystallization as a function of temperature.
Figure 4-10  Microstructure of the cold-rolled Fe-C-Mn-Mo steel heated at 1 °C/s and quenched out at various temperatures.
Figure 4-11 Microstructure of the cold-rolled Fe-C-Mn-Mo steel heated at 10 °C/s and quenched out at various temperatures.
Figure 4-12 Microstructure of the cold-rolled Fe-C-Mn-Si steel heated at 10 °C/s and quenched out at various temperatures.
and then numerically integrating. Clearly, good agreement can be observed between the model and experimental data for the Fe-C-Mn-Si steel in Figure 4-13, however, the model slightly underestimate the fraction recrystallized for the Fe-C-Mn-Mo steel at the heating rates of 1 and 10 °C/s.

Figure 4-14 illustrates the model predictions for the effect of heating rate on the progression of recrystallization. It can be observed that for both Fe-C-Mn-Mo and Fe-C-Mn-Si steels, as the heating rate increases, the recrystallization is retarded to higher temperatures. It is interesting to note that the $T_{ac}$ temperature calculated under OE condition falls well below the recrystallization finish temperatures at higher heating rate (e.g. 100 °C/s). Therefore, the interaction between recrystallization and austenite formation has to be considered. Important implications on the austenite formation may be expected when it occurs simultaneously with ferrite recrystallization, as will be discussed in detail in the next chapter which deals with the austenite formation.

4.4 Summary

The recrystallization kinetics of ferrite in the cold-rolled Fe-C-Mn-Mo and Fe-C-Mn-Si steels has been experimentally determined by a combination of softening measurements and quantitative metallography. It was found that a good mathematical description of the data could be made using the Avrami equation. It was observed that the possibility of an overlap between ferrite recrystallization and austenitization exists in the Fe-C-Mn-Mo and Fe-C-Mn-Si steels if higher heating rates are employed for annealing. This can have a significant impact on the austenitization process and thereby affect the final microstructure and properties for intercritically annealed steels.
Figure 4-13 Recrystallization behaviour for the a) Fe-C-Mn-Mo and b) Fe-C-Mn-Si steels under continuous heating condition; symbols represent experimental data and lines model predictions.
Figure 4-14 Effect of initial heating rate on the progressing of recrystallization with respect to the onset of austenite formation for the a) Fe-C-Mn-Mo steel and b) Fe-C-Mn-Si steel.
Chapter 5 Study of Austenite Formation

In the previous chapter, it was shown that for the cold-rolled steels, the recrystallization process could potentially overlap with the austenite formation region under certain reheating conditions. In this chapter, the austenite formation in the Fe-C-Mn-Mo and Fe-C-Mn-Si steel will be examined in detail and the interaction between the recrystallization and austenite formation processes will be addressed.

5.1 Experimental Procedures

First, the austenite formation kinetics for the hot-rolled steel under continuous heating conditions (1, 10 and 100 °C/s) was determined to establish the base line for this study. The dimension of the sample was the same as that used in the recrystallization study (see Figure 4-1). Heating was conducted with the Gleeble 1500 and a dilatometer was attached onto the sample during the thermal cycle to measure the dimensional change in the width direction. Figure 5-1 gives an example of the dilation-temperature relationship for the hot-rolled Fe-C-Mn-Mo steel heated to 1000 °C at a constant heating rate of 1 °C/s. The first linear portion in the curve represents the thermal expansion of the ferrite/pearlite mixture, which can be fitted with a linear line AA'. The point where the dilation curve deviates from the linear line AA' gives the austenite transformation start temperature. As the austenite and ferrite have different specific volumes, when the transformation to austenite progresses, the dilation of the sample decreases, until it reaches the beginning of the second linear portion FF' in the curve at
Figure 5-1  An example of the dilation-temperature relationship for hot-rolled Fe-C-Mn-Mo steel heated at 1 °C/s.
higher temperatures, which is related to the thermal expansion of the high temperature austenite phase. The onset of the second linear portion in the curve gives the austenite transformation finish temperature. The volume fraction of the austenite phase at intermediate temperatures can be determined using the lever-rule\textsuperscript{[113]}:

\[
V_y = \frac{A_O}{A_F} \quad (5-1)
\]

After the continuous heating austenite transformation tests were completed, the austenite transformation tests with an isothermal holding period were carried out. During the experiments, the samples were heated into the intercritical region at various heating rates (1, 10 and 100 °C/s) and held for a pre-determined time at selected temperatures. For microstructure analysis, the sample was quenched using helium gas after various holding times. For dilatometric analysis, the heating of the sample was resumed and the temperature raised to 1000 °C after the isothermal stage in order to complete the austenite transformation and obtain the FF' line for the data analysis. An example of the dilation-temperature relationship for the isothermal austenite transformation is given in Figure 5-2. In this case, when the intercritical holding temperature is reached, the austenite volume fraction is given by:

\[
V_y (1) = \frac{AO_1}{AF} \quad (5-2a)
\]

and at the end of holding at this temperature it has increased to:

\[
V_y (2) = \frac{AO_2}{AF} \quad (5-2b)
\]

The microstructures of all samples were also examined using optical microscopy. Samples were cut in the appropriate plane to be analyzed and then mounted, mechanically grounded and polished, with the final polishing step using 0.05 micron colloidal silica solution.
Figure 5-2 An example of the dilation-temperature relationship for the isothermal austenite transformation at 800 °C.
The etching method was the same as the one used in the recrystallization study as described in Section 4.1. This etching method reveals ferrite grain boundaries and tints the martensite darker than the ferrite matrix. The volume fraction of martensite was determined using a Clemex image analysis system, in which the martensite in the microstructure was traced out of the ferrite matrix and the area measured. The volume fraction of martensite can then be calculated by taking the ratio of martensite area vs. the total area in the micrograph. In the intercritically annealed samples, the volume fraction of martensite was assumed to represent the austenite volume fraction at the end of the isothermal hold.

5.2 Experimental Results

5.2.1 Continuous Heating Experiments

The results for the austenite formation kinetics under continuous heating condition (1, 10 and 100 °C/s) for the hot-rolled and cold-rolled Fe-C-Mn-Mo and cold-rolled Fe-C-Mn-Si steels are summarized in Figure 5-3. For comparison, the orthoequilibrium and paraequilibrium volume fraction of austenite (see Section 4.1 for details on this calculation) is shown in these graphs. For the hot-rolled Fe-C-Mn-Mo steel, the kinetics of austenite formation are similar for the cases of 1 and 10 °C/s heating rates, albeit the austenite fraction clearly trails the equilibrium fraction significantly. However, increasing the heating rate to 100 °C/s results in the expected behavior that the amount of austenite fraction at a given temperature is decreased.

For the cold-rolled Fe-C-Mn-Mo steel, an even more unusual result can be observed in Figure 5-3b. Here, there is essentially no difference in the austenite transformation start and
Figure 5-3  Austenite formation kinetics for the a) hot-rolled, b) cold-rolled Fe-C-Mn-Mo steel and c) cold-rolled Fe-C-Mn-Si steel under continuous heating conditions, and ortho-equilibrium (OE) and para-equilibrium (PE) austenite fractions.
finish temperature in the cold-rolled Fe-C-Mn-Mo steel for all the heating rates investigated (1, 10 and 100 °C/s). The austenite transformation rate at 100 °C/s heating rate in the cold-rolled Fe-C-Mn-Mo steel is increased significantly compared to the case of hot-rolled Fe-C-Mn-Mo steel.

Finally, for the cold-rolled Fe-C-Mn-Si steel, the austenite formation behavior under continuous heating condition is shown in Figure 5-3c. The results are qualitatively similar to the austenite formation behavior observed in the hot-rolled Fe-C-Mn-Mo steel (Figure 5-3a), i.e. the degree of austenite transformed at a given temperature is decreased for the highest employed heating rate (100 °C/s) only.

Table 5-1 summarizes the observed austenite transformation start and finish temperatures for the Fe-C-Mn-Mo and Fe-C-Mn-Si steels and those calculated from the Thermo-Calc software with FE-2000 data base, under orthoequilibrium condition.

<table>
<thead>
<tr>
<th>Material</th>
<th>$T_{Ac1}$ (Thermo-Calc)</th>
<th>$T_{Ac1}$ (1 °C/s)</th>
<th>$T_{Ac1}$ (10 °C/s)</th>
<th>$T_{Ac1}$ (100 °C/s)</th>
<th>$T_{Ac3}$ (Thermo-Calc)</th>
<th>$T_{Ac3}$ (1 °C/s)</th>
<th>$T_{Ac3}$ (10 °C/s)</th>
<th>$T_{Ac3}$ (100 °C/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-C-Mn-Mo</td>
<td>669</td>
<td>722</td>
<td>724</td>
<td>724</td>
<td>821</td>
<td>860</td>
<td>868</td>
<td>873</td>
</tr>
<tr>
<td>Fe-C-Mn-Si</td>
<td>603</td>
<td>651</td>
<td>651</td>
<td>777</td>
<td>848</td>
<td>920</td>
<td>920</td>
<td>948</td>
</tr>
</tbody>
</table>
5.2.2 Ramp and Hold Experiments

To examine the effect of heating rate in more detail, a series of annealing tests were conducted where the material was heated at different rates and then held isothermally at various temperatures in the intercritical region for 10 minutes. Figure 5-4 compares the results for austenite volume fraction obtained from dilatometry and quantitative metallography. The lever rule applied in the dilation data analysis (Equation 5-1 and 5-2) has a tendency to overestimate the austenite volume fraction transformed, since the volume change due to phase transformation is superimposed by the austenite volume change caused by carbon redistribution in the austenite phase. However, good agreement between the results from dilatometry and quantitative metallography observed in Figure 5-4 indicates that for the Fe-C-Mn-Mo steel, which is low in carbon content, the carbon redistribution during phase transformation has a minor effect on the dilation measurement. Therefore, the volume fraction of austenite from dilatometry can be used without correction. Repeating dilatometry experiments showed that the austenite volume fraction could be measured within ± 4%. Table 5-2 summarizes the results for austenite fraction after 10 minutes at 750 °C obtained from quantitative metallography and from the analysis of dilation data, which are found to be in good agreement with each other. For example, in the hot-rolled steel, the volume fraction of austenite was measured to be 0.19 and 0.21 for 1 °C/s and 0.27 and 0.29 for 100 °C/s by metallography and dilatometry measurements, respectively. Similar agreement was found for the cold-rolled samples.
Figure 5-4 Comparison of the austenite volume fraction for the Fe-C-Mn-Mo steel heated at 100 °C/s and then held at 750 °C from the dilatometry and metallurgy techniques. Solid lines represent the results from dilatometry and symbols represent the results from metallography.
Figure 5-5 compares the results from the analysis of dilatometry data for heating rates of 1 and 100 °C/s to an isothermal hold at 750 °C for the hot and cold-rolled Fe-C-Mn-Mo steel. It can be seen that for the hot-rolled material, the volume fraction of austenite phase increases with increased annealing time, as expected. However, the most significant observation that can be made is that for a given isothermal temperature, the samples with higher heating rates have a higher austenite volume fraction at a given annealing time. For example, at the beginning of the isothermal hold the fraction of austenite is 0.09 and 0.11 for the 1 and 100 °C/s heating rates, respectively, but these fractions increase to 0.21 and 0.29 after 10 minutes of holding at 750 °C. For the cold-rolled material, the effect of heating rate is even more significant. In this case, the fractions of austenite at the beginning of the isothermal hold are 0.12 and 0.14 for the 1 and 100 °C/s heating rates, respectively (i.e. slightly higher than for the hot-rolled material). At the end of the isothermal hold, these fractions increase to 0.17 and 0.34 for heating rates of 1 and 100 °C/s, respectively, i.e. the final fraction of austenite is twice as high for the higher heating rate even though the initial fractions are essentially the same.

Table 5-2  Austenite volume fraction for the Fe-C-Mn-Mo steel after 10 min at 750 °C.

<table>
<thead>
<tr>
<th>Heating rate (°C/s)</th>
<th>Austenite volume fraction</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Hot-rolled</td>
<td>Cold-rolled</td>
</tr>
<tr>
<td></td>
<td>Metallography Dilatometry</td>
<td>Metallography Dilatometry</td>
</tr>
<tr>
<td>1</td>
<td>0.19 ± 0.03 0.21 ± 0.04</td>
<td>0.17 ± 0.03 0.17 ± 0.04</td>
</tr>
<tr>
<td>100</td>
<td>0.27 ± 0.02 0.29 ± 0.04</td>
<td>0.34 ± 0.03 0.33 ± 0.04</td>
</tr>
</tbody>
</table>
Figure 5-5  Effect of initial heating rate on the isothermal austenite formation kinetics at 750 °C for hot-rolled and cold-rolled Fe-C-Mn-Mo steel.
The microstructure evolutions for the hot-rolled Fe-C-Mn-Mo steel annealed at 750 °C for 10 min with heating rates of 1 and 100 °C/s are illustrated in Figure 5-6 and Figure 5-7. The dark phase in these microstructure pictures is martensite, which was transformed from the high temperature austenite phase during the quench. Therefore, the austenite volume fraction and distribution at intercritical temperature are reflected in the observations of martensite at room temperature. It can be seen that for the slower heating rate (1 °C/s, Figure 5-6), the austenite grains are blocky in morphology, while for the higher heating rate (100 °C/s, Figure 5-7), the austenite grains are finer. Closer observations also suggest that more often ferrite grain boundary area is covered by the austenite phase for the 100 °C/s sample vs. 1 °C/s sample.

The austenite formation behaviour for the cold-rolled Fe-C-Mn-Mo steel during the intercritical annealing is also quite different from that for the hot-rolled material, as illustrated in Figure 5-8 and Figure 5-9. For the 1 °C/s heating rate case, the austenite forms a fine network covering most of the ferrite grain boundaries. In comparison, the 100 °C/s heating rate leads to a ‘banded’ austenite morphology resembling the original pearlite band structure in the as-cold-rolled material (see Figure 4-3b). There is virtually no other austenite formed in between the bands of the austenite phase.

To further appreciate the transformation behaviour in the cold-rolled steel, Figure 5-10 shows the isothermal austenite formation kinetics at 775 and 800 °C with initial heating rates of 1, 10 and 100 °C/s. Similar to the transformation data at 750 °C, the transformation in the isothermal stage is strongly affected by the heating rate. Increasing the heating rate increases the transformation rate at holding temperature. As expected, the volume fraction of austenite
phase increases with increased annealing temperature and time.

The effects of heating rate and temperature are similar for the cold-rolled Fe-C-Mn-Si steel as shown in Figure 5-11 for annealing at 750 °C and 785 °C with heating rates of 1 and 100 °C/s.

Figure 5-6 Microstructure evolution for the hot-rolled Fe-C-Mn-Mo steel heated at 1 °C/s and held at 750 °C.
Figure 5-7 Microstructure evolution for the hot-rolled Fe-C-Mn-Mo steel heated at 100 °C/s and held at 750 °C
Figure 5-8  Microstructure for the cold-rolled Fe-C-Mn-Mo steel reheated at 1 °C/s and held at 750 °C for various times.
Figure 5-9 Microstructure for the cold-rolled Fe-C-Mn-Mo steel reheated at 100 °C/s and held at 750 °C for various times up to 600 s.
Figure 5-10 Effect of initial heating rate on the isothermal austenite formation kinetics of cold-rolled Fe-C-Mn-Mo steel during holding at 775 °C and 800 °C, respectively.
Figure 5-11  Effect of initial heating rate on the isothermal austenite formation kinetics of the cold-rolled Fe-C-Mn-Si steel during holding at 750 °C and 785 °C, respectively.
5.2.3 Cooling from Intercritical Temperatures for the Fe-C-Mn-Mo Steel

Following intercritical annealing, the Fe-C-Mn-Mo steel is quenched to room temperature to produce ferrite-martensite microstructure. The dilatometric curves for the cold-rolled Fe-C-Mn-Mo steel cooled from temperatures above and below $T_{Ae}$ are presented in Figure 5-12. In Figure 5-12a, the holding temperature was 650 °C, which is below the $T_{Ae}$ temperature of 669 °C. A linear dilatometric curve for the cooling section was observed, which indicates that no austenite formed, as expected. In Figure 5-12b, where the holding temperature of 750 °C was higher than the $T_{Ae}$, the linear section for the heating side of the curve deviated at temperature above 722 °C, indicating austenite formation took place. While the linear trend for the cooling side of the curve remained till the temperature reached 329 °C. The similar observations can be made for the case of cooling from 775 °C. In this case, the cooling curve deviated from the linear trend at a temperature of 465 °C. The microstructures for the cold-rolled Fe-C-Mn-Mo steel after the 1 °C/s cooling following the 120 seconds holding stage at 650 °C, 750 °C and 775 °C are presented in Figure 5-13. In Figure 5-13a, the dark phase was identified as pearlite with some degree of spheroidization, while the light phase was ferrite. This confirms that no austenite formation has occurred under the heat treatment condition, as indicated previously by the dilatometry data (Figure 5-12a). In Figure 5-13b and Figure 5-13c, the light phase was identified as ferrite and the dark phase as martensite.
Figure 5-12 Dilation curves for the cold-rolled Fe-C-Mn-Mo steel cooled at 1 °C/s cooling rate after 2 min of holding at a) 650 °C, b) 750 °C and c) 775 °C.
Figure 5-13  Microstructures of the cold-rolled Fe-C-Mn-Mo steel after the 1 °C/s cooling to room temperature following the a) 650 °C, b) 750 and c) 775 °C / 120 s heat treatment.
5.3 Discussion

It has been shown in Section 4.3 that the ferrite recrystallization process may extend into the austenite formation region (Figure 4-14). However, this discussion was based on the equilibrium condition. In practice, the onset of austenite formation can be delayed with higher heating rate, so that the austenite transformation occurs at a temperature higher than the $T_{Ae1}$ (see Table 5-1). Figure 5-14 shows a comparison of the non-isothermal recrystallization kinetics derived using the model described in Section 4.3 and the temperature, $T_s$, at which austenite formation in ferrite was first experimentally observed as a function of heating rate (i.e. after the rapid pearlite to austenite reaction which corresponds e.g. to the first 11 % transformed in Fe-C-Mn-Mo steel). For the Fe-C-Mn-Mo steel, it can be observed that there is a significant overlap between ferrite recrystallization and austenite formation at higher heating rates. For example, at heating rates of 10 °C/s and 100 °C/s, the ferrite is 50 % and 90 % unrecrystallized, respectively, when austenite formation in ferrite is first observed. On the other hand, Figure 5-14b, shows that for the Fe-C-Mn-Si steel, ferrite recrystallization is completed before the austenite formation in ferrite for all investigated heating rates. Thus, for the Fe-C-Mn-Si steel heating rates in the order of 1000 °C/s appear to be required for substantial overlap between ferrite recrystallization and austenite formation consistent with the recent experimental results of Petrov et al.\textsuperscript{[141]} The fact that the overlap between recrystallization and austenite formation occurs at lower heating rates for the Fe-C-Mn-Mo steel is related to two observations, i) recrystallization is slower in this steel and ii) $T_s$ is lower. Both these effects can be attributed to the differences in chemistry of the two steels, i.e. i) Mo delays recrystallization and ii) Si is a ferrite stabilizer and thus increases $T_s$. 

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Figure 5-14 Effect of heating rate on the progressing of recrystallization with respect to the onset of austenite formation for the a) Fe-C-Mn-Mo and b) Fe-C-Mn-Si steel.
It might be expected that austenite formation behaviour may change when it occurs simultaneously with ferrite recrystallization as will be discussed below. However, the effect of heating rate on the subsequent isothermal transformation was also observed for hot-rolled material (Figure 5-5), i.e. in the absence of recrystallization. Thus, it appears that there is an important intrinsic effect of thermal history on austenite formation.

Figure 5-15 summarizes the volume fraction of austenite observed in the Fe-C-Mn-Mo steel after 1 and 10 minutes of holding at various temperatures with different heating rates being employed to reach the temperature. The experimental results are compared to ortho-equilibrium and para-equilibrium predictions from Thermo-Calc. The limited number of result from the hot-rolled steel annealed at 750 °C is also displayed in Figure 5-15, and shows the same trend as the data for the cold-rolled material. As the heating rate increases, a larger fraction of austenite is obtained after holding. All the other trends of the results shown in Figure 5-15 are consistent with expectations. The volume fraction of austenite increases with intercritical annealing temperature and holding time. The largest observed austenite fractions appear to coincide with ortho-equilibrium predictions. However, this cannot be taken as proof that redistribution of substitutional alloying elements has taken place. At the lower temperatures (<775 °C), ortho- and para-equilibrium yield similar austenite fractions. At higher temperatures, the austenite fraction in para-equilibrium is significantly larger than that in ortho-equilibrium. Further, using diffusion data reported for Mn\textsuperscript{[142]}, typical diffusion distances for a holding time of 10 minutes in the temperature range 750 – 800 °C are estimated to be 0.5 μm in ferrite and less than 0.05 μm in austenite. This estimate replicates the detailed austenite growth analysis by Wycliffe et al.\textsuperscript{[143]}. Their analysis predicts some Mn partitioning such that a non-
Figure 5-15 Summary of austenite fractions in the Fe-C-Mn-Mo steel for a) 1 min and b) 10 min of holding at various temperatures after different heating rates to holding temperature, and orthoequilibrium (OE) and paraequilibrium (PE) fractions.
uniform Mn distribution is established in relatively short times. However, much longer holding times in the order of hours would be required to obtain complete redistribution. Another work by the present author and co-workers on a 0.2 wt.% carbon steel have shown that this process can take more than 14 hours to complete[119].

To understand the effect of heating rate on the volume fraction and morphology of the austenite formed during the intercritical annealing, it is worthwhile to recall the basic phenomena of the austenite formation which is a diffusion controlled transformation. Classical nucleation and growth theory applies to this process. The transformation starts from the pearlite colonies by nucleation at the pearlite-ferrite interface followed by quick growth consuming the dissolving pearlite. Subsequently, austenite may nucleate at ferrite grain boundaries in competition with austenite growth from the prior pearlite areas. It is the delicate interplay between austenite formed at these different sites which appears to be responsible for the marked heating rate effect on the transformation kinetics. Slower heating rates will favour substantial growth of austenite nucleated at pearlite sites whereas faster heating rate will promote additional nucleation at ferrite grain boundaries. Re-examination of the microstructures shown in Figure 5-6 - Figure 5-9 appear to support this general trend for the hot-rolled (Figure 5-6b, Figure 5-7b) Fe-C-Mn-Mo steel, but not for the cold-rolled Fe-C-Mn-Mo steel (Figure 5-8e and Figure 5-9f).

Figure 5-16 provides schematics of the austenite distribution in the hot- and cold-rolled Fe-C-Mn-Mo steel after 600 seconds of isothermal holding at 750°C. In the hot-rolled material, large austenite islands are evident for 1°C/s heating rate but not for 100°C/s heating rate. The distribution of these islands replicates the distribution of pearlite in the as-hot-rolled
Figure 5-16 Schematic illustration for the microstructure evolution of cold-rolled Fe-C-Mn-Mo steel annealed with different heating rate.
material and confirms substantial growth of pearlite nucleated austenite. However, for the cold-rolled material much larger austenite islands which coincide with the as-received pearlite distribution are recorded for 100°C/s heating rate than for 1°C/s heating rate. Moreover, there is an absence of austenite formed at ferrite grain boundaries for the high heating rate case in the cold-rolled steel. Austenite nucleation at ferrite grain boundaries is observed in all other cases. For the slow heating rate, grain boundary and pearlite nucleated austenite remain distinct and compete during growth. This competition contributes to the comparatively slow transformation rates during holding. For the high heating rate case in the hot-rolled material, the extent of austenite nucleation at ferrite grain boundaries is such that an almost complete network of austenite along the boundaries connecting to the pearlite nucleated austenite emerges. Thus, there is no growth competition between pearlite and grain boundary nucleated austenite thereby promoting larger transformation rates at holding temperature.

Similarly, there is no growth competition in the high heating rate case for the cold-rolled material. However, the mechanisms here are quite different since ferrite recrystallization occurs simultaneously with austenite formation. The sequence of micrographs of Figure 5-9 a), b) and c) clearly confirms that ferrite still recrystallizes when austenite has nucleated in the pearlite colonies and starts to grow from there. The moving ferrite grain boundaries do not provide suitable nucleation sites for austenite. Thus, there are only very few austenite particles located at the ferrite grain boundaries. The pearlite nucleated austenite can then grow without significant competition from grain boundary austenite. This growth is apparently fast enough to eliminate the driving pressure required for austenite nucleation at ferrite grain boundaries before the completion of ferrite recrystallization. As a result, a block-like austenite distribution
forms parallel to the rolling direction with the largest transformation rate of the four considered cases.

This interpretation appears to be in stark contrast to that of Yang et al. [60] who also found austenite bands parallel to the rolling direction but attributed them to austenite nucleation primarily occurring on ferrite grain boundaries before completion of recrystallization. Closer inspection of their results may suggest that these locations coincide with the presence of spheroidized cementite colonies along the boundaries of the elongated ferrite grains, i.e. like proposed here, preferred nucleation at the pearlite-ferrite and cementite-ferrite interfaces, respectively, appears to occur.

The same principles explain the unusual austenite formation behavior under continuous heating conditions (Figure 5-3). In the hot-rolled material, increasing the heating rate leads to an enhanced austenite nucleation at ferrite boundaries. Associated is a gradual decrease of growth competition between pearlite and boundary nucleated austenite until a network connecting the different types of austenite is formed, i.e. the growth geometry changes for intermediate heating rates. As a result, the temperature dependence of the fraction transformation is similar for 1 and 10 °C/s heating rates and only for even higher heating rates the transformation is delayed to higher temperatures, as expected from a diffusional transformation with a given growth geometry. The interaction of recrystallization and austenite formation in the cold-rolled Fe-C-Mn-Mo steel for higher heating rates (>10 °C/s) extends the apparent heating rate independence of the transformed austenite fraction to even higher heating rates, i.e. 100 °C/s.

The latter is not observed for the cold-rolled Fe-C-Mn-Si steel. As indicated, in Figure
5-14b, ferrite recrystallization is sufficiently fast that no overlap with austenite formation occurs at the highest investigated heating rate of 100 °C/s. Consequently, the Fe-C-Mn-Si steel displays in the investigated heating rate range trends in terms of austenite formation similar to those of the hot-rolled Fe-C-Mn-Mo steel.

The discussion so far has focused on the ferrite to austenite transformation. However, there are also important implications of the heat treatment path on the pearlite to austenite formation transformation which precedes the ferrite to austenite transformation. This can be seen for the case of the Fe-C-Mn-Si steel intercritically annealed at 750 °C where even when initially the austenite fraction remains below the pearlite fraction of 25 %, a clear heating rate dependence is found. It can be expected that slower heating rates promote spheroidization of pearlite. The early work of Roberts and Mehl \[62\] found that spheroidized pearlite transforms to austenite more slowly than unspheroidized pearlite which is consistent with the results found in the current work (Figure 5-11).

The final point of interest is to consider the possible decomposition of austenite during cooling after intercritical annealing. For the Fe-C-Mn-Mo steel, in order to obtain the ferrite-martensite microstructure, a critical cooling rate has to be applied when cooling the material from the intercritical region to avoid the pearlite/bainite formation. The results shown in Figure 5-12 suggest that the critical cooling rate for the Fe-C-Mn-Mo steel considered in this work is less than 1 °C/s. In Figure 5-12b, a small amount (15 %, see Figure 5-5) of austenite phase is formed in the Fe-C-Mn-Mo steel heated at a rate of 1 °C/s and held at 750 °C for 2 min. Therefore the carbon concentration in the austenite phase is relatively high, assuming that all carbon is in the austenite phase. Calculated from the mass balance, the carbon concentration is
0.4 wt.%, resulting in a $M_s$ temperature of 313 °C (from the Andrew’s equation 2-5). This is consistent with the observation that the cooling curve in Figure 5-12b deviated from the linear trend at 329 °C. The microstructure obtained from the 1 °C/s cooling consists only of ferrite and martensite (Figure 5-13b), which confirms that the critical cooling rate is less than 1 °C/s for the austenitization condition considered. For the case of 775 °C, the amount of austenite formed after the 2 min hold with an initial heating rate of 1 °C/s is larger, thus the carbon concentration is diluted. As a result, the $M_s$ temperature increases, which is consistent with the observation that for a cooling rate of 1 °C/s, the dilation curve deviates earlier from the linear trend (Figure 5-12c). The ferrite-martensite microstructure (Figure 5-13c) obtained under this condition indicates that 1 °C/s cooling rate is enough to produce a dual phase microstructure.

5.4 Summary

The austenite formation kinetics and resulting austenite morphology and distribution are strongly affected by the heating rate employed to reach the soaking temperature during intercritical annealing. Higher heating rates lead to faster austenite formation rates at the holding temperature. For cold-rolled materials, an overlap of ferrite recrystallization and austenite formation occurs at sufficiently high heating rates resulting in a banded austenite morphology.

The interaction of recrystallization and austenite formation needs further investigation. It appears to be an additional metallurgical tool to design tailored microstructures. In particular, it may contribute to refine microstructures. This will be of particular interest for ultra fast heating routes. However, as the present study suggest, this interaction may occur at moderate heating rates similar to those which can be employed in conventional continuous annealing.
lines when the steel contains alloying elements such as Mo, Nb or B which are known to delay recrystallization substantially.
Chapter 6  Study of Bainite Formation in a TRIP Steel  using Neutron Bragg-edge Transmission (BET)  Technique

The final stage in the processing of TRIP steel is the low temperature bainite transformation stage, in which austenite transforms to bainite. As the carbide precipitation in bainite is inhibited by alloying elements such as Si or Al in the steel, carbon is rejected into the remaining austenite phase. The carbon enrichment stabilizes the austenite phase, since carbon has the strongest effect, among other alloying elements in steel, in decreasing the \( M_s \) temperature. The carbon enriched austenite phase can survive the final cooling to room temperature without transforming to martensite, if the \( M_s \) temperature is below room temperature. The retained austenite in the steel contributes to the TRIP effect, thus gives the material a good combination of strength and ductility.

With the understanding that carbon plays an important role in retaining austenite in TRIP steel, therefore, it is of interest to investigate the carbon redistribution during the low temperature bainite transformation process. Traditionally, metallography, X-ray diffraction and dilatometry techniques are used to characterize the structure during the transformation\[^{144}\]. However, they either are off-line techniques, or give volume fraction information only. In this Chapter, the application of a new technique, the neutron Bragg-edge Transmission (BET) technique for the study of bainite transformation in a TRIP steel with a model chemistry will be described. It offers on-line, \textit{in-situ} information about the phase transformation kinetics and
unique information on carbon concentration in the participating phases (austenite and bainite in this case).

### 6.1 Introduction to the neutron Bragg-edge Transmission (BET) technique

Neutron BET technique is closely related to the neutron diffraction technique i.e. both share the same operational principle: Bragg’s law, $\lambda = 2d_{hkl} \sin \theta$\[^{[116]}\], where $\lambda$ is the wavelength of the incident beam, $d_{hkl}$ the lattice spacing for the crystal plane \{h k l\} and $\theta$ the diffraction angle. When an incident neutron beam with appropriate energy spectrum enters a crystalline sample, it can be split into two parts: the diffracted beam and the transmitted beam. In the diffraction technique, the information carrier is the diffracted neutron, which has a characteristic energy distribution. Since the neutron absorption cross-section is small (neutron reacts with the nuclei of the atom, not the outer shells of electron due to the difference in mass between neutron and electron), the diffraction events are relatively sparse. The majority of neutron in the incident beam travel through the sample volume without being diffracted. In addition, a regular diffraction detector normally covers only a fraction of the diffraction space. Therefore, the collected intensity for the diffracted neutron beam is low, which translates to longer data acquisition times. Furthermore, rigid diffraction geometry has to be observed in neutron diffraction setup, restricting the flexibility in manipulating the sample environment.

While neutrons of specific energy levels in the incident beam are diffracted by the sample (which forms the characteristic diffraction spectrum), the remaining (and majority) pass through the sample and form the transmission spectrum, which also has a characteristic energy distribution. Therefore, the transmitted neutron can also serve as information carrier for the
structure of the material. Since the intensity of the transmitted neutron beam is higher than of the diffracted beam, higher time resolution can be achieved with the BET technique, compared to the conventional diffraction technique. In a neutron BET experiment setup, the sample is placed in between the neutron source and the transmission detector. A schematic of the neutron BET setup is illustrated in Figure 6-1. In contrast to a diffraction experiment setup, neutron source, sample and detector are located on a common axis.

![Figure 6-1 Schematic of the neutron BET transmission setup. Neutron beam travels from left to right.](image)

Due to the wave properties of neutrons, Bragg's law[^116] is applicable when neutrons interact with crystalline solids. From this law it becomes obvious that no radiation of a wavelength greater than \(2d_{hkl}\) can be diffracted by a particular set of lattice planes \(\{hkl\}\). Therefore, the transmitted intensity rises abruptly at \(\lambda = 2d_{hkl}\), forming a so-called Bragg-edge in the transmission spectrum at that wavelength. The set of lattice planes \(hkl\) is excluded from the elastic coherent scattering for neutrons of greater wavelength. The wavelength of this abrupt rise immediately allows one to measure d-spacings, from which the lattice parameter can be derived. Using polychromatic neutrons of a pulsed neutron source allows to measure the
neutron wavelength conveniently from its time-of-flight. Therefore, no time-consuming wavelength analysis is necessary.

The absorption of neutrons in most materials is low enough that a large fraction of the incident intensity travels through a sample of a thickness of several centimeters without any interaction. Compared for example to TEM or x-ray techniques, this allows one to probe relatively large sample volumes in the order of cubic centimeters. The height and width of a Bragg-edge follow the same laws as are valid for the intensity and width of a diffraction peak. Accordingly, a transmission spectrum contains in principle the same information as a conventional diffraction pattern, i.e. crystallographic structure, lattice parameter, volume fraction, etc. In practice, the general application of the Bragg-edge transmission technique is limited by the fact that small changes (edge-heights) of a large signal (transmitted intensity) have to be measured. This restricts Bragg-edge transmission experiments to systems of high symmetry which consist of nuclei with a high scattering length and low absorption cross-section. Such system will produce higher Bragg edges for analysis at a given incident neutron beam. This requirement is perfectly met by steels.

When combined with a novel remotely controlled sample maneuvering system (SMS) developed in this work, the Bragg-edge transmission technique allows one to investigate the phase transformations of steel during multi-stage heat treatment. The SMS moves the sample between two furnaces setting at different heat treatment temperatures, for example, one at the austenitization temperature and another at the bainite formation temperature, while the neutron beam penetrates the sample. This setup allows for the acquisition of crystallographic information on bulk steel samples during a full heat treatment cycle. Allowing such motion of
the sample along the neutron beam path without changing the resulting signal is a unique capability of the neutron BET technique.

The Bragg-edge transmission technique allows splitting the cylindrical sample into several thin disks, further increasing the possible rate of temperature changes. The rapid temperature change potential with such a setup is hardly achievable with a bulk sample on a diffraction setup. For neutrons, the required large bulk sample volumes make it difficult to homogeneously change the temperature rapidly. Synchrotron radiation allows smaller sample volumes, hence fast temperature change, but one might see only a few grains due to the small beam and therefore kinetic information obtained in such conditions might be not representative for bulk material.

6.2 Experimental

6.2.1 Neutron Facility

Neutron BET experiments were conducted at flightpath 5 of the Los Alamos Neutron Science Centre (LANSCE)\textsuperscript{[145, 146]} in the Los Alamos National Laboratory. LANSCE is a pulsed neutron source in which high flux neutrons are produced in a spallation process\textsuperscript{[147]}. The neutron pulse rate was 20 Hz. The flightpath consists of an evacuated beam tube between moderator and the shielded experimental room, followed by a second evacuated beam tube leading to the detector station. A beam dump is located behind the detector system.

The first beam tube starts close to the high-flux water moderator and has a length of 6.73 m. In direction of the neutron beam, a changeable collimator made of polyethylene and steel with holes of converging diameter is installed in the tube to collimate the incident neutron
beam. For the experiments presented here, an exit diameter of 40 mm was installed. The second beam tube has a diameter of 74.3 cm and runs over a length of 45.7 m to the detector station. Both tubes are made of steel and covered at both ends with aluminum plates. Holes covered with thinner aluminum sheets are drilled into the plates where the highest neutron intensity is expected (‘neutron windows’). The evacuation of both tubes (less than 50 Pa, equivalent to less than 0.1 % of local atmospheric pressure) prevents the severe intensity losses of approximately 5% per meter in air. For the total flightpath, the distance between moderator and detector was 58.56 m. The shielded experimental room, the ‘cave’, has a dimension of 2(H) x 2(W) x 6(L) m. The experimental setup is accommodated in the cave.

6.2.2 Experimental Setup for the Neutron BET Experiments

In order to simulate the thermal cycle applied to the bainite transformation, two tube furnaces were employed, aligning up with the neutron beam. Figure 6-2 illustrates schematically the layout of the experimental setup. The first tube furnace (105 cm in length) was set at austenitization temperature and the second one (60 cm in length) down the neutron beam was set at bainite transformation temperature. A gas quench unit and a remotely controlled sample maneuvering system (SMS) were designed and prepared. The gas quench unit was made of a 6.35 mm square spray nozzle, a $\phi 76.2 \times 150$ mm guiding funnel, solenoid control valve and tubing. Helium gas was supplied by a compressed gas cylinder. The gas pressure was regulated at 275 kPa (40 psi) during quench. The sample maneuvering system moves the sample assembly between the furnaces and the quench unit at a speed of 12 cm/s. The furnaces, quench unit and the sample maneuvering system were lined up with the help of a
laser beam so that the centre region of the sample was covered by the neutron beam (40 mm in diameter).

Figure 6-2 Schematic illustration of the experimental set-up used in the neutron BET experiment.

6.2.3 Test Material and Sample Assembly

The model steel has a nominal chemistry of 0.4C - 3Mn - 2Si (in wt.%). It was chosen as previous data by Bhadeshia\cite{148} has shown that the material has a slow bainite reaction rate (ideal for testing the technique) and a TRIP microstructure can be produced. The material came in two batches for two experimental runs. The first batch of material was exhausted during the first experimental cycle in year 2001. Then a second batch was prepared for the second run cycle in year 2002. The chemical composition of the material is shown in Table 6-1 (note the slight variation in the chemistry for the two batches of material).
Table 6-1 Chemical composition of the model steel (wt. %).

<table>
<thead>
<tr>
<th>Steel</th>
<th>C</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Si</th>
<th>Cu</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>V</th>
<th>Nb</th>
<th>N</th>
<th>B</th>
<th>Ti</th>
<th>ASA*</th>
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</thead>
<tbody>
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<td>3.08</td>
<td>.008</td>
<td>.001</td>
<td>2.19</td>
<td>.006</td>
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<td>.002</td>
<td>.002</td>
<td>.004</td>
<td>.002</td>
<td>.004</td>
<td>.002</td>
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<tr>
<td>Batch B</td>
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<td>.016</td>
<td>.002</td>
<td>1.95</td>
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<td>.002</td>
<td>.002</td>
<td>.005</td>
<td>.002</td>
<td>.005</td>
<td>.042</td>
<td></td>
</tr>
</tbody>
</table>

ASA: Acid Soluble Aluminum.

The material was casted into a 80 kg ingot, then hot-rolled to 60mm wide, 1.6 mm thick stripes. After removing the surface oxidation layers formed during hot-rolling, the material was machined into 55 mm diameter disks of approximately 1 mm thickness, then assembled to produce samples for the neutron BET experiment of 15 disks. Figure 6-3 shows schematically the sample assembly. Two stainless steel rings connected by bolts form a frame. Sample disks are held in place by the bolts and separated by 1.5 mm thick stainless steel washers. The sample assembly was designed in a way that the sample disks are not constrained rigidly by the frame so that no thermal stress should develop during the applied thermal cycle.

Figure 6-3 Schematic illustration of the sample assembly used for the neutron BET experiment. The length of the sample assembly between the supporting rings is 60 mm.
The two holding rings had a 45 mm diameter clearance in the centre for 40 mm diameter collimated neutron beam. A run with the frame without steel disks showed no Bragg-edges from the frame material. For temperature recording during the experiment, a type K thermocouple was spot-welded on the centre of the centre disk in the sample assembly.

To examine the homogeneity of sample disk temperature in the assembly, extra type K thermocouples were also attached to the edges of the centre plate, as well as in the centre of the plates towards the neutron beam source and the detector for selected experiment runs. The results are given in Table 6-2.

<table>
<thead>
<tr>
<th>Furnace Temperature</th>
<th>Disk Upper Edge Temperature</th>
<th>Disk Lower Edge Temperature</th>
<th>Disk Centre Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000 °C</td>
<td>1001 °C</td>
<td>995 °C</td>
<td>1002 °C</td>
</tr>
<tr>
<td>350 °C</td>
<td>353 °C</td>
<td>355 °C</td>
<td>347 °C</td>
</tr>
<tr>
<td>330 °C</td>
<td>334 °C</td>
<td>338 °C</td>
<td>338 °C</td>
</tr>
</tbody>
</table>

The greatest difference observed was 7 °C between the top and bottom edge of the centre disk at a sample temperature of 1000 °C. Therefore, the temperature in the sample disks was considered to be sufficiently homogeneous.
6.2.4 Neutron BET Experiment Procedures

To conduct an experimental run, the sample assembly was loaded in the centre of the austenitization furnace with 1.5 l/min helium gas flow to avoid oxidization at austenitization temperature. Then the neutron cave was swept and neutron shutter was opened to allow the neutron beam going through the sample disks. The sample temperature was raised to the austenitization temperature and held for 30 minutes. Then the sample assembly was pulled into the quench unit where it is cooled rapidly by the helium gas flow. Quench rates up to 16 °C/s were achieved with this setup, while the minimum quench rate was 6 °C/s. When the target bainite temperature was reached, the sample assembly was pulled away from the quench unit and into the second tube furnace, which had been preheated and held at bainite transformation temperature. The sample stayed at the bainite reaction temperature for up to 10 hours before it was finally pulled out and quenched to room temperature.

The thermal cycles applied to the material are summarized in Table 6-3. An example of the recorded experimental thermal cycle is given in Figure 6-4. For 1000 °C/335 °C and 1000 °C/400 °C run cycles, which were carried out in year 2001, materials from batch A were used. For all other run cycles, which were conducted in year 2002, materials from batch B were used.

The neutron BET patterns were taken at 5 seconds intervals. It took another 7 seconds for the computer data acquisition software to store the data. Hence a total of 12 seconds is required for a single data acquisition cycle. Neutron patterns were recorded throughout the bainite transformation processes. For a single experimental run, as a result of the short data acquisition time, the number of neutron BET patterns generated ranges from
Table 6-3 Heat treatment parameters for the material.

<table>
<thead>
<tr>
<th>Austenitization Condition</th>
<th>Bainite Transformation Temperature °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>1000 °C /30 min.</td>
<td>283</td>
</tr>
<tr>
<td></td>
<td>335</td>
</tr>
<tr>
<td></td>
<td>355</td>
</tr>
<tr>
<td></td>
<td>400</td>
</tr>
<tr>
<td></td>
<td>405</td>
</tr>
<tr>
<td></td>
<td>436</td>
</tr>
</tbody>
</table>

Figure 6-4 An example of the thermal cycle applied to the steel.
1900 to 2700 patterns, depending on the time length of the isothermal bainite transformation holding, which ranges from 4 to 10 hours in this study. The data analysis is based on a full pattern fitting against a model, taking the Rietveld approach\textsuperscript{[149]}. Phase volume fraction information can be extracted from the Bragg edge height parameter, lattice parameter information can be extracted from the edge position, and edge width parameter can be obtained from the edge profile. The details can be found in ref. [128].

6.2.5 Related XRD, Metallography and Dilatometry Experiments

After the heat treatment cycle, selected samples were measured using XRD at room temperature in a Siemens D5000 diffractometer to obtain the phase volume fraction in order to compare with the neutron BET results. The XRD sample of 1.25 cm\textsuperscript{2} was cut out and polished down with the final step using 1 μm diamond. The experimental parameters for the XRD step scan were as follows: Cu kα radiation, 40 kv, 30 mA, 2θ scanning range: 40° to 85°, step size: 0.01 ° and dwell time 1 s. The XRD data was analyzed taking the Rietveld approach\textsuperscript{[149]} using the GSAS (General Structural Analysis System) program\textsuperscript{[150]}. Austenite volume fraction can then be extracted from the data analysis.

Microstructure of the sample was also characterized using conventional optical metallography. The sample was mounted and mechanically grounded, followed by 6 μm and 1 μm diamond polishing, finished with 0.05 μm colloidal silica polishing. The etching was carried out by swabbing the sample with 2 % Nital, rinsing, followed by immersing the sample in 10 % sodium meta-bisulfite aqueous solution. Quantitative volume fraction analyses were done on selected samples using the Clemex imaging analysis system.
To verify that no phase transformation occurs during the intermediate cooling from the austenitization temperature to the bainite transformation temperature, dilatometric experiments were carried out on a Gleeble 1500 thermo-mechanical simulation system. A steel sample of 60 × 12 × 1.2 mm³ in the as-received condition was given the same austenitization treatment as the neutron BET runs, i.e. 1000 °C/30 min, using a resistance heating mechanism. Then a controlled cooling was applied to the sample with a cooling rate of 6 °C/s, which was the minimum cooling rate recorded during the neutron BET runs. Dilatometry and temperature data were acquired for the Gleeble simulation run.

One of the neutron BET experiment runs (1000 °C/335°C) was also simulated on the Gleeble system, in which an as-received steel sample of 60 × 12 × 1.2 mm³ was heat treated at 1000 °C for 30 min., the cooled at 10 °C/s to 335 °C, followed by isothermal holding for 1 hour. Dilation data was recorded during the experiment.

6.3 Results

Examining the as-received material using optical microscope revealed a microstructure consisting of pearlite and martensite (Figure 6-5). The starting microstructure will be replaced by a single austenite phase microstructure with an austenite grain size of ~110 μm during the austenitization process in the neutron BET experiments.

Before going into the detailed results from the neutron BET experiments, the result from the Gleeble cooling simulation is presented. This was done to verify that no ferrite or pearlite transformation should form during the cooling from the austenitization temperature.
Figure 6-5 Microstructure of the steel in the as-received state showing a mixture of pearlite and martensite microstructure.
Figure 6-6 Dilatometry curve for the steel heated at 10 °C/s and held at 1000 °C for 30 min., then cooled at 6 °C/s.
to the bainite transformation temperature under the cooling conditions of the neutron BET runs. The dilation vs. temperature diagram is shown in Figure 6-6. It can be seen that at 6 °C/s cooling rate, which was the minimum cooling rate recorded for the neutron BET runs, the dilatometric behaviour of the material follows a linear manner until the temperature is lower than 280 °C. This means that the cooling capacity of the quench unit used for the neutron BET experiments is sufficient to suppress any ferrite or pearlite formation during cooling of the investigated material.

The following section describes the results from the neutron BET experiments. Figure 6-7 presents a set of snapshots for the neutron BET patterns acquired during an experimental run, in which a series of characteristic Bragg-edges can be observed. Note that the height of the austenite edges is decreasing while height of the bainite edges is increasing with increasing isothermal transformation time.

The patterns were analyzed in a batch process\textsuperscript{[151].} Figure 6-8 shows an example of the analysis, with details of the neutron Bragg edges shown in the inset. In this neutron BET pattern, edges from a FCC phase and a BCC phase can be identified and indexed. For example, the inset in Figure 6-8 shows a FCC (220) and BCC (211) edge being indexed. In this case, the FCC edges are associated with the austenite phase and the BCC edges are associated with the bainitic ferrite phase. The small amplitude in the difference curve indicates that the pattern is well described by model. The lattice parameter can be determined using the information from the position of the Bragg edges and volume fraction from the height of the Bragg edges.
Figure 6-7 Neutron BET patterns acquired for a steel sample isothermally transformed at a bainite transformation temperature of 335 °C, after 1000 °C/30 min. austenitization treatment. The lower row of marker for the austenite edge, the upper row for the bainite edge.
Figure 6-8 Example of neutron BET pattern analysis. The bottom curve represents the difference between the pattern and analysis.
The volume fraction change as a function of the isothermal bainite transformation time is shown in Figure 6-9 to Figure 6-13. Note the gaps in Figure 6-9 and Figure 6-11 were due to neutron beam failure during the experimental runs, thus no data was available for that period. From these figures (except Figure 6-13), it can be seen that the austenite volume fraction decreases rapidly in the first 1 to 2 hours of isothermal bainite transformation time and then the transformation rate decreases as the transformation continues. For example, for the 335 °C bainite transformation (Figure 6-10), the volume fraction of austenite of initially 100% decreases continuously throughout the transformation to a final value of 30.5 ± 0.3 %. The ± 0.3 % is a typical value observed from the analysis for the uncertainty in the volume fraction measurement in the neutron BET experiment. The transformation rate slows down with increasing transformation time. A value of 50% was reached after about 28 minutes of transformation, after one hour the austenite volume fraction decreased to 36%.

The transformation at 436 °C is different from other investigated temperatures. No transformation occurs throughout the holding period, as seen in Figure 6-13, which shows a constant austenite volume fraction of 100 %.

For the transformation at 405 °C, an additional experiment was conducted to examine repeatability. The results given in Figure 6-14 show that excellent repeatability was observed.
Figure 6-9 Volume fraction evolutions for the austenite and bainite during the 283 °C isothermal holding.
Figure 6-10 Volume fraction evolutions for the austenite and bainite during the 335 °C isothermal holding.
Figure 6-11 Volume fraction evolutions for the austenite and bainite during the 355 °C isothermal holding.
Figure 6-12 Volume fraction evolutions for the austenite and bainite during the 405 °C isothermal holding.
Figure 6-13 Volume fraction evolutions for the austenite and bainite during the 436 °C isothermal holding.
Figure 6-14 Comparison of the results from two independent experimental runs – bainite isothermal temperature 405 °C.
The information about the changes in lattice parameter during the thermal cycles is presented in Figure 6-15 to Figure 6-19. The following observations can be made:

1. At lower bainite transformation temperatures (283 and 335 °C), there is a large increase in the austenite lattice parameter at the early stage of isothermal holding period.

2. At higher bainite transformation temperatures (355 and 405 °C), the increase in the austenite lattice parameter becomes less pronounced.

3. At 436 °C of isothermal holding, there is virtually no change in the austenite lattice parameter. At this temperature, the BCC phase is not present, as indicated by the volume fraction analysis; therefore, there is no information about the bainite lattice parameter.

4. The change of BCC lattice parameter is less significant compared to the change of austenite lattice parameter.

5. It is noteworthy that there is a small hump in the BCC lattice parameter curve at the initial stage of the 283 °C and 335 °C isothermal holding.

Figure 6-20 presents the neutron BET edge width parameter $\sigma_1$ for austenite phase during the bainite transformations at various temperatures. The edge width concept which is equivalent to the peak width in a XRD spectrum can be illustrated schematically in Figure 6-21. For an ideal crystal, there is no variation in the lattice parameter, therefore, the Bragg edge is vertical, shown as solid line in Figure 6-21. However, in the real world, the variation in the lattice parameter can arise due to strain, inhomogeneity in chemical composition and dislocations, etc. This causes the vertical Bragg edge to diffuse out, as shown in Figure 6-21.
Figure 6-15 Changes in the austenite and bainite lattice parameters during the isothermal bainite transformation at 283 °C.
Figure 6-16 Changes in the austenite and bainite lattice parameters during the isothermal bainite transformation at 335 °C.
Figure 6-17 Changes in the austenite and bainite lattice parameters during the isothermal bainite transformation at 355 °C.
Figure 6-18 Changes in the austenite and bainite lattice parameters during the isothermal bainite transformation at 405 °C.
Figure 6-19 Changes in the austenite lattice parameter during the isothermal bainite transformation at 436 °C.
Figure 6-20 Edge width parameter for austenite phase during the bainite transformation at temperatures of a) 283 °C, b) 335 °C, c) 355 °C, d) 405 °C and e) 436 °C.
Figure 6-21  Schematic illustration for the edge width parameter. Solid line for ideal iron α (211) Bragg edge, dots for the real α (211) edge.
Instrument factor can also contribute to the edge broadening. The edge width parameter $\sigma_1$ describes the broadening due to the instrument and the inhomogeneity in the lattice parameter of the sample material. It can be seen that the edge width parameter for austenite varies with the bainite transformation temperatures. For example, for the lower bainite transformation temperature (Figure 6-20a, 283°C), there is a large variation in the edge width parameter. As the transformation temperature increases, the variation in the edge width parameter for austenite decreases.

The microstructure of the steel at the end of the 283°C isothermal bainite transformation is illustrated in Figure 6-22. For the lower bainite transformation temperature (283°C), the final structure consists of fine bainite packets with retained austenite in between. The XRD measurements were made to characterize the retained austenite in the samples at the end of the test. The XRD spectrum is shown in Figure 6-23, in which the dots represent the experimental results, the solid line is the fitted model from the Rietveld analysis. The bottom curve represents the difference between the experimental data and the model, below which the first row of the edge marker corresponds to the austenite edge and the second row for the ferrite edge. It can be seen that the XRD spectrum is well described by the Rietveld model, which in turn gives reliable results for the volume fraction analysis. Room temperature XRD measurements give a retained austenite volume fraction of 19%, which is in good agreement with the neutron BET result (20%, see Figure 6-9).

The microstructure of the steel at the end of the 8 hour bainite transformation at 335°C is presented in Figure 6-24. It can be seen that the microstructure consists of bainite
Figure 6-22 Microstructure of the steel after 9 hours of isothermal bainite transformation at 283 °C showing fine bainite packets (B) and retained austenite (RA).
Figure 6-23 Room temperature XRD spectrum for the steel heated and held at 1000 °C, then rapidly cooled to 283 °C and held for 9 hours, followed by quenching to room temperature.
packets, a small amount of martensite (gray islands surrounded by white area in the micrograph) and retained austenite (white area) after the thermal cycle. The presence of the retained austenite phase is confirmed by the XRD measurement. Figure 6-25 gives the room temperature XRD spectrum for the sample after the heat treatment. The same type of legend is used in this diagram as in Figure 6-23. XRD phase volume fraction analysis from the Rietveld approach gives the austenite volume fraction of 25 \%, which is less than the quantitative metallography result of 28 \pm 3 \% and the neutron BET result of 30 \% (see Figure 6-10). This discrepancy will be discussed later.

For the 405 °C bainite transformation temperature, the microstructure of the material at the end of the heat treatment figures a course bainitic structure in the martensite matrix which was transformed from austenite during cooling to room temperature, as can be seen in Figure 6-26. The XRD spectrum (Figure 6-27) shows only a trace of the austenite (200) peak. The volume fraction analysis from the XRD measurement gives an austenite volume fraction of less than 1 \%, whereas the volume fraction from the neutron BET result is 75 \%, while the quantitative metallography gives a austenite volume fraction of 75 ± 2 \%. Again this discrepancy will be discussed in the later section.

Figure 6-28 shows the microstructure of the material after the Gleeble simulation heat treatment cycle of 1000 °C/335 °C – 1 hr. The microstructure feature packets of bainite, with martensite (dark gray area in the background) dotted with retained austenite phase (white area). Quantitative metallographic analysis gives a bainite volume fraction of 61 ± 2 \%, while the corresponding neutron BET result for the bainite volume fraction is 64 \%.
Figure 6-24 Microstructure of the steel after 8 hours of isothermal bainite transformation at 335 °C showing fine bainite packets, martensite (M) and retained austenite (RA).
Figure 6-25 Room temperature XRD spectrum for the steel heated and held at 1000 °C, then cooled to 335 °C and held for 8 hours, followed by quenching to room temperature.
Figure 6-26 Microstructure of the steel after 6 hours of isothermal bainite transformation at 405 °C showing fine bainite packets and retained austenite.
Figure 6-27 Room temperature XRD spectrum for the steel heated and held at 1000 °C, then rapidly cooled to 405 °C and held for 6 hours, followed by quenching to room temperature.
Figure 6-28  Microstructure of the material after Gleeble simulation heat treatment cycle of 1000 °C/335 °C – 1 hr. RA: retained austenite; M: martensite.
Figure 6-29 shows a comparison of the bainite transformation kinetics at 335 °C measured from the neutron BET and dilatometry techniques. The dilation data was analysis using the lever rule and the bainite volume fraction at the end of the 1-hour transformation time was forced to the value of 61 % (obtained from the quantitative metallographic measurement, i.e. 61 ± 2%). It can be seen that the result from dilatometry agrees well with the result from neutron BET in the first and last 15 min of transformation time during the first hour of transformation. However, the bainite volume fraction measured from dilatometry is higher than that from neutron BET from 15 to 45 min of transformation. In fact, the volume change of the sample measured by the dilatometry is caused not only by the volume change due to the phase transformation, but also by the carbon redistribution in the austenite phase, which can not be ignored for the steel of high carbon content (i.e. 0.4 wt.%). As a result, the dilatometry technique tends to overestimate the volume fraction transformed.

Finally, Table 6-4 summaries the phase volume fraction measurement at different bainite transformation conditions from the neutron BET, XRD and metallographic techniques.

<table>
<thead>
<tr>
<th>Bainite transformation condition</th>
<th>Neutron BET %</th>
<th>XRD %</th>
<th>Metallography %</th>
</tr>
</thead>
<tbody>
<tr>
<td>283 °C/9 hr.</td>
<td>20 ± 0.3 (austenite)</td>
<td>19 ± 0.7 (austenite)</td>
<td>-</td>
</tr>
<tr>
<td>335 °C/1 hr.</td>
<td>64 ± 0.3 (bainite)</td>
<td>-</td>
<td>61 ± 2 (bainite)</td>
</tr>
<tr>
<td>335 °C/8 hr.</td>
<td>30 ± 0.3 (austenite)</td>
<td>25 ± 0.6 (austenite)</td>
<td>28 ± 3 (austenite)</td>
</tr>
<tr>
<td>405 °C/5.8 hr.</td>
<td>75 ± 0.2 (austenite)</td>
<td>&lt; 1 (austenite)</td>
<td>75 ± 3 (austenite)</td>
</tr>
</tbody>
</table>
Figure 6-29 Comparison of the bainite formation kinetics at 335 °C measured from the neutron BET and dilatometry.
6.4 Discussion

At 1000 °C, well above the Thermo-Calc predicted $T_{Ae3}$ temperature of 768 °C, the material is in fully austenitic condition, with all carbon in the austenite phase. It is not surprising to see that no transformation occurs during the intermediate cooling from the austenitization temperature to the bainite transformation temperatures down to 283 °C. In fact, an estimation of the $M_S$ temperature from the Andrews equation (equation 2-5) gives a value of 280 °C, which is below the bainite temperatures applied in this study.

A TTT diagram for the bainite transformation can be generated from the volume fraction information obtained from the neutron BET measurements, as shown in Figure 6-30. The $B_S$ temperature of 441 °C was calculated from equation (2-4). Considering the data scatter when applying the equation, this is in good agreement with the fact that no bainite is detected at the temperature of 436 °C after 4 hours of holding from the neutron BET technique (Figure 6-13).

Many factors affect the austenite and ferrite lattice parameters. Carbon, being an interstitial solute element, is obviously a major contributor. Literature data for the linkage of austenite carbon concentration and lattice parameter are found mainly for room temperature$^{152, 153, 154}$ and high temperatures (above $T_{Ae1}$)$^{155}$. The general form of such relationship is commonly presented by the following equation:

$$a_{\gamma} = a_0 + \beta x_c^\gamma$$  \hspace{1cm} (6-1)

where $a_{\gamma}$ is the austenite lattice parameter, $a_0$ the austenite lattice parameter at zero carbon concentration, $\beta$ a constant and $x_c^\gamma$ the carbon concentration in austenite phase. For example, the following carbon concentration dependence of the austenite lattice parameter has been reported:

133
Figure 6-30 TTT diagram for the bainite transformation.
\[
\begin{align*}
a_\gamma &= 3.5550 + 0.044 \text{C}^{[156]} \\
a_\gamma &= 3.6003 + 0.044 \text{C}^{[157]} \\
a_\gamma &= 3.5780 + 0.044 \text{C}^{[158]} \\
a_\gamma &= 3.572 + 0.033 \text{C}^{[159]}
\end{align*}
\]
where \( a_\gamma \) is in Å and carbon concentration in wt.%. A summary of the relationships can be found in ref. [160].

At high temperatures, the effect of thermal expansion is taken into consideration and the relationship becomes:

\[
a_\gamma (T) = a_{\text{ref}} [1 + \text{CTE}(T - T_{\text{ref}})]
\]  (6-6)

where \( \text{CTE} \) is the coefficient of thermal expansion for austenite, \( T \) is temperature, \( T_{\text{ref}} \) the reference temperature and \( a_{\text{ref}} \) the austenite lattice parameter at the reference temperature. For ferrite, a similar relationship can be established. For example, Onink et al\(^{[155]} \) found the following set of relationships between the lattice parameters and carbon concentrations of austenite and ferrite phases at high temperatures, for a number of iron-carbon binary alloys:

\[
a_\gamma (X_c, T) = (0.36308 + 0.00075X_c) [1 + (24.9 - 0.6X_c) \times 10^{-6} (T - 1000K)]
\]  (6-7)
\[
a_a (T) = 0.28863 \times [1 + 17.5 \times 10^{-6} (T - 800 K)]
\]  (6-8)

where the lattice parameters are in nm, \( X_c \) the number of carbon atoms per 100 iron atoms, temperature \( T \) in Kelvin.

For alloyed steels like the one used in this study, the alloying elements such as Mn, Si, etc also affect the lattice parameter of austenite and ferrite phase. However, these are substitutional solute elements and their effects on the lattice parameter are orders of magnitude smaller comparing to those of the interstitial solute element such as carbon in steel\(^{[152, 161]} \). Furthermore, it was found\(^{[88]} \) that the substitutional elements do not partition even on the finest conceivable scale during the bainite transformation, as the ratio of substitutional to iron atoms
remains constant everywhere including across the interface, until prolonged holding at the bainite transformation temperature. Therefore, the effect of the substitutional elements on the lattice parameters can be neglected.

However, the interstitial solute element carbon is considered mobile during the bainite transformation, according to Stark, et al.\cite{162}. Therefore, during isothermal bainite transformation, carbon concentration change must be accounted for the change of lattice parameters. The transformation strain may also contribute to the change of lattice parameters. But this is insignificant comparing to the change of carbon concentration based on the estimation by Hall et al.\cite{163}.

In summary, the lattice parameters of austenite and ferrite are affected by carbon concentration and thermal expansion only. Following Roberts\cite{153}, the relationship can be expressed as:

\begin{align*}
a_r(x^r_C, T) = (a_{y, ref.} + \beta_r x^r_C)[1 + CTE_r(T - T_{ref.})] \quad \text{(6-9)} \\
a_a(x^a_C, T) = (a_{a, ref.} + \beta_a x^a_C)[1 + CTE_a(T - T_{ref.})] \quad \text{(6-10)}
\end{align*}

where $a_r$ is the austenite lattice parameter, $a_{y, ref.}$ the austenite lattice parameter for zero carbon concentration at the reference temperature $T_{ref.}$, $\beta_r$ a constant, $x^r_C$ the carbon concentration in austenite and $CTE_r$ the coefficient of thermal expansion for austenite. The $\alpha$ symbol denotes the parameters for the bainitic ferrite in equation (6-7).

To determine the $CTE_p$, the initial lattice parameter of austenite at the beginning of the isothermal transformation as a function of the transformation temperature is plotted, as illustrated in Figure 6-31. It is assumed that initial austenite is un-transformed, hence carries the nominal carbon concentration. Therefore, the lattice parameter of austenite at this point is a
Figure 6-31 Lattice parameter of austenite phase at the beginning of isothermal bainite transformation as a function of the transformation temperature.
function of the transformation temperature only. A linear line $a_T = kT + \text{const.}$ is fitted to the data points in Figure 6-31. The good agreement indicates that the above assumption is valid.

Combining the fitting equation into the expression $CTE_T = \Delta a_T / (a_T \Delta T) = (a_T - a_{ref}) / a_{ref}(T - T_{ref})$, the $CTE_T$ can be calculated as $CTE_T = k / (kT_{ref} + \text{const.})$. Taking the reference temperature at 20 °C, the $CTE_T$ of $22.3 \times 10^{-6} \text{°C}^{-1}$ is obtained. This value is in good agreement with the literature data of $21.15 \times 10^{-6} \text{°C}^{-1}$ [164] and the macroscopic CTE from dilatometry measurement ($23.4 \times 10^{-6} \text{°C}^{-1}$).

Taking various $\beta_T$ values from the literature [152, 153, 154, 160], the carbon concentration in the austenite phase varies in a wide range, as illustrated in Figure 6-32, taking the 335 °C transformation as an example. In this figures, the dashed line represents the required austenite carbon concentration to maintain the carbon mass balance in the system. For the $\beta_T$ value of 0.044, the $a_{T,ref}$ at the reference temperature of 20 °C is determined to be 3.571 Å. While for the $\beta_T$ value of 0.033, the $a_{T,ref}$ at the reference temperature of 20 °C is determined to be 3.575 Å by forcing the carbon concentration of austenite at the beginning of bainite transformation stage to be the nominal composition, i.e. 0.4 wt.%. It can be seen that the $\beta_T$ value of 0.044 better describes the effect of carbon on the lattice parameter of austenite for this transformation temperature case.

The austenite carbon concentration at the end of the isothermal transformation vs. the transformation temperature is summarized in Figure 6-33. It can be seen that for all transformation temperatures, $\beta_T$ value of 0.033 gives carbon concentrations in austenite higher than the calculated values based on mass balance, while $\beta_T$ value of 0.044 results in consistent
Figure 6-32 Variation in austenite carbon concentration during the 335 °C isothermal transformation.
Figure 6-33 Comparison of the austenite carbon concentration at the end of the isothermal holding at various temperatures based on different $\beta_\gamma$ coefficient to the austenite carbon concentration calculated from the mass balance.
carbon concentration with the calculated values based on mass balance. Therefore, the $\beta_\gamma$ value is taken as 0.044 for the calculation of carbon concentration in austenite from the lattice parameter.

The $CTE_\alpha$ and $a_{\alpha,ref}$ for ferrite can be determined in a similar fashion. The $\beta_\alpha$ value of 0.030$^{[153]}$ was adopted. As a result, the effect of carbon concentration and thermal expansion on the lattice parameters of austenite and ferrite phase can be presented by the following equations and are shown in Figure 6-34 to Figure 6-38.

$$a_{\gamma}(T, x_{c,\gamma}^\gamma) = (3.571 + 0.044x_{c,\gamma}^\gamma)[1 + 22.3 \times 10^{-6}(T - 20^\circ C)]$$ (6-11)

$$a_{\alpha}(T, x_{c,\alpha}^\alpha) = (2.863 + 0.030x_{c,\alpha}^\alpha)[1 + 14.7 \times 10^{-6}(T - 20^\circ C)]$$ (6-12)

where $a_\gamma$ and $a_\alpha$ are the lattice parameter for the austenite and bainite in Å, respectively, $x_{c,\gamma}^\gamma$ and $x_{c,\alpha}^\alpha$ the carbon concentration in the austenite and bainite in wt.%, respectively.

There is a clear trend of carbon enrichment in the austenite phase during the bainite transformation stage, as shown in Figure 6-34. Looking back in Figure 6-9, in which the evolution of the austenite and bainite volume fraction at 283 °C is presented, one can see that the austenite volume fraction drops rapidly from 100 % to about 25 % in the first two hours of transformation, while the bainite volume fraction increases from 0 to 75 % in the same period. Initially, carbon is located in the austenite phase when the material is given the austenitization treatment. After cooling to the bainite transformation temperature, austenite starts to transform into bainite, thus its volume fraction decreases. Consequently, carbon in the austenite phase has to be rejected and redistributed. High silicon concentration in this material prevents carbide precipitation$^{[30,48,165]}$, since silicon has no solubility in cementite. Therefore, carbon can not be absorbed by bainite. This results in the carbon being rejected to the remaining austenite phase,
Figure 6-34 Changes of carbon concentration in the austenite and bainite during isothermal bainite transformation at 283 °C.
Figure 6-35 Changes of carbon concentration in the austenite and bainite during isothermal bainite transformation at 335 °C.
Figure 6-36 Changes of carbon concentration in the austenite and bainite during isothermal bainite transformation at 355 °C.
Figure 6-37 Changes of carbon concentration in the austenite and bainite during isothermal bainite transformation at 405 °C.
Figure 6-38 Change of carbon concentration in the austenite phase during isothermal bainite transformation at 436 °C.
causing the carbon content in the austenite to increase, which is nicely captured by the neutron BET technique. At this bainite transformation temperature (i.e. 283 °C), the carbon in the austenite phase has been enriched to such a high level that the $M_S$ may be postponed to below room temperature. Therefore, all the austenite phase at the end of the thermal cycle can survive the final quench to room temperature and present in the microstructure as retained austenite. This justifies the good agreement between the 'off-line' room temperature XRD measurement and the 'on-line' neutron BET measurement for the austenite volume fraction in the material.

For the microstructure of the steel heat treated at 335 °C, it is interesting to notice that the martensite islands are enclosed by austenite (Figure 6-24). This indicates that there was a carbon gradient in the remaining austenite grain during the bainite phase transformation process. When bainite is formed, carbon is rejected into the surrounding remaining austenite, making the carbon concentration higher in this region and leaving the interior of the remaining austenite phase at relatively lower level of carbon content. This leads to a higher $M_S$ temperature for the interior region of the remaining austenite. During final quench to room temperature, the martensite transformation occurs in the interior of the remaining austenite, while the outer area of the remaining austenite survives as the enriched carbon concentration pushes the $M_S$ temperature to below room temperature. Thus, the martensite island is produced within the retained austenite phase in the microstructure. This also explains the discrepancy in the austenite volume fraction measured from neutron BET and room temperature XRD in this case. The room temperature XRD measurement gives lower austenite volume fraction than the neutron BET at the end of the thermal cycle because part of the austenite phase transforms to martensite during the quench.
At higher bainite transformation temperature (405 °C), less austenite is transformed into bainite, therefore, the carbon enrichment during the transformation is reduced, as seen in Figure 6-37. Consequently, all austenite transforms to martensite during the final quench to room temperature. This explains the discrepancy in the austenite volume fraction between the room temperature XRD measurement and the neutron BET measurement.

In an extreme case, i.e. 436 °C of transformation temperature, since no austenite is transforming into bainite (see Figure 6-13), obviously no carbon enrichment in the austenite occurs and the carbon concentration in the austenite phase remains constant at the material's nominal carbon concentration, as illustrated in Figure 6-38. This observation also justifies the assumption made previously that the initial austenite is un-transformed at the beginning of the isothermal bainite transformation. In addition, this observation confirms that the cooling rate applied is fast enough so that no transformation occurs during the intermediate cooling from the austenitization temperature of 1000 °C to the bainite transformation temperature.

The carbon inhomogeneity information in the austenite phase during the bainite transformation is also captured by the neutron BET edge width parameter $\sigma_1$, as illustrated in Figure 6-20. In this work, the instrument factor that contributes the broadening of the Bragg edge is constant for the experimental runs; therefore, the edge width parameter $\sigma_1$ reflects the variations in the lattice parameter. In this case, this variation in the austenite lattice parameter is caused by the inhomogeneity of carbon concentration in austenite. Large value in the edge width parameter $\sigma_1$ for the lower bainite transformation temperature (Figure 6-20b, for example) indicate a higher degree of carbon inhomogeneity in the austenite phase, which is reflected in the final microstructure (Figure 6-24).
It is worth noting that at the early stage of the bainite transformation, there is a hump in the bainitic ferrite carbon concentration curve for the 283 and 335 °C transformation temperatures (Figure 6-34, Figure 6-35). This suggests that the bainitic ferrite initially transformed has higher carbon concentration. A number of factors may contribute to this, eg. i) the composition of the bainitic ferrite may follow the equilibrium between $\alpha$ and $\gamma$ phase which is higher than for the $\alpha/\text{Fe}_3\text{C}$ equilibrium and therefore precipitation of carbides in ferrite may occur at later stages of the transformation; ii) the solubility for carbon in the bainitic ferrite could be increased if the radius at the tip of the bainite ferrite is small, due to the Gibbs-Thompson effect, or iii) possibly the bainite transformation may follow the displacive mechanism\textsuperscript{[88,166]} with the carbon initially supersaturated. At later stage the excess carbon may be rejected into the residual austenite (carbon is mobile at the bainite transformation temperatures\textsuperscript{[162]}). However, at this point, these observations can only be noted as preliminary. Further neutron BET experiments are planned to obtain detailed information about the bainite transformation behaviour of this material at temperature range of 280 to 350 °C and also to test repeatability and significance. At higher bainite transformation temperatures (i.e. 355 and 405 °C), the carbon supersaturation in the bainite ferrite is not noticeable.

6.5 Summary

1. Neutron BET technique can be used to study ‘in-situ’ the phase transformation kinetics in steels during two stage thermal cycle.

2. Evolution of volume fractions and lattice parameters in the FCC and BCC
phases can be extracted from the data analysis.

3. Carbon enrichment in the austenite phase during isothermal bainite transformation is clearly observed from this technique.
Chapter 7 Conclusions and Outlook

In this work, a systematic experimental study on the microstructure evolution in Fe-C-Mn-Mo dual phase and Fe-C-Mn-Si TRIP steels during intercritical annealing and intermediate temperature holding (for the Fe-C-Mn-Si steel) was conducted. Metallography, dilatometry, XRD and neutron BET techniques were employed to follow the microstructure evolution in the material. The metallurgical phenomena examined include recrystallization of ferrite, austenite formation during heating and austenite decomposition during step cooling. For the cold-rolled materials, recrystallization of ferrite took place during the reheating to the austenitization temperature. A mathematical description of the ferrite recrystallization phenomenon could be made using the Avrami equation for both isothermal and non-isothermal temperature paths. It was found that an overlap between ferrite recrystallization and austenite formation was possible at high heating rates.

Very significant effects of heating rate on austenite formation were observed including the fraction of austenite and its spatial distribution and morphology. For both Fe-C-Mn-Mo and Fe-C-Mn-Si steels, high heating rates lead to higher fraction of austenite formed at a given intercritical temperature and time. These observations are of importance since intercritical annealing represents the first processing step during annealing of Fe-C-Mn-Mo or Fe-C-Mn-Si steels. The microstructure produced in this step is inherited through the rest of the process and is thus reflected in the final microstructure.

Fundamentally, heating rate clearly affects the nucleation and growth of austenite both for hot-rolled and cold-rolled materials, although the effect is greater for cold-rolled materials.
The interaction between the microstructure of ferrite/pearlite mixture and austenite formation is complex and requires further understanding. The basic trends can be rationalized by the competing mechanisms for nucleation and growth of austenite and how these depend on the starting microstructure (i.e. spatial distribution of pearlite and the degree of ferrite recrystallization). The interaction between ferrite recrystallization and austenite formation is strong and it affects not only the rate of austenite formation but also the spatial distribution and morphology of austenite.

From an industrial point of view, the range of heating rates examined (1 to 100 °C/s) is typical of continuous annealing lines used for galvanizing such that these effects are expected to be of industrial relevance. The morphology of the final microstructure is of particular interest since the highly anisotropic martensite distribution that are produced under high heating rates from cold-rolled starting materials would be expected to have significant effects on the final mechanical properties. Furthermore, it appears that it is possible to tailor microstructures by control of processing conditions and alloy design. The level of cold rolling is expected to be important since it determines the initial spatial distribution of carbon which is predominately found in the pearlite islands and will also affect the rate of ferrite recrystallization. The interaction between ferrite recrystallization and austenite formation can be controlled by changing heating rates but also by alloy design. For example, the addition of elements such as Mo, Nb or B which are known to retard ferrite recrystallization would promote the overlap between these phenomena.

A novel technique, neutron Bragg-edge transmission (BET) was applied to study the bainite reaction, which occurs when the material is cooled to an intermediate temperature after the austenitization process. The neutron BET technique not only gives volume fraction
information for the participating phases, but also reveals unique, "in-situ", time-resolved carbon redistribution information from the lattice parameter, which is not available from other techniques. The volume fraction results were compared to the results from traditional XRD, dilatometry and optical metallography. Excellent agreement between these results was observed. In addition, carbon enrichment in the austenite phase during bainite transformation is clearly observed from this technique. Preliminary results suggest that for lower bainite transformation temperatures, carbon supersaturation is observed in the bainitic ferrite at the early stage of transformation, indicating that the transformation seems to follow the displacive mechanism.

For the future, it is important to extend the current understanding of the basic phenomena which occur during processing into a mathematical model. However, it is still challenging to develop an integrated process model that tailors sub-models for recrystallization, austenite formation and austenite decomposition. Ultimately, the process model will link the microstructure and mechanical behaviour of the material to provide predictions for the mechanical properties.

Finally, it has been demonstrated that the neutron BET is a unique tool to study phase transformations in steels and more interestingly, "in-situ", time-resolved carbon redistribution. Further neutron BET experiments are planed to study this phenomenon in more detail. Moreover, the extension of this work to industrial material may be beneficial to improvement in process control and material properties.
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