MICROSTRUCTURAL EVOLUTION IN THE HAZ OF X80
LINEPIPE STEEL: PHASE FIELD MODELLING

by

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Abstract

During welding, the heat affected zone (HAZ) of X80 linepipe steel is subjected to very steep spatial variations in temperature and concentration of Nb bearing particles which results in a strongly graded microstructure. Therefore, models on the length scale of the microstructure, i.e. the so-called mesoscale, are useful to simulate microstructure evolution in the HAZ. Among mesoscale models, phase field modelling (PFM) is selected because it is based on diffusional time steps and it is a robust tool to capture complex morphologies, e.g. bainitic ferrite.

A PFM is developed for austenite grain growth in 2D and 3D that is applicable to rapid heat-treatment cycles taking the pinning/dissolution effects of Nb bearing particles into account by using an effective mobility concept. In addition, a PFM is developed for the austenite decomposition to predict the simultaneous formation of polygonal ferrite and bainite. PFM is coupled with a carbon diffusion model and an effective interface mobility is introduced to implicitly account for the solute drag effect of Nb. For simplicity, the formation of carbide-free bainite is considered and a suitable anisotropy approach is proposed for the austenite-bainite interface mobility.

The model is first applied to a TRIP steel in which ferrite and bainite form separately, and bainite can be considered carbide-free bainite. Then the model is applied to simulate the microstructural evolution in the HAZ of the X80 linepipe steel accounting for the thermal and microstructural gradients and validated with microstructure observations made in a weld trial.
Preface

This research work was conducted as a part of an NSERC grant awarded to The University of British Columbia in collaboration with TransCanada PipeLines and Evraz Inc. All publications listed below have been prepared from work presented in the dissertation. I am the primary contributor to all of them, and the contributing co-author is my supervisor Professor Matthias Militzer.


- "Phase Field Modelling of Microstructure Evolution in the HAZ of X80 Linepipe Steel." M. Toloui, M. Militzer, 9th International Pipeline Conference, Calgary, Canada. September 2012

- "3D Phase Field Simulation of Austenite Grain Growth in Microalloyed Linepipe Steel." M. Toloui, M. Militzer, 4th International Conference on Recrystallization and Grain Growth, Sheffield, UK. July 2010

Three more papers are in progress to be submitted for publication as refereed journal papers.

The experimental tests included in this document were supplied by Dongsheng Liu (Liu 2007), Jennifer Reichert (Reichert 2012), and Michael Gaudet (Gaudet 2012). The experimental contribution of this thesis was to perform additional quantitative analysis on Reichert and Liu’s micrographs as well as to prepare and quantitatively analyze metallographic samples from Gaudet’s welding samples.
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Nomenclature

Latin symbols

\[ A \] Constant

\[ B \] Constant

\[ b \] Empirical parameter

\[ Bs \] Bainite start temperature (°C)

\[ C \] Constant

\[ c \] Carbon chemical concentration

\[ D \] Diffusivity

\[ d \] Average volumetric grain size

\[ d_{\alpha\beta} \] Jump distance

\[ d_\alpha \] Ferrite grain size EQAD

\[ d_\gamma \] Prior austenite volumetric grain size

\[ dV_B \] Incremental volumetric growth

\[ d_{lim} \] Limiting volumetric grain size

\[ f \] Volume fraction of particles

\[ F \] Free energy functional

\[ Fs \] Ferrite start temperature (°C)

\[ f_F \] Ferrite fraction

\[ f \] Relative fraction of Nb(CN) precipitated

\[ f_B \] Bainite fraction

\[ I \] Nucleation rate per volume
$K$  Solubility product

$k$  Empirical parameter

$k_{st}$  Static anisotropy coefficient

$k_{kin}$  Kinetic anisotropy coefficient

$m$  Empirical parameter

$M$  Atomic weight

$Ms$  Martensite start temperature (°C)

$n$  Particle density

$N_{3D}^j$  Volumetric density of $j$ nucleation sites in 3D

$N_{2D}^j$  Volumetric density of $j$ nucleation sites in 2D

$P$  Driving force for grain growth

$P^*$  Pinning pressure

$Q$  Activation energy

$R$  Universal gas constant

$r$  Average radius of particles

$r_i$  Inscribed radius of the actual grain geometry

$r_c$  Circumscribed radius of the actual grain geometry

$\Delta S$  Driving force proportionality

$T$  Absolute temperature

$t$  Time

$T_p$  Peak temperature (°C)

$\Delta T_c$  Critical nucleation undercooling
$\Delta T_{i}$  & Critical nucleation undercooling at $i$ boundary  \\
$\Delta T_{Tj}$  & Critical undercooling at triple junctions  \\
$\Delta T_{R}$  & Nucleation temperature range at PAGBs  \\
$T_s$  & Transformation start temperature in Kelvin  \\
$\Delta T_{\gamma\gamma}$  & Critical undercooling type I  \\
$\Delta T_{\gamma\alpha}$  & Critical undercooling type II  \\
$\Delta T_{\gamma\beta}$  & Critical undercooling type III  \\
$T_{eq}$  & Equilibrium transformation temperature  \\
$V_u$  & Unit volume of subunit  \\
v  & Grain boundary velocity  \\
$\dot{v}$  & Debye frequency  \\
$\dot{\gamma}$  & Adjustable temperature dependent grain growth kinetics parameter  \\
$X$  & Mean solute mole fraction  \\
$\dot{X}$  & Fraction transformed  \\
x  & Distance from fusion line (μm)  \\
$X^p$  & Molar composition  \\
$Z$  & Empirical parameter  \\

Greek symbols  \\
$\alpha$  & Geometrical factor in 3D  \\
$\dot{\alpha}$  & Empirical parameter  \\
$\beta$  & Topological factor  \\
$\eta$  & Interfacial width
<table>
<thead>
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<th>Symbol</th>
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<tbody>
<tr>
<td>$\eta_o$</td>
<td>Maximum interfacial width</td>
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<tr>
<td>$\theta$</td>
<td>Crystallographic orientation</td>
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<tr>
<td>$\kappa$</td>
<td>Anisotropy intensity</td>
</tr>
<tr>
<td>$\kappa_g$</td>
<td>Mean curvature</td>
</tr>
<tr>
<td>$\Lambda$</td>
<td>Ratio of matrix to precipitate atomic volumes</td>
</tr>
<tr>
<td>$\mu$</td>
<td>Mobility</td>
</tr>
<tr>
<td>$\mu_o$</td>
<td>Pre-exponential factor</td>
</tr>
<tr>
<td>$\mu^p$</td>
<td>Mobility of unpinned boundaries</td>
</tr>
<tr>
<td>$\mu^{\text{eff}}$</td>
<td>Effective mobility</td>
</tr>
<tr>
<td>$\mu_o^{\beta\text{YOR}}$</td>
<td>Mobility pre-exponential factor for compatible orientation</td>
</tr>
<tr>
<td>$\mu_o^{\beta\text{YNOR}}$</td>
<td>Mobility pre-exponential factor for incompatible orientation</td>
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<tr>
<td>$\xi$</td>
<td>Parameter representing the density of nucleation sites</td>
</tr>
<tr>
<td>$\rho$</td>
<td>Density</td>
</tr>
<tr>
<td>$\sigma$</td>
<td>Interfacial energy</td>
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<tr>
<td>$\sigma_o$</td>
<td>Maximum interfacial energy</td>
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<tr>
<td>$\sigma^*$</td>
<td>Interface stiffness</td>
</tr>
<tr>
<td>$\phi_i$</td>
<td>Phase field parameter in phase $i$</td>
</tr>
<tr>
<td>$\psi$</td>
<td>Geometric factor in nucleation model</td>
</tr>
<tr>
<td>$\omega$</td>
<td>Fitting parameter for grain growth kinetics</td>
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**Abbreviations**

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<tr>
<td>AFM</td>
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<tr>
<td>API</td>
<td>American Petroleum Institute</td>
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</tr>
<tr>
<td>BCT</td>
<td>Body-centered tetragonal</td>
</tr>
<tr>
<td>BM</td>
<td>Base metal</td>
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<tr>
<td>CCT</td>
<td>Continuous cooling transformation</td>
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<td>CA</td>
<td>Cellular Automata</td>
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<td>CF</td>
<td>Continuum-field</td>
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<td>CGHAZ</td>
<td>Coarse-grained HAZ</td>
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<td>DP</td>
<td>Dual Phase</td>
</tr>
<tr>
<td>EQAD</td>
<td>Equivalent area diameter</td>
</tr>
<tr>
<td>FGHAZ</td>
<td>Fine-grained HAZ</td>
</tr>
<tr>
<td>GMAW</td>
<td>Gas Metal Arc Welding</td>
</tr>
<tr>
<td>HAZ</td>
<td>Heat affected zone</td>
</tr>
<tr>
<td>HIC</td>
<td>Hydrogen induced cracking</td>
</tr>
<tr>
<td>ICHAZ</td>
<td>Inter-critical HAZ</td>
</tr>
<tr>
<td>ISS</td>
<td>Inter-sheaf spacing</td>
</tr>
<tr>
<td>JMAK</td>
<td>Johnson–Mehl–Avrami–Kologoromov</td>
</tr>
<tr>
<td>KS</td>
<td>Kurdjumov–Sachs</td>
</tr>
<tr>
<td>MA</td>
<td>Martensite/austenite island constituents</td>
</tr>
<tr>
<td>MC</td>
<td>Monte Carlo</td>
</tr>
<tr>
<td>MPF</td>
<td>Multi-phase field</td>
</tr>
<tr>
<td>NW</td>
<td>Nishiyama–Wassermann</td>
</tr>
<tr>
<td>PAGB</td>
<td>Prior austenite grain boundaries</td>
</tr>
<tr>
<td>PFM</td>
<td>Phase field model</td>
</tr>
<tr>
<td>PMZ</td>
<td>Partially melted zone</td>
</tr>
<tr>
<td>Acronym</td>
<td>Description</td>
</tr>
<tr>
<td>---------</td>
<td>-------------</td>
</tr>
<tr>
<td>SCHAZ</td>
<td>Sub-critical HAZ</td>
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<tr>
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<td>Specified minimum yield strength</td>
</tr>
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<td>SSC</td>
<td>Stress corrosion cracking</td>
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<tr>
<td>TMCP</td>
<td>Thermomechanical Controlled Processing</td>
</tr>
<tr>
<td>TRIP</td>
<td>Transformation Induced Plasticity</td>
</tr>
<tr>
<td>WM</td>
<td>Weld metal</td>
</tr>
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</table>
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First, I give thanks to God who gave me health and ability to do the work. I would like to express my sincere gratitude to my supervisor, Professor Matthias Militzer. Words cannot express how grateful I am to his sincere support and kind guidance throughout the progression of this work. His advice and understanding on my research, career, and personal life have been priceless.

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Special thanks are owed to my beloved parents and my brother, for all the love, encouragement, and support throughout my life.
1 Introduction

Iron forms up to 35 % of the mass of the earth and therefore is one of the most abundant metals on our planet (Weast 1989). The most common alloy of iron is steel, which is made of iron and carbon as the basic elements with other alloying additions. There is no doubt that steel is the most versatile alloy among all the industrial alloys. Steel is also the most dominant engineering material and will remain so in the near future. The versatility of steel results primarily from the multiplicity of microstructures and properties that can be attained through alloying and thermomechanical processing. For example in low carbon steels the austenite-ferrite transformation can be used to tailor microstructures, i.e., the size, shape, distribution, composition and crystal structure of the microstructural constituents that essentially control the final properties of any given steel. Nowadays microstructural engineering of steels includes the design of the microstructure with Multi-phase, Meta-stability and Multi-scale constituents on the length scales from micron to nano, i.e. so-called M3 microstructure (Figure 1-1) (Dong 2012).

One of the applications of advanced steels is the pipeline industry. Since the oil and gas fields are frequently located in remote areas, long distance pipelines are the most economic, safe, and environmentally friendly delivery systems to transport oil and gas from the field to the consumers. For example, there is an increased need to explore new and strategically accessible sources of oil and gas in the Arctic. In order to reduce the construction cost of pipelines, enhance transmission efficiency, and
reduce transportation cost, the operating pressures and diameters of pipelines continue to increase, which requires enhanced properties of the linepipe steels.

The property demands are particularly pronounced in the Arctic as pipelines are exposed to very low temperatures and a number of geo-hazards such as frost heave, thaw settlement, and soil movement, which will impose various external loads on the pipeline in addition to the internal pressure and other stress inducing loads that non-arctic pipelines typically experience. The loads induce longitudinal strain at levels well above the linear elastic limit, which deform the pipeline and if not properly designed lead to local buckling or wrinkles. The conventional stress-based pipeline design approach does not fully exploit plastic properties of pipe material and often requires demanding measures under such load conditions, which leads to cost-ineffective design solutions (Cimbali 2002). However, an alternative approach, i.e. the strain-based method that is based on a limit state and displacement controlled load, can provide safe and economically attractive solutions. The use of the “strain based” design criterion in offshore pipeline technology has widely increased in recent years (Cimbali 2002). For the strain-based design of pipelines for traverse unstable permafrost territory, sufficient uniform elongation is required which requires high strength, high fracture toughness at low operating temperatures and weldability. As a result, development of advanced linepipe steels continues to receive tremendous attention.

The desire for improved welding productivity, by reducing the number of welding teams and weld stations working in extreme remote environments, raised the interest for more efficient joining methods. One of the traditional manual metal-arc techniques is Stovepipe welding. This involves applying all passes in the vertical
down direction using covered electrodes and V butt joint design. Such welding techniques are very prone to welder-induced defects and have a low efficiency. Therefore, traditional techniques have been recently replaced by the latest welding techniques such as application of multi-torch systems with semi-automatic welding procedures, which increase the processing speed, and in parallel, by improving the integrity of the welds and weld properties (Dolby 2000). Among these methods, semi-automatic dual torch Gas Metal Arc Welding (GMAW) is the technique that is proposed by TransCanada for the welding of pipelines in the Arctic.

Figure 1-1- Multi-phase, Meta-stability, and Multi-scale are used in microstructural design of steels (Dong 2012).

In every arc welding process, the material can be divided into three main zones: weld metal (WM), heat-affected zone (HAZ) and base metal (BM). WM usually has
higher level of alloy content and superior properties compared to the HAZ and BM because of the alloying design of the filler material. The BM has a carefully designed microstructure that is obtained through Thermomechanical Controlled Processing (TMCP) leading to a very well defined set of mechanical properties. The HAZ, however, may have potentially inferior properties resulting from the very rapid heat treatment experienced by this region during weld thermal cycles (Easterling 1992). This is a concern as no chemical control can be applied in the HAZ. Despite significant achievements over the past decades regarding the development of novel thermomechanical processing methods, which led to improved mechanical properties, there has been comparatively little development in welding processes to control the microstructural evolution in the HAZ. Therefore, there is a great demand for studies that find solutions for improvements of the HAZ properties.

As strain-based design relies on the ductility of pipe material and the girth welds, feasibility studies of new pipelines must include a proper understanding of microstructural evolution in the critical regions of the pipeline especially the HAZ of girth welds to control their mechanical properties. Therefore, it is necessary to develop microstructural models that can yield a better understanding of grain growth, ferrite formation and bainite formation in the HAZ where steep temperature gradients result in significant microstructure variations on the length scale of the microstructure. In order to capture gradients, it would be beneficial to use the so-called mesoscale models. Among mesoscale models, phase field modelling (PFM) is particularly well suited as it is based on diffusional time steps and it is a robust tool to capture complex morphologies, e.g. bainitic ferrite.
In this work, it is aimed to describe the microstructural evolution in the HAZ of pipeline girth welds. This work is part of a larger collaborative research and development project sponsored by NSERC, TransCanada PipeLines, and Evraz, to develop a microstructure-property model for the HAZ of X80 line-pipe steel proposed for applications in the Arctic.

Here, a critical part is bainite modelling. As a preparatory step, the phase transformation model was applied to describe continuous cooling transformation in a selected TRIP steel in which ferrite and bainite formation are significantly separated and the bainite structure is a carbide free structure.
2 Literature Review

2.1 Microstructure Evolution in Low-Carbon Steels

2.1.1 Austenite Grain Growth

Upon heating above $A_{c1}$, the microstructure starts to transform to a face centered cubic phase, which results in austenite. Austenite is the parent of the microstructures in low carbon steels, which result from austenite decomposition upon cooling and provides the framework for some of their characteristics. Shape and size of the austenite grains will determine the rates of transformation in terms of nucleation and growth.

The austenitic microstructure evolves by decreasing the total surface energy of grain boundaries by increasing grain size. Grain growth maintains the volume conservation and surface tension equilibrium between grains (Thorvaldsen 1992). To minimize the energy larger grains grow at the expense of smaller grains. If grain growth occurs with a scaling grain size distribution, it is named normal grain growth. Otherwise, if normal grain growth is inhibited, abnormal grain growth occurs which leads to a non-uniform grain structure (Cotterill 1976). Dissolution of second phase particles is one of the well-known causes, which may lead to abnormal grain growth (Gladman 1997).
2.1.2 Austenite Decomposition

2.1.2.1 Continuous Cooling Transformation

Austenite transforms into a variety of microstructure constituents upon cooling. The types of transformation products that form depend on cooling rate, austenite grain size, and chemical composition of the steel. The most widely used way to represent the austenite decomposition is in form of continuous cooling transformation (CCT) diagrams. Figure 2-1 shows a schematic CCT diagram for the weld metal of low carbon steel (Kou 2002). As shown in this figure, CCT diagram describes how microstructural constituents evolve at given cooling rates.

![Continuous cooling transformation diagram](image-url)

Figure 2-1- Continuous-cooling transformation diagram for weld metal of low-carbon steel (Kou 2002)
2.1.2.2 Austenite Decomposition to Ferrite

At sufficiently slow cooling rates below the $\text{Ae}_3$ temperature, austenite transforms to ferrite. Austenite decomposition to ferrite consists of two stages nucleation and growth.

Ferrite nucleation usually occurs at austenite grain boundaries where different nucleation sites, e.g. grain corners, edges, faces, etc., are available. If ferrite nuclei are assumed to have a shape of spherical caps, then the activation energy for nucleation at grain corners is most favoured, followed by grain edges and grain faces. Homogeneous nucleation within austenite grain requires the highest activation energy and is therefore the least favoured way for nucleation (Johnson 1975). In an investigation by Enomoto et al. (Enomoto 1986), nucleation rates at grain faces were compared to nucleation rates at grain edges. They concluded that at small undercoolings, grain edge nucleation is dominant over grain face nucleation, while at larger undercoolings, the grain face nucleation is dominant. Sectioning experiments by Huang and Hillert (Huang 1996) showed that the dominance of grain corners as nucleation sites is so strong that it may be suggested that the nucleation at grain edges have occurred at special positions, which are energetically similar to the grain corners. These experiments also prove that the number of active nucleation sites is very low, for instance, ferrite nuclei occupy only about 5-7% of all grain corners. Regardless of the site, there are usually Kurdjumov–Sachs (KS) and/or Nishiyama–Wassermann (NW) orientation relationships between ferrite nuclei and the parent austenite phase (Ray 1994, Adachi 2005). When ferrite nucleates at sites along prior austenite grain boundaries (PAGBs), the activation energy for nucleation is
minimized by having an orientation relationship either with both or with one of the two austenite grains and get randomly orientated with respect to the other austenite grain. Experimental studies show that, generally, 90% of ferrite grains have a close orientation relationship with one or both of the neighbouring austenite grains (Landheer 2009). These orientation relationships define the shape and interfaces of the ferrite nuclei. Unfortunately, there is no conclusive model in the literature that can thoroughly describe the shape and interfaces of ferrite nuclei. A first description is the spherical-cap model with incoherent austenite/austenite grain boundaries and incoherent austenite/ferrite phase boundaries. Experimental results reveal that this model under-predicts the experimental results of the rate of nucleation (Lange 1988). A more advanced model, i.e. the disk shaped pillbox model, describes nuclei with partially or fully coherent interfaces. This model yields a lower interface-energy configuration and thereby can match the experimentally observed nucleation rates. However, this model fails to explain the temperature dependence of the nucleation rate that was observed by in-situ synchrotron measurements (Offerman 2002).

After nucleation at a PAGB, polygonal ferrite usually grows isotropically within each neighbouring parent austenite grain. However, between two austenite grains, it selectively grows most rapidly into one of the austenite grains depending on the orientation relationship. Figure 2-2 shows an experimental case reported by Bhadeshia (Bhadeshia 2001) in which ferrite grain formed on a PAGB and a bainite plate nucleated at the ferrite/austenite interface. According to the author, the ferrite, $\alpha$, has an orientation relationship with one austenite grain, $\gamma_1$, but is randomly orientated with respect to the other austenite grain, $\gamma_2$, therefore it grew faster within
γ₂. It also shows that the bainite plate has been able to nucleate at one side of the austenite/ferrite interface where the orientation is suitable.

![Micrograph of austenite and ferrite phases](image)

Figure 2.2- Ferrite selectively grows most rapidly into one of the austenite grains depending on the orientation relationship and bainite plate nucleates at the austenite/ferrite interface only on the side where the orientation is suitable (Bhadeshia 2001).

2.1.2.3 Austenite Decomposition to Widmanstätten Ferrite

For larger undercooling, in grades with sufficiently high carbon content, there is an increasing tendency for the ferrite to grow as Widmanstätten plates, which is another potential product of austenite decomposition. Nucleation of Widmanstätten ferrite usually occurs at the prior austenite grain boundaries and its growth often has a steady-state plate-like fashion, especially in the later stages of transformation (Yamanaka 2007). Widmanstätten plates consist of coherent broad sides with low
interfacial energy and mobility and incoherent tips with a much higher interfacial energy and mobility. Widmanstätten ferrite can be considered as the intermediate phase between polygonal ferrite and bainite.

2.1.2.4 Austenite Decomposition to Bainite

Bainite is another decomposition product that may form when austenite is cooled past a critical temperature. The temperature range for transformation to bainite is between those of ferrite and martensite.

Bainite grows in the form of sheaves. A sheaf of bainite has a complex morphology consisting of many subunits (Figure 2-3), and within each sheaf the subunits are parallel and of identical crystallographic orientation and habit plane (Bhadeshia 2001). Bainite can be commonly classified into upper and lower bainite (Mehl 1938) (Figure 2-4). These two variants have similar lath substructures, (Honeycombe 1980) but they are different in terms of carbide distribution. At higher temperatures upper bainite forms where upon formation of ferrite lath the excess carbon in supersaturated ferrite is partitioned into the residual austenite such that carbide precipitates between the laths (Bhadeshia 1990). The ferrite laths grow into the austenite in a similar way to Widmanstätten side-plates. However, at low temperatures lower bainite forms where during the formation of ferrite plates carbon cannot partition rapidly, therefore carbide dispersion is finer and aligned at approximately the same angle to the plane of the ferrite plate (Bhadeshia 1983, Sandvik 1982, Porter 2009).
Generally, bainite nucleation takes place through a diffusional mechanism in three steps at the following nucleation sites:

- **Type I:** Nucleation at PAGBs.
- **Type II:** Nucleation on ferrite formed at PAGBs.
- Type III: Nucleation within austenite grains at the tip of bainite sheaves (autocatalytic sympathetic nucleation).

Nucleation sites at PAGBs have the lowest activation energies, i.e. highest nucleation temperature, therefore there is a preference that the first set of bainite nuclei form at these sites (Yamamoto 1995) acquiring as close as possible orientation relationships with respect to both sides of the PAGB. However, the orientation relationship with respect to austenite matrix never exactly coincides with the exact KS. Angular deviations of close packed planes between bainite and austenite increase with the transformation temperature while variations in angular deviations for close packed direction with temperature are small (Takayama 2012). As shown in Table 2-1, twenty-four different bainitic ferrite orientations (variants) can be formed within a single austenite grain in the KS orientation relationship (Takayama 2012). However, the orientation deviations of bainite with respect to one of the adjacent austenite grains is much more than the other one. Therefore, bainite sheaf does not grow into the austenite grain with higher orientation deviation (Spanos 1996, Furuhara 2008). Furthermore, bainite sheaves stop growing when they impinge with a hard obstacle such as a grain boundary or another plate.

The second step (Type II) involves the nucleation of bainite on ferrite formed along PAGBs (Aaronson 1956). Early experimental investigations based on low magnification optical microscopy (<2000X) observations did not capture this type of nucleation (Zackay 1962). Instead, they suggested secondary bainitic ferrite was a product of instabilities at the growing ferrite/austenite boundaries of ferrite allotriomorphs in a manner similar to instability-driven dendrite formation during
solidification. This was also supported by early modeling work by Townsend and Kirkaldy (Townsend 1968) who adapted the Mullins-Sekerka theory of morphological instability in solidification (Mullins 1963) to the solid-solid case. Nevertheless, later investigations using higher magnification microscopy proved that the type II nucleation is responsible for the formation of bainitic ferrite on allotriomorphs. Spanos et al. (Spanos 1996) demonstrated that bainitic ferrite and the allotriomorphs from which they evolve are not single crystals but are instead composed of multiple crystals formed by individual nucleation events. Even some early low magnification optical microscopy observations (Aaronson 1956) confirm that there is a clear interface between the bainitic ferrite and the allotriomorphic ferrite, suggesting that type II nucleation was responsible for their formation. Type II nucleation is also supported by numerical calculations (Aaronson 1995) showing that at the growing ferrite/austenite interface there is sufficient driving force available at the terraces of ledges to permit nucleation of bainitic ferrite. Bainite nuclei can only form on regions of the austenite/ ferrite interface where the orientation lies within the so-called Bain region, which encompasses the KS and NW relationships (Bhadeshia 2001) (Figure 2-2). The experimental study of Guo et al. shows that in the austenite grain which has a very close KS relationship with the ferrite, bainitic plates select the same variant as the ferrite; and because bainite plates form a low energy interface with the ferrite, bainite nucleates faster and quickly covers the entire surface of the ferrite. In contrast, in the austenite grain, which has an orientation several degrees deviated from the KS relationship, bainite choose the variants that
have a small misorientation angle with the austenite; and the nucleation is slower (Guo 2012).

Table 2-1- Variants of the KS orientation relationships (Takayama 2012).

<table>
<thead>
<tr>
<th>Variant no.</th>
<th>Plane parallel</th>
<th>Direction parallel</th>
<th>CP group</th>
<th>Bain group</th>
<th>Misorientation from V1 (°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>V1</td>
<td>(111)_a // (011)_a</td>
<td>(101)_a // [111]_a</td>
<td>CP1</td>
<td>B1</td>
<td>60.0</td>
</tr>
<tr>
<td>V2</td>
<td>(101)_a // [111]_a</td>
<td>(101)_a // [111]_a</td>
<td>B2</td>
<td>60.0</td>
<td></td>
</tr>
<tr>
<td>V3</td>
<td>(011)_a // [111]_a</td>
<td>(101)_a // [111]_a</td>
<td>B3</td>
<td>60.0</td>
<td></td>
</tr>
<tr>
<td>V4</td>
<td>(111)_a // (011)_a</td>
<td>(110)_a // [111]_a</td>
<td>B1</td>
<td>10.5</td>
<td></td>
</tr>
<tr>
<td>V5</td>
<td>(101)_a // [111]_a</td>
<td>(110)_a // [111]_a</td>
<td>B2</td>
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<td></td>
</tr>
<tr>
<td>V6</td>
<td>(110)_a // [111]_a</td>
<td>(110)_a // [111]_a</td>
<td>B3</td>
<td>49.5</td>
<td></td>
</tr>
<tr>
<td>V7</td>
<td>(111)_a // (011)_a</td>
<td>(101)_a // [111]_a</td>
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<td>B2</td>
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<td>B1</td>
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<td>B3</td>
<td>57.2</td>
<td></td>
</tr>
<tr>
<td>V13</td>
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<td>B1</td>
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<td>(101)_a // [111]_a</td>
<td>B1</td>
<td>21.1</td>
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</tr>
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</table>

In the late stages of the bainitic transformation, corresponding to higher undercooling, nucleation of bainite occurs within austenite grains through nucleation type III. This type of nucleation mainly includes face-to-face nucleation of bainite sheaves atop previously existing sheaves (autocatalytic sympathetic nucleation) (Spanos 1996, Bhadeshia 1990).

Although there is a consensus on the diffusional nucleation mechanism of bainite, the underlying growth mechanism is controversially debated. In general, two competing hypothesizes are discussed: military, displacive (Bhadeshia 1979), or civilian, diffusional ledge-wise growth (Reynolds 1991). In military transformations, many atoms homogeneously move and the chemical composition is maintained,
whereas in civilian transformations atoms do not move homogenously leading to a chemical composition change.

Diffusional theory describes both nucleation and growth of bainitic ferrite via a reconstructive mechanism. In this approach, the growth of bainite is described by the propagation of a series of steps on the growing interface and ledges, whilst carbide is assumed to form and grow simultaneously with growth of bainite ledges. Diffusional theory has been supported by several experimental observations including:

- Metallographic evidences of similarities between bainite and other structures known to form via diffusion (Borgenstam 2009)
- High-resolution measurements of slow and continuous bainite growth rates that are compatible with diffusional growth processes (Hehemann 1972, Nemoto 1974)
- Atomic force microscopy (AFM) measurements of a subunit showing a surface relief characteristic of a ledge mechanism (Fang 2002)

Displacive theory considers a diffusion-less transformation description via the autocatalytic nucleation and growth of successive subunits. In this approach, it is assumed that a subunit of bainitic ferrite nucleates with carbon partitioning into parent austenite and grows displacively until growth is ceased by plastic relaxation in the adjacent austenite. Carbon is then rejected from the supersaturated ferrite by diffusion and carbide starts to form. In this theory, growth occurs by nucleation and growth of new subunits on the tips of old subunits. According to this theory, the extent
of bainitic transformation of carbide free bainite formation is limited by the $T_0$ curve\(^1\) resulting in incomplete transformation. Displacive theory has been supported by several experimental observations, including:

- Measurements showing surface relief and plastic relaxation in the austenite characteristic of displacive transformation (Nemoto 1974, Swallow 1996, Caballero 2013)
- High subunit growth rates measurements that are compatible with non-diffusional processes (Bhadeshia 1984)
- X-ray diffraction measurements showing that retained austenite contents match the $T_0$ curve that is consistent with one of the main assumptions of the displacive theory (Caballero 2013, Garcia-Mateo 2003)
- Carbon partitioning during nucleation that is revealed by synchrotron studies (Stone 2008) and carbon supersaturation in bainitic ferrite during transformation measured by atom probe (Caballero 2013, Caballer 2010) that is in line with the non-simultaneous carbide formation assumption

In short, bainitic transformation has a complex mechanism that is not yet clear, and there is no comprehensive model that can describe all the features of the bainitic transformation.

\subsection{2.1.2.5 Austenite Decomposition to Martensite}

At very high cooling rates, austenite transforms to martensite. Martensite is formed by a displacive mechanism, which involves the cooperative movement of

\(^1\) $T_0$ encompasses all the compositions and temperatures under which ferrite and austenite have the same Gibbs free energy.
atoms to form a body-centered tetragonal (BCT) crystal structure. Because of accumulation of elastic strain, martensite formation is always incomplete accompanied by a fraction of retained austenite. Prior to reaching the martensite start temperature, austenite may be enriched by carbon that has been rejected by the partitioning during ferrite and bainite formation. Prior carbon enrichment makes austenite more stable, thereby lowering the martensite start and martensite finish temperatures. Therefore, because of the prior carbon enrichment, austenite may further not completely transform to martensite upon cooling. Thus, the martensitic transformation is typically a partial transformation forming martensite/austenite island constituents (MA) (Wang, 1992, Davis 1993). MA microstructural constituents usually appear in four different morphologies including blocky particles/islands, connected particles at PAGBs, elongated stringer particles along bainite subunits and MA-C constituents consisting of MA and carbides (Li 2010).

2.2 Advanced Steels

2.2.1 TRIP Steels

The microstructure of TRIP (Transformation Induced Plasticity) steels is very complex consisting of four constituents: ferrite, bainite, martensite, and a considerable fraction of retained austenite (0.10-0.20). The presence of 10-20% retained austenite in the final microstructure leads to an attractive combination of strength and elongation (Figure 2-5). This excellent property combination is due to the TRIP effect that is associated with the strain-induced transformation of retained austenite to martensite during deformation.
In these steels, due to Si and/or Al addition, carbide precipitation is retarded during the bainite transformation stage such that so-called carbide-free bainite forms. Consequently, during bainite formation the remaining austenite is further enriched with carbon leading to high stability of austenite, which can be retained at room temperature.

### 2.2.2 Linepipe Steels

Linepipe steels are a group of micro-alloyed steels that normally have low carbon content (<0.14wt.%) and total alloy content in the range of 2-3 wt.% (Collins 1983). Pipelines can be prone to failure in the HAZ or WM under conditions of longitudinal traction and internal pressure. Stress corrosion cracking (SSC) and hydrogen induced
cracking (HIC) also preferentially affect the girth weld regions, especially in exposure to sour gas. Therefore, pipeline steels are required to have high resistant to the initiation and propagation of cracks in order to avoid failure. Thus, as defined in API 5L, linepipe steels with very low carbon equivalent are required to facilitate welding and improve the properties of the weld deposit and the HAZ (Booth 2001). In addition to low carbon equivalent, the resistance to SCC and HIC are required to be improved by addition of alloying elements, which avoid elongated non-metallic inclusions (Hudson 2004).

Because the pipeline industry seeks to make longer pipelines with greater carrying capacity and higher operating pressure in increasingly more remote areas such as the Arctic, there has been a tendency to increase the strength (equivalent to API grade) of linepipe steels as shown in Figure 2-6. API X52\textsuperscript{ii} steel grades were one of the earliest grades that were developed using the traditional hot rolling and normalizing processing. The microstructure of this steel mainly consists of ferrite and pearlite. In the late seventies API X70 grades were developed through thermo-mechanical processing gaining higher strength and toughness. X70 is strengthened by grain refinement, but similar to X52, has a ferrite and pearlite microstructure (Dittrich 1992, Gray 1988). State-of-the-art processing methods, consisting of thermo-mechanical rolling plus subsequent accelerated cooling, emerged in the

\textsuperscript{ii}For pipeline steels, the most common classification is according to the American Petroleum Institute (API), where a number designates the specified minimum yield strength (SMYS) in ksi. In Canada, Canadian Standards Association (CSA) suggests a similar classification using "Grade" instead of "X" and a number designating the SMYS in MPa. Therefore API X80 used in this thesis would be equivalent to CSA Grade 550 (CSA 2002).
eighties leading to X80 steels (Hillenbrand 2001). These steel grades have bainitic-ferritic microstructures along with martensite/austenite constituents (MA) (Okatsu 1997, Asahia 1995). Recent additions of molybdenum, copper, and nickel enabled the strength to be raised to that of grades X100 and X120, but these grades are still at the scale of laboratory research. The microstructure of these grades mainly consists of bainite with lath and granular morphology and MA. The key element controlling microstructure evolution and final microstructure of linepipe steels is Nb.

Figure 2-6- Development of high strength linepipe steels (Hillenbrand 2001).

### 2.2.3 Effect of Nb on Microstructure Evolution

Nb is considered an effective micro-alloying element in controlling the microstructure of linepipe steels and other TMCP steels that are usually microalloyed with Nb. Nb atoms have a strong affinity with carbon and nitrogen atoms. Therefore,
NbC, NbN, and/or Nb carbonitride (Nb(CN)) particles can form. Nb bearing precipitates control/ inhibit recrystallization and grain growth in austenite by pinning the migrating boundaries (Zhang 2008, Wang 2008, Zurob 2002). The remaining Nb in solution causes a strong solute drag effect that also results in the retardation of austenite recrystallization and grain growth (Maruyama 1998, Bai 1992). The dissolution of Nb(CN) suppresses polygonal ferrite formation such that it promotes the formation of bainitic microstructures (Thomas 1982) i.e. Nb in solid solution delays the austenite decomposition thereby encouraging microstructural refinement and promoting the appearance of non-equiaxed microstructural products (Poole 2010). For example, bringing all Nb in solution in the X80 steel that is studied here, can shift the transformations to lower temperatures by up to 100 °C, and thereby promotes the formation of bainite over ferrite (Poole 2010). As shown in Figure 2-7 the critical cooling rate, which is the cooling rate required to achieve a mainly martensitic microstructure, significantly decreases as the Nb content in solid solution is increased (Fossaert 1995).

These strong effects of Nb in microalloyed steels are not attributed to the thermodynamics of the system, as the total quantity of niobium is too small to noticeably alter the free energies of ferrite, bainite, and austenite. In essence, Nb in solution mostly affects the kinetics of the decomposition through retardation of either nucleation or growth. Different suggestions are available in the literature about the mechanism by which the kinetics is affected. In particular, it is suggested that Nb atoms strongly segregate to the prior austenite grain boundaries and at the inter-phase boundaries, therefore, they:
• Exert solute drag on the migrating inter-phase boundary (Thomas 1982);
• Decrease the diffusivity of carbon and lead to a decrease in the growth rate (Thomas 1982, Rees 1995);
• Lower the grain boundary energy to reduce the nucleation of transformation products (Fossaert 1995).

![Diagram](image)

Figure 2-7- Critical cooling rate required to achieve a microstructure with the martensite fraction greater than 0.95 as a function of the amount Nb in solid solution (Fossaert 1995).

Similar to Nb in solution, Nb bearing precipitates can also interfere with the migrating interfaces by exerting a pinning force but the retardation effect of Nb in solution is stronger. Therefore, precipitation of niobium-containing particles lowers the overall retarding effect and leads to the formation of higher temperature transformation products (Fossaert 1995, Rees 1995). This effect considerably
intensifies the microstructural variations and formation of a graded microstructure in the HAZ of linepipe steels.

2.2.4 HAZ Structure of Linepipe Steels

In the HAZ, steels undergo all the phase transformations described in the previous section in every weld thermal cycle, i.e. austenite formation, austenite grain growth, and austenite decomposition.

A prime feature of the HAZ is the steep thermal gradient on the length scale of a few grains. Thermal gradient can strongly affect grain growth. Experimental in-situ observations in an organic alloy revealed a grain drift toward the high temperature region under the presence of a temperature gradient (Figure 2-8) (Lee 2005). The drift speed has been found to be proportional to the thermal gradient. Other experimental observations suggested that a thermal gradient could also lead to the confinement of grain growth, known as "thermal pinning," as first reported by (Alberry 1977). A morphological justification of thermal pinning is that grains tend to grow non-uniformly, resulting in a change of shape from equiaxed to pear shaped (Figure 2-9). The corresponding increase in surface to volume ratio effectively represents an increase in energy, thereby neutralizing the reduction in energy present in normal grain growth (Easterling 1992, Mishra 2006). However, these studies are not conclusive and further investigations are required on the effect of steep temperature gradients on grain growth.
Controlling the austenite grain size in the HAZ is of paramount importance as it provides the initial condition for the subsequent austenite decomposition. Large austenite grains are of particular concern as they push the phase transformation towards lower temperatures, thereby increasing the possibility of lower temperature transformation products such as martensite. Martensite formation in the region adjacent to the fusion zone significantly lowers the weld toughness and resistance to HIC (Easterling 1992, Kou 2002, Zhang 1999, Eroglu 2000, Ashby 1982, Ion 1984). Large grains in the HAZ can also influence the susceptibility to cold cracking, reheat cracking and even may affect the grain size in the WM through epitaxial growth of grains in the WM from the HAZ grains (Guthrie 1990). Therefore, in order to avoid excessively large austenite grains, microalloying additions that result in second phase precipitates, e.g. carbides and nitrides, are introduced in many advanced steels (Kanazawa 1976). Second phase precipitates, e.g. TiN and Nb(CN), are meant to provide Zener pinning to slow-down grain growth, respectively. Titanium nitride is traditionally considered as the most stable compound of all the microalloy carbides.
and nitrides (Gladman 1997). Figure 2-10 illustrates that a dispersion of TiN precipitates, of appropriate size and volume fraction, can hinder grain growth even at elevated temperatures. However, it has been observed that in the HAZ, under certain circumstances, Ostwald ripening of the TiN particles occurs (Ichikawa 2011). Therefore, more advanced steels exploit oxides and sulphide pinning particles to hinder excessive austenite grain growth. A recent study revealed that ultra-fine oxide or sulphide particles with magnesium or calcium could withstand dissolution at temperatures as high as 1400°C (Ichikawa 2011).

Figure 2-9- Grain growth under steep temperature gradient in HAZ may lead to changes in shapes of grains, which in turn may hinder grain growth: arrows indicate direction of motion of grain boundaries (Easterling 1992).

Banerjee et al. (Banerjee 2010) investigated the grain growth behaviour in the Nb-Ti microalloyed line-pipe steel that was studied in this work. Their results show that depending on the size, Nb(CN) precipitates may dissolve fully or partially upon heating. As a result, the pinning pressure may change with time and position in the HAZ due to partial dissolution of precipitates. In contrast, TiN particles are relatively stable. Employing appropriate alloying strategies can be used in designing a dense
population of TiN particles (with an average size of \(~50\text{nm}\)) as an efficient way to control austenite grain growth in the HAZ.

![Austenite grain size vs. mean distance between TiN precipitates](image)

**Figure 2-10**- Austenite grain size vs. mean distance between TiN precipitates (Matsuda 1978).

Hence, because of the spatial variation of the thermal cycles a graded microstructure forms along the HAZ. In general, the HAZ of steels can be divided into five sub-zones corresponding to the peak temperature experienced during the thermal cycle (Figure 2-11):

- Partially melted zone (PMZ) that had a peak temperature between the solidus and liquidus lines in the phase diagram.
- Coarse-grained HAZ (CGHAZ) that had a peak temperature higher than the dissolution temperature of precipitates (Figure 2-12-left) such that austenite grain growth substantially occurred. Austenite decomposition
from these coarse grains leads to the formation of low-temperature products like bainite, and martensite (Collins 1983, Losz 1990).

- Fine-grained HAZ (FGHAZ) with a peak temperature higher than $\text{Ar}_3$ but below the dissolution temperature of precipitates (Figure 2-12-right). In this region sufficient pinning by precipitates prevails to retard austenite grain growth, therefore during cooling, fine-grained austenite promotes ferrite formation in most parts of this sub-zone (Losz 1990).

- Inter-critical HAZ (ICHAZ) that was subjected to peak temperatures between $\text{Ar}_1$ and $\text{Ar}_3$. Upon heating, because of partial austenitization, carbon content of austenite is higher than in other regions. Upon cooling these austenite grains, depending on the cooling rate, may transform to martensite-retained austenite (MA) or bainite that are local brittle zones potentially deteriorating the fracture toughness.

- Sub-critical HAZ (SCHAZ) that had a peak temperatures below $\text{A}_1$. Microstructural changes that likely occur in this zone, e.g. tempering, cannot be detected by optical microscopy but may affect mechanical properties.
Figure 2-11- Schematic diagram of the different weldment zones corresponding to the peak temperature experienced. Modified picture from (Easterling 1992).

Figure 2-12- Schematic illustration of the events occurring due to extreme thermal cycles along the fully austenitized HAZ corresponding to the regions close to and far from the fusion line.
2.3 Analytical Modelling

2.3.1 Ideal Grain Growth

Coarsening of grains which is only driven by their curvature (i.e. ideal grain growth) may be described by the concepts of mobility, $\mu$, and the grain boundary velocity, $v$, as

$$v = \mu P$$

(2-1)

In their seminal work, Burke and Turnbull (Burke 1952) suggested that the driving force for grain growth, $P$, arises from the curvature of the boundaries. Then, for each section of the boundary with mean curvature of $\kappa_g$ and interfacial energy of $\sigma$ the driving force is given by

$$P = \sigma \kappa_g$$

(2-2)

Burke and Turnbull assumed that the radius of curvature is proportional to the mean radius of grains

$$P = \alpha \frac{\sigma}{d}$$

(2-3)

where $\alpha$ is a geometrical factor and $d$ is the average volumetric grain size. Using the assumption made in Eq. (2-3), Eq. (2-1) can be rewritten as

$$\frac{\partial d}{\partial t} = \mu \sigma \frac{\alpha}{d}$$

(2-4)

Integration of this equation leads to the parabolic grain growth law as:

$$d^2 = d_0^2 + wt$$

(2-5)

where $d_0$ is the initial grain size and $w$ is $2\mu \sigma \alpha$. Temperature-dependence of $w$ can be described with an Arrhenius mobility relationship as
where $\mu_o$ and $Q$ represent the pre-exponential factor and the activation energy of the grain boundary mobility. Although in Eq. (2-5) the grain growth exponent is 2 for ideal curvature driven growth (Mullins 1989) as a result of solute drag and particle pinning, values larger than 2 are generally obtained for normal grain growth in most systems, even in pure iron (Antonione 1977, Riontino 1979). Thus the general form of

$$d^n = d_0^n + \dot{w}t$$

has usually been adopted to empirically describe grain growth where $\dot{w}$ is an adjustable temperature dependent parameter that is used to replicate the experimental grain growth rate.

### 2.3.2 Particle Pinning

Particles will exert a retarding force or pressure on migrating boundaries, which may have a profound effect on grain growth. For that reason, grain growth models must account for the pinning pressure exerted by precipitates. In the pioneering work of Zener and Smith (Smith 1948), it was proposed that the pinning mechanism arises from the reduction in the grain boundary area and the associated decrease in grain boundary energy per volume when a particle intersects the boundary. Zener pinning pressure due to randomly distributed particles with a volume fraction of $f$ and a mean radius of $r$ is given by (Humphreys 2004):

$$P^* = \frac{3f\sigma}{2r}$$
Hence, in the physical picture, the total grain growth driving pressure is dependent on pinning pressure and can be obtained by subtracting the pinning pressure, \( P^* \), from the driving pressure which originates from the mean curvature of grain boundaries, i.e.

\[
P = \sigma \kappa_g - P^* \tag{2-9}
\]

Grain growth will cease whenever the curvature based driving pressure reaches the pinning pressure, i.e.

\[
\sigma \kappa_g = P^* \tag{2-10}
\]

in addition, this defines a limiting grain size as (Humphreys 2004):

\[
d_{lim} = \frac{4\hat{\alpha}r}{3f_v} \tag{2-11}
\]

where \( \hat{\alpha} \) is an empirical parameter. Many modifications have been made to the model proposed by Zener (Manohar 1998). For instance, quantitative agreement with the limiting grain size has been obtained by using Rios's modifications to Zener's pinning pressure (Rios 1987) for a case with stable particle size distribution. Limiting grain size often can be quantified in the general form of

\[
d_{lim} = 2Z \frac{r}{f_v} \tag{2-12}
\]

where \( Z \) and \( m \) are parameters depending on the underlying assumptions of the model, e.g. \( Z \) has been quantified by Rios (Rios 1987) to be 1/6 and \( m=1 \) when the volume fraction of pinning particles is smaller than 0.05. Figure 2-13 summarizes the effect of volume fraction and size of particles on the experimentally observed limiting grain size (Manohar 1998).
Figure 2-13- Ratio of limiting grain size to particle radius as a function of volume fraction of particles (Manohar 1998).

Banerjee et al. (Banerjee 2010) investigated austenite grain growth in the X80 linepipe steel studied here during heating at rates of 10, 100 and 1000 K/s. They developed a phenomenological grain growth model incorporating a pinning term that was established based on modelling the dissolution of NbC particles. Taking the grain boundary mobility as the only adjustable parameter, they were able to obtain a reasonable agreement with the experimentally measured grain sizes (Figure 2-14).
2.3.3 Grain Growth under Thermal Gradient

Early modeling approaches of grain growth in the HAZ (Alberry 1977, Ashby 1982) were generally based on the use of mean-field approaches and integration over the thermal cycle. Potential effects of thermal gradients cannot be captured by such analytical techniques where each position is assumed homogenous. Under steep temperature gradients, e.g. in the HAZ, this assumption is not correct, especially closer to the fusion zone where the grains are the coarsest and the temperature gradient is the steepest. Therefore, according to Eq. (2-7), the quantity \( w \) changes across each grain, and hence, the grain growth rate can be significantly different from the estimates obtained by Eq (2-5).

To capture the effect of steep temperature gradients a simulation approach on the length scale of the microstructure is required, i.e. the so-called meso-scale. MC simulations have been particularly attractive for studying grain growth in a
temperature gradient because the spatial variation of grain boundary mobility can be incorporated into the MC algorithm by site selection probability (Radhakrishnan 1995, Sista 2000, Kong 2009, Wei 2007). This probability, which dictates the rate of grain growth, can be adjusted according to different peak temperatures at different positions. Figure 2-15 shows that rate of grain growth simulated by Monte Carlo (MC) is much slower in gradient heating than in bulk heating.

The influence of thermal gradients on grain growth is not certainly known yet and the concept of thermal pinning is still under debate. Some 2D (Holm 1998) and recent 3D MC simulations (Garcia 2008) with linear change in grain boundary mobility have suggested that grain growth kinetics under the mobility gradient is locally normal and smaller or larger adjacent grains cannot considerably influence the local grain growth rate.

Figure 2-15- 3D MC results for the kinetics of grain growth at distance of 1 mm from fusion plane in HAZ (gradient heating) and bulk material subjected to the same thermal cycle (bulk heating) (Mishra 2004).
### 2.3.4 Austenite Decomposition


\[
\hat{X} = 1 - \exp(-bt^k) 
\]  

(2-13)

where \(\hat{X}\) is the fraction transformed and \(b\) and \(k\) are empirical parameters. \(k\) is usually assumed temperature independent; however, it is quite sensitive to phase transformation conditions such as deformation and carbon enrichment (Cho 2011, Sun 2008). Kinetics of both ferrite and bainite formation can be captured using proper values for \(k\)-value (Tamura 1988). The \(b\) accounts for the temperature dependence and the effect of initial austenite grain size on the transformation kinetics (Umemoto 1980).

The JMAK approach can be applied to non-isothermal conditions, like continuous cooling, when \(k\) is constant such that the additivity principle holds. Although the additivity preconditions usually hold for ferrite formation, i.e. \(k\) is approximately constant; it is usually of limited use for the bainite reaction (Umemoto 1982). In order to get a decent picture of the overall phase transformation, the formation of different transformation products has to be considered in the JMAK analysis. Extensions of the JMAK approach have been proposed under the assumptions of either simultaneous (Jones 1997) or sequential (Umemoto 1992) formation of different transformation products.

In order to develop more physically based models than JMAK for ferrite formation, transformation mechanisms should be considered, i.e. ferrite
transformation should be described by a model that is based on the long-range diffusion of carbon. In an early approach by Zener (Zener 1949); based on the assumptions of local equilibrium at the interface and infinite medium, Ficks’s equation was used to describe diffusional phase transformation. Infinite medium means that the chemical composition of the matrix at far locations is always equal to the initial composition. Soft impingement, which is caused by the carbon build up in the parent austenite, cannot be captured by infinite medium assumption. Therefore, Vandermeer considered spherical growth geometries. (Vandermeer 1990). In their model it was assumed that nucleation of ferrite on PAGBs occurs as site saturated, which leads to the formation of a thin layer of the new phase (ferrite), which thickens as the interfaces move towards the grain centers of the parent austenite. Vandermeer’s analytical solution was obtained through simplifications like a constant carbon diffusion coefficient. To avoid these simplifications, Kamat et al. (Kamat 1992) incorporated numerical methods into a similar model. Kamat et al. compared the planar growth geometry with spherical geometry and proved that spherical geometry leads to a more reasonable prediction of the overall austenite to ferrite transformation in Fe-C alloys. Diffusion controlled conditions hold when interface mobility is high enough; otherwise, the transformation is at least in part interface-controlled (Kop 2000). This becomes increasingly important when the carbon content is lowered. In essence, the ferrite transformation in low carbon steels depends on both long-range carbon diffusion and the interface reaction, i.e. it is of mixed-mode character. Therefore, mixed mode growth models have also been proposed (Sietsma 2004).
More physically based models for bainite include diffusional and displacive models. In the displacive model, individual subunits are assumed to grow without diffusion and the excess carbon is then partitioned into the residual austenite. The overall kinetics is mainly controlled by the rate of nucleation of sub-units at an austenite grain surface in early stages (type I) (Matsuda 2004) and the subsequent autocatalytic nucleation (type III) at later stages (Rees 1992). Bainite nucleation is assumed to not follow the classical nucleation theory, but similarly to that of martensite, the nucleation is related directly to the driving force i.e. nucleation starts when the nucleation driving force ($\Delta G_m$) exceeds the critical driving force. On the other hand, growth is assumed to start when the diffusionless driving force exceeds the stored energy of bainite ($\sim 400$ J/mol) (Turnball 1981). The bainitic phase transformation stops when one of these two criteria is not satisfied (Bhadeshia 1981).

In these types of models the nucleation rate per volume, $I$, and the incremental volumetric growth, $dV_B$, are usually described by (Bhadeshia 2001):

$$ I = A \exp \left( -\frac{B}{RT} \left( 1 + \frac{\Delta G_m}{C} \right) \right) \quad (2-14) $$

$$ \xi dV_B = V_u \epsilon ld t \quad (2-15) $$

where $A$, $B$ and $C$ are constants, $R$ is the universal gas constant and $T$ is the temperature. $V_u$ and $\epsilon$ represent volume of subunit and a parameter representing the density of nucleation sites, respectively. Bainitic transformation is an incomplete transformation and $\xi$ shows the reaction limit. In the initial stages, $I$ and $\epsilon$ represent the nucleation events at the austenite grain boundaries and at later stages, they represent autocatalytic nucleation.
In contrast to the displacive model, diffusional models usually claim that site-saturation nucleation assumption is not valid and the nucleation is likely to proceed during the entire duration of the transformation while the nucleation rate decreases with the decrease in temperature (Quidort 2001). In this approach the classical nucleation theory is applied to describe the nucleation rate, the pillbox model is used to describe the shape of the nuclei and the JMAK model is used to describe the transformation kinetics (Quidort 2001).

2.3.4.1 Empirical Models for TRIP Steels

In a simple approach, Minote et al. (Minote 1996) employed the JMAK method to predict the overall kinetics of ferrite in a Fe-0.2C-1.5Mn-1.5Si TRIP steel. For bainite formation, they compared the displacive approach (Rees 1992) and the diffusional approach (Kaufman 1962). They showed that bainitic transformation kinetics at higher temperatures follows the diffusional model while it follows the displacive model at lower temperatures. The microstructural evolution in the TRIP steel of interest in this work has been modeled before by Liu et al. (Liu 2007). They proposed a microstructure model that is comprised of individual sub-models for austenite decomposition into ferrite and bainite. In the model, the ferrite start is estimated by a diffusion-controlled criterion and its growth is described by JMAK. For bainitic transformation, bainite start is determined by the critical driving force and the growth rate is described by a diffusional Zener-Hillert approach. In a more recent model, Fazeli and Militzer proposed (Fazeli 2012) a more comprehensive description of the diffusional bainite nucleation and growth (Figure 2-16). As Figure 2-16 shows Fazeli and Militzer’s model is not suitable for high cooling rate and large prior
austenite grain size thermal cycles where rejected carbon does not completely redistribute, in particular at the lower temperatures of bainite formation.

The above-mentioned analytical approaches are useful tools to describe the overall kinetics of ferritic and bainitic transformations, however, they are incapable of considering the morphological effects. In order to incorporate the complex morphological features of ferrite and bainite, meso-scale methods should be used.

Figure 2-16- Comparison of predictions of the empirical model of Fazeli and Militzer with the experimental kinetics for the bainitic portion of the phase transformation for the TRIP steel of interest in this work (Fazeli 2012).
2.3.4.2 Empirical Models for the HAZ of Linepipe Steels

Recently Militzer et al. (Militzer 2011) proposed a phase transformation model for the linepipe steel of interest in this work. The model consists of five sub-models for ferrite start temperature, ferrite growth kinetics, bainite start temperature, bainite growth kinetics and martensite/austenite fraction. The ferrite model includes a diffusion-controlled nucleation model for ferrite, while the growth is described by the JMAK model. The bainite start is determined by the critical driving force criterion and bainite growth is described with a JMAK model. Using this model, they were able to generate microstructure maps describing the microstructural change along the HAZ. One of the unique features of this model is that it accounts for the effects of Nb in solution on the microstructural evolution (Figure 2-17). The quantitative accuracy of these comparisons has not yet been analyzed with the microstructure in real HAZ.

Empirical models like Militzer’s model, make predictions for individual portions in the HAZ without accounting for thermal gradients, i.e. the thermal cycle of this position is applied to the microstructure evolution as if it were to occur in a bulk sample with homogeneous temperature. However, in order to account for the gradient in the microstructure, mesoscale models like PFM should be applied.
2.4 Phase Field Modelling

2.4.1 Why PFM?

For grain growth and austenite to ferrite transformation, meso-scale models are now widely available including

- Phase field models (PFM) (Krill 2002, Mecozzi 2006)
- Monte Carlo (MC) methods (Anderson 1984, Tong 2004)
- Cellular Automata (CA) (Geiger 2001, Lan 2004)

One of the major issues with the application of conventional meso-scale models, such as MC and probabilistic Cellular Automata, is due to the difficulty of converting the numerical time-step to the physical time-step (Godfrey 1995, Lan 2006). It is not
an issue with PFMs because they are formulated on the time scale of diffusional processes. In addition to this, PFM is a more suitable modeling tool for the simulation of complex microstructure formation, as it is not required to explicitly track the macroscopically sharp interfaces. Therefore, PFM is the method of choice to simulate microstructural evolution in the HAZ of linepipe steels.

### 2.4.2 PFM formulation

Phase field models belong to the category of diffuse interface models that originated near the end of the 19th century (van der Waals 1894). For these models, it is argued that the interfaces between different coexisting phases have a finite thickness through which the physical properties, such as density and concentration, change smoothly between two equilibrium values. Accordingly, the expression of the diffuse interface approach by the Ginzburg-Landau theory of superconductivity (Ginzburg 1950) inspired the development of the first generation of phase field models by Cahn and Hilliard (Cahn 1958) for non-uniform systems and Allen and Cahn (Allen 1979) for the anti-phase boundary motion. In phase field models, the interface between the phases is treated as a region of finite width having gradual variation of the different state variables. Even though the interfacial width, $\eta$, is chosen to be much smaller than the scale of the microstructure, it is still significantly larger than the actual interface thickness of a few atomic distances. In order to perform meaningful quantitative calculations, the model is developed in a fashion to become independent of phase field interfacial width, but still capture the physics on the meso-scale. There are two main types of PFMs. The first type originates from the
work of Khachaturyan (Khachaturyan 1983) that was applied in a continuum-field (CF) phase field formulation (Chen 1991). In this formulation, phase field variables are related to long-range order parameter fields. The second type of PFMs was developed by Langer et al. (Langer 1986) and based on the work of Hohenberg and Halperin (Hohenberg 1977). Steinbach et al. (Steinbach 1996) and Garcke et al. (Garcke 2004) expanded this model to multi-phase systems. In Steinbach’s model, similar to all PFMs of the Allen–Cahn or Ginzburg–Landau type, the microstructure is described with non-conserved field variables that represent, for example, the local structure and orientation. The equations contain parameters that are related to physical properties of the system, such as interface energy and mobility (Moelans 2009).

In all PFMs, the evolution of the phase field variables are described as the path that leads to the minimization of the total free energy based on kinetic principles. In the case of a multi-phase system, the phase field evolution is given by a set of equations

$$\frac{d\phi_i}{dt} = \sum_{i \neq j} \mu_{ij} \left( \frac{\delta F(\phi_i, c)}{\delta \phi_j} - \frac{\delta F(\phi_i, c)}{\delta \phi_i} \right)$$

(2-16)

where $\phi_i$, $t$, $\mu$, $c$ and $F$ represent the phase field parameter, time, mobility, concentration and free energy functional. In order to perform physical predictions, model parameters should be related to physical quantities in the conventional sharp-interface equations while assuring to obtain similar results through asymptotic analysis. Steinbach et al. (Steinbach 1996, Tiaden 1998) in their phenomenological approach, proposed following formulations:
\[
\frac{d\phi_i}{dt} = \sum_{i\neq j} \mu_{ij} \left( \sigma_{ij} \left[ \phi_j \nabla^2 \phi_i - \phi_i \nabla^2 \phi_j \right] + \frac{\pi^2}{2\eta^2} (\phi_i - \phi_j) \right) + \frac{\pi}{\eta} \sqrt{\phi_i \phi_j \Delta G_{ij}}
\]  

(2-17)

where \(\eta\) represents the interfacial width, \(\Delta G_{ij}\) is the chemical driving force between phase \(i\) and \(j\) and the concentration dependency is introduced into the standard phase field equation by applying the locally linearized phase-diagram data as (Tiaden 1998)

\[
\Delta G_{ij} = \Delta S_{ij} (T - T_{eq})
\]

(2-18)

where \(\Delta S_{ij}\) and \(T_{eq}\) are driving force proportionality factor and equilibrium transformation temperature, respectively. A similar approach can be taken to obtain elastic strain evolution equation (Steinbach 2006). The PFM can be coupled with a diffusion model to calculate the evolution of the concentration field. Tiaden et al. (Tiaden 1998) proposed the following equations for a binary alloy with two phases

\[
\frac{dc}{dt} = \nabla \left[ (1 - \phi_j) D_i \nabla c_i + \phi_j D_j \nabla c_j \right]
\]

(2-19)

where

\[
c = (1 - \phi_i) c_i + \phi_j c_j
\]

(2-20)

### 2.4.3 PFM of Austenite Formation and Grain Growth

For grain growth, the most frequently applied phase field approaches in the literature are the continuum-field (CF) model developed by Chen and Yang (Chen 1994) and Fan and Chen (Fan 1997), and the multi-phase field (MPF) model (Steinbach 1998). Besides these two approaches, other PFM for multi-grain
structures are less popular (Kobayashi 1998, Pusztai 2005). In-depth comparisons of these models for grain growth (Moelans 2009) have shown that these models yield similar results when identical model parameters are applied. Furthermore, if a sufficiently large number of grains are included in simulations (Krill 2002, Kim 2006); PFM are capable of replicating the results of mean field theories for normal grain growth, i.e. parabolic grain growth.

There is a significant body of work in which particle pinning is incorporated into PFM. Attempts to model the pinning effect for grain growth by phase field have been primarily performed by resolving the interaction of individual precipitates in the phase field mesh (Moelans 2005, Moelans 2006, Suwa 2006, Chang 2009, Apel 2009). Moelans et al. (Moelans 2005) modified the CF formulation to describe the interaction between a single particle and a grain boundary for grain growth. Using this approach, they performed 2D simulations for Zener pinning in polycrystalline systems (Moelans 2006). Suwa et al. (Suwa 2006) used a similar method to simulate particle pinning in 3D. Chang et al. (Chang 2009) studied the effect of the average size, size distributions, and morphology of second-phase particles on grain growth kinetics in two dimensions. In all these phase field approaches individual particles were directly included in the simulation mesh. Thus, these simulations are in practice limited to cases where particle spacing is comparable to grain size.

2D grain growth systems are fundamentally different from 3D grain growth. In case of pinning this difference is more pronounced as 2D systems pining leads to the straightening of grain boundaries which eventually stagnates grain growth, whereas in 3D systems grain boundaries never become totally flat (Hillert 1988).
The situation of interest in the X80 linepipe steel, i.e. a finely dispersed particle distribution within a given grain, requires modified approaches. Due to the large difference in size scale between the particles and the austenite grains, the mesh size should be fine enough to resolve the particles and the shape of the grain boundaries. On the other hand, the calculation domain must also contain a large number of grains in order to obtain statistically reliable results. The technique of resolving the particles in the phase field mesh in such conditions would require extensive computer resource that makes it impractical. Furthermore, explicit introduction of pinning particles into the calculation domain, especially in 2D simulations, leads to the straightening of the boundaries that violates the physics of grain growth stagnation. Apel et al. (Apel 2009) have proposed an alternative. Based on the simulation results of micro-scale interaction of precipitates with moving grain boundaries, Apel et al. formulated a driving pressure dependent effective mobility as

\[ \mu = \mu^p \exp\left(-\frac{\omega P^*}{P - P^*}\right) \]  \hspace{1cm} (2-21)

where \( \mu^p \) is the mobility of unpinned boundaries and \( P^* \) is the critical pinning parameter that can be associated with the pinning force and \( \omega \) is a fitting parameter. Apel et al. incorporated this effective mobility into mesoscopic phase field calculations (Figure 2-18a). This approach could not be verified as it is implemented in MICRESS® as shown in Figure 2-18. This approach is not general and for some circumstances, it gives physically unreasonable results, i.e. limiting grain size at zero pinning force as well as the crossovers between different pinning forces. Recently, a friction force approach has been proposed by Shahandeh et al. (Shahandeh 2012) to simulate grain growth with numerical efficiency to capture the pinning pressure on
the grain boundaries. Similar to Apel’s model this model also does not resolve the particles. In this approach, the pinning pressure applied to a single migrating grain boundary is calculated in small-scale simulations, and then the pinning pressure is applied as a uniform friction pressure to all the grain boundaries in larger scale simulations. Their results show that grain growth does not stop when the average driving pressure is equal to the pinning pressure, but it stops when the maximum driving pressure reaches the pinning pressure.

Figure 2-18- Simulated grain size evolution for different particle densities or, correspondingly, critical pinning forces (Apel 2009) (a) and simulated evolution of grain size for different critical pinning parameters with unpinned mobility of 5 cm$^4$/Js and surface energy of 0.00005 J/cm$^2$ (b).

2.4.4 PFM of Austenite Decomposition

2.4.4.1 Polygonal ferrite

Coupled with a diffusion model, PFM is distinctively capable of providing insight with respect to the mixed mode character of the ferrite formation. Previous phase
field simulations (Mecozzi 2005b) have demonstrated that austenite decomposition at the early stages is interface-mobility-controlled and then transfers to the diffusion-controlled mode at the late stages. In the phase field method, chemical driving pressure can be found from the local chemical composition within the interface. Most of the diffusion coupled PFM approaches for austenite to ferrite transformation assumed that only carbon can diffuse, i.e. para-equilibrium, is adopted (Mecozzi 2005b, Takahama 2008, Thiessen 2006, Thiessen 2007).

The phase field formulation developed by Wheeler et al. (Wheeler 1992) was the first PFM used to simulate the isothermal austenite to ferrite transformation in a Fe-Mn-C system (Yeon 2001). However, perhaps because of its ease of relating model parameters to physical properties, MPF soon became the most recognized and widely used formulation to simulate the austenite to ferrite transformation (Takahama 2008, Thiessen 2006, Mecozzi 2005b, Militzer 2006). Pariser et al. (Pariser 2001) were the first group who applied MPF for modeling the austenite-to-ferrite transformation.

The first step in PFM of austenite decomposition is to consider the formation of new nuclei. As a first approach, Thiessen et al. (Thiessen 2007) used the classical nucleation theory to incorporate temperature-dependent nucleation rates into the PFM. Prior austenite grain boundaries are the most important ferrite nucleation sites. However as PFM simulation of Huang et al (Huang 2006) indicated, ferrite could also precipitate on inclusions and dislocations, i.e. what actually happens is something between grain boundary nucleation and random nucleation (Figure 2-19). Furthermore, not all sites on grain boundaries have the same nucleation potency but there are preferential sites, i.e. grain corners and edges. For slower cooling rates there
will be time for ferrite nuclei to significantly grow at small undercooling on preferred sites such as grain corners. At larger undercooling nucleation extends to large areas of the grain boundaries. For moderate cooling rates, nucleation is assumed to occur only at triple points (Mecozzi 2005a) and for higher cooling rates, it is assumed to take place preferentially at the triple points and subsequently at the grain boundaries (Mecozzi 2006). Different potential nucleation modes are normally distinguished by critical undercooling (Apel 2009). Usually, in PFM simulations, experimentally observed kinetics of decomposition is replicated by using appropriate interface mobility, nucleation rate and nucleation undercooling/range relationships (Mecozzi 2005b, Takahama 2008, Thiessen 2006). Traditionally the nucleation behaviour in PFM simulations is taken as either site-saturation or occurring with a constant nucleation rate (Mecozzi 2005b, Militzer 2006).

Figure 2-19- Phase-field prediction of ferrite fraction during continuous cooling of Fe-0.176at.%C–0.747at.%Mn under random nucleation (dotted line), grain boundary nucleation (solid line) compared to experimental data (solid data points) (Huang 2006).
For a given nucleation scenario, along with the nucleation density which defines growth distances, the microstructural evolution in PFM is governed by diffusion and interfacial reactions. The austenite/ferrite interface mobility is the main parameter that determines interfacial reactions. In general, the mobility can be described by an Arrhenius relationship as

\[ \mu = \mu_0 \exp \left( -\frac{Q}{RT} \right) \]  \hspace{1cm} (2-22)

where \( Q \) represents the activation energy. Usually the pre-exponential factor of the mobility (\( \mu_0 \)) is considered as adjustable parameter to fit the simulated ferrite fraction curves to experimental data (Huang 2006, Mecozzi 2005a). Generally, there are two options to fit the mobility. The first option is to adjust the pre-exponential factor, which is similar to the approach proposed by Mecozzi et al. (Mecozzi 2008), for austenite decomposition. Alternatively, the mobility activation energy, similar to the approach of (Savran 2009) for austenite formation, can be adjusted to the experimental data for different conditions, while the mobility pre-exponential factor is found from an analytical relationship such as (Thiessen 2007):

\[ \mu_0 = \frac{d_{\alpha\beta} \dot{\nu}}{kT} \]  \hspace{1cm} (2-23)

where \( d_{\alpha\beta} \) and \( \dot{\nu} \) are the jump distance and Debye frequency, respectively.

As an alternative, transformation fraction–temperature relationships can also be fitted to the experimental ones adjusting the nucleation rate in each time step (Huang 2006). However, fitting the parameters to the experimental data is not a trivial task. As Figure 2-20 illustrates, these parameters are interrelated and cannot be adjusted separately (Mecozzi 2008). For instance high nuclei density leads to a fine final
structure and verse versa. Therefore, nuclei density should not violate the expected final ferrite grain size and the undercooling range must lead to a final grain size distribution that matches the experimental one. As a pragmatic approach, the nucleation density and sequence can be matched with the size distribution of ferrite grains (Mecozzi 2008). As an alternative approach, for the sake of simplicity, some researchers e.g. (Mecozzi 2006) suppressed ferrite grain growth by choosing a very low value for grain boundary energy of ferrite to obtain a direct correlation between the density of ferrite nuclei and the ferrite grain size in the final microstructures.

![Figure 2-20- Interface mobility as a function of the nucleation temperature spread to reproduce the reference transformation kinetics for two nucleation regimes (Mecozzi 2008).](image)

Even though some researchers like Takahama et al. found good agreement between 2D and 3D simulations, (Takahama 2008) it is evident that proper nucleation sites, such as grain edge and corners, cannot be represented in 2D
simulations. In order to embrace the nucleation at corners and edges in 2D calculations, volumetric density of these sites \((N_j^{3D})\) can be found in a 3D structure and subsequently converted into an area density \((N_j^{2D})\) (Thiessen 2007):

\[
N_{2D}^j = N_{3D}^j 2^{2/3}
\]  

(2-24)

In addition to the nucleation deficiency of 2D simulations, Militzer et al. (Militzer 2006) have shown that more realistic grain shapes can be obtained by 3D simulations (Figure 2-21). Further, for a given mobility, the transformation in 2D is faster as a direct consequence of the columnar growth in 2D compared to more spherically growth in 3D. For simple geometries, e.g. homogeneous equiaxed ferrite structures, similar results can be obtained by using a mobility 3D/2D conversion factor (Militzer 2006).

Figure 2-21- 2D microstructures (a) with the associated cuts through the 3D microstructure (b), white represents ferrite, orange austenite and blue the interfaces (Militzer 2006).
2.4.4.2 Widmanstätten ferrite

Coupling the single-phase field formulation presented in (Loginova 2003) with proper anisotropy models, Widmanstätten ferrite formation has been studied using PFM (Yamanaka 2007, Loginova 2004). Loginova et al. (Loginova 2004) used an isotropic ferrite/austenite interface mobility and anisotropic ferrite/austenite interface energy and thickness for Widmanstätten ferrite. They introduced an anisotropy function, $A(\theta)$, to correlate the interfacial energy ($\sigma$) and the interface thickness ($\eta$) with the orientation of the phase boundary

$$\sigma = \sigma_o A(\theta) \quad (2-25)$$

$$\eta = \eta_o A(\theta) \quad (2-26)$$

where $\theta$ represents the angle between the interface normal and a reference axis. The anisotropy function leads to thinner coherent interfaces with lower interfacial energies compared to incoherent interfaces.

Their simulations revealed that the anisotropy in the surface energy and interface thickness plays a key role in determining whether Widmanstätten growth is possible or not, i.e. Widmanstätten plates can only grow if the anisotropy is large enough. Widmanstätten plates simulated by this model look very realistic, but their sides are rather smooth compared to experimentally observed plates (Figure 2-22). Yamanaka et al. (Yamanaka 2007) have also modeled Widmanstätten formation in a similar fashion but with a different anisotropy formulation (Yamanaka 2006).
2.4.4.3 Martensite and Bainite

At sufficiently high cooling rates, austenite transforms to martensite by a military transformation. To describe the evolution of the martensitic transformation, the transformation-induced strain energy has to be integrated into the phase field theory. To do so, Khachaturyan’s phase field micro-elasticity theory (Khachaturyan 1967, Khachaturyan 1969) can be used where the strain energy is described as a functional of the continuous fields. Incorporating this theory into the first type of PFMs (section 2.2), displacive martensitic transformation was first modeled for a ceramic system by Wang et al. (Wang 1997) and then for the FCC to BCT transition in a metallic system (Artemev 2001b). These models are far from making quantitative predictions for martensitic transformation in conventional steels.

There are many challenges in modelling the bainite formation because its underlying mechanism is under debate and its microstructure strongly depends on the temperature at which it forms, i.e. upper vs. lower bainite. As phase field models...
have successfully been used for simulation of both displacive and diffusional transformations, PFM appears to be a suitable approach to describe the bainitic transformation. Although there are PFM s for martensitic transformation, PFM has not yet been applied for the quantitative description of the displacive aspect of bainite formation because it is dependent on complex stress states and crystallographic orientations. However, coupled with a proper anisotropy model, phase field appears to be a suitable tool to study the diffusional aspect of bainitic growth.

It has been observed that carbide and ferrite in bainite have an orientation relationship with the grain in which they are growing. Hence, bainitic growth could be considered as an anisotropic growth controlled by chemical and elastic driving forces. The solid/liquid interface anisotropy can be introduced into PFM by the dependency of the interfacial properties on the lattice orientation. Recently, an anisotropy model has been used to develop a preliminary 2D phase field model for bainitic transformation in which the driving pressure was calculated in the same fashion as for austenite to ferrite phase transformation (Mukherjee 2010). This model is oversimplified because this simple phase field anisotropy approach cannot rigorously be extended to 3D growth (Eiken 2010) and bainitic transformation is a solid/solid transformation where the growth direction does not only depend on the normal orientation of the growing phase, but also depends on the relative orientations with the parent austenite grains.
2.4.5 PFM for HAZ

With their first attempt, Thiessen and Richardson (Thiessen 2006) applied phase field modelling to the evolution of grain size in the HAZ of an austenitic stainless steel. Later the same research group, Thiessen et al. (Thiessen 2006), extended the application to the austenitization and reversion to ferrite in the heat affected zone (HAZ) of carbon steel using physically based derivations for interface mobility (Eq. (2-23)) and the driving force. Figure 2-23 shows the predicted kinetics of austenite formation and decomposition at different locations along the HAZ in comparison with the synchrotron experimental measurements.

![Image of Figure 2-23](image_url)

Figure 2-23- Austenite fraction as a function of welding time at various positions from the weld center. Red lines are phase field results and blue dotted lines are synchrotron measurements (Thiessen 2006).

All these simulations followed the classical approach to consider different positions throughout the HAZ without taking into account the thermal gradients for each position. Although, for a system with singular transformation product, this approach provides good agreement with the experimental data especially for the
section close to the fusion zone, for more complex systems refined models have yet
to be developed to take care of the influence of gradients in temperature, chemical
composition and initial structure.
3 Objectives

Designing appropriate pipelines will be greatly aided by an in-depth quantification of the effect of welding conditions on the microstructure evolution in the HAZ and related mechanical properties. This work seeks to develop a novel meso-scale model to simulate all stages of microstructure evolution in the HAZ of an X80 steel using PFM. The proposed new model must explicitly account for the gradients in temperature and dissolution of Nb bearing particles in the HAZ. In addition, a novel way has to be developed to model the evolution of bainite using PFM. Furthermore, the model is aimed at accounting for bainite and ferrite formation that occur simultaneously where the final microstructure is the shared product of the competition of these transformations.

The individual sub-objectives can be summarized as follows:

- To develop a PFM for austenite grain growth in 2D and 3D that is applicable to rapid heat-treatment cycles taking the pinning/dissolution effects of Nb bearing particles into account
- To extend the grain growth model to the HAZ of X80 steel with steep temperature gradients
- To adapt a phase field ferrite formation model to describe the ferrite formation in the studied steels
- To develop a phase field modelling approach for bainite formation
• To design a framework to predict the simultaneous evolution and morphologies of the transformation products, i.e. polygonal ferrite and bainite

• To validate model predictions with experimental results for continuous cooling transformation and HAZ microstructures of X80 steel
4 Methodology

4.1 Introduction

The purpose of this study is to model the microstructural evolution in the HAZ of X80 linepipe steel. Therefore, a comprehensive phase field model is developed to describe austenite grain growth and austenite decomposition. Mechanical properties of the HAZ strongly depend on the microstructural-scale variation in size, morphology, and distribution of microstructural constituents. In order to capture these variations, an integrated meso-scale model is developed comprising of sub-models for grain growth, ferrite formation, and bainite formation. A critical part of the model is to capture the simultaneous bainite and ferrite formation. To aid the model development, continuous cooling transformation in a TRIP steel was also considered as a case where the bainite formation is clearly separated from the ferrite formation.

Based on the evaluation presented in section 2.4.1, PFM was selected for the modelling of complex morphologies, such as bainite, instead of alternative models (CA and MC). For PFM calculations, a MPF formulation as represented by Eq. (2-17) (Steinbach 1998) was selected from the available formulations because it allows easy correlation of the model parameters to the physical quantities. Solution of the diffusion equation (Eq. (2-19)) was needed to determine redistribution of carbon associated with austenite decomposition. Commercial software MICRESS® was deemed as a proper choice for numerical calculations. According to its developers, MICRESS® is a numerically efficient phase field software and its applicability to
industrial cases has been widely proven (MICRESS). Finite Phase-Field Method (Eiken 2012) that has recently been introduced into MICRESS® was used for numerical calculations. Finite Phase-Field Method is a modified FDM calculation for diffuse interface problems that minimizes the discretization error associated with FDM solution and decreases the deviation of the solution from the sharp-interface solution. Because of these advantages, the usage of MICRESS® was preferred to developing an in-house code.

The numerical model consists of a set of adjustable parameters describing nucleation and growth of microstructural constituents. A group of parameters were taken from data available in the literature and the rest were found by fitting with continuous heating and continuous cooling test results. Fitted parameters have subsequently been validated using the simulations with other independent experimental data (e.g. from welding trials). For grain growth calculations, fitting included finding an effective mobility that considers the pinning effect of Nb(CN) particles from continuous heating tests. For austenite decomposition calculations, the interface mobility, the nucleation behaviour (nucleation temperatures, number of nuclei, and distribution of nuclei) and bainite anisotropy parameters were adjusted to fit the simulated transformation kinetics and resulting experimental microstructure data available from continuous cooling tests.

Although 3D simulations are more appropriate than 2D simulations for the simulations of phase transformations, considering the numerical artefact associated with 2D simulations, because of the prohibitive computational cost of the 3D calculations of bainite formation, all the austenite decomposition simulations were
performed in 2D. For the case of grain growth, limited 3D calculations were performed to examine the reliability of 2D simulations.

To facilitate the simulations, a main program (Appendix A) along with subroutines (Appendix B, Appendix C, Appendix D and Appendix E) were developed in MATLAB to generate text input files (run file) for MICRESS. The subroutines were used to calculate the initial parameters required by MICRESS® such as nucleation locations of ferrite and bainite, time-temperature profile, boundary conditions, and dissolution profile. The subroutines are interconnected through the main program, e.g. thermal profile subroutine (Appendix B) provides the thermal history to be used in the dissolution subroutine (Appendix D). This feature enables the model to account for the effect of the gradient in fraction of Nb bearing precipitates and Nb level in solution on grain growth and austenite decomposition, respectively.

4.2 Grain Growth

The X80 steel of interest in this work contains a fine dispersion of complex precipitates containing Ti, Nb, and Mo (Banerjee 2010). The Nb-rich particles are in the form of Nb(CN) as the Ti microalloying content is insufficient to take all N out of solution. The equilibrium fractions of the three families of particles, i.e. TiN, NbC, and Mo2C that are the basic forms of the complex particles in the microstructure are compared in Figure 4-1. These thermodynamic calculations describe a system with chemical composition of the steel studied in this thesis. Figure 4-1 suggests that the Mo-rich particles dissolve in ferrite prior to austenite formation (Maalekian 2012). Therefore, Mo bearing precipitates were neglected in the model evaluating austenite
grain growth in a weld HAZ as all Mo-rich precipitates in the HAZ will have dissolved before austenite forms. In contrast, TiN particles are far more stable than other particles at high-temperatures, and thus, particle dissolution/coarsening occurs less for TiN than for other particles (Grong 1997). Therefore, TiN was assumed to have a stable particle size distribution and the dissolution of these particles was neglected in the model. At different locations of the HAZ, no dissolution, partial dissolution, or full dissolution of Nb bearing particle may occur depending on the thermal cycle experienced. Therefore, in contrast to TiN that is expected to have a uniform distribution, Nb(CN) particles are expected to have a significant spatial variation along the HAZ.

![Stability of the precipitates (TiN, NbC and Mo2C) in the X-80 steel with respect to temperature calculated in the equilibrium state (i.e. infinitely large precipitates) using MatCalc (Maalekian 2012).](image)

Figure 4-1- Stability of the precipitates (TiN, NbC and Mo2C) in the X-80 steel with respect to temperature calculated in the equilibrium state (i.e. infinitely large precipitates) using MatCalc (Maalekian 2012).
4.2.1 Effective Mobility

Grain growth simulations were carried out by the numerical solution of the MPF equation (Eq. (2-17)). For simplicity, all grain boundaries were assumed to have the same grain boundary energy (σ) and grain boundary mobility (μ).

The rate of grain growth depends on how easily grain boundaries can migrate. Grain boundary migration is controlled by thermal activation and hence the rate at which the grain boundary migrates is temperature dependent. In addition, the migration rate is controlled by resisting effects such as Zener pinning. The grain boundary mobility is the main parameter that represents the rate of grain boundary migration, and it is typically assumed that the mobility follows an Arrhenius relationship.

In the case of grain growth in the HAZ in the linepipe steel, the grain growth occurs within a few seconds which is too short a period to reach the limiting grain size resulting from Zener pinning. Thus, an effective grain boundary mobility, \( \mu^{\text{eff}} \), is assumed. Then Eq. (2-1) can be rewritten as:

\[
\nu = \mu^{\text{eff}} \sigma \kappa
\]

\( \mu^{\text{eff}} \) depends on the magnitude of particle pinning. Pinning pressure is typically described as function of average radius and fraction of particles. Particle coarsening can be neglected for the rapid thermal cycles in the HAZ, i.e. pinning pressure, are only controlled by the fraction of pinning particles (f). Therefore, one can describe \( \mu^{\text{eff}} \) as a function of temperature and fraction of Nb(CN) particles (f) i.e.:
\[
\mu^\text{eff} = \mu_o(f) \exp\left(\frac{-Q(f)}{RT}\right)
\]

(4-2)

### 4.2.2 Dissolution Estimation of Nb Bearing Particles

A mean-field dissolution model was used in order to estimate the extent of particle dissolution as a function of thermal history. Banerjee et al. (Banerjee 2010) showed that the mean radius dissolution model developed by Perez et al. (Perez 2008) is capable of predicting the dissolution behaviour of Nb-rich precipitates in the X80 linepipe steel if a proper set of input parameters is used.

The analysis of precipitates by Banerjee et al. (Banerjee 2010) showed that in the X80 steel studied, Ti-rich and Mo-rich precipitates, are more or less of the same size, however, Nb-rich precipitates have a bimodal size distribution consisting of large Nb-rich particles (\(r_{\text{NbCN(l)}}=69\) nm) and small Nb-rich particles (\(r_{\text{NbCN(s)}}=2\) nm). In the dissolution model, a superscript of \(j\) is introduced to indicate the large (\(l\)) and small (\(s\)) Nb(CN) precipitate families, respectively. The initial particle densities \(n_j\) are determined from the experimentally estimated precipitate radii, while the initial fractions are obtained assuming that all microalloying elements are precipitated in the as-received steel (Banerjee 2010):

\[
n_j = \frac{3f_j \pi r_j^3}{4}
\]

(4-3)

where \(r\) and \(f\) are particle radius and volume fraction of precipitates of type \(j\). The mean field model predicts the variation of \(r_j\). It is assumed that \(n_j\) is constant and \(f_j\) changes in accordance with the \(r_j\) variations at each time step. The model is based on Zener’s model for the diffusion-controlled shrinkage rate of spherical precipitates in
a solid solution assuming small super-saturation and local equilibrium (Zener 1949).

If interstitials (C and N) are homogeneously distributed in the matrix and Nb diffusion is the rate controlling process, then the variation in mean particle radii $r_j$ may be described by the following equations (Perez 2008):

$$\frac{dr_j}{dt} = \frac{D_{Nb}}{r_j} \frac{X_{Nb} - X_{Nb}^j(r_j)}{A_{NbCN}X_{Nb}^p - X_{Nb}^j(r_j)}$$ (4-4)

$$X_{Nb}^j(r_j) \times 10^4 = \frac{K_{NbCN}}{X_{C+N}} \exp \left( \frac{2\sigma_{NbCN}(M_{Nb} + M_C + M_N)}{\rho_{NbCN}RT_j} \right)$$ (4-5)

Here, $A_{NbCN}$ is the ratio of matrix to precipitate atomic volumes (mean volume per atom), $K_{NbCN}$ is the solubility product, $X_{Nb}^p$ is the molar composition of Nb precipitated, $X_{Nb}$ is the mean solute mole fraction of Nb in the matrix, and $X_{Nb}^j$ is the equilibrium solute mole fraction of Nb at the precipitate matrix interface. $M_i$ represents the atomic weight of elements and $\rho_{NbCN}$ is the density of Nb(CN) particles. $A_{NbCN}$ is given by

$$A_{NbCN} = \frac{M_{Fe}/\rho_{Fe}}{(M_{Nb} + M_C + M_N)/(2\rho_{NbCN})}$$ (4-6)

where $\rho_{Fe}$ represents the density of iron (matrix). The interfacial energy ($\sigma_{NbCN}$) was assumed to be 0.66 J/m² as suggested by Maalekian et al. (Maalekian 2012). The rate of dissolution is controlled by the diffusivity of the controlling element (here Nb), which is described with an Arrhenius relationship in m²/s as (Janssens 2007):

$$D_{Nb} = 1.5 \times 10^{-5} \exp \left( \frac{-286 \times 10^3}{8.314 \times T} \right)$$ (4-7)

in addition, the solubility product is by given by (Janssens 2007):

$$\log_{10} K_{NbCN} = -\frac{10345.6}{T} + 4.5$$ (4-8)
Nitrogen is much more stable in TiN than in Nb(CN), therefore the total nitrogen content available for Nb(CN) precipitation/dissolution is equal to the nitrogen content remaining after TiN formation i.e.

\[ X_{N}^{@\text{TiN}} = X_{Tl}^{o} \]  \hspace{1cm} (4-9)

\[ X_{N}^{@\text{Nb(CN)}} = X_{N}^{o} - X_{N}^{@\text{TiN}} \]  \hspace{1cm} (4-10)

At each time step, based on the value of \( r_{b} \), the solute fractions are updated to ensure mass conservation

\[ X_{Nb} = X_{Nb}^{o} - \frac{A_{NbCN}}{2} (f_{NbCN(s)} + f_{NbCN(l)}) \]  \hspace{1cm} (4-11)

\[ X_{C+N} = (X_{C}^{o} + X_{N}^{o} - X_{N}^{@\text{TiN}})^{c} \]
\[ - \frac{A_{NbCN}}{2} (f_{NbCN(s)} + f_{NbCN(l)}) \]  \hspace{1cm} (4-12)

It has been observed that the set of input parameters (thermodynamic and diffusion data) collected by Maalekian et al (Maalekian 2012) from the literature are more relevant than those of Banerjee et al. (Banerjee 2010) when the dissolution model is applied to the HAZ.

The initial volume fraction of Nb(CN) precipitates were estimated by assuming that in the as-received material an equilibrium fraction of precipitates exists

\[ f_{NbCN(\text{total})}^{o} = f_{NbCN(\text{total})}^{eq} = \frac{2X_{Nb}^{o} M_{Fe}}{A_{NbCN} M_{Nb}} \]  \hspace{1cm} (4-13)

while 74.3% of the total volume of Nb(CN) precipitates is large Nb(CN) and the rest are small ones (Maalekian 2012). The initial particle sizes were adapted from the study of Banerjee et al. (Banerjee 2010). The details of the mean-field Nb(CN) dissolution code written in a MATLAB subroutine can be found in Appendix D.
4.2.3 Incorporation of Effective Mobility in MICRESS

Let us define “fraction of Nb precipitated” \( \dot{f} \) as the ratio of total instantaneous fraction of Nb(CN) precipitates \( f_{NbCN(total)} \) to the total equilibrium fraction of precipitates in the base metal at room temperature \( f_{eqNbCN(total)} \). The purpose of this parameter is to facilitate the comparison of two extreme conditions, i.e. Nb bearing particles fully dissolved and fully precipitated. Then, according to Eq. (4-2), mobility is a function of both \( T \) and \( \dot{f} \). However, mobility can only be defined as a unary function (a look-up table with one variable) in MICRESS. In order to create a unary look-up table that accounts for \( T \) and \( \dot{f} \) for any given thermal path the following steps were taken.

As a first step, the dissolution profile \( (\dot{f} - T) \) is calculated for the given thermal history \( (T-t) \). Then the temporal variation of EQAD \( (d_{2D}-t) \) is found by combining the thermal history \( (T-t) \) and the experimental \( d_{2D}-T \) data. The rate of grain growth \( (\dot{d}_{2D}) \) at each temperature, \( T_i \), is determined from the local slope of the empirical \( d_{2D}-T \) fit, implementing a central difference numerical differentiation technique using Eq.(2-4) and the chain rule i.e. \( \frac{dd_{2D}}{dt} = \frac{d d_{2D}}{dT} \frac{dT}{dt} \). Effective mobility data can finally be extracted as a function of temperature; using the assumption made in Eq. (2-1) i.e. Eq. (2-4) can be rewritten in 2D as

\[
\frac{\partial d_{2D}}{\partial t} = \mu_{2D} \sigma \frac{\alpha_{2D}}{d_{2D}} \quad (4-14)
\]

where \( \mu_{2D} \), \( d_{2D} \) and \( \alpha_{2D} \) are the grain boundary mobility, EQAD and corresponding geometrical factor for 2D grain growth, respectively. The 2D effective mobility can be obtained from
\[ \mu_{2D}^{\text{eff}}(T_i) = \frac{d_{2D}(T_i) \dot{d}_{2D}(T_i)}{\sigma a_{2D}} \]  

(4-15)

This gives the back-calculated effective mobility profile as a function of the temperature \( (\mu_{2D}^{\text{eff}} - T) \) that describes the transition from low temperature corresponding to no dissolution \((f = 1)\) to high temperatures with full dissolution \((f = 0)\). If it is assumed that the activation energy is a constant and the pre-exponential factor is the only parameter that depends on pinning pressure, the high temperature region (full dissolution of Nb(CN) particles) mobility can be described as

\[ \mu_{2D}^{f=0} (T) = \mu_{2D}^{o,f=0} \exp\left(-\frac{Q}{RT}\right) \]  

(4-16)

Combination of the dissolution profile \((f - T)\) and back-calculated effective mobility profile \((\mu_{2D}^{\text{eff}} - T)\) yields a \((\mu_{2D}^{0} - f)\) relationship. Therefore, for every thermal history, the above-mentioned steps lead to a look-up table of mobility versus temperature to be implemented in the MICRESS® calculations (Figure 4-2). It is worthwhile mentioning that this approach is only applicable to continuous heating and continuous cooling conditions.

In order to apply the model to the HAZ, the non-linear gradients of \( T \) and \( f \) and corresponding mobility variations must be taken into account. In MICRESS®, mobility is defined only as a function of temperature \( (T) \). Non-linear thermal gradients can be directly implemented into MICRESS® by defining temperature distribution at each time step as an input. However, as mentioned earlier, MICRESS® does not recognize \( \dot{f} \) and it only takes a single-variable mobility look-up table. To overcome this problem mobility was defined as a function of an effective temperature \( (\tau) \) which represents the variations of \( T \) and \( \dot{f} \). First, as shown in Figure 4-2, the distribution of mobility in
the HAZ at each time step, \( \mu_{2d}(t, x) \), was found based on the outputs of the thermal model, \( T(t, x) \), and the dissolution model, \( \dot{f}(t, x) \). Then, as shown in Figure 4-3, the distribution of the effective temperature at each time step, \( \tau = \tau(t, x) \), was found by comparing the look-up table of \( \mu_{2d} - \tau \) with temporal nonlinear distribution of mobility along the section, \( \mu_{2d}(t, x) \).

\[
\mu = \mu(f, T) = \mu_0(f, T) \exp \left( -\frac{Q}{RT} \right)
\]

\[
T = T(t)
\]

\[
\mu_0 = \mu_0(f)
\]

\[
f = f(T)
\]

\[
\mu = \mu(f, T) = \mu_0(T) \exp \left( -\frac{Q}{RT} \right)
\]

Figure 4-2- Mobility was defined as a unary function of T for the given thermal in accordance with corresponding thermal profile and mobility profile.
4.2.4 2D versus 3D

Since grain growth occurs in 3D, it is necessary to investigate the correlation between 2D and 3D simulations and to evaluate whether or not the experimentally observed grain growth could be replicated with 2D simulations.

The mean equivalent area diameter (EQAD), $d_{2D}$, which is frequently used to experimentally quantify grain sizes in 3D structures, can be correlated to the volumetric grain size, $d$, by a topological factor of $\beta = 1.2$, i.e. $d = \beta d_{2D}$ (Giumelli 1999). Thus, Eq. (2-5) may be rewritten as

$$\frac{\partial d_{2D}}{\partial t} = 2 \frac{\mu}{\beta^2 \sigma} \frac{\alpha}{d_{2D}} \quad (4-17)$$

As will be shown in section 6.2, using a modified effective mobility, $\mu_{2D}^{\text{eff}}$, experimental EQADs can be reproduced by 2D simulations. By comparing Eq. (2-4)
and Eq. (4-17), a relationship between the effective mobility in 2D and 3D can be obtained, i.e.,

$$\mu_{2D}^\text{eff} \alpha_{2D} = \frac{2}{\beta^2} \mu_{\alpha}^\text{eff}$$  \hspace{1cm} (4-18)

In order to find the mobility conversion factor, the ratio of 2D and 3D geometric factors, $\alpha_{2D}/\alpha$, needs to be analyzed. These geometrical parameters represent the deviation between the idealized geometries (cylinder and sphere) and the actual geometries of individual grains. These geometrical parameters are postulated to be proportional to the ratio of inscribed radius, $r_i$, and circumscribed radius, $r_c$, of the actual grain geometry, i.e.

$$\alpha_{2D} = q \frac{r_i^{2D}}{r_c^{2D}}$$  \hspace{1cm} (4-19)

$$\alpha = q \frac{r_i}{r_c}$$  \hspace{1cm} (4-20)

where $q$ is a constant. The 2D phase field calculations by Kim et al. (Kim 2006) show that each grain has on average 6 neighbours. In addition, they noted that grains with 5, 6 and 7 neighbours are the major contributors to the overall grain size distribution. Thus 2D phase field grains may be considered as hexagons (Figure 4-4 a) with $r_i^{2D} = \sqrt{3}a/2$ and $r_c^{2D} = a$ where $a$ is the length of a hexagon side. Similarly, it has been shown (Kim 2006) that for 3D grains the number of neighbours is on average close to 14. Therefore, a truncated octahedron may be considered as grain shape with $r_i = 9\sqrt{10}a/20$ and $r_c = \sqrt{10}a/2$  (Figure 4-4 b). These geometrical shapes give $\frac{\alpha_{2D}}{\alpha} \approx 1$. Considering $\beta = 1.2$ as determined from experimental data (Giumelli 1999) Eq. (4-18) yields
\[ \mu_{2D}^{\text{eff}} = \frac{\mu^{\text{eff}}}{0.7} \]  

Therefore, to obtain identical ideal grain growth kinetics in 2D and 3D, the 3D mobility should be lowered by a factor of 0.7 to compensate for higher levels of driving force (curvature) in 3D.

![Figure 4-4 - Hexagon (a) Truncated octahedron (b)](image)

### 4.3 Austenite Decomposition

Austenite decomposition modelling was conducted by coupled numerical solution of MPF (Eq. (2-17)) and diffusion (Eq. (2-19)) equations to take into account the long-range diffusion of carbon in addition to the interfacial phenomena (Tiaden 1998). Because of low diffusion rate of substitutional atoms, it was assumed that only carbon can diffuse i.e. Fe–C para-equilibrium was used for thermodynamic calculations. Thermodynamic parameters for the phase-field calculations, i.e. chemical driving force, were calculated by Thermo-Calc® using LinearTQ. The LinearTQ approach provides a simplified coupling of MICRESS with Thermo-Calc® by
locally linearizing the phase diagram. This approach is different from a traditional linearized phase diagram method where a temperature is selected as a reference point with reference concentration for both phases and the slopes of the phase boundaries for all elements as well as a constant proportionality factor, $S$, to determine the driving force as a function of undercooling. In the LinearTQ approach, additional parameters are introduced to account for the local slope change of the phase boundaries. In particular, $S$ is replaced by the temperature derivatives of the driving force with fixed composition in phase 1 and phase 2.

For the linepipe steel, because of the significant role of the Nb bearing precipitates and Nb atoms in solid solution similar to the grain growth calculations, the Nb(CN) dissolution model (Appendix D) was used to provide the input dissolution profile for the austenite decomposition calculations in MICRESS®.

Austenite decomposition consists of two stages: ferrite formation and bainite formation. As a starting point, it is preferred to develop the model for a simpler system where ferritic and bainitic transformations occur separately and the corresponding products, ferrite and bainite, are clearly distinguishable in the microstructure. Thus, prior to the application of the model to the linepipe steel the model was applied to a laboratory grade, Mo TRIP steel where:

- in the microstructure, ferrite and bainite are clearly different in appearance whereas this is not the case for the linepipe steel
- ferritic and bainitic transformations are clearly separated in the CCT curves forming two distinct humps, in contrast to very smooth CCT curves in the linepipe steel (Figure 4-5)
• the bainite formation is carbide free in contrast to conventional bainite in the linepipe steel with concurrent carbide formation. This means the type of bainite that forms in TRIP steels are ferrite lathes with negligible carbide formation.

Figure 4-5- Comparison of the typical CCT curves of a TRIP steel (a) and a X80 steel (b)

4.3.1 Initial Microstructure

The first step in austenite decomposition modelling is to establish the starting austenite grain structure. In cases where austenite decomposition was preceded by austenite grain growth, the outputs of the grain growth model were simply adopted as the starting austenite structures. For the simulation of austenite decomposition where the average grain size of austenite is predetermined, e.g. in the TRIP steel, the starting austenite structure was generated by Voronoi construction followed by initialization to eliminate artefacts of the Voronoi construction e.g. straight grain boundaries. The built-in initialization procedure in MICRESS® includes the solution of Eq. (2-17) for the grain growth using numerically stable time steps. The
initialization was applied for 25 iterations to ensure complete removal of the straight grain boundaries. The distribution of Voronoi seeds was used as an adjustable parameter to generate a desired austenite grain size and distribution. For further details, please refer to chapter 7.

In the model, every microstructural object has its attributes, i.e. phase/transformation product and crystallographic orientation by which different sheaves or grains are distinguished among microstructural constituents. A simplified approach was taken to represent the crystallographic orientations of the microstructure constituents. In this approach, starting from a fully austenitic initial microstructure, one of the austenite grains with a particular crystallographic orientation is taken as the “seed grain”. Then the crystallographic orientations of the rest of the microstructure constituents are described as orientation angles with respect to the crystallographic orientation of the seed grain. Because it was assumed that the initial austenite structure is a non-textured structure, the relative angle of the rest of austenite grains with respect to the seed grain were uniformly distributed in the range of 0 to 180 degrees (in 2D). Figure 4-6 illustrates an initial austenitic structure where the seed grain with the reference orientation is shown in the middle. PAGB lines, in Figure 4-6, correspond to the prior austenite grain faces in a 3D structure. The crystallographic misorientation at each PAGB is obtained by the difference between the relative angles of the neighbouring austenite grains. In Figure 4-6, different levels of grain boundary misorientation are represented with different colors, e.g. blue, green, and orange represent high, medium, and low misorientation grain boundaries. When ferrite and/or bainite nucleation takes place,
each nucleus is also assigned a relative orientation angle with respect to the seed grain that reflects the corresponding orientation relationships of ferrite and/or bainite with respect to the parent austenite phase.

4.3.2 Nucleation

Each of the austenite decomposition sub-models proposed in this work consists of nucleation and growth stages. The PFM approach represented by \( \text{Eq.}(2-17) \) only describes the growth stage; therefore, nucleation must be dealt with separately.

Considering preferred nucleation sites, each nucleation site in the microstructure has a varied potency for nucleation and thus a specific critical nucleation driving force is set such that if exceeded nucleation commences. One can assume that the driving
force, $ΔG(c,T)$, is proportional to the transformation undercooling, $ΔT(c,T)$, such that, at any given site, nucleation commences when the local transformation undercooling exceeds a specific critical undercooling i.e.

$$ΔT(c,T) > ΔT_c(c,T) \quad (4-22)$$

The critical undercooling method was employed in MICRESS® for both ferrite and bainite. The critical undercooling for each nucleation mode was considered as a fitting parameter to match their corresponding estimated experimental transformation start temperatures.

Implementation of the nucleation criterion can be done by using the nucleation subroutine of MICRESS® or by a separate subroutine to define the critical undercooling and locations of the nuclei. The built in nucleation subroutine of MICRESS® leads to considerable numerical artefacts like clustering of the nuclei. Thus, a separate subroutine was developed in MATLAB to check phase field calculations and generate the nucleation input for phase-field calculations. The nucleation procedure and the method of combining the subroutine with phase field calculations can be found in Appendix E. This subroutine effectively mitigates the clustering prevalent in the built-in nucleation subroutine of MICRESS® by setting a minimum spacing for the nuclei.

### 4.3.3 Polygonal Ferrite Sub-model

The austenite-to-ferrite phase field model employed in this work is an enhanced version of the previously developed model by Mecozzi et al. (Mecozzi 2008) with the major distinction that it considers the crystallographic orientations. Similar to the
previous approach, the mobility and the nucleation behaviour (number of nuclei and nucleation temperature range) were used to fit the simulated ferrite fraction kinetics and grain size distribution to experimental data.

The most important nucleation sites for ferrite formation are grain boundaries, i.e. grain faces, edges and corners. In 2D simulations, triple points and grain boundaries are considered as the nucleation sites. Nucleation sites at triple junctions have lower critical undercooling than the nucleation sites at PAGBs, therefore the first set of ferrite nuclei form at triple junctions. The experimental study by Lange et al. (Lange 1979) shows that for a 0.13wt% C steel the number of ferrite nuclei at austenite grain edges (corresponding to triple junctions in 2D) is 4 to 13 times higher than the number of nuclei formed at austenite grain faces (corresponding to grain boundaries in 2D). Therefore, for the sake of simplicity, it was assumed that ferrite nucleation at sites along PAGBs will not commence until all the triple junction sites are occupied by ferrite nuclei, considering the minimum allowed nucleation spacing (Figure 4-7a). It was assumed that all the triple points have the same critical undercooling ($\Delta T_{TJ}$).

PAGBs in the austenite structure have different misorientation. PAGBs were categorized into three misorientation groups (say a, b and c) assuming all the PAGBs within each group have identical critical nucleation undercooling. However, different groups were assumed to have different critical nucleation undercooling, $\Delta T_{GB,a}$, $\Delta T_{GB,b}$ and $\Delta T_{GB,c}$. This assumption allows nucleation of ferrite to take place over a temperature range that is consistent with experimental observations (Offerman 2002). Furthermore, this assumption is consistent with previous studies (Landheer
that demonstrate that ferrite nucleation on austenite/austenite grain faces with higher misorientation is more efficient than nucleation on lower misorientation grain faces. In order to decrease the number of adjustable parameters, it was assumed that $\Delta T_{GB,b}$ is equal to the mean of $\Delta T_{GB,a}$ and $\Delta T_{GB,c}$. Therefore, ferrite nucleation temperatures at PAGBs are controlled by the critical undercooling at PAGBs with the highest misorientation ($\Delta T_{GB,a}$) and the nucleation undercooling range at PAGBs ($\Delta T_{R} = \Delta T_{GB,c} - \Delta T_{GB,a}$).

Ferrite nuclei were introduced at equidistant positions along the PAGBs (Figure 4-7b). As illustrated in Figure 4-7, the orientation of ferrite nuclei is determined by the crystallographic orientation angle of one of the neighbouring austenite grains, i.e. ferrite is confined to select one of the two variants within a compatible orientation relationship with neighbouring austenite grains. In the MATLAB subroutine, the random generator function is used to choose one of the available crystallographic orientations. It was assumed that all ferrite nuclei at triple points get different orientations while PAGB ferrite nuclei appear with the same orientation repeated along the length of each segment of austenite grain boundary.

Once the austenite grain structure and their orientations is created in MICRESS®, either from grain growth or Voronoi, the microstructure is then exported as an ASCII file. Nucleation subroutine in MATLAB reads the initial structure from the ASCII file and generates a MICRESS® script in which ferrite nucleation is only allowed in confined separated squares within the microstructure in order to obtain the required ferrite nuclei spacing along the PAGBs (Appendix E).
For each ferrite nucleus, nucleation is followed by growth where ferrite nuclei are assumed to grow isotropically into the austenitic matrix. The growth of ferrite is described as mixed-mode while the mobility of the migrating inter-phase boundaries is taken as an effective mobility, which is essentially used to replicate the experimental transformation kinetics. For the X80 linepipe steel, it assumed that the interfacial mobility is a function of the level of Nb in solid solution. Therefore, dissolution calculations are performed prior to the generation of the austenite decomposition script in order to obtain the values of interfacial mobilities.

At later stages of the ferritic phase transformation, growing ferrite grains may impinge. Then, all the ferrite grains formed along a given grain boundary line will coalesce into one long ferrite rim along the PAGB (Figure 4-7c). The main reason of coalescence is that ferrite grains have identical crystallographic orientations. The coalescence of ferrite grains along the PAGBs is widely observed in experimental studies and theoretical justifications are available in the literature (Pariser 2001, Bengochea 1998). Coalescence is believed to be one of the main causes of the deviation of the number density of final ferrite grains from the experimentally estimated number density of ferrite nuclei. Coalescence was made possible in simulations by using the “categorization” option in MICRESS®. This option lets grains to merge when they have identical phase and crystallographic orientations.
4.3.4 Bainite Sub-model

Here, the austenite-to-bainite PFM describes the growth of bainite sheaves through a diffusion-based mechanism. Bainite is considered as ferrite in terms of thermodynamics, but bainite and ferrite are different in terms of nucleation,
crystallographic orientation, and growth. In particular, it was assumed that the undercooling for bainite nucleation is significantly larger than that of ferrite. Further, bainite growth is assumed to depend on the orientation with respect to the austenite phase.

In the model, some features of the bainitic microstructure like subunits and potential carbide formation were neglected and bainite sheaves were considered homogeneous microstructural constituents with a single crystallographic orientation, i.e. carbide-free bainite. Before the application of the model to steels with conventional bainite, e.g. X80 steel, the model is applied to the TRIP steel for which this assumption is particularly fulfilled.

As shown in Figure 4-8, bainite nucleation is assumed to occur at austenite grain boundaries (type I), austenite/ferrite interfaces (type II), and within austenite grains (type III). Each of these nucleation sites has their own specific critical nucleation undercooling.

Experimental observations show that bainitic ferrite and its parent austenite always hold a KS crystallographic orientation relationship (Spanos 1996, Furuhara 2008). Thus, in this work, crystallographic orientations of bainite sheaves were related to the crystallographic orientations of the parent austenite grains. Furthermore, for the sake of simplicity, bainite sheaves were assumed to have a single orientation variant. As a result, when bainite sheaves form within a parent austenite grain, i.e. type III, all the sheaves acquire one single variant among 24 KS crystallographic variants with respect to that parent austenite grain. Getting the same crystallographic variant is not equivalent to having exactly the same crystallographic
orientation. It should be noted that in reality the crystallographic orientations do not perfectly match with the theoretical variants (Table 2-1) for all sheaves. Instead, there are some minor deviations from the perfect variants, which hinder their coalescence upon impingement. In simulations coalescence of bainite sheaves were avoided by toggling off the “categorization” option in MICRESS®.

It was assumed that when a bainite nucleus forms at the austenite grain boundary, type I, it acquires a KS compatible orientation relationship with the crystallographic orientation of only one of the austenite grains. This is consistent with the experimental observation of Furuhara et al. (Furuhara 2008) who found that bainite lath has a very close KS orientation relationship with one side of the grain boundary into which it grows and the opposite side, into which it does not grow, shows a significant deviation in orientation relationship. In the MATLAB subroutine, at each location along the PAGB, the crystallographic orientation of one of the parent austenite grains is selected using the random generator (Appendix E).

For type II nucleation at ferrite/austenite interfaces, experimental observations (e.g. Figure 2-2) reveal that there is only bainite formation on one side of the ferrite rim. That is attributed to the difference in the ferrite/austenite orientation relationships at two opposite sides of the ferrite rim. In order to incorporate this feature into the model, in the nucleation subroutine in MATLAB (Appendix E), type II nucleation is confined to take place only on one side of the ferrite rim with orientation compatibility (e.g. Figure 4-8b).

---

iii The difference between the crystallographic orientations of the bainite sheaves is so small that cannot be effectively distinguished in the schematic representation (Figure 4-8).
Figure 4-8- Schematic representation of the microstructure a) bainite nucleation type I, b) bainite nucleation type II, c) bainite nucleation type III

For any given case, the ferrite model in MICRESS® is run and stopped once the temperature of the system reaches the temperature at which bainite can form, i.e. \( (T_{\text{Ae3}} \textless \text{critical undercooling of bainite nucleation at PAGBs} \) for the given
chemical composition). At this stage, the austenitic-ferritic microstructure is exported from MICRESS® again to an ASCII file to be read by the nucleation subroutine in MATLAB for bainite nucleation. As presented in Figure 2-3, the average number of bainite sheaves per unit length of the microstructure perpendicular to the growth direction of bainite sheaves (inter-sheaf spacing) can be used to quantify the nucleation spacing for any given processing conditions. The subroutine translates the inter-sheaf spacing to shield distance, which defines the minimum allowed spacing of nuclei in the nucleation code of MICRESS®. At each nucleation site, nucleation only occurs if the critical undercooling is locally surpassed while the critical undercooling criterion (Eq. (4-22)) automatically accounts for the effects of local chemistry changes.

Bainite sheaves, usually, do not grow into the austenite grain with higher orientation deviation and stop growing when they impinge with a hard obstacle, such as a grain boundary (Spanos 1996, Furuhara 2008). Therefore, an “orientation compatibility dependent interface mobility effect” is included in the model. Here, it was assumed that a bainite sheaf at a PAGB in contact with two austenite grains grows 100 times slower into the austenite grain with incompatible orientation (\(\beta\gamma\text{NOR}\)) compared to the one with compatible orientation (\(\beta\gamma\text{OR}\)). The factor of 100 is sufficient to ensure negligible growth of bainite sheave in an austenite grain with incompatible crystallographic orientation, i.e.

\[
\frac{1}{100} \mu_o^{\beta\gamma\text{OR}} = \mu_o^{\beta\gamma\text{NOR}} \quad \text{(4-23)}
\]

Figure 4-9a schematically shows how this effect gives rise to a lower growth rate of the bainite sheaf into austenite grain 2 than into austenite grain 1.
In addition to the “orientation compatibility dependent interface mobility effect”, bainite grows under the influence of a strong anisotropy with respect to the crystallographic orientation of the parent austenite grain. The crystalline faceted anisotropy model (Steinbach 1995), which was originally developed for solid-liquid systems, is employed here to describe the anisotropic character of the bainite sheave growth in a 2D section. In a faceted anisotropic system, every local crystallographic orientation of the crystal has an individual growth rate. Therefore, some planes form the slow growing macroscopic dominant facets, whereas other planes form the fast growing facets that disappear during the evolution and form corners of the structure (e.g. Figure 4-9b). In the faceted anisotropy model (Steinbach 1995), surface energy and mobility depend on the angle of the interface normal to the crystallographic orientation ($\theta$) by

\[
\sigma^* = \sigma_0^* k_{st}^2 (k_{st}^2 \cos^2 \theta + \sin^2 \theta)^{-1.5} \tag{4-24}
\]

\[
\mu = \mu_{\text{eff}} \left( k_{kin} + \frac{1 - k_{kin}}{\kappa} \tanh \left( \frac{\kappa}{\tan \theta} \tan \theta \right) \right) \tag{4-25}
\]

where $\sigma^*$ is the interface stiffness that is defined as the sum of the interface energy and its second derivative with respect to the interface inclination. The sharpness of the facet is controlled by $\kappa$ and the anisotropy intensity is determined by $k_{st}$ and $k_{kin}$, static and kinetic anisotropy coefficients, respectively. Anisotropy coefficients have values between 0 and 1, where 0 means full intensity and 1 means no intensity of the anisotropy of the facet\textsuperscript{iv}. Because of the anisotropy effect, bainite

\textsuperscript{iv} Full intensity is equal to the $\sigma_0^*$ and $\mu_{\text{eff}}$ respectively whereas no intensity corresponds to 0.
sheaves residing in a parent austenite grain are assigned a preferred growth direction. Anisotropy coefficients determine the aspect ratio of the bainite sheaves. These parameters were used to match the aspect ratio of the bainite sheaves with the experimentally observed bainite morphology.

Figure 4-9- a) Schematic representation of the growth after type I nucleation and b) schematic 3D representation of the faceted growth of a crystal.

4.4 Fitting Procedure

The transformation model consists of 5 adjustable parameters for ferrite (Table 4-1) and 8 parameters for bainite (Table 4-2) that need to be tuned. These parameters will enable the model to describe the nucleation and growth stages.

Figure 4-10 shows the overall flow of the fitting procedure. Two experimental cases need to be chosen in which the variation of one parameter caused a change in the microstructure from a mainly ferritic microstructure (one case with a higher than 50% ferrite) to a mainly bainitic microstructure (one case with higher than 50% bainite). In the TRIP steel, the cooling rate causes the change in the microstructure
whereas in the linepipe steel, cooling rates fall within a narrow range pertinent in the HAZ, therefore the level of Nb in solid solution is the dominant variable that controls microstructural changes.

Figure 4-10- Flow chart of fitting procedure.
Table 4-1- List of fitting input parameters of ferrite.

<table>
<thead>
<tr>
<th>Nucleation</th>
<th>Growth</th>
</tr>
</thead>
<tbody>
<tr>
<td>1- Spacing of ferrite nuclei, $S_\alpha$</td>
<td>5- Mobility pre-exponential factor, $\mu_o$</td>
</tr>
<tr>
<td>2- Critical undercooling at triple junctions, $\Delta T_{TJ}$</td>
<td></td>
</tr>
<tr>
<td>3- Critical undercooling at PAGBs type a, $\Delta T_{GB_a}$</td>
<td></td>
</tr>
<tr>
<td>4- Nucleation range at PAGBs, $\Delta T_R$</td>
<td></td>
</tr>
</tbody>
</table>

Table 4-2- List of fitting input parameters of bainite.

<table>
<thead>
<tr>
<th>Nucleation</th>
<th>Growth</th>
</tr>
</thead>
<tbody>
<tr>
<td>6- Minimum spacing of nuclei, $S_\beta$</td>
<td>10- Mobility pre-exponential factor of incompatible orientation, $\mu_o^{BYNOR}$</td>
</tr>
<tr>
<td>7- Critical undercooling type I, $\Delta T_{\gamma\gamma}$</td>
<td>11- Interface sharpness, $\kappa$</td>
</tr>
<tr>
<td>8- Critical undercooling type II, $\Delta T_{\gamma\alpha}$</td>
<td></td>
</tr>
<tr>
<td>9- Critical undercooling type III, $\Delta T_{\gamma\beta}$</td>
<td>12- Anisotropy kinetic intensity, $k_{kin}$</td>
</tr>
<tr>
<td></td>
<td>13- Anisotropy static intensity, $k_{st}$</td>
</tr>
</tbody>
</table>

Figure 4-11 schematically shows the total fraction of transformation products (ferrite fraction plus bainite fraction) as a function of the transformation temperature (CCT curve). The critical driving force for the nucleation at triple points, $\Delta T_{TJ}$, was tuned in a fashion that the nucleation temperature of the triple points matches the experimental start temperature of the ferritic phase transformation, which can be obtained from the CCT curve. This is done by running the program with different values of critical undercooling and trying to match the 5% transformation temperature (Figure 4-11) with the temperature at which ferrite forms 5% of the microstructure for the case with a higher fraction of ferrite. As depicted in Table 4-1, $S_\alpha$ is the spacing of the equidistant ferrite nuclei along PAGBs and triple junctions. The case with higher fraction of ferrite was used to fit this parameter. The ferrite nuclei spacing was adjusted to replicate the experimental ferrite grain size in the linepipe.
steel and the experimental width of ferrite rim for the TRIP steel in which pronounced ferrite rims form along PAGBs.

Experimental transformation kinetics curve of the case with highest level of ferrite was also used to find the critical undercooling at PAGBs with highest level of misorientation ($\Delta T_{GB,a}$) and the nucleation range at PAGBs ($\Delta T_R$). As will be described in chapter 7, for the linepipe steel with a mainly uniform ferritic structure, for simplicity, $\Delta T_R$ was assumed to be zero. In the TRIP steel with partial ferrite fraction, $\Delta T_R$ was tuned to mimic the morphology of ferrite rims. It was assumed that nucleation fitting parameters ($S_\alpha$, $\Delta T_{Tf}$, $\Delta T_{GB,a}$ and $\Delta T_R$) are independent of processing variables (i.e. prior austenite grain size and level of Nb in solution for the linepipe steel and cooling rate for the TRIP steel) within the considered processing range.

Figure 4-11- Schematic representation of a CCT curve used for fitting.
In contrast to nucleation parameters, the pre-exponential factor of the ferrite/austenite interface mobility, \( \mu_o \), was assumed to be cooling rate dependent in the TRIP steel and dependent on the level of Nb in the linepipe steel. Values of this parameter at two extremes of cooling rate in TRIP steel and level of Nb in the linepipe steel were obtained by finding the best matches of the PFM kinetic curves with the experimental CCT curves of both cases at the mostly ferritic temperature ranges (Figure 4-11).

The critical undercooling of bainite nucleation at PAGBs, \( \Delta T_{\gamma/\gamma} \), was used to match the temperature at which type I bainite nuclei form with the experimental bainite nucleation temperature for the case with highest fraction of bainite. The minimum allowed spacing of bainite nuclei (\( S_\beta \)), the critical undercooling at austenite/ferrite grain boundaries (\( \Delta T_{\gamma/\alpha} \)), and the critical undercooling within austenite grains (\( \Delta T_{\gamma/\beta} \)) were determined by fitting the simulated and the experimental inter-sheaf spacing and the bainite fraction for the case with highest level of bainite.

Similar to ferrite it was assumed that bainite nucleation parameters are independent of the processing conditions and the pre-exponential factor of bainite/austenite interface mobility, \( \mu_o^{\beta\gamma^{NOR}} \), is the only parameter that depends on the cooling rate in the TRIP steel and the level of Nb in solution in the linepipe steel. The pre-exponential factor was adjusted to obtain the best matches between the simulated and experimental CCT curves of the mostly bainitic transformation range for both fitting cases (Figure 4-11). Bainite anisotropy input parameters (\( \kappa \), \( k_{kin} \) and \( k_{st} \)) were defined in accordance with experimental morphology of bainite sheaves (Appendix H).
During fitting of ferrite and bainite pre-exponential factors, the fit was graded by finding the optimum coefficient of determination (R-squared error) for the overall kinetics curve, along with obtaining the closest morphology parameters (ferrite grain size, ferrite fraction, bainite inter-sheaf spacing, and bainite fraction) to the experimental values. In summary, fitting procedure was aimed at obtaining the minimum acceptable deviation in terms of the fractions of microstructural products, morphology of products, and kinetics.
5 Experimental Work

The experimental tests included in this document were mainly supplied by Dongsheng Liu (Liu 2007) for CCT of the TRIP steel, Jennifer Reichert (Reichert 2012) for CCT of X80 steel and Michael Gaudet (Gaudet 2012) for welding of X80 steel. The experimental contribution of this work includes

- quantitative analysis of micrographs of Liu
- quantitative analysis of micrographs of Reichert
- preparation of the metallographic samples and quantitative analysis of welding samples of Gaudet

5.1 Materials

The TRIP steel studied in this work was laboratory cast steel with the chemistry shown in Table 5-1. The materials supplied were hot forged bars from ArcelorMittal Dofasco (Hamilton, ON).

| Table 5-1- Chemical composition of the TRIP steel (wt. %) |
|-----------------|-------|------|-------|-------|
| C | Mn | Si | Mo |
| 0.19 | 1.5 | 1.6 | 0.2 |

The linepipe steel used in this work was in the form of hot-rolled API 5L X80 pipes with wall thickness of 16.9mm supplied by Evraz Inc. NA (Regina, SK). Table 5-2 gives the chemical composition of the studied X80 steel. The starting microstructure of the X80 steel is composed of acicular ferrite and MA (Figure 5-1). As verified by
Maalekian et al. (Maalekian 2012), most of the micro alloying elements in the pipe material were precipitated during hot rolling. TEM-Energy-dispersive X-ray spectroscopy analysis of Banerjee et al. (Banerjee 2010) showed that the precipitates in the starting microstructure are mainly of three families: Nb-rich, Ti-rich and Mo-rich particles.

Table 5-2- Chemical composition of the X80 steel (wt. %)

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Mn</th>
<th>Nb</th>
<th>Mo</th>
<th>N</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.06</td>
<td>1.65</td>
<td>0.034</td>
<td>0.240</td>
<td>0.005</td>
<td>0.012</td>
</tr>
</tbody>
</table>

Figure 5-1- Optical micrograph of the starting microstructure of the X80 linepipe steel (Tafteh 2010)
5.2 Experimental Contribution

The main body of experimental work was available from other studies performed at UBC (Liu 2007, Reichert 2014, Gaudet 2012). To supplement the experimental data from literature required for the validation of the PFM, additional quantitative metallography techniques were performed. The experimental work undertaken for this thesis can be grouped into five measurements types:

- ferrite grain size
- ferrite area fraction
- bainite inter-sheaf spacing
- prior austenite grain size
- spacing of thermocouples form fusion line

Analyses were undertaken on micrographs prepared by Liu (Liu 2007) and Reichert (Reichert 2014) for CCT samples and on micrographs prepared in this work from Guadet’s welded samples (Gaudet 2012). For the welding samples, surface preparation was conducted on each sample including grinding with silicon carbide and polishing with diamond solutions. The samples were etched with 2 % Nital solution and examined with the Nikon EPIPHOT 300 series optical microscope. Image analysis of all the CCT and welding micrographs was conducted using the Clemex Vision PE image analyzing software and ImageJ open-source software (ImageJ 2015).
5.2.1 Quantitative Analysis of Ferrite

To measure the average diameter of ferrite grains one micrograph of welding samples and 3 micrographs of CCT samples were analyzed. The welding samples were taken at different locations in the HAZ.

Because of the difficulty of the recognition of ferrite grains at microstructures with very low ferrite fraction, only cases with more than 20% ferrite were analyzed. Ferrite grain boundaries were manually outlined on micrographs. Approximately 100 grains were examined for each sample. Micrographs were analyzed using the Clemex software. The EQAD was estimated in accordance with ASTM standard E 1382-97. Figure 5-2 shows a micrograph of a CCT sample along with the highlighted ferrite grain boundaries for image analysis.

Figure 5-2- An example of a micrograph for which ferrite grain size was measured a) Experimental micrograph and b) ferrite grain structure obtained through image analysis.
5.2.2 Quantitative Analysis of Bainite

An important parameter for the bainite transformation that required further analysis was the average inter-sheaf spacing\(^v\) as shown in Figure 2-3. Here the average inter-sheaf spacing of bainitic microstructure constituent was measured for all X80 and TRIP steel samples.

Inter-sheaf spacing measurements were undertaken on representative areas of the etched samples, and a selection of these is presented in Figure 5-3. The sheaf-shaped objects in the microstructure were marked manually on the micrographs. For each colony of sheaves, an identifier line was drawn on the micrograph perpendicular to the growth direction of sheaves. Identifier lines are indicated by the red lines (AB, CD, and EF) in Figure 5-3. Inter-sheaf spacing was calculated as the length of identifier lines divided by the number of sheaves intersecting that line. A total number of 50 sets of bainite sheaves in the final microstructure were investigated for each heat-treatment to ensure reasonable statistics for this evaluation.

\(^v\) The average number of bainite sheaves per unit length of the microstructure perpendicular to the growth direction of bainite sheaves.
Figure 5-3- Bainite inter-sheaf spacing measurement from the experimental micrographs (here, micrograph of the X80 linepipe).

5.2.3 Quantitative Analysis of Prior Austenite Grain Size

To determine the prior austenite grain sizes in the welded samples, 2% Nital etchant was used to highlight PAGBs. Microscopic examinations were carried out on regions that displayed mostly bainitic microstructures. Prior austenite grain boundaries were highlighted, and marked on the micrographs, and the EQAD and linear intercept were measured through image analysis of 50 grains using Clemex software.

5.2.4 Estimation of Temperature Profile

The thermal cycle experienced during welding is a practical example of continuous heating followed by continuous cooling heat treatment. In order to model
the microstructure evolution, the thermal profiles, \( T(t) \), need to be established for all positions in the HAZ.

In this work, the measured time-temperature data obtained from experimental welding trials of the X80 steel were analyzed and an empirical temperature fit was developed to describe the thermal profile along the HAZ. Due to the considerable level of noise in the measured data, a Savitzky-Golay smoothing filter was applied to data, using MATLAB (MATLAB 2015). In this approach each value of the series are replaced with a new value which is obtained from a polynomial fit to \( 2n+1 \) neighbouring points (including the point to be smoothed), with \( n \) being equal to, or greater than the order of the polynomial (Figure 5-4).

![Figure 5-4- Savitzky-Golay smoothing method.](image)

50 iterations of 4 and 2-degree polynomials were used for the Savitzky-Golay smoothing using 400 and 200 data points, respectively. For each smoothed thermal profile, the peak temperature was extracted and the relative location of the thermal profile, with respect to the fusion line, was measured from the micrographs. For these measurements, first, all samples were cut and repeatedly polished up to the point
where the mid-section of the thermocouple was revealed. The samples were then etched with 2 % Nital solution for 3-4 minutes until the boundaries of HAZ with fusion line and BM are delineated. Then measurements were undertaken using Clemex software. As Figure 5-5 shows, the thermocouple distance was measured from the middle section of the thermocouple tip to the closest point on the fusion line.

![Image](image.jpg)

Figure 5-5- The spatial distribution of peak temperatures were measured from the distance of thermocouple tip to the closest point on the fusion line.

5.3 Experimental Results

5.3.1 TRIP Steel CCT

For the TRIP steel, experimental data was mainly taken from the study of Liu et al. (Liu 2007). CCT tests were conducted by heating up the sample to different reheating temperatures to yield different initial austenite grain sizes and then cooling at different cooling rates. Table 5-3 depicts the heat treatment scenarios in terms of
reheating temperature, resulting austenite grain size, and subsequent cooling rate. The fractions of ferrite and bainite depicted in this table were obtained by image analysis as reported in (Fazeli 2012, Li 2007). In this work, for the verification of bainite PFM model, inter-sheaf spacing of bainite constituents were measured in accordance with the procedure described in the previous chapter.

Table 5-3- Selected heat treatment scenarios and experimentally measured microstructural data for the Mo TRIP steel.

<table>
<thead>
<tr>
<th>Case</th>
<th>EQAD</th>
<th>CR</th>
<th>Fs</th>
<th>( f_f )</th>
<th>Bs</th>
<th>( f_B )</th>
<th>Ms</th>
<th>ISS</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>24</td>
<td>1</td>
<td>755</td>
<td>0.55</td>
<td>597</td>
<td>0.33</td>
<td>285</td>
<td>2.4</td>
</tr>
<tr>
<td>2</td>
<td>24</td>
<td>5</td>
<td>725</td>
<td>0.23</td>
<td>616</td>
<td>0.53</td>
<td>370</td>
<td>1.9</td>
</tr>
<tr>
<td>3</td>
<td>34</td>
<td>1</td>
<td>740</td>
<td>0.43</td>
<td>590</td>
<td>0.41</td>
<td>300</td>
<td>2.5</td>
</tr>
<tr>
<td>4</td>
<td>34</td>
<td>5</td>
<td>740</td>
<td>0.08</td>
<td>618</td>
<td>0.66</td>
<td>375</td>
<td>2.7</td>
</tr>
<tr>
<td>5</td>
<td>52</td>
<td>1</td>
<td>750</td>
<td>0.35</td>
<td>604</td>
<td>0.53</td>
<td>315</td>
<td>2.7</td>
</tr>
<tr>
<td>6</td>
<td>52</td>
<td>5</td>
<td>720</td>
<td>0.11</td>
<td>621</td>
<td>0.74</td>
<td>360</td>
<td>3.2</td>
</tr>
</tbody>
</table>

EQAD: Prior austenite grain size (µm)
CR: Cooling rate (°C/s)
Fs: Ferrite start temperature (°C)
\( f_f \): Ferrite fraction
Bs: Bainite start temperature (°C)
\( f_B \): Bainite fraction
Ms: Martensite start temperature (°C)
ISS: Inter-sheaf spacing (µm)

All the data but the inter-sheaf spacing is taken from (Fazeli 2012).

5.3.2 Linepipe Steel CCT

The experimental grain growth study on the linepipe steel was conducted by Banerjee et al. (Banerjee 2010). They measured the austenite grain growth during continuous heating at rates of 10, 100 and 1000 °C/s to peak temperatures of 950,
1150 and 1350 °C using a Gleeble 3500 thermo-mechanical simulator. To reveal prior austenite grains, samples were quenched from the above peak temperatures and EQAD\textsuperscript{vi} were measured by metallography. In all cases, the residence time at peak was approximately 0.5 s (Banerjee 2010).

Austenite decomposition was studied in the linepipe steel by Reichert et al. (Reichert 2012) using a Gleeble 3500 thermo-mechanical simulator to represent CGHAZ and FGHAZ conditions in terms of austenite grain size and the amount of Nb in solution. Heat treatments consisted of two steps (Figure 5-6). The samples were first solutionized at 1300 °C to dissolve all Nb(CN), followed by a gas quench to ambient temperature. In the second step, samples were heated with a heating rate of 100 °C/s to 950 °C and 1250 °C, respectively, to obtain different austenite grain sizes while maintaining the Nb in solution. Then samples were cooled with a cooling rate of 180 °C/s to 900 °C, either after 20 minutes or after no holding time to fully re-precipitate Nb or keep Nb in solution, respectively. Then, starting from 900°C, the CCT tests were conducted using a cooling rate of 10 °C/s which is consistent with the average cooling rate observed in the transformation temperature range (800 – 500 °C) of the thermal profiles of the trailing torch in a linepipe weld (as will be shown in section 5.3.3). The CCT conditions of Reichert et al. are summarized in Table 5-4. The fractions of ferrite/bainite and MA in this table were obtained by image analysis of the micrographs of the samples etched with 2 % Nital and Le Pera solutions, respectively (Reichert 2012). Then additional quantitative metallographic analyses

\textsuperscript{vi}the diameter of a circle having an area equal to the mean area of the measured grains in experimental micrographs.
were conducted to measure the ferrite grain size and the bainite inter-sheaf spacing in the samples of Reichert et al. (Reichert 2012).

Table 5-4- Heat treatment scenarios representing potential combination of processing parameters (Reichert 2012).

<table>
<thead>
<tr>
<th>Case</th>
<th>Cooling rate (K/s)</th>
<th>Austenite EQAD (μm)</th>
<th>Fraction Nb Precipitated</th>
<th>MA Fraction</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10</td>
<td>5</td>
<td>1</td>
<td>0.06</td>
</tr>
<tr>
<td>2</td>
<td>10</td>
<td>26</td>
<td>0</td>
<td>0.13</td>
</tr>
<tr>
<td>3</td>
<td>10</td>
<td>80</td>
<td>1</td>
<td>0.14</td>
</tr>
<tr>
<td>4</td>
<td>10</td>
<td>26</td>
<td>1</td>
<td>0.06</td>
</tr>
</tbody>
</table>

Figure 5-6- Schematic representation of the heat treatments for CCT tests (Reichert 2012).

5.3.3 Linepipe Steel Welding

Welding trials on X80 grade pipes were conducted by Gaudet et al. (Gaudet 2012). Tests included single torch and dual torch welding using an automatic GMAW
robot at Evraz Inc. (Regina, SK). The dual torch weld procedure is a relatively new process that has a second welding torch that trails the lead torch at a set distance. Figure 5-7 schematically compares thermal cycles of a traditional single torch and a dual torch procedure. Direct temperature measurements were made by Gaudet et al. embedding thermocouples at different locations of the HAZ. In many welding trials excessive welding heat damaged thermocouple tips leading to unreliable data. Therefore, for the analysis of the HAZ, only 2 welding trials were selected for which the most reliable time-temperature data were recorded.

![Figure 5-7- Schematic thermal cycle curves from a) single and b) dual torch welds.](image)

Figure 5-7 shows the peak temperatures obtained from smoothed welding time-temperature curves versus the measured distances from the fusion line (Figure 5-5). From these measurements a linear spatial variation in the peak temperature along the HAZ was concluded such that

\[ T_p = 1538 - 0.48x \quad (5-1) \]

where \( x \) and \( T_p \) represent distance (\( \mu \text{m} \)) and peak temperature (\( ^\circ \text{C} \)), respectively.
An empirical fit was obtained based on the smoothed thermal profiles. As shown in Figure 5-9, extrapolation of the fit provides an estimate of the thermal profile along the HAZ. In Figure 5-9, the black dots represent the experimentally measured temperature history at a distance of 1050 µm from fusion line; and the pink curve shows the thermal profile obtained for the same location using the estimated thermal profile. This analysis was used in a MATLAB code (Appendix B) to find the temporal and spatial distribution of temperature during the weld thermal cycles required for PFM calculations of chapter 8.

![Graph showing peak temperatures and distances](image)

Figure 5-8- Distribution of the peak temperatures of the second peak along the HAZ for dual torch welding trails based on the experimentally measured distances of thermocouple tips from the fusion line (F.L.).

Although welded samples and initial time-temperature data were available from the experimental work of Gaudet et al. (Gaudet 2012), all the sample preparations, metallographic analyses, and noise filtering were conducted in this work. The results of microstructural analysis can be found in chapter 8.
Figure 5-9- Estimated thermal profiles for the trailing torch in dual torch welding at different positions in the HAZ including experimental data measured 1050 µm away from the fusion line.
6 Grain Growth

This chapter is devoted to the simulation of austenite grain growth during continuous heating tests of the X80 linepipe steel. As described in chapter 4, grain growth simulation includes numerical solution of Eq. (2-17). Here, austenite was assumed to be a non-textured structure, therefore, there is no driving force other than grain boundary curvature as described by Eq. (2-2). The grain boundary energy of austenite was set to a constant value of 0.5 J/m$^2$ (Zurob 2002). Mobility was considered to be an effective mobility that reflects the pinning effect of Nb(CN) particles and only depends on temperature.

6.1 Effective Mobility

The effective mobility of austenite grain growth was determined by analyzing the continuous heating data of Banerjee et al. as reported in (Banerjee 2010). In their work, austenite grain growth was measured during continuous heating at rates of 10, 100 and 1000 °C/s to peak temperatures of 950, 1150 and 1350 °C using a Gleeble 3500 thermo-mechanical simulator. To reveal prior austenite grains, samples were quenched from specific temperatures and $d_{2D}$ (equivalent area diameter: the diameter of a circle having an area equal to the mean area of the measured grains in experimental micrographs) were measured by metallography. In all the continuous heating experiments of Banerjee et al., the residence time at the peak temperature was approximately 0.5 s. In the case of 10°C/s heating rate, unlike the other cases, the residence time at peak is negligible compared to the heating time. Therefore, this case is a suitable experiment for analyzing grain growth during continuous heating.
Figure 6-1 shows the \( d_{2D} - T \) fit obtained by Banerjee et al. for the variation of the EQAD \( (d_{2D}) \) with temperature at a heating rate of 10 °C/s. This fit was obtained by a phenomenological description of grain growth as a function of Zener pinning where pinning parameters were considered as fitting parameters to replicate the experimentally measured grain size data. For details regarding this fit, please refer to (Banerjee 2010).

Figure 6-1- Empirical fit to experimental measurements of austenite grain growth during continuous heating at 10 K/s (Banerjee 2010).

Figure 6-2 shows the predicted variation of the fraction of Nb precipitated \( (f=\frac{f_{NbCN(total)}}{f_{eq, NbCN(total)}}) \) for the continuous heating test of Banerjee et al. According to Figure 6-2, the completion of the dissolution of small size Nb(CN) particles occurs very quickly at temperatures as low as 1300 K while the full dissolution of large particles does not occur until a temperature of 1590 K.
Figure 6-2- Predicted dissolution profile for the 10°C/s continuous heating test with a dwell time of 0.5s at peak temperature ($\dot{f}$ vs $1/T$)

Figure 6-3 shows the variations of the back-calculated effective mobility with temperature in an Arrhenius plot obtained using the procedure described in section 4.2.3. There are two distinct linear regimes at lower and higher temperatures corresponding to full dissolution of the particles and no dissolution. The mean austenite grain size increases moderately with temperature in the low temperature region, while it significantly increases in the high temperature region. This trend can be associated with the reduction in the pinning force exerted by the Nb(CN) precipitates due to partial or full dissolution of these precipitates at higher temperatures. At low temperatures, above $7.7\times10^{-4}$ $1/K$ ($1025$ °C), the low mobility branch reflects strong pinning effect of precipitates on austenite grain boundaries ($\dot{f}>0.76$). At high temperatures, below $6.3\times10^{-4}$ $1/K$ ($1315$°C), the high mobility branch is consistent with weak pinning corresponding to full dissolution of Nb
bearing particles ($\dot{\gamma} \sim 0$). TiN particles are responsible for the weak pinning effects at higher temperatures. In addition to pinning, the Nb solute drag effect decreases at higher temperatures as the segregation of Nb to the boundaries decreases.

Figure 6-3- Effective mobility values back calculated from the empirical grain growth kinetics fit.

In the temperature range of full dissolution, an activation energy of 260 kJ/mole was extracted from the slope of the Arrhenius plot of the back-calculated effective mobility data in accordance with Eq. (4-16). Mobility pre-exponential fit as a function of $\dot{\gamma}$ was obtained by the comparison of the back-calculated pre-exponential mobilities (data points at Figure 6-3) with the instantaneous fractions of Nb bearing precipitates from the dissolution profile (Figure 6-2). The best fit obtained for the thermal path of 10°C/s continuous heating starting from 950°C is given by

$$
\mu^0_{2D}(\dot{\gamma}) = 4.1\dot{\gamma}^2 - 4.3\dot{\gamma} + 1.8 \text{ (m}^4\text{J}^{-1}\text{s}^{-1})
$$
Figure 6-3 compares the mobility fit obtained through this procedure with the back-calculated mobility values.

### 6.2 2D Grain Growth

Simulations were performed on a regular square grid in 2D with a domain size of 250 µm × 250 µm, employing periodic boundary conditions. For the X80 steel, at 950 °C, an average volumetric austenite grain size of approximately 5 µm was measured independently of heating rate (Banerjee 2010). Therefore, as the initial condition, 3850 austenite grains were introduced into the calculation domain to give an initial EQAD of 4.8 µm for all phase field simulations. The initial grain structure was generated by a Voronoi construction that is built in MICRESS®. Voronoi leads to a polygonal grain structure with flat grain boundaries. 25 primary iterations for initialization were applied to convert the Voronoi structure to a topologically reasonable grain structure. The calculations were performed with a grid size of 0.2 µm and a grain boundary width of 0.8 µm. The starting grain size and distribution were kept the same for all the simulations. The numerical convergence of grain growth simulations has been confirmed by a set of calculations that are described in Appendix F.

As shown in Figure 6-4, the phase field simulations quantitatively replicate the grain growth kinetics during continuous heating. Figure 6-5 compares the optical micrographs of the austenite grains with PFM results at different heating rates. Predictions lie within the error range of experimental measurements of the grain size.
that supports the good agreement between the simulated and observed grain structures.

Figure 6-4- Simulated and measured grain growth kinetics during continuous heating to 950, 1150 and 1350 °C and holding for 0.5 s at different peak temperatures. Error bars show the range of grain sizes measured from the micrographs.
Figure 6-5 - Comparison of PFM results with experimental micrograph for continuous heating tests up to peak temperature of 1350 °C and heating rates of 10 K/s (a, b) and 1000 K/s (c, d). Measured average austenite grain size from experiments: a) 51±8 μm and c) 27±4 μm and simulations: b) 65 μm and d) 21 μm.

6.3 3D Grain Growth

In a set of 3D simulations, the proposed effective mobility approach was used for continuous heating and cooling conditions in bulk samples. Simulations were started with a 29 μm × 29 μm × 29 μm domain employing periodic boundary conditions. As an initial condition, 200 austenite grains were introduced into the calculation domain by a Voronoi construction to give an initial 3D average grain diameter of 6.2 μm that corresponds to an EQAD of 5.1 μm that replicates the experimental observations (Banerjee 2010). The calculations were performed with an initial grid size of 0.1 μm and a grain boundary width of 0.6 μm. As stability calculations presented in Appendix
G show, the simulation results in 3D are numerically stable when there are more than 5 grid points within the interface.

For proper statistical analysis, it is required to maintain at least 70 grains in the domain (Appendix G). To keep a sufficient number of grains in the domain, simulations were repeatedly interrupted to rescale the domain size, number of grains and grid size. After each interruption a Voronoi construction, in addition to 25 initialization iterations, were used to generate the initial microstructure for the next step with an average grain size replicating that of the previous step. This technique effectively reduces the numerical cost of the 3D simulations while maintaining a statistically significant number of grains in the calculation domain. Appendix G presents the considerations taken into account in rescaling with regards to grain size distribution and also provides an example to test the reliability of rescaling. In order to compare the 3D simulation results with the experimental data, and the previous 2D simulations, the EQAD was extracted from the 3D simulation by making 2D cuts through the 3D simulation data. To have good statistics in determining the average EQAD, 29 equally spaced 2D cuts were analyzed for selected times. Figure 6-6 compares the 3D simulation results in terms of EQAD with the calculated kinetics of 2D grain growth and experimental measurements in samples subjected to continuous heating and cooling. The EQAD values measured from sections of 3D phase field calculations are very close to the calculated 2D grain sizes, with less than 5% difference, when a mobility conversion factor of 0.7, Eq. (4-21), is used. In Figure 6-7 and Figure 6-8, the same comparisons are made by showing the final austenite micrographs revealed by metallography and microstructures obtained in 2D and a
representative 2D cross section of the 3D simulation. Both 2D and 3D simulation results are in good agreement with the experimental observations.

Figure 6-9 shows the variation of $\beta$ for 3D grain growth simulations at different scales. The simulations confirm that $\beta$ is equal to 1.2 for the ratio of average volumetric grain size and EQAD as illustrated in Figure 6-9. The average value of $\beta$ in the simulations is approximately 1.23 and $\beta$ asymptotically approaches 1.2. The initial values for each calculation are in the range 1.24 – 1.27 and these slightly increased $\beta$ values result from the grain geometries generated by using a Voronoi construction. The phase field calculations then predict a change in grain topology that is consistent with experimental observations, i.e. $\beta$ approaches 1.2.
For the investigated bulk grain growth with short growth time (a few seconds), 2D and 3D simulations give the same results by introducing an appropriate conversion factor for the effective mobilities. However, such a simple translation of 2D simulations into 3D predictions is in general not possible, e.g. when pinning particles must be explicitly considered in the simulations.
Figure 6-8- Experimentally observed (a) (Banerjee 2010), 2D simulated (b) and 3D simulated (c) grain size structures for the sample heated at 1000 K/s, held for 0.5 s at peak temperature of 1150 °C (c, d and e) and cooled at 100 K/s.

The present simulations emphasize rapid heating and cooling scenarios that replicate thermal cycles in the HAZ. Reduction in pinning pressure due to dissolution of NbC precipitates has been taken into account using the concept of an effective mobility. In essence, if this simulation method was applied to an isothermal holding it would predict parabolic grain growth in contrast of attaining a limiting grain size that is observed for grain growth under the presence of pinning particles. Therefore, the proposed effective mobility scenario is only valid for short grain growth periods where deviations from parabolic grain growth are not significant.
Figure 6-9 - Variation of $\beta$ with number of grains in the simulation domain at the various simulation stages with different grid sizes, $dx$, for the grain growth case shown in Figure 6-6a. Rescaling was conducted by changing the grid size. Simulations yield a mean value of $\beta = 1.23$.

In summary, 3D phase field simulations have been carried out for austenite grain growth during rapid heating and cooling. The 3D simulations are in agreement with 2D simulations by employing a conversion factor of 0.7 between 3D and 2D mobilities. The simulation results are in good agreement with experimental data in bulk samples.
7 Austenite Decomposition

This chapter is devoted to analyze the austenite decomposition into ferrite and bainite. As mentioned in section 4.3, these simulations require numerical solution of the phase field (Eq. (2-17)) and diffusion (Eq. (2-19)) equations. The model is applied to CCT tests of the TRIP steel and the X80 linepipe steel. Periodic boundary conditions were applied for simulations because bulk samples experience negligible thermal gradient during CCT tests.

The model consists of a set of input parameters. One portion of input parameters, including interface energies and activation energies for interface mobility and diffusivities of carbon in austenite and ferrite/bainite, were taken from the literature. Ferrite and bainite interfaces with austenite were assumed to have identical interface energies and interface mobility activation energies. Interface energy was set to 0.5 J/m² which is a typical value taken from an earlier study (Mecozzi 2008). The activation energy of interface mobility was set equal to 140 kJ/mol, which was the value found by Krielaart and Van der Zwaag for binary Fe–Mn alloys (Krielaart 1998). It was assumed that the diffusivity of carbon in bainite is identical to ferrite. The diffusivities of carbon in the austenite and ferrite were calculated as the function of chemistries using the open-source subroutine of Babu et al. vii (Babu 1995). The rest of the input parameters were subjected to a fitting process as described in section 4.4.

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viihttp://www.msm.cam.ac.uk/map/steel/subs/diffus-b.html (June 2015)
7.1 Application to the TRIP Steel

The austenite decomposition model was used to simulate the CCT tests conducted by Liu et al (Liu 2007) on TRIP steel. In this experimental work, samples were subjected to different reheating temperatures, which led to different initial austenite grain sizes and then cooled at different cooling rates (Table 5-3).

Calculations started with a fully homogenous austenitic structure as the initial condition at a temperature of 1058 K. This temperature is the highest experimentally observed ferrite start temperature in the study of Liu et al (Liu 2007). As mentioned in section 4.3.1, for the sake of simplicity, austenite grains are assigned orientation angles. Orientation angles of austenite grains were evenly chosen between 0 and 180 degrees. Three domain sizes of (36.8 μm)$^2$, (52.2 μm)$^2$ and (79.6 μm)$^2$ were considered to correspond to the initial austenite grain sizes, EQAD, of 24 μm, 34 μm and 52 μm (Table 5-3). In accordance with the convergence analysis (refer to Appendix H) a grid size of 0.18 μm was chosen for each domain. Larger domains, (52.2 μm)$^2$ and (79.6 μm)$^2$, were created by magnifying the (36.8 μm)$^2$ structure by using more nodes while keeping the grid size constant. During continuous cooling, both ferritic and bainitic transformations progress and contribute to the total fraction of the transformation products, therefore ferrite and bainite models were running during the entire transformation temperature range.

7.1.1 Fitting Procedure Results

Ferritic and bainitic transformation parameters were fit using the procedure described in section 4.4. Case 1 (950 °C reheating temperature with 1 °C/s cooling
rate) and Case 6 (1100 °C reheating temperature with 5 °C/s cooling rate) were chosen as the two fitting heat-treatment conditions yielding the highest fractions of ferrite and bainite constituents, respectively (see Table 5-3).

Spacing of the equidistant ferrite nuclei along PAGBs, $S_\alpha$ in Table 4-1, was adjusted to obtain the closest ferrite morphology to Case 1 in terms of the average width of the ferrite rims. In cases with relatively larger prior austenite grain sizes and higher cooling rates, e.g. Case 6, coalescence is limited; therefore, $S_\alpha$ determines the final number density of ferrite grains. In contrast, at relatively smaller austenite grain sizes and lower cooling rates, e.g. Case 1, the total number density of ferrite grains in the final PFM microstructure does not vary with variation of $S_\alpha$. In that instance, no matter how many ferrite nuclei form along a PAGB, all the nuclei coalesce into one elongated ferrite rim and $S_\alpha$ only changes the width of the rim. For simplicity, it was assumed that the spacing of ferrite nuclei is the same for the entire range of processing conditions included in Table 5-3.

Critical nucleation undercooling, $\Delta T_{\text{J}}$ and $\Delta T_{\text{GB,a}}$, were determined in accordance with the transformation start temperature obtained from the experimental transformation kinetics curve in Case 1 (see Table 7-1). Nucleation range at PAGBs, $\Delta T_R$, was adjusted to match the experimental ferrite morphology in Case 6 consisting of coalesced and non-coalesced ferrite grains. Similar to the approach of Mecozzi et al. (Mecozzi 2008), the pre-exponential factor of the ferrite/austenite interface, $\mu_0$, was assumed to depend on the cooling rate. This parameter was determined by finding the best match between PFM and experimental CCT curves at different cooling rates for the ferritic transformation ranges of Cases 1 and 6.
Experimentally measured inter-sheaf spacing values in Table 5-3 imply that for the entire given range of processing conditions inter-sheaf spacing stays almost invariant with a value of 2.7 µm. Generally, the average sheaf width decreases with bainite start temperature and increases with the carbon content in the austenite phase (Bhadeshia 2001). The apparent independence of the inter-sheaf spacing from the heat treatment conditions can be attributed to the opposite trends of these two effects. Therefore, for the envisaged range of heat-treatment conditions, a minimum allowed bainite nuclei spacing was chosen which leads to an inter-sheaf spacing of 2.7 µm.

\[ \Delta T_{\gamma/\gamma}, \Delta T_{\gamma/\alpha}, \text{and} \Delta T_{\gamma/\beta} \] (critical undercooling for bainite nucleation at PAGBs austenite/ferrite grain boundaries and within austenite grains) were set in accordance with the experimental CCT kinetic data in the bainite formation range of Case 6. The resulting nucleation parameters are summarized in Table 7-1. Similar to ferrite, \( \mu_o^{\beta_NOR} \) (pre-exponential factor of bainite/austenite interface mobility) was assumed to be cooling-rate dependent and it was used to replicate the experimental kinetic data for Cases 1 and 6 in the bainitic transformation ranges.

Table 7-1- Critical undercooling describing ferrite and bainite nucleation (K) in the TRIP steel

<table>
<thead>
<tr>
<th>Ferrite ( \Delta T_{T1} )</th>
<th>Ferrite ( \Delta T_{GB_a} )</th>
<th>Ferrite ( \Delta T_{GB_b} )</th>
<th>Ferrite ( \Delta T_{GB_c} )</th>
<th>Bainite ( \Delta T_{\gamma\gamma} )</th>
<th>Bainite ( \Delta T_{\gamma\alpha} )</th>
<th>Bainite ( \Delta T_{\gamma\beta} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>70</td>
<td>105</td>
<td>140</td>
<td>150</td>
<td>160</td>
<td>205</td>
</tr>
</tbody>
</table>
The anisotropy parameters $\kappa$, $k_{kin}$ and $k_{st}$ were obtained by finding the best match between PFM sheaf morphologies as represented in Appendix H and the experimentally observed morphology of bainite sheaves. The model response to the input parameters shows a spectrum where anisotropy parameters lead to unrealistically narrow sheave morphology at one end and unrealistically broad morphology at the other end. Choosing a set of parameters (Table 7-2) in between these two extremes yields a morphology that compares well with the experimental bainite sheaf morphologies.

Figure 7-1 shows the best match obtained between experimental and PFM CCT kinetic curves for the fitting cases (1 and 6). Here, the variation of the total fractions of transformation products (ferrite + bainite) is plotted as a function of transformation temperature. It should be noted, that because martensite was not included in the total fraction, none of these curves ended up with a total overall fraction of 1 at lower temperatures.

<table>
<thead>
<tr>
<th>$k_{st}$</th>
<th>$k_{kin}$</th>
<th>$\kappa$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.04</td>
<td>0.04</td>
<td>1.0</td>
</tr>
</tbody>
</table>

Table 7-2- Bainite faceted anisotropy parameters.

Figure 7-2 shows the obtained pre-exponential factors of ferrite and bainite as a function of the cooling rate. Increasing the cooling rate from 1 to 5 °C/s results in the pre-exponential factor increasing from 5.7 to 11.0 cm$^4$//(Js) for ferritic transformation and from 90 to 370 cm$^4$//(Js) for bainitic transformation, respectively. The apparent
cooling rate dependence of the pre-exponential factor is associated with the solute-drag effect of Mn and Si atoms that segregate at the moving interfaces. This dependence was also observed in earlier PFM studies (Mecozzi 2005b, Mecozzi 2008).

Figure 7-3 compares the PFM micrographs for these two fitting cases with the experimental micrographs. In this figure, in order to capture a reasonable picture of the variations in the microstructure based on the definition of periodic boundary conditions, 4 identical simulated microstructures were stitched together to be compared with 4 times larger experimental micrographs. It is worth mentioning that in the PFM micrographs the red areas correspond to untransformed austenite. In this TRIP steel, the experimental studies of Liu et al. (Liu 2007) showed that during the late stages of the CCT tests all the retained austenite transforms into martensite. Since the proposed PFM does not account for the martensitic transformation, all the red regions can be interpreted as martensite regions in the final microstructure. From these representations, it is evident that the model gives an acceptable description of the morphological features of ferrite and bainite and these results, hence, prove the capability of the PFM in capturing the kinetics and the microstructural features of the phase transformations in the studied material. In addition, PFM simulations for ferrite and bainite fractions are in good agreement with experimental data (Table 5-3), i.e. 61% and 19% versus 55% and 33% in case 1 and 10% and 62% versus 11% and 74% in case 6 (Figure 7-3).
Figure 7-1- Experimental kinetics data for Case 1 and Case 6 along with the PFM kinetics obtained from fitting.

Figure 7-2- Linear interpolation of the mobility pre-exponential factor as a function of cooling rate.
Figure 7-3- Representative predicted microstructures of austenite decomposition modelling fitting. a,b) Case1, c,d) Case6. Red, yellow and white regions represent austenite (which eventually transform to martensite), ferrite and bainite, respectively. Figure b is obtained through stitching 4 identical simulated microstructures together while the actual simulation domain is outlined with dotted square.
7.1.2 Results

In order to test the predictive capability of the model, the set of parameters obtained by fitting were used, without any further adjustment, to predict the phase transformations in the rest of the cases from Table 5-3.

Figure 7-4 shows predicted and experimentally measured total fraction of ferrite and bainite as a function of transformation temperature. It is found that Cases 2, 3, 4 and 5 give coefficients of determination of 0.96, 0.93, 0.96 and 0.97, respectively which implies that the simulated kinetics agree reasonably well with the experimental kinetics.

Figure 7-5 compares the predicted microstructures with the experimental micrographs. Here, Figure 7-5 b, d and f are obtained through stitching 4 identical simulated microstructures together, while the actual simulation domain is outlined with a purple square. An in-depth look at Figure 7-5 shows that in both the simulation and experiment, ferrite forms long rims along PAGBs. In addition, not all the rims are of the same size and there is considerable variation in the width of the ferrite rims. The morphology of bainite, in both the simulation and experiment represents a single variant needle-like structure. The main deviations of the ferritic structure from experiment are dog bone shaped features (indicated by a black arrow in Figure 7-5f) and small ferrite islands (indicated by green arrow in Figure 7-5f).

Dog bone shaped ferrite morphology was formed because carbon was trapped at the intersection of two growing ferrite grains. If fast diffusion paths along the phase boundaries were taken into account, carbon could easily diffuse away through the interphase boundaries leading to a ferrite rim with a smoother structure.
Figure 7-4- Experimental data obtained from (Liu, Fazeli & Militzer 2007) and PFM calculations for continuous cooling transformation at different combinations of austenite grain size and cooling rate. Coefficients of determination of 0.96, 0.93, 0.96 and 0.97 were obtained for Cases 2, 3, 4 and 5.

Small ferrite islands along PAGBs in the microstructure are associated with ferrite nucleation along low misorientation PAGBs at later stages of transformation. At the time when these nuclei formed, the temperature was far too low to allow ferrite nuclei to grow to a considerable extent. Very low carbon diffusion rates freeze the partially grown ferrite nuclei, i.e. neither let them grow nor vanish. Considering the fact that these tiny ferrite grains have a high surface to volume ratio, these artefacts can also be removed by carbon diffusion through interphase boundaries (fast diffusion routes), which would lead to the dissolution of small ferrite nuclei and coarsening of larger ones.
Figure 7-5- Representative predicted microstructures of austenite decomposition modelling. a,b) Case2, c,d) Case3, e,f) Case4, g,h) Case5. Red, yellow and white regions represent austenite (which eventually transform to martensite), ferrite and bainite, respectively.
Figure 7-6 shows fractions of microstructural constituents extracted from PFM along with fractions obtained from image analysis of experimental micrographs (Fazeli 2012). This comparison shows that the proposed overall transformation model provides a tool to predict not only the morphology and relative kinetics of ferrite and bainite transformation in the studied material but also the relative fractions of the microstructural constituents.

Figure 7-6 - Calculated and measured fractions of transformation products for all the processing conditions of the TRIP steel.

Figure 7-7 quantitatively compares the measured and simulated number density of ferrite grains for microstructures with higher than 20% ferrite constituent (cases 1, 2, 3, and 5). Because quantitative analysis of microstructures, with less than 20%
ferrite, does not lead to reliable results cases 4 and 6 were excluded. Experimental numbers were obtained from the measured average final ferrite grain size, EQAD, which was given by (Liu 2007):

$$d_\alpha = \left\{ f_F \exp \left( 7.85 \times d_\gamma^{0.11} - \frac{3610}{T_s} \right) \right\}^{1/3} \mu m$$  \hspace{1cm} (7-1)

where $d_\gamma$ is the prior austenite volumetric grain size in μm, $f_F$ is the measured ferrite fraction in the final microstructure, and $T_s$ is the transformation start temperature in Kelvin (Table 5-3).

![Figure 7-7](image.png)

Figure 7-7- Expected number of ferrite grains in the simulation domain and the model predictions.

At first glance, the predicted total number of ferrite grains (light grey bars) appears to be far from the measured value (black bars) in Case 2. In addition, comparison of experimental results for case 1 and 2 shows a counterintuitive trend;
at higher cooling rate the number density of ferrite grains is smaller. These issues can be seen from the following aspects:

1. The total count of simulated ferrite grains was higher at cooling rate of 5°C/s (case 2) than cooling rate of 1°C/s (case 1) because the total number of nuclei activated and formed was proportionally higher. Considering that at higher cooling rates more nucleation sites are activated, it can be inferred that ferrite coarsening was pronounced and this cancelled out the higher nucleation rate effect. Elongated ferrite rims in experimental micrographs (Figure 7-5) may be interpreted as the sign of considerable ferrite coarsening. Although Figure 7-5b also shows that ferrite coarsening in the form of coalescence occurred to a considerable degree (denoted by a black arrow), the fact that the model does not account for the fast diffusion paths along the PAGBs limits the extent of coarsening; leaving isolated ferrite islands (denoted by green arrow).

2. If microstructural features smaller than 2μm are filtered out from the analysis of Figure 7-7, the agreement between PFM (dark grey bars) and experiment (balk bars) is significantly improved. This may imply that the absence of small features, potentially missed during experimental image analysis, has intensified the discrepancies observed between PFM and experimental results.

These discussions demonstrate that the presented austenite decomposition model is a reasonable first approach to obtain a satisfactory quantitative description of the simultaneous formation of ferrite and bainite phases.
In order to examine the degree to which simulation outputs are affected by input parameters a sensitivity analysis was conducted. As shown in Figure 7-8, final ferrite and bainite fractions as main outputs of the model were calculated for 5 equally spaced values of selected input parameters. In this sensitivity analysis, Case 4 from Table 5-3 with the simulation conditions shown in Figure 7-5f was taken as the reference case.

In order to demonstrate the relative effect of each parameter on the model response, sensitivity was described as a dimensionless parameter

\[ \text{Sensitivity} = \frac{\Delta F}{\frac{F_m}{\Delta x}} \]  

(7-2)

Here $\Delta F$ is the total fraction of ferrite/bainite variation, $\Delta x$ is the input parameter variation, $F_m$ is the fraction of ferrite/bainite of the reference case, and $x_m$ is the input parameter of the reference case. Figure 7-9 compares the sensitivity of the ferrite and bainite fractions to the input parameters. As Figure 7-9 shows, the ferrite fraction only depends on ferrite parameters and it is relatively independent of bainite parameters, whereas bainite fraction has a comparable sensitivity to both ferrite and bainite parameters. This can be associated with the fact that in TRIP steels bainite transformation does not commence until ferrite transformation has progressed to a considerable extent. Therefore, bainitic transformation is strongly affected by the carbon enrichment in parent austenite due to ferrite formation. On the other hand, ferritic transformation is mostly affected by bainite nucleation since formation of bainite nuclei will quickly lead to the end of ferritic transformation.
Figure 7-8- The dependence of final fractions of ferrite and bainite constituents on the parameters for case 4 from Table 5 3: a) Ferrite Nucleation Spacing, b) Bainite Nucleation Spacing, c) Ferrite Pre-exponential Factor, d) Bainite Pre-exponential Factor, e) Grid Size and f) Bainite Kinetic Anisotropy Factor.
Figure 7-9 - Sensitivity of final ferrite and bainite fractions to simulation input parameters.

In short these results show that ferrite fraction only depends on ferrite parameters and it is relatively independent of bainite parameters, whereas bainite fraction has a comparable sensitivity to both ferrite and bainite parameters. Furthermore, model shows minimal sensitivity to grid size, which implies the numerical stability of the model in the range relevant to these calculations.

7.2 Application to Nb-Ti Microalloyed Steel

After applying the austenite decomposition model to a TRIP steel that exhibits a carbide-free bainite structure, the model was then applied to the complex microstructure in the linepipe steel consisting of conventional bainite and quasi-polygonal ferrite where the formation of the ferrite rim is not obvious.
7.2.1 Fitting Process

In the application of the PFM to the microalloyed steel, the significant retardation of the austenite decomposition due to Nb solute drag cannot be neglected. Therefore, the extent of Nb(CN) dissolution was predicted and the model parameters for the two extreme conditions, i.e. when Nb is completely in solution and when Nb is present as precipitates were determined. In order to find the model parameters for these two extreme conditions, CCT tests conducted by Reichert et al. (Reichert 2012) were simulated (Table 5-4). Because the final aim of this work is to predict the microstructural evolution in the HAZ, Case 1 and 2 heat-treatments (Table 5-4) were selected for fitting which represent the CGHAZ and the FGHAZ conditions in terms of austenite grain size, the amount of Nb in solution and cooling rate. The cooling rate of 10 °C/s in these heat treatments is consistent with the average cooling rate observed in the transformation temperature range (800 – 500°C) of the thermal profiles in the HAZ (Figure 5-9).

Two domains of 52μm² were used for the PFM calculations of these cases. In the starting microstructures, austenite grains were randomly assigned orientations; and the grid spacing was 0.184 μm. For the purpose of ferrite nucleation fitting, the case with higher fraction of ferrite, Case 1 (prior austenite grain size of 5μm, Nb precipitated and cooling rate of 10 °C/s) was used. The minimum allowed spacing of ferrite nuclei along PAGBs and triple junctions (Sα) was found by replicating the total number density of ferrite grains of Case1 where a ferrite nuclei density of 0.01 μm⁻² was measured. The ferrite grain size distribution is rather narrow such that it can be assumed that nucleation undercooling at all PAGBs is independent of their
misorientation, i.e. the same critical undercooling was applied for the nucleation of ferrite at all the PAGBs ($\Delta T_R=0$). Because $\Delta T_R$ was assumed to be zero, $\Delta T_{TJ}$ and $\Delta T_{GB,a}$ represent the critical undercooling at triple junctions ($\Delta T_{TJ}$) and PAGBs ($\Delta T_{GB,a}$), respectively. These parameters were determined from the experimental transformation kinetic curve of Case 1 in a similar fashion to what was done for the TRIP steel. Prior to PAGB nucleation, ferrite nucleates at triple junctions that lead to a non-uniform distribution of carbon concentration along PAGBs. Considering that nucleation temperature strongly depends on the local carbon concentration, although $\Delta T_R$ was assumed to be zero, all the nucleation sites along PAGBs will not commence at the same temperature.

In contrast to the TRIP steel, the X80 steel was only studied at a range of processing conditions applicable to the HAZ, therefore the dependence of the pre-exponential factor of the ferrite/austenite interface mobility, $\mu_o$, on cooling rate was neglected. Instead, to replicate the significant impact of Nb solute drag on ferritic phase transformation, $\mu_o$ was related to the level of Nb in solution. As described in section 4.4, $\mu_o$ was used as a fitting parameter to replicate the ferritic transformation range of the experimental CCT curve for case 1. For case 2, the fraction of ferrite was negligible and the pre-exponential mobility term was set to the maximal value that yields a ferrite fraction less than 0.01.

Case 2, with more than 85% bainitic microstructural constituent, was used to find bainite nucleation parameters. The spacing of the bainite nuclei, $S_B$, was estimated by finding the value that resulted in a bainite morphology with an average bainite inter-sheaf spacing of 4.6 $\mu$m as measured for Case 2. Values of critical undercooling of
bainite nucleation at PAGBs ($\Delta T_{\gamma/\alpha}$), austenite/ferrite inter-phase boundaries ($\Delta T_{\gamma/\alpha}$) and within austenite grains ($\Delta T_{\gamma/\beta}$) were found by fitting the portion of the mainly bainitic transformation range of the PFM kinetic curve to the experimental CCT curve of Case 2. Table 7-3 summarizes the adjustable nucleation parameters obtained from fitting. Bainite anisotropy input parameters ($\kappa, k_{\text{kin}}$ and $k_{\text{st}}$) were assumed to be the same as those of the TRIP steel (Table 7-2). These parameters were assumed to be constant for the range of processing conditions studied in this work.

Similar to ferrite, the pre-exponential factor of the bainite/austenite interface mobility, $\mu_0^{\beta/\gamma\text{NOR}}$, was defined as a function of the level of Nb in solid solution. This parameter was used to find the best match between the PFM and experimental CCT curves of Case 1 and 2 in the bainitic transformation ranges.

Table 7-3- Critical undercooling describing ferrite and bainite nucleation (K) in the X80 steel

<table>
<thead>
<tr>
<th>Ferrite $\Delta T_{\gamma}$</th>
<th>Ferrite $\Delta T_R$</th>
<th>Ferrite $\Delta T_{\text{GB,a}}$</th>
<th>Bainite $\Delta T_{\gamma\gamma}$</th>
<th>Bainite $\Delta T_{\gamma\alpha}$</th>
<th>Bainite $\Delta T_{\gamma\beta}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>0</td>
<td>40</td>
<td>130</td>
<td>160</td>
<td>160</td>
</tr>
</tbody>
</table>

Figure 7-10 shows the fitted values of ferrite/austenite and bainite/austenite mobility pre-exponential factors as a function of the level of Nb precipitated. Dissolution of Nb bearing precipitates (higher level of Nb in solid solution) leads to higher level of Nb solute drag, which results in a decrease in the mobility of ferrite/austenite and bainite/austenite interfaces. The retardation effect of Nb solute drag on austenite decomposition has been widely observed in experimental studies (Fossaert 1995, Reichert 2012). In contrast to austenite decomposition, the effective
austenite grain boundary mobilities increase by dissolution of Nb bearing particles as
the reduced pinning leads to higher rates of grain growth (Figure 6-3),

As Figure 7-2 and Figure 7-10 show, this modelling work indicates that the
interface mobility for bainite formation is much higher than that of ferrite formation.
The interface mobility for bainite is 2 orders of magnitude larger than that for ferrite
in both the TRIP and linepipe steels. Considering the fact that the excessive energy of
bainite due to formation of dislocations was neglected in this work, these results show
that at least in the range of materials studied in this work, bainite forms because of
the kinetic advantage over ferrite.

Figure 7-10- Linear interpolation of the mobility pre-exponential factor as a function the
fraction Nb precipitated.

Figure 7-11 shows the best fit obtained for the CCT curves of the two cases. These
curves represent the total fraction of austenite decomposition products (ferrite +
bainite) as a function of temperature. The temperature range in this figure does not encompass the martensite start temperature, i.e. phase transformations were incomplete (fraction transformed < 1). The data reported by Reichert (Reichert 2012) spans from 0 to 1 in that temperature range. Therefore, to account for the experimentally measured MA fractions of 0.06 and 0.12, the predicted fraction transformed for case 1 and 2 were corrected by multiplying by 0.94 and 0.88, respectively.

Figure 7-11- Experimental data and PFM calculations for continuous cooling transformation at 10 °C/s for two initial conditions: Case 1: Austenite grain size of 5 µm and Nb precipitated, Case 2: Austenite grain size of 26 µm and Nb in solution.

Figure 7-12 and Figure 7-13 compare the final microstructures obtained by PFM with corresponding experimental micrographs for Cases 1 and 2, respectively. In the PFM micrographs, white, gray, and back regions show bainite sheaves, ferrite grains, and MA, respectively. Experimental micrographs were obtained by Nital etching of
the samples. In Case 1, Figure 7-12a, because of the small prior austenite grain size resulting in a very large quantity of triple junctions, ferrite nucleation has only occurred at triple junctions and no nuclei have formed on the PAGBs. Therefore, no two ferrite grains had identical crystallographic orientations and coalesced. However, because of the abundance of triple junctions, nucleation has only occurred at 14.8% of all the available triple junctions.

Figure 7-12- Representative microstructures of Case 1: a) PFM microstructure, b) Experimental micrograph using Nital etchant (Reichert 2012). Figure a was obtained through stitching 4 identical simulated microstructures together.

Figure 7-14 compares the resultant final fractions of microstructural constituents of the fitting cases. As shown in Figure 7-10, the Nb in solid solution exerts a solute drag effect on both austenite/ferrite and austenite/bainite interfaces, however as Figure 7-14 shows, higher level of Nb in solution led to an increase in
bainite fraction and a decrease in ferrite fraction. This effect has been widely reported in the literature (Thomas 1982, Poole 2010).

![Figure 7-13- Representative microstructure of Case 2: a) PFM microstructure, b) Experimental micrograph using Nital etchant (Reichert 2012). Figure a was obtained through stitching 4 identical simulated microstructures together.](image)

These results show that because of the fitting, experimental and simulated fractions of ferrite and bainite are very close and microstructures look similar especially because ferrite nuclei density and inter-sheaf spacing have been matched. One of the remarkable similarities is the effect of “orientation compatibility dependent interface mobility effect” (section 4.3.4). As Figure 7-13a shows, this effect enabled the model to replicate experimentally observed features of the growth of bainite sheaves in Figure 7-13b where bainite sheaves did not grow into the austenite grain with higher orientation deviation and stopped growing when they impinged with PAGBs. These comparisons show that the model provides a fair description of
the microstructural evolution in terms of kinetics, fraction, and morphology of the microstructural constituents for the fitting cases.

Figure 7-14- Measured fractions of transformation products compared to predicted values using fitting parameters.

### 7.2.2 Results

The set of fit parameters obtained by fitting to Cases 1 and 2 was used for the simulation of other transformation scenarios. The description of the MA structure is of a critical importance as it has a strong influence on the fracture toughness of the weldment (Li 2001). Therefore, as the first step, the model was used to simulate the morphology of MA in all available four Cases, 1, 2, 3 and 4, from Table 5-4. MA morphology is essentially a by-product of the model as none of the fitting parameters directly controls the morphology of MA.

Figure 7-15 compares the simulated carbon concentration profile in carbon-rich MA constituents in the microstructure with the experimental micrographs obtained by LePera etching that only reveals MA constituents. In order to examine the
predictive capability of the model, the morphology of simulated and experimentally obtained micrographs were quantitatively analysed. Table 7-4 shows the final fraction and average area of MA particles obtained by the quantitative analysis. Particles in the simulations that are of comparable size to the grid size cannot be considered reliable because of the associated discretization numerical errors. Therefore, for the sake of consistency, all the MA particles smaller than 0.1μm² surface area were excluded from the analysis of both PFM and experimental microstructures. Experimental results in Table 7-4 were obtained by filtering small particles out from size distribution data provided by Reichert et al. (Reichert 2012).

Table 7-4- Comparison of predicted and experimentally measured MA contents for four cases represented in Table 5-4. Here all the MA particles with area less than 0.1μm² were excluded from the image analysis results of Reichert et al. (Reichert 2012).

<table>
<thead>
<tr>
<th>Case</th>
<th>Fraction Percentage (%)</th>
<th>Average area (μm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Experiment</td>
<td>PFM</td>
</tr>
<tr>
<td>Case 1 (5 μm, f' = 1)</td>
<td>5.8</td>
<td>4.3</td>
</tr>
<tr>
<td>Case 2 (24 μm, f' = 0)</td>
<td>12.7</td>
<td>13.9</td>
</tr>
<tr>
<td>Case 3 (80 μm, f' = 1)</td>
<td>13.7</td>
<td>8.4</td>
</tr>
<tr>
<td>Case 4 (24 μm, f' = 1)</td>
<td>6.3</td>
<td>8.3</td>
</tr>
</tbody>
</table>
Figure 7-15 PFM MA morphology (lightness represents carbon concentration in MA regions) and experimental MA structure obtained by LePera etchant (Reichert 2012) for cases 1 (a and b), 2 (c and d), 3 (e and f) and 4 (g and h). For cases 1, 2 and 4 PFM structures obtained through stitching 4 identical simulated microstructures together.
These results show that the model captures the main specifications of MA occurrence in the studied steel as reported by Reichert et al. (Reichert 2012), i.e.

- mainly ferritic microstructures consist of lower fractions of MA particles compared to mainly upper bainitic microstructures and
- the MA particles that form in mainly ferritic microstructures just reside within the prior austenite grains whereas in mostly bainitic microstructures MA particles exist both within the prior austenite grains and along the PAGBs.

To gain a more in-depth look at the predictive capability of the model, size and aspect ratio distributions of, the MA particles are compared in Figure 7-16 and Figure 7-17. Figure 7-16 represents the total area fraction of particles with individual areas within the specified ranges. Likewise, Figure 7-17 shows the total area fraction of particles at specified aspect ratio ranges. These comparisons show that PFM results are in good agreement with the experimental ones. The largest discrepancy of PFM predictions from experimental results is observed in Case 3 which can be attributed to the considerable difference between the prior austenite grain size of this case and two fitting cases which made nucleation assumptions (e.g. nucleation spacing of ferrite and bainite) less accurate. Tails at the high aspect ratio end of Figure 7-17 b, c, and d stem from elongated MA layers trapped between bainite sheaves. Comparison of Cases 2 and 4 (Figure 7-16b and d) shows that higher level of Nb in solution increased the area fraction of MA, but it did not affect the average size or the aspect ratio of the MA particles. Therefore, unlike cases where ferrite is the dominant
microstructural constituent, MA morphology is dictated by bainite structure when bainite has a considerable fraction like in Case 2 and 4.

Figure 7-16- Comparison of the calculated and experimentally measured (Reichert 2012) size distribution of MA particles for: a) case 1, b) case 2, c) case 3 and d) case 4. MA particles smaller than 0.1\(\mu m^2\) surface area were excluded.

As the next step, simulations were performed for Gleeble heat treatments that replicate the HAZ thermal cycles for a single-torch welding scenario (Militzer 2011). In their experimental work, Militzer et al. tried to mimic the thermal cycle of the second torch of a dual-torch welding practice with a single heating cooling path (single torch). Three thermal cycles were employed with different peak
temperatures, 1000, 1200 and 1350 °C, to represent different positions in the HAZ. Figure 7-18a illustrates the time-temperature paths of the employed heat treatments. Phase-field calculations of these heat treatments include austenite grain growth in parallel with the dissolution of particles, followed by the austenite decomposition.

Figure 7-17- Comparison of the calculated and measured (Reichert 2012) aspect ratio area distribution of MA particles for: a) case 1, b) case 2, c) case 3 and d) case 4. MA particles smaller than 0.1μm² surface area were excluded.

Three calculation domains of (52μm)² size with initial EQAD of 4.2 μm in accordance with the findings of Banerjee et al. (Banerjee 2010) were considered for the calculations of these cases. The austenite grains were randomly assigned
orientations; the grid spacing was 0.184 μm and periodic boundary conditions were applied all around the calculation domains for phase field and diffusion calculations. Figure 7-18b shows the predicted fractions of Nb precipitates obtained from dissolution calculations. The phase field simulations of grain growth give final EQADs of 4.8 μm, 10.4 μm and 26.3 μm, for the corresponding final $f$ values of 0.99, 0.67 and 0 and peak temperatures of 1000, 1200 and 1350 °C respectively. Clearly increasing the peak temperature resulted in more dissolution of Nb bearing particles and coarser austenite grain structure.

![Figure 7-18](image)

The values of mobility pre-exponential factors of ferrite and bainite for two extremes of all Nb precipitated and all Nb in solid solution are already known from Figure 7-10. In order to simulate these thermal paths with partial dissolution of Nb precipitates it has been assumed that pre-exponential factor varies linearly with fraction of Nb bearing precipitates. Figure 7-19 provides the comparison of the
predicted and the measured fractions of transformation products (fraction of ferrite + fraction of bainite) as a function of transformation temperature. In this figure, the experimental data points reported in (Militzer 2011) which spanned from 0 to 1 were normalized with the experimental MA fractions of 0.10, 0.08, and 0.05 for the peak temperatures of 1000, 1200, and 1350 ºC, respectively. In order to examine the predictive capability of the model in terms of kinetics, coefficients of determination (R-squared error) were calculated for PFM curves in Figure 7-19 with respect to experimental ones. For the peak temperatures of 1000, 1200, and 1350 ºC, R-squared error (coefficients of determination) of 0.92, 0.68 and 0.87 were obtained, respectively. Thus, although the model reasonably predicts the trends observed in the laboratory tests corresponding to Nb fully in solution and Nb fully precipitated, it fails to describe the case with partial dissolution. This discrepancy may be associated with the assumption of linear dependence of the mobility pre-exponential factor on the fraction of Nb bearing precipitates (Figure 7-10).

Figure 7-20 compares the predicted microstructures with the experimental micrographs for these three different heat treatment cycles. For the peak temperature of 1000 ºC a combination of ferritic and bainitic constituents was obtained. Increasing the peak temperature leads to an increase in bainitic microstructure such that for a peak temperature of 1350 ºC a completely bainitic microstructure was obtained. PFM microstructures show 4%, 14%, and 15% of MA constituent for the peak temperatures of 1000, 1200, and 1350 ºC, respectively. Predicted MA fractions are in the same order as the experimentally measured values (0.10, 0.08, and 0.05 for the peak temperatures of 1000, 1200, and 1350 ºC). Cooling
rates of these experiments (Figure 7-18a) are almost twice the 10 °C/s cooling rate applied for fitting cases. The discrepancy in MA fractions may be attributed to the higher cooling rate, compared to fitting cases, which altered the characteristics of the bainite. Because quantitative analysis of ferrite constituent in microstructures with less than 20% ferrite does not lead to reliable results, quantitative metallography was performed on the only experimental micrograph with higher than 20% ferrite constituent. A ferrite fraction and average grain size of 20% and 4.3μm were obtained by the image analysis of the experimental micrographs for the case with the peak temperature of 1000 °C (Figure 7-21). These values are in good agreement with PFM predictions, i.e. ferrite fraction and average grain size of 19% and 3.5μm.

Figure 7-19- The comparison of the experimental kinetics data and PFM calculations for simulated HAZ heat treatments.
Figure 7-20- Experimental micrographs from the simulated HAZ at different peak temperatures and the corresponding simulated microstructures at $T_p$: 1350 °C (a and b), 1200 °C (c and d) and 1000 °C (e and f). In PFM microstructures, white, gray, and black regions represent upper bainite, ferrite, and MA, respectively. Figures a, c and e are obtained through stitching 4 identical simulated microstructures together.
Although there are some anticipated discrepancies between predictions and the experimental data, it is evident that as a first approach the model makes reasonable predictions.

Figure 7-21- Ferrite grains were manually outlined and analysed using ImageJ image analysis software for the simulated HAZ thermal cycle with the peak temperature of 1000°C.

7.3 Summary and Conclusion

An integrated diffusional austenite decomposition model was developed that is capable of describing the simultaneous ferritic and bainitic transformations in advanced steels. Because martensitic transformation is a military transformation, it was excluded from the current model and analysis. In the model, the crystallographic
orientation relationships of austenite/ferrite and austenite/bainite were addressed in a simple/pragmatic fashion.

As a first step, the model was applied to TRIP steel in which high Si content retards carbide precipitation thus forming carbide-free bainite. Whilst most of the experimental data was available from previous studies at UBC (Fazeli 2012), some additional microstructure analyses were needed, e.g. an experimental parameter, inter-sheaf spacing was introduced to quantify bainite nuclei density. The model provides a reasonable description of the diffusional bainite formation.

It was shown that the model framework that was developed for the TRIP steel, in which carbide formation was neglected, could be applied to the linepipe steel with some minor adjustments. In the microalloyed steel, the model provides good description of the fraction of all transformation products and can describe the morphological features of the microstructural constituents, especially those of MA particles.

The model predictions were in agreement with the experimental kinetics and morphology observations. This multi aspect capability of the model, in providing fair predictions for both microstructure and kinetics, shows the model is a proper tool to deal with relevant thermal cycles where just ferrite, upper bainite and MA form as microstructural constituents.

Limitations of the integrated model are as follows:

- Fast diffusion paths are not taken into account
- Strain effects and displacive aspects of the bainitic transformation are neglected
• Only one variant of bainite formation is considered (i.e. upper bainite)

The ways to overcome these limitations are described in the future work section.

In the next chapter, model predictions will be validated with independent experimental data from welding trials.
8 Application to the HAZ

By following the procedure shown in the previous chapter, the PFM is employed to study the microstructural evolution in the HAZ of X80 linepipe steel. An experimental dual torch welding trial was considered for which reliable experimental time-temperature profiles were recorded and an estimation of the thermal profile was generated. The thermal fit is available in Appendix B. Simulations include grain growth and then simultaneous austenite decomposition to ferrite and bainite. Initial conditions for the calculations include thermal profile and dissolution profile along the HAZ.

8.1 Grain Growth

The PFM was used to predict the austenite grain structure in a section of HAZ. It was assured that the chosen section of the HAZ encompasses extreme conditions at both ends of the HAZ (Figure 2-12), i.e. all Nb bearing particles dissolved at CGHAZ and no dissolution at FGHAZ. At the same time, it was ensured that the calculation domain is large enough to contain a statistically sufficient number of austenite grains. Figure 5-9 shows the variations in the thermal cycles along the selected section. The starting volumetric grain size was 5µm, therefore, for the sake of numerical accuracy the upper limit of allowable grid size for grain growth calculations was 0.3µm while the average width of HAZ for dual torch welding technique was about 2.5mm. Because of the limitation of computational resources and also because of the relatively large size of the required calculation domain (850µm×83µm) compared to the grid spacing, 3D simulations of the entire HAZ were computationally too expensive to perform.
using MICRESS®. Therefore, all simulations were performed in 2D. In addition, the calculation domain was limited to a portion of the HAZ where full austenitization occurs during both passes and precipitate dissolution and grain growth occurs to a considerable extent, i.e. the HAZ portion in which grain growth is not considerable was neglected. Banerjee et al. (Banerjee 2010) showed that for the steel of interest in this work, upon full austenitization of the as-received material at 950 °C, the equivalent grain area diameter (EQAD) is approximately 4.2 μm regardless of the heating rate. Therefore, the austenite formation stage was neglected and all the simulations of austenite grain growth started with a volumetric grain size of 5 μm.

As mentioned earlier, in the simulated section of the dual torch welding HAZ, it was assumed that the material went through complete austenitization during both passes such that the second pass wiped out all the microstructural features of the first pass except for the dissolution of the precipitates. In fact, the first pass was not included in the phase field simulation and only the dissolution was calculated for the first pass. The dissolution profile obtained from the first pass was used as the initial conditions for the dissolution calculations of the second pass. A grid size of 0.3 μm and grain boundary width of three grid points was chosen for the calculations using the finite phase field numerical approach. Test simulations verified stability and convergence of the calculations.

To numerically solve the phase field equations for the HAZ with its significant thermal gradients (Figure 5-9), appropriate boundary conditions must be defined at the domain boundaries. In the direction parallel to the fusion line, there is negligible gradient, therefore as for the bulk simulations in section 6.2, periodic boundary
conditions were applied. In the direction perpendicular to the fusion zone, because of the thermal gradient, periodic boundary conditions cannot be used and an insulating boundary condition was applied instead as illustrated in Figure 8-1. There are two options available for introducing insulating boundary conditions depending on the choice of the order parameter assumed in the ghost node \( \phi_{j+1} \) that would be the neighbour of a node with an order parameter of \( \phi_j \) located at the domain boundary, i.e.

- **Gradient:** where \( \phi_{j+1} \) is approximated from the linear extrapolation of \( \phi_{j-1} \) and \( \phi_j \)
- **Insulated:** where \( \phi_{j+1} \) is assumed to have the same value as \( \phi_j \)

For both versions of these boundary conditions, some artefacts were observed in the 2D grain growth simulations. The insulated condition leads to the formation of elongated grains at the domain boundary. Although in 2D grain growth experiments, similar behaviour at heat source boundaries has been observed (Lee 2005), this appears to be less realistic for grain growth that occurs in 3D. The gradient boundary condition leads, on the other hand, to the formation of tiny grains with a size of one grid spacing at the domain boundary that stems from linear extrapolation that is equivalent to zero convexity.

Running constant temperature test simulations with different boundary conditions (Appendix C) show that the results obtained by insulated boundary condition show smaller deviation with respect to periodic boundary conditions compared to gradient boundary conditions. Therefore, it can be concluded that the insulated boundary conditions leads to a lesser number of numerical artefacts. Thus
in the present simulations, insulated boundary conditions were used at both sides of the calculation domain parallel with the fusion line (Figure 8-1).

![Figure 8-1- Schematic representation of the boundary conditions applied to the HAZ.](image)

For grain growth calculations, the effective grain boundary mobility coupled with the Nb(CN) dissolution models were used to replicate time and temperature dependent particle pinning and solute drag effects.

The initial volume fraction of Nb(CN) precipitates in the base material was assumed to be the equilibrium fraction i.e. \( f^* = 1 \). The re-precipitation of Nb(CN) during cooling was neglected. This assumption is in accordance with the experimental observations (Collins 1983) that showed that when the cooling rate is sufficiently high, Nb would remain in solution and re-precipitation of Nb(CN) particles during cooling is a slow process.
Figure 8-2 shows the thermal profiles and the dissolution behaviour at three representative locations along the studied section of the HAZ in the selected dual torch scenario. For simplicity, it was assumed that along the section, the first torch peak temperatures were equal to the peak temperatures of the second torch (Figure 8-2a). Data from the welding trials indicates this to be a reasonable assumption. Although, peak temperatures for the first and second torch can vary depending on the position in the HAZ relative to the welding pass location, in the case of interest for this work they usually were within ±100 °C.

Figure 8-2- Temporal variations in temperature (a) and fraction Nb precipitated (b) at three locations along the HAZ.

Figure 8-3a shows the predicted prior austenite grain structure for the considered portion of the HAZ. This simulation predicts the gradual decrease in the austenite grain size across the HAZ from CGHAZ to FGHAZ. To quantitatively determine the extent of grain growth along the HAZ, one can measure the variation of the grain size based on the linear intercept at each section as shown in Figure 8-3b.
These calculations show that the linear intercept\textsuperscript{viii} decreases from about 50 to 4\(\mu\text{m}\) along the HAZ.

Considering the thermal and dissolution profiles (refer to Figure 8-2), two expected scenarios are:

- High peak temperature and full dissolution (shown by red lines): sufficiently high peak temperature near the WM/HAZ border to dissolve Nb(CN) precipitates in austenite leading to significant austenite grain growth, i.e. coarse grained region (CGHAZ) near the fusion line (corresponding to the left side of the Figure 8-3a)

- Low peak temperature and small dissolution (shown by blue lines): peak temperature above complete austenitizing temperature but lower than the dissolution temperature of Nb(CN) particles with very minor grain growth occurring, i.e. fine grained region (FGHAZ) further away from the fusion line (corresponding to the right side of the Figure 8-3a)

Obviously, there are transitional scenarios in between these two extreme cases in which some of the Nb(CN) precipitates dissolve and grain growth occurs at intermediate rates.

\textsuperscript{viii}Linear intercept \(\sim 0.8\times\text{EQAD}\) (Militzer 1995)
In order to examine the predictive capability of the model, the grain size in a region close to the fusion zone was analyzed in the simulated HAZ section, i.e. 100 μm away from the fusion line represented with a green dotted rectangle in Figure 8-3. This region is of particular interest as it displays coarse austenite grains that may be...
most detrimental to weld properties. The PFM results (Figure 8-3b) for this region suggest an average linear intercept of 28μm. Because a statistically sufficient number of grains were not captured in this simulation (Figure 8-3a), additional simulations with the same initial number of grains but with larger height to width ratios embracing the CGHAZ section were conducted (Figure 8-4). Here domains 2, 3, 4, and 5 lead to mean linear intercepts of 32.3, 27.6, 23.7, and 30.9μm, respectively. The average grain size obtained from these simulations was 29±6μm. In the studied section of HAZ in the welding trial this region displays a bainitic microstructure that permits, at least to a first approximation, estimation of prior austenite grain sizes (Figure 8-3c). For this region, the average experimental linear intercept was estimated to be 29μm. These results show that when gradients in temperature and dissolution were taken into account simulations yield a good description of the experimental observations.

Figure 8-4- a) the relative shape of simulation domains, b) Polynomial fit to simulation 1 (red curve) versus data points of simulation 5 (green dots).
8.2 Austenite Decomposition

The set of parameters found by fitting for the linepipe steel (section 7.2.1) without adjustment were used to predict the microstructure of the HAZ from the selected weld trial. The inputs of the austenite decomposition model include the thermal profile (Figure 8-2a), dissolution profile (Figure 8-2b), and austenite grain structure (Figure 8-3b) along the chosen section of the HAZ. Austenite decomposition calculations are much more computationally expensive than grain growth calculations, especially since they are coupled with diffusion and anisotropy calculations. Therefore, it was computationally unaffordable to model the austenite decomposition in the whole section depicted in Figure 8-3b. Subsequently, the output of the grain growth calculations (austenite grain structure, crystallographic orientations and dissolution profile) was sub-divided into ten sub-domains with the size of $(82.8\mu m)^2$ to act as ten inter-dependent calculation domains for the austenite decomposition calculations. For both phase field and diffusion calculations, insulated boundary conditions were applied to the boundaries between sub-domains that are parallel to the fusion line and periodic boundary conditions were applied to both sides perpendicular to the fusion line. Nonlinear thermal gradients along with nonlinear dissolution gradients were applied to each simulation domain.

Figure 8-5 shows the simulated microstructure along the HAZ where a gradual transition from the bainitic microstructure in the CGHAZ to ferritic microstructure in the FGHAZ is predicted.
Figure 8-5 - a) simulated microstructure in the portion of the HAZ perpendicular to the fusion line, b) the model input, austenite structure. In PFM microstructure, white, gray, and black regions represent upper bainite, ferrite, and MA, respectively.

To quantitatively the variation of microstructure along the HAZ, one can measure the fractions of microstructural constituents along five equally spaced vertical lines (Figure 8-6). According to PFM predictions for dual torch welding a predominantly bainitic microstructure forms in the region close to the WM (CGHAZ). This prediction is consistent with the general observations reported by the industrial partners of the research project (TransCanada and Evraz). Furthermore, PFM predicts that considerable amount of MA forms in the CGHAZ (coarse-grained heat affected zone) of the dual torch welding. This is in line with the experimental observations in the literature (Fossaert 1995) that shows that critical cooling rate for martensite formation significantly decreases as Nb content in solid solution is increased (e.g. Figure 2-7).
Figure 8-6- Simulated variation of microstructural constituents along the HAZ (data point represents the values obtained at each section and the solid line represents the trend line).

Figure 8-7 compares the model predictions with the corresponding microstructures in the HAZ at certain distances from the fusion line (1, 2, and 3). The thermal and dissolution histories at these locations were already shown with three curves in Figure 8-2a and Figure 8-2b, respectively.

Location 1 in Figure 8-7 was subjected to very high peak temperatures at first and second torch welding thermal cycle; therefore, all the Nb(CN) particles were dissolved leading to a coarse austenite structure. Coarse prior austenite grain structure provided limited nucleation site density at grain boundaries for ferrite and bainite. Therefore, most of the nucleation occurred at lower temperatures within parent austenite grains. Because diffusion rate significantly drops at lower
temperatures and high level of Nb in solution suppresses austenite decomposition by exerting significant solute drag force on migrating interphase boundaries, a relatively large fraction of MA was formed in location 1; and ferrite formation was to a considerable extent retarded and bainite was the dominant product of the austenite decomposition. Location 2 ended up with a finer structure than location 1, which can be attributed to the finer prior austenite structure, which supplied more nucleation sites along PAGBs in addition to a lesser amount of Nb in solution, which resulted in lesser solute drag force. In location 3, because most of Nb bearing precipitates were not dissolved and also there was a very fine prior austenite structure, ferrite nucleation and growth was highly promoted that resulted in a mostly ferritic microstructure.

Because quantitative analysis of ferrite constituent in microstructures with less than 20% ferrite does not lead to reliable results, quantitative metallography was performed on the only experimental micrograph with higher than 20% ferrite constituent (Figure 8-8). For location 3, PFM gives an average ferrite grain size and fraction of 7μm and 39% versus experimental values of 5μm and 52%, respectively. Considering the intrinsic uncertainty in the recognition and classification of microstructural constituents in such complex microstructures, model simulations are in reasonable agreement with the experimental observations of the HAZ microstructure.
Figure 8-7- Experimental micrographs of the studied section in the HAZ and the corresponding simulated microstructures at CGHAZ (a and b), intermediate region (c and d) and FGHAZ (e and f). In PFM microstructures, white, gray, and black regions represent upper bainite, ferrite, and MA, respectively.

Comparison of the quantitative analyses made for the actual dual torch welding in here with the single torch heat treatment (section 7.2.2) shows that the model made fair predictions for both cases and the level of deviations in both cases were more or less of the same order. Applied thermal cycle of the dual torch welding was based on estimation whereas the exact thermal cycles of the single torch heat treatments were available from the thermocouple measurements of the Gleeble
machine. Uncertainty in thermal profile leads to a significant uncertainty in model predictions. On the other hand, in the dual torch welding, average cooling rate in the range of austenite decomposition transformation was close to the cooling rate of fitting cases (10°C/s) whereas for the single torch heat treatments cooling rate was almost double that of the fitting cooling rate. A higher cooling rate increases the potency for the formation of lower bainite, which did not exist in fitting cases; therefore, affects the accuracy of the model predications.

Figure 8-8- Quantitative image analysis of the polygonal ferrite constituent in the experimental micrograph of location 3. Polygonal ferrite grains were manually outlined and analysed by ImageJ software.

8.3 Summary and Conclusions

HAZ is characterized by steep temperature and dissolution gradients that result in microstructural gradients. The unique capability of the present modelling work was to make predictions for the entire HAZ that accounts for the prevalent gradients. This work also presented the phase-field models as tools to deal with the complex
morphologies, e.g. the microstructural phenomena that occur in the HAZ of the studied X80 steel including austenite grain growth and austenite decomposition into ferrite and bainite. Present simulations show that an effective mobility approach can be combined with a dissolution model to describe the effects of variation in pinning pressure and solute drag on grain growth and phase transformation.
9 Overall Conclusions

A phase field modelling approach has been developed to describe the microstructural evolution in advanced low-carbon steels under laboratory and industrial process conditions. The overall goal of the model was to describe the microstructural evolution in the HAZ of X80 linepipe steel. To explicitly address all the underlying microstructural events, the model integrates a number of sub-models. Each sub-model was fitted with continuous heating and cooling test data. Integration of these sub-models enables the model to deal with the overlapping phase transformations that occur in most of industrial processes. In general, the proposed integrated model is unique from the following aspects:

- It is the first comprehensive meso-scale model that describes austenite to bainite transformation
- It accounts for concurrent austenite grain growth, ferrite formation, bainite formation and ferrite coarsening
- It provides a multi-aspect description of the phase transformation including transformation kinetics, diffusion profile and morphologies of each microstructural constituent
- It is the first modelling approach to describe the microstructural evolution in the HAZ, taking the effect of gradients in temperature and level of particle dissolution into account

The overall achievements of this work are as follows:
To replicate austenite grain growth during rapid heating and cooling of microalloyed linepipe steel under the influence of Zener pinning and dissolution of Nb(CN) particles, the concept of effective grain boundary mobility as a function of temperature and Nb in solution is used, coupled with the dissolution model. It is suggested that computationally less expensive 2D simulations can replicate 3D results when a 2D to 3D mobility conversion factor of 0.7 is used. Furthermore, the proposed model proved that it is capable of accurately describing the austenite grain structure in the HAZ.

To replicate the austenite decomposition in TRIP steel, a simple approach for the implementation of the crystallographic orientation relationships of austenite/ferrite and austenite/bainite was used. A diffusional bainite model is introduced in which the carbide formation is neglected such that is suitable for the bainitic transformation in TRIP steels. In the ferrite model both experimental and simulation results show that despite higher nucleation rates at higher cooling rate, the number density of ferrite grains is lower. This can be associated with the ferrite-coarsening phenomenon (coalescence) that was included in the model. An experimental parameter, bainite inter-sheaf spacing, was introduced to quantify bainite nucleation.

To deal with the microstructural evolution that occur in the HAZ of X80 steel under spatial variations in thermal profile and dissolution conditions an effective mobility approach is taken that takes into account
the effect of spatial variation in the level of Nb(CN) dissolution on the phase transformations. It was shown that the current model makes a proper description of the MA fraction and morphology that is an essential factor in determining the mechanical properties of the HAZ. The unique capability of the present work is that it can make predictions for the entire HAZ accounting for the thermal and microstructural gradients.
10 Future Work

To improve the predictive capability of the PFM simulations it will be required to address the following points. Grain growth in the HAZ should be simulated in 3D rather than in 2D. This will eliminate some of the potential artefacts in the predicted grain morphologies, e.g. elongated grains near the fusion line. Further, particle pinning should be incorporated in a more realistic way, e.g. by making the effective mobility a function of both temperature and driving pressure.

To enhance PFM for the HAZ a sub-model on austenite formation will be needed to model the microstructure evolution in the ICHAZ and potential intercritical annealing of CGHAZ with a subsequent torch and/or welding pass. These regions are of special importance as some studies show that they have inferior fracture properties compared to the rest of the HAZ (Bott 2013). The PFM on austenite formation could adopt the approach proposed by Savran (Savran 2009).

A PFM is needed to account for the elastic/plastic interaction of bainite and the matrix. The model should be able to simulate different types of bainite. To do so, especially for bainite that forms at lower temperatures, displacive bainitic growth aspects must be included through considering the elastic part of the free energy density using a similar approach as that proposed for martensitic transformation (Artemev 2001a). An outcome of the improved bainite model would be a more thorough prediction of the MA constituent as the size and morphology of MA constituents depend on the bainite type and have a critical effect on the overall mechanical properties of the HAZ.
Fast diffusion paths, a more comprehensive crystallographic description of ferrite and bainite and incorporation of 3D aspects are further considerations that may improve the predictive capability of the model. It would also be very important to conduct 3D phase transformation simulations. This will be of particular significance for bainite formation with complex morphology.

There is a wealth of experimental data available for the steel under investigation. However, additional experiments may be required to develop and validate models. Further experimental tests may be useful to evaluate the role of steep spatial temperature gradients on grain growth in the HAZ. Heating capabilities of the Gleeble thermo-mechanical simulator can be used to generate the thermal gradient. As an option, thermal gradients in the Gleeble sample can be adjusted using iso-quench samples.

A critical aspect, however, of making quantitative predictions for the HAZ is the exact knowledge of the time-temperature profiles for given positions in the HAZ as input into the PFM simulations. Advancing knowledge on these thermal cycles can be expected from additional temperature measurements in welding trials, preferably in conjunction with developing improved temperature models for the HAZ.

Further, the microstructure needs to be connected to a structure-property model to predict the mechanical properties of the HAZ, in particular fracture toughness. For this purpose, it will be required to develop structure-property relationships that can be applied to individual positions in the HAZ and subsequently integrated over the graded microstructure of the HAZ.
References


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Appendices

Appendix A  Main Code

The following MATLAB code shows an example of microstructural simulation for the HAZ. Here the thermal profile subroutine and dissolution subroutine are called to obtain the thermal and dissolution profiles.

```matlab
% The following MATLAB code shows an example of microstructural simulation for the HAZ. Here the thermal profile subroutine and dissolution subroutine are called to obtain the thermal and dissolution profiles.
```

```
% The following MATLAB code shows an example of microstructural simulation for the HAZ. Here the thermal profile subroutine and dissolution subroutine are called to obtain the thermal and dissolution profiles.
```
Appendix B Thermal Fit

The details of the thermal fit can be found in the following subroutine

```c
function ThermoFit(T,Temp,Time,TorchNo) [N,R]=size(Time);
if TorchNo==2 Dual torch
  T=1.53907-1.58767e-4*T; V_m=0.29499+373.88251*exp(-T/100.2567); 
  v=0.1;
end
if V_m<=0.1
  V_m=0.1;
end

else
  A_m=-1/(3*V_m+1); B_m=(500-T)/T-500; C_m=1/(3*V_m+1)*T-500/(3*V_m);
  C_v=231.35737-0.07873*T; D_v=-0.00386+0.43044e-4*T; 
  s_v=0.00218+0.00976*T; F_v=-0.0095-0.73382e-6*T; C_v=C_v/C_v; C_v=C_v';
end

else Single torch
  C_v=0.00057*exp(-T/12.30994)-0.69903; V_m=1;
  B_m=-3195.5718+832.321*T; A_m=1/(3*V_m+1)*T-500/(3*V_m);
  C_v=233.3344*exp(-T/133.52279); apv=358533.33958*exp(-T/68.20125)-126.38653;
  V_m=0.1;
  V_m=max(apv, apv/2);
  else
    V_m=0.1;
  end

  b_p=0.275686+2527.61e-4*exp(T/185.87088); bp=91.5386*exp(-T/71.3878)+0.73166;
  else
    V_m=0.1;
  end

  bp=min(bp1, bp2);
  else
    V_m=0.1;
  end

if T<1000
  T_500=2.34*exp(-T/30.78139)+242.19909;
  else
    T_500=1000;
  end

if v<0.1
  T_500=exp(-T/30.78139)+242.19909;
  else
    T_500=1000;
  end

for i=1:5
  time(i)=0; Q=per1/60;
  end

if TorchNo==2 Dual torch
  Q=Q/2*Time/m_period
else
  Q=Q/2*Time/m_period
end

if T<1000
  Q=Q11;
end
if TorchNo==2 Dual torch
  Q=Q29*exp(F(time(i)+T_500));
else Single torch
  Q=Q29*exp(F(time(i)+T_500));
end
if T<1000
  Q=Q29*exp(F(time(i)+T_500));
else
  Temp(i)=Temp(i)+T_500;
end
```

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Appendix C Boundary Conditions

Figure C-1 shows a set of identical 2D grain growth simulations with different boundary conditions.

Figure C-1- Average grain radius of a multigrain system with a square calculation domain as a function of time for different boundary conditions.

Here the grid size was 0.2 μm, starting average grain size was 7 μm, domain size was 450 μm × 450 μm and model parameters were set in accordance with those of X80 steel. All of these boundary conditions are predefined in MICRESS®.

For all versions of these boundary conditions, some artefacts were observed in two dimensional (2D) grain growth simulations. Insulated boundary conditions lead to the formation of elongated grains at domain boundaries. The gradient boundary conditions lead, on the other hand, to the formation of tiny grains with a size of one grid spacing at the domain boundary, which stems from linear extrapolation that is
equivalent to zero convexity. This effect leads to a significantly lower average grain radius of the simulation with gradient boundary conditions compared to other cases (insulated and periodic boundary conditions). These simulations show that the results obtained by insulated boundary condition shows smaller deviation with respect to periodic boundary condition, therefore it can be concluded that the insulated boundary conditions leads to a lesser extent of numerical artefacts.
Appendix D Dissolution

The MATLAB subroutine for dissolution is called by the thermal code. Inputs include time, temperature, torch number (first torch or second torch in dual torch welding), initial fractions and radii of TiN, small NbC, and large NbC particles. Outputs include final fractions and radii of TiN, small NbC, and large NbC particles.

function [F_TiN,F_NbC1,F_NbC2,F_NbC3,F_NbC4,F_TiN,F_NbC1,F_NbC2,F_NbC3,F_NbC4]

MATLAB subroutine for dissolution is called by the thermal code. Inputs include time, temperature, torch number (first torch or second torch in dual torch welding), initial fraction and radii of TiN, small NbC, and large NbC particles. Outputs include final fractions and radii of TiN, small NbC, and large NbC particles.

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Appendix E Nucleation

The nucleation subroutine in MATLAB imports the ASCII file of the fully austenitic microstructure for ferrite and austenitic ferritic microstructure for bainite. Then based on the given values for the spacing of nuclei and critical nucleation undercooling, it generates a text file that can directly be used in the nucleation section of the MICRESS® run file. Once the MICRESS® run file was updated by this subroutine, the MICRESS® run can be re-started.
for j=1:size
    for k=1:size
        Loc_Orrient(j,k)=zero(3,1);
        Loc_Orrient(1,1)=k(1,j);
    end
end
for v=1:w
    if v>size(Loc_Orrient);
        x=v;
        if x>size(Loc_Orrient)
            x=v;
            if x>size(Loc_Orrient)
                x=v;
            end
        end
    end
    Loc_Orrient(1,1)=k(prnd(ic,j,1),size,v);
    v=x;
end
if v>size(Loc_Orrient)
    x=v;
    if x>size(Loc_Orrient)
        x=v;
        if x>size(Loc_Orrient)
            x=v;
            if x>size(Loc_Orrient)
                x=v;
            end
        end
    end
end
for j=1:size(Loc_Orrient)
    end
end
for v=1:w
    if v>size(Loc_Orrient)
        x=v;
        if x>size(Loc_Orrient)
            x=v;
            if x>size(Loc_Orrient)
                x=v;
            end
        end
    end
end
end
for j=1:size
    end
end
for v=1:w
    if v>size(Loc_Orrient)
        x=v;
        if x>size(Loc_Orrient)
            x=v;
            if x>size(Loc_Orrient)
                x=v;
            end
        end
    end
end
end
for j=1:size
    end
end
for v=1:w
    if v>size(Loc_Orrient)
        x=v;
        if x>size(Loc_Orrient)
            x=v;
            if x>size(Loc_Orrient)
                x=v;
            end
        end
    end
end
end
for j=1:size
    end
end
for v=1:w
    if v>size(Loc_Orrient)
        x=v;
        if x>size(Loc_Orrient)
            x=v;
            if x>size(Loc_Orrient)
                x=v;
            end
        end
    end
end
end
for j=1:size
    end
end
for v=1:w
    if v>size(Loc_Orrient)
        x=v;
        if x>size(Loc_Orrient)
            x=v;
            if x>size(Loc_Orrient)
                x=v;
            end
        end
    end
end
end
for j=1:size
    end
end
for v=1:w
    if v>size(Loc_Orrient)
        x=v;
        if x>size(Loc_Orrient)
            x=v;
            if x>size(Loc_Orrient)
                x=v;
            end
        end
    end
end
end
for j=1:size
    end
end
for v=1:w
    if v>size(Loc_Orrient)
        x=v;
        if x>size(Loc_Orrient)
            x=v;
            if x>size(Loc_Orrient)
                x=v;
            end
        end
    end
end
end
for j=1:size
    end
end
for v=1:w
    if v>size(Loc_Orrient)
        x=v;
        if x>size(Loc_Orrient)
            x=v;
            if x>size(Loc_Orrient)
                x=v;
            end
        end
    end
end
end
for j=1:size
    end
end
for v=1:w
    if v>size(Loc_Orrient)
        x=v;
        if x>size(Loc_Orrient)
            x=v;
            if x>size(Loc_Orrient)
                x=v;
            end
        end
    end
end
end
for j=1:size
    end
end
for v=1:w
    if v>size(Loc_Orrient)
        x=v;
        if x>size(Loc_Orrient)
            x=v;
            if x>size(Loc_Orrient)
                x=v;
            end
        end
    end
end
end
for j=1:size
    end
end
for v=1:w
    if v>size(Loc_Orrient)
        x=v;
        if x>size(Loc_Orrient)
            x=v;
            if x>size(Loc_Orrient)
                x=v;
            end
        end
    end
end
end
for j=1:size
    end
end
for v=1:w
    if v>size(Loc_Orrient)
        x=v;
        if x>size(Loc_Orrient)
            x=v;
            if x>size(Loc_Orrient)
                x=v;
            end
        end
    end
end
end
for j=1:size
    end
end
for v=1:w
    if v>size(Loc_Orrient)
        x=v;
        if x>size(Loc_Orrient)
            x=v;
            if x>size(Loc_Orrient)
                x=v;
Appendix F  Grain Growth Convergence

As shown Figure G-1, convergence of grain growth simulations for a case representing the simulation conditions of Figure 6-6b has been confirmed by systematically varied boundary widths and grid sizes keeping the grain size and the ratio of the grain boundary width to the grid size constant.

![Figure G-1](image)

Figure G-1- Final average grain size as a function of grid size and number of grid points at grain boundary.

2D stability calculations show that when there are more than 3 grid points through the interface, the count of grid points has a negligible effect on the simulation results.
Appendix G Grain Growth Stability

In 3D grain growth calculations it is essential to make sure that statistically enough number of grains exists in the calculation domain. Figure H-1 shows the variations of the standard deviation of lognormal grain size distribution at different grid sizes taken at different stages of rescaling during grain growth calculations.

![Graph showing variations of the standard deviation of lognormal grain size distribution at different grid sizes](image)

Figure H-1- Grain size standard deviation as a function of number of grains remaining in the calculation domain for varying grid sizes at different stages of rescaling. Arrows represent the rescaling process.

These simulations were obtained by altering grid sizes at simulations shown in Figure 6-6a. A sufficient number of grains must be present to be sure that grain growth evolution accurately represents the grain growth statistics. Figure H-1 shows that at least 70 grains should be in the domain. Black arrows in Figure H-1 show how grain size standard deviation changed during rescaling. During every rescaling step, increasing the grid size, parameters of Voronoi construction in MICRESS® (minimum
allowed seed size, maximum allowed seed size, and minimum seed spacing) were adjusted to generate a structure with very close average volumetric grain size and standard deviation. Figure H-2 compares a section of rescaled simulation of Figure 6-6a (1st and 2nd), with a third simulation. These results show that rescaling with the considerations taken in this work does not affect the reliability of 3D simulations.

Figure H-2- Volumetric grain size as a function of time for three sets of 3D grain growth calculations for simulations shown in Figure 6-6a.
Appendix H Bainite Anisotropy

Figure I-1 shows the effect of the anisotropy parameters ($\kappa$, $k_{\text{kin}}$ and $k_{\text{st}}$) on the morphology/aspect ratio of a single bainite sheaf that grows perpendicular to an arbitrary PAGB.

![Figure I-1](image)

Figure I-1- Effect of anisotropy parameters on the morphology of a binate sheaf that nucleated at a PAGB. $k_{\text{st}}=0.04$, $k_{\text{kin}}=0.04$ (a), $k_{\text{st}}=0.3$, $k_{\text{kin}}=0.04$ (b), $k_{\text{st}}=0.08$, $k_{\text{kin}}=0.3$ (c), $k_{\text{st}}=0.3$, $k_{\text{kin}}=0.3$ (d), $k_{\text{st}}=0.01$, $k_{\text{kin}}=0.04$ (e), $k_{\text{st}}=0.08$, $k_{\text{kin}}=0.01$ (f), $k_{\text{st}}=0.01$, $k_{\text{kin}}=0.01$ (g). Here, red, blue, and yellow represent austenite, interface, and bainite, respectively.

Here the single sheaf has the same orientation as topside austenite grain and different orientation than the bottom-side grain and the sheaf has been assigned a single-orientation faceted anisotropy. The model response to the input parameters shows a spectrum where at one end the there is an unrealistic narrow feature and on the other end there is an unrealistic broad feature. The set of parameters shown in Figure I-1a was selected which lies in between these two extremes leading to a morphology that compares well with the experimental bainite sheaf morphologies.