MICROSTRUCTURE DEVELOPMENT AND FATIGUE CRACK GROWTH STUDIES ON HIGH-CARBON PLATE-MARTENSITE AUSTENITE MICROSTRUCTURES

by

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ABSTRACT

Carburized components such as gears and drive shafts experience bending fatigue loading during service. At stresses above the endurance limits, fatigue cracks nucleate at an early stage and most of the fatigue life of the component is spent in the fatigue crack propagation regime, particularly in the case region of a carburized component. The goal of this PhD work was to study the influence of microstructural features on the fatigue crack growth (FCG) resistance of high-carbon plate martensite austenite microstructures for potential application in carburized and bearing steels. Using commercial high-carbon 52100 steel (1 wt pct carbon), high-carbon plate-martensite austenite microstructures (microstructures comparable to the case regions of carburized steel) were developed, fatigue tested and characterized. Thermal processing strategies, namely thermal cycling and step-quenching were implemented and the subsequent refinement in prior austenite grains size (PAGS), plate-martensite and RA was quantitatively characterized. Lower austenitizing temperatures and shorter hold times resulted in the greatest PAGS refinement as well as refinement in plate-martensite and RA constituent size. Multiple thermal cycling did not further refine the PAGS but resulted in a narrower PAGS distribution. Step-quenching (interrupted quenching below $M_s$ temperature) mechanically and/or thermally stabilized the RA, resulting in a refinement in plate-martensite size and an increase in the stability of RA. The mode I fatigue crack growth (FCG) behavior of high-carbon plate-martensite austenite microstructures with varying amounts and stability of RA was investigated. The interaction between the fatigue cracks and the surrounding microstructure was characterized using microscopy, electron backscatter diffraction and quantitative fractography. Higher initial amounts of RA, mechanical stability of RA, and greater degree of martensite tempering improved FCG resistance of high-carbon plate martensite austenite microstructures. Stress-assisted RA to martensite transformation was observed in the vicinity of the fatigue cracks in the microstructures with substantial amounts of RA (more than 15 vol pct). The fatigue fracture surfaces of the different microstructures exhibited varying combinations of IG fracture, mixed-ductile brittle (MDB) and TG (cleavage, quasi-cleavage, and ductile striations) fracture as a function of stress intensity range at
the crack tip. Microstructure conditions showing greater fractions of MDB, and TG fracture had better fatigue crack growth resistance. The presence of a higher initial RA content suppressed brittle IG fracture and promoted TG fracture, potentially due to the transformation-toughening associated with RA to martensite transformation or transformation induced micro-crack coalescence in the vicinity of the fatigue crack. Finally, transformation toughening models were developed to predict the role of deformation induced martensite transformation (DIMT) on the FCG behavior of high-carbon plate-martensite austenite microstructures. The model predictions of FCG rates were highly sensitive to small variations in the amount and distribution of DIMT, suggesting that transformation toughening may be used as a microstructure design strategy for improving FCG resistance of high-carbon plate-martensite austenite microstructures.
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CHAPTER 1
INTRODUCTION

Carburized components are used in vehicle powertrains and power generation equipment where high strength is required in addition to good fatigue resistance [1.1]. The high-carbon case region provides strength and wear resistance, whereas the low-carbon core provides toughness to the carburized components. The case regions contain plate-martensite, retained austenite (RA) and occasionally undissolved cementite, whereas the core regions contain lath martensite, ferrite or bainite [1.2]. Along with the carbon composition gradient, carburized components also have a gradient in hardness and RA content along the depth of the component [1.3]. The difference in austenite to martensite transformation kinetics between the case and core during quenching introduces compressive residual stresses at the surface [1.4]. Additionally, the RA in the case region is susceptible to deformation-induced martensite transformation during service [1.2].

Fatigue is a common mode of failure in carburized components. During service, regions such as a root of a gear tooth experience bending loads. Microstructural features such as grain size, amount of RA, and residual stresses affect the bending fatigue resistance of carburized components [1.2]. At stresses above the endurance limits (presumably, the maximum value of stress below which, the material can withstand infinite number of fatigue cycles), fatigue cracks nucleate at an early stage and most of the fatigue life of the component is spent in the fatigue crack propagation regime. The microstructure in the case region plays an important role in crack initiation and propagation during bending fatigue failure [1.1]. Hence, an improved understanding of the interaction between fatigue cracks and high-carbon plate-martensite austenite microstructures can help better optimize processing and performance of carburized components.
1.1 Research Objectives

The overarching goal of this PhD work was to study the influence of microstructural features on the fatigue crack growth (FCG) resistance of high-carbon plate martensite austenite microstructures for potential application in carburized and bearing steels. While the case region of a carburized component contains a gradient of carbon content, RA phase fraction, martensite morphology and processing induced residual stress, this study specifically focuses on the interaction between the fatigue crack and the plate-martensite and RA in the absence of high residual stresses. Using commercial high-carbon 52100 steel (1 wt pct carbon), microstructures consisting of plate martensite, RA and undissolved cementite were developed, fatigue tested and characterized. The 52100 steel without the surface compressive residual stress was prone to cracking during quenching. Consequently, it was necessary to limit the heat treatments to develop microstructures comparable to the case regions of carburized components. The differences in the FCG behavior of the carburized steel and the high-carbon microstructures developed in this study may stem from the higher carbon content of 52100 steel (1 wt pct) than typically observed in the case regions of carburized components (0.8 to 0.9 wt pct). However, the findings of this study are applicable to microstructures present in carburized components and bearing steels. To systematically address the goals of this research, the following research objectives were defined -

1. To Develop Microstructure Refinement Strategies for High-Carbon Plate-Martensite Austenite Microstructures

   Research Question: How do thermal processing strategies, specifically, thermal cycling and step-quenching, influence the refinement of prior austenite grains, plate martensite and RA?

   Two thermal processing strategies, (i) thermal cycling and (ii) step quenching, were implemented to develop microstructures containing plate martensite and RA. The resulting microstructures were characterized using dilatometric analysis, quantitative stereological methods, and neutron diffraction.

2. To Study the Fatigue Crack Growth Behavior of High-Carbon Plate-Martensite Austenite Microstructures

   Research Question: How do fatigue cracks interact with the high-carbon plate martensite austenite
microstructure?

High-carbon plate-martensite austenite microstructures with varying amounts and stability of RA were developed and their mode I FCG behavior was investigated. The fatigue crack path and fracture surfaces were characterized to rationalize the influence of microstructure and the deformation induced RA to martensite transformation on FCG behavior.

3. To Develop Experimentally Informed Fatigue Life Prediction Models for High-Carbon Plate-Martensite Austenite Microstructures

Research Question: How does the transformation of RA to martensite in the vicinity of fatigue crack affect FCG rates in high-carbon plate martensite austenite microstructures?

Existing transformation toughening models[1.5, 1.6] were modified to predict the influence of RA to martensite transformation on FCG rates in high-carbon plate-martensite austenite microstructures. Experimentally determined transformation data were used to calculate the reduction in stress intensity at the crack tip due to the volume expansion associated with RA to martensite transformation.

1.2 Thesis Outline

The background literature pertaining to the carburizing process, carburized microstructures, and bending fatigue behavior of carburized steels are presented in Chapter 2. A general overview of fatigue crack propagation in metals, and special topics on fatigue crack propagation in embrittled steels, and the influence of deformation induced martensite transformation on FCG behavior are also described. Relevant background is also presented at the start of each chapter and throughout the thesis where discussion of existing literature was necessary.

Chapter 3 presents the experimental methods and microstructure characterization results of thermal processing strategies aimed at microstructure refinement [1.7]. The trends in refinement of prior austenite grain size (PAGS), plate martensite and RA constituents during thermal cycling and step quenching are discussed.
Chapter 4 describes the development and FCG testing of high-carbon plate-martensite austenite microstructures with varying RA content. The interaction between fatigue cracks and the surrounding microstructure was characterized using microscopy, electron backscatter diffraction and stereological fractography. Pertinent failure mechanisms, the influence of RA on FCG rates, and the implication of the study on high-carbon plate-martensite austenite microstructures are discussed in detail.

Chapter 5 presents implementation of existing transformation toughening models \[1.5, 1.6\] for high-carbon plate-martensite austenite microstructures. The experimentally informed model predicts the influence of RA to martensite transformation on FCG rates in high-carbon plate-martensite austenite microstructures.

The key findings and conclusions of this research are summarized in Chapter 6. Finally, Chapter 7 suggests some recommendations for future research based on specific results from this work.

1.3 References


CHAPTER 2
LITERATURE REVIEW

The following chapter presents background information on the key concepts relevant to this PhD thesis. Section 2.1 presents an introduction to the carburizing process. Microstructures resulting from the carburizing process are discussed in Section 2.2. Bending fatigue behavior of carburized steels is discussed in Section 2.3 with an emphasis on the influence of prior austenite grain size, RA and residual stress on the fatigue performance of carburized microstructures. A general overview of fatigue crack propagation in steels is presented in Section 2.4. Specifically, discussions on FCG in embrittled steels and the influence of deformation induced martensite transformation (DIMT) on fatigue crack propagation in steels is presented in Subsections 2.4.1 and 2.4.2 respectively.

2.1 Carburizing Process

During the process of carburization, the component is heated to an elevated temperature in the austenite range and is exposed to a carbon-rich atmosphere. This atmosphere can be a solid like graphite in case of packed carburizing or gases like CO, CO$_2$ and CH$_4$ in case of gas carburizing. At extended holdings at high temperatures, carbon diffuses into the surface layer thereby establishing a concentration gradient of carbon along the depth of the component on top of the pre-existing carbon levels [2.1]. Often, alternating boost and diffuse cycles are performed to tailor the depth of this concentration profile; a typical profile is shown in Figure 2.1(a) [2.2]. The component is subsequently quenched to obtain a martensitic microstructure in the high-carbon region. The core may develop a lath martensitic, ferritic, bainitic or pearlitic microstructure. Optionally, the component may be tempered at moderate temperatures (150 °C-200 °C) to obtain low temperature tempered (LTT) martensite which has transition carbide precipitates for high strength and bending/rolling contact fatigue resistance [2.3]. A carburizing heat treatment cycle is schematically shown in Figure 2.1(b). The processing conditions directly govern features like carbon concentration, case depth, and case and core hardness.
2.2 Carburized Microstructures

The austenitized and quenched carburized microstructures predominantly contain martensite. Due to the carbon content gradient along the depth of the component, the martensite in the high-carbon case and the low-carbon core vary in morphology, amount, and properties. Furthermore, other microstructural constituents such as RA, undissolved cementite, and non-martensitic transformation products may be present [2.4].

The microstructure in the core region depends on the carbon content, hardenability, and thermal gradients during quenching. If the alloy has high hardenability, the low-carbon core transforms to martensite and if the alloy has low hardenability, the core may transform to a mixture of ferrite and pearlite, bainite, and martensite. The low-carbon martensite exists as lath martensite morphology which has a hierarchical structure. Each prior austenite grain consists of several packets, which consist of parallel blocks of fine sub-micron wide laths of martensite crystals [2.5]. Figure 2.2 (a) shows an optical micrograph of a lath martensite microstructure in the core region of 8719 gas carburized steel (0.19 pct C, 1.06 pct Mn, 0.52 pct Cr, 0.50 pct Ni, and 0.17 pct Mo) [2.4]. Fine martensite laths cannot be resolved.
using an optical microscope, but the packets can be observed. In the core region, lath martensite is desirable due to its high strength and toughness as compared to a ferrite pearlite microstructure.

During carburizing, the steel is heated to 930 °C and carbon diffuses from the surface into the steel. Control of temperature above $A_{cm}$ and the carbon atmosphere limits the possibility of precipitating cementite particles during carburizing. During quenching, in the high-carbon region, displacive transformation of austenite results in martensite which has a plate-like morphology. The martensite plates differ from laths in that they do not form parallel to each other. The first plate martensite spans the length of the prior austenite grain. Subsequently, finer martensite plates form by partitioning the remaining austenite constituents, resulting in a large range of plate sizes [2.6]. Figure 2.2 (b) shows an optical micrograph of a martensitic microstructure in the case region of direct quenched SAE 4120 steel [2.4]. The dark regions correspond to the needle shaped (in 2 dimensions) plate martensite.

![Light optical micrographs etched using nital showing carburized microstructures](image)

(a) (b)

Figure 2.2 Light optical micrographs etched using nital showing carburized microstructures, (a) lath martensite in the low-carbon core region of gas carburized 8719 steel, and (b) plate-martensite (dark region) and retained austenite (light regions) in high carbon case region of 4120 steel.

The carbon-rich case regions are associated with incomplete austenite to martensite transformation resulting in RA. The light regions in Figure 2.2 (b) show the RA constituents. The quenching process determines the RA phase fraction particularly as given by the Koistinen and Marburger...
where $V_\gamma$ is RA phase fraction, and $M_s$ and $T_Q$ are martensite start and quench temperature in Kelvin, respectively [2.7]. The $M_s$ temperature is an indicator of the thermal stability of austenite, and it depends on a multitude of factors like composition, prior austenite grain size and stress states [2.8]. Carbon is an austenite stabilizer and suppresses the $M_s$ temperature [2.4]. At room temperature, significant amounts of RA between 15 pct and 30 pct may be present in the microstructure in the case region.

The RA constituents in the case region are susceptible to mechanically-induced martensite transformation. Figure 2.3 (a) and (b) shows optical micrographs of AISI 40126 steel (1.26 wt pct carbon) in the as carburized and post cyclic deformation conditions respectively [2.9]. The dark regions in Figure 2.3 (a) are plate martensite while the light regions are RA constituent. After 12 strain cycles at 0.5 pct strain amplitude, the microstructure showed the presence of martensitic plates (dark features) resulting from mechanically induced phase transformation (Figure 2.3 (b)).

Figure 2.3 Light optical micrographs of oil quenched 40126 steel under cyclic strain of $\Delta \varepsilon = 0.01$ (a) as carburized undeformed (b) deformed after 12 cycles. Magnification is 850 x [2.9].

In the literature, two mechanisms have been proposed for mechanically-induced martensite transformation: (i) stress assisted martensite transformation [2.10] and (ii) strain-induced martensite
transformation [2.11]. According to the stress-assisted martensite transformation mechanisms, the mechanical work done on or by the transforming region provides the thermodynamic driving force for martensite transformation. The strain-induced martensite transformation mechanism suggests that during plastic deformation, intersecting shear bands give rise to martensite embryos and thus result in martensite transformation. The key differences between the two mechanisms are that in stress-assisted martensite transformation, martensite nucleates at existing sites in the parent phase, while in strain-induced transformation, new nucleation sites are formed during plastic deformation. Additionally, stress-assisted transformation is possible at stress below the yield strength of the material whereas strain-induced transformation occurs at stresses greater than yield strength.

During the quench step of the carburizing treatment, the case of the component cools faster than the core. At elevated temperature, the surface experiences axial and circumferential tension as it tries to shrink against the hot core. However, as the core cools, the rigid shell applies tensile stresses on a shrinking core and experiences compression on itself. Thermal gradients are further accompanied by a time lag in the austenite to martensite transformation between the high carbon and low carbon regions. The austenite in the low carbon core (higher $M_s$ temperature) transforms to martensite at higher temperatures than the high carbon case (lower $M_s$ temperature). A volume expansion (~4 pct) is associated with this transformation and consequently the residual stresses in a component depend on a complex interplay of thermal and carbon content gradients during the processing stages [2.12].

The final step of the carburizing process is low temperature tempering (LTT) between 150 °C and 200 °C. During tempering, fine transition carbides precipitate in the carbon super-saturated martensite. The toughness of the microstructure improves, and residual stresses relax. At temperatures below 200 °C, the RA does not decompose, and compressive residual stresses in the case region are preserved [2.4].

2.3 Bending Fatigue Behavior of Carburized Steels

Carburized components, especially gear steels, are subjected to bending and rolling contact sliding fatigue in their service lives. In this PhD thesis, microstructural aspects pertaining to the bending
fatigue behavior of carburized steels are investigated. Bending fatigue behavior of components is measured in terms of endurance limits which are evaluated by means of S-N plots where S is maximum stress during fatigue cycling and N is the number of cycles. The endurance limit is the maximum value of stress below which the component is said to endure infinite number of stress cycles. Figure 2.4 (a) shows a stress versus number of cycles to failure plot (S-N plot) for a 0.02 wt pct Nb containing 8620 steel that was vacuum carburized at 1050 °C [2.2]. Each open triangle data point represents samples that failed in less than 1 million cycles, while each solid triangle data point represents a sample that did not fail in over 10 million cycles. The endurance limit for the steel was determined to be 758 MPa [2.2]. Bending fatigue endurance limits are a good representation of high-cycle fatigue strength of the alloy. At stress amplitudes greater than endurance limits, within a certain number of stress cycles, test specimens fail by a sequence of crack nucleation, stable crack growth and unstable crack growth stages [2.3]. This sequence is sensitive to microstructural gradients, residual stresses, and experimental conditions.

Cracks can potentially nucleate at surface defects, internal metallurgical or phase/grain boundaries. Intergranular (IG) cracking at prior austenite grain boundaries (PAGBs) is a common mode of fracture in high-carbon case-carburized steels. Intergranular cracks initiate when the bending stresses overcome compressive surface residual stresses and cohesive forces of prior austenite grain boundaries. The crack then propagates in stable transgranular mode until a critical crack length is reached which is followed by an unstable fracture. In case-hardened steels, crack initiation and stable crack growth regions are relatively small due to the low toughness of high-carbon LTT martensite. A fracture surface is shown in Figure 2.4 (b) where the three different regions of crack nucleation, stable crack growth, and unstable crack growth are labelled. The sections below discuss the influence of critical microstructural features on fatigue behavior of carburized steels.
Figure 2.4 (a) Schematic S-N curves for Nb modified 8620 carburized steel tested in bending fatigue [2.2]. (b) An overview of a typical fracture surface from these bending fatigue tests showing crack nucleation, stable and unstable crack propagation regions [2.3].

2.3.1 Prior Austenite Grain Size and Bending Fatigue

The prior austenite grain size (PAGS) affects both crack nucleation and crack propagation aspects of bending fatigue resistance of carburized steels. Generally, finer PAGS leads to better fatigue performance because of the following reasons:

1. A finer PAGS upon quenching results in finer martensite and hence, a high strength case and better high-cycle fatigue resistance

2. Larger grain boundary area leads to dilution of phosphorus concentration along grain boundaries and hence, reduced susceptibility to IG crack initiation along PAGBs

3. Martensite transformation in high carbon austenite produces microcracks along sites where plates impinge, particularly in microstructures with coarse PAGS.

Figure 2.5 shows endurance limits as a function of PAGS (to the power -1/2) for carburized 4320 steels [2.2]. Greater fatigue endurance limits were observed for finer PAGS in the case region. Reheating below the $A_{cm}$ temperature after the carburizing treatment has been effectively used as a microstructure
refinement strategy. The presence of undissolved cementite in the microstructure suppresses PAG coarsening at low austenitizing temperatures. During reheat treatments, austenite reversion takes place. At elevated temperatures, austenite grains nucleate and grow on PAG boundaries and at martensite/austenite interfaces. They subsequently impinge against one another resulting in fine austenitic grains and hence a fine quenched final microstructure. Austenite grain size along the depth of carburized specimen (carburized at 930 °C) is shown in Figure 2.6 for as carburized and multiple reheat cycles (820 °C) [2.13]. Triple reheating treatments led to three times reduction in grain size and small cementite precipitates, which was consistent along the depth from the surface.

![Graph](image)

**Figure 2.5** Hall-Petch relationship between case grain size and endurance limit for gas- and vacuum-carburized 4320 and 8620 gear steel. Endurance limit data were obtained from a cantilever bending modified Brügger geometry [2.2].

Apple and Krauss performed tension-tension fatigue testing on as-carburized (927 °C for 6 h), single-reheat (fully austenitic, 843 °C for 1 h) and double-reheat (inter-critical region, 788 °C for 1 h) specimens [2.14]. Progressively finer microstructures (Figure 2.7 (a)-(c)) were observed for single and double reheat treatments owing to finer PAGS. The tendency towards microcracking during quenching dropped significantly in finer microstructures, and fine microstructures showed dramatically improved endurance limits, as seen in the S-N plots in Figure 2.7 (d).
2.3.2 Residual Stress and Bending Fatigue

Residual stresses shift the mean stress experienced by a component during fatigue cycles. Hence, under tensile residual stress, endurance limits are drastically reduced. Surfaces are prone to crack initiation, and high tensile residual stresses may even induce static fracture in some components [2.3]. Alternatively, fatigue lives can be significantly improved by introducing compressive residual stresses in the surface by optimizing the carburizing process or by post-processing approaches like shot-peening. During crack growth, compressive residual stresses can lead to crack closures and improved fatigue performance. To illustrate the effects of residual stresses on fatigue behavior, Figure 2.8 (a) and (b) show residual stresses for as-carburized and shot-peened (after carburizing) samples as a function of depth in carburized 4320 steel and the corresponding bending fatigue S-N curves, respectively [2.3]. Various shot-peening techniques induce compressive stresses near the surface and, as a result, the endurance limit is significantly increased.
Figure 2.7 Light optical micrographs for nital etched case microstructures of gas-carburized 8620 steel subjected to reheating treatments. (a) As-carburized (927 °C/6 h) and direct quenched (b) single reheat treatment (fully austenitic, 843 °C/1 h) and (c) a subsequent double reheat treatment (intercritical, 788 °C/1 h). (d) S-N curves for the microstructures above [2.14].
Figure 2.8 Role of residual stresses on fatigue behavior of gas-carburized, direct quenched and various shot peened 4320 steel; (a) residual stress profile along the depth and (b) corresponding bending fatigue S-N curves [2.3].

Experimental investigations on the role of residual stresses are rather complex because residual stresses are difficult to predict and measure, and it is difficult to isolate residual-stress effects from other related parameters like case depth and case hardness. In a study by Funatani, the influence of peak compressive stress on endurance limit was investigated for varying case depths and a constant surface hardness [2.15]. Figure 2.9 shows trends in endurance limit as a function of effective case depth for chromium-molybdenum steels; the peak compressive stresses are labeled by an arrow on each curve [2.12]. The study used steels of different hardenability and thus the steels had varying hardness profiles along the depth despite having the same surface hardness. While the endurance limit does not monotonically increase with respect to case depth, higher peak compressive stresses generally resulted in improved endurance limits [2.12].
2.3.3 Retained Austenite and Bending Fatigue

RA is a critical component of the high-carbon case microstructure. Carbon, along with some common alloying elements like nickel, is an austenite stabilizer and hence rapid quenching results in incomplete austenite to martensite transformation and significant amounts of RA. Increased amounts of RA are detrimental to high-cycle fatigue lives but are known to improve low cycle fatigue performance. In high-cycle fatigue situations, the presence of RA lowers the yield strength thereby diminishing fatigue performance. On the other hand, in low cycle fatigue, RA provides improved ductility and enhances the ability to accommodate plastic strains. Additionally, under sufficient stress, RA transforms to martensite, which is accompanied by a volume expansion that resists crack growth during fatigue loading [2.3].

Richman and Landgraf performed fully reversed strain controlled fatigue testing of high-carbon steels to deduce the influence of RA on fatigue behavior of case-hardened steels [2.16]. Quenched-and-tempered 40126 alloys containing 1.26 wt pct carbon were used to simulate the plate martensite and RA microstructure of a carburized case. Figure 2.10 (a) shows peak compressive and tensile stress as a function of cycle number for various strain amplitudes for oil-quenched 40126 [2.16]. The magnitude of
stable compressive stress was greater than the magnitude of stable tensile stress after cycling, suggesting that mean compressive stresses developed during strain-controlled cycling due to the RA to martensite transformation. Figure 2.10 (b) shows strain amplitude versus number of reversals to failure data for oil quenched 40126 steel (high initial RA content) and liquid N$_2$ quenched 40126 steels (low initial RA content). The oil-quenched 40126 was more ductile (strain amplitude corresponding to one reversal to failure) and was more fatigue resistant. They suggested that the transformation of RA to martensite influenced the mechanical behavior in the following three ways:

1. The stress/strain induced martensite transformation shear provides a deformation mechanism for otherwise brittle high-carbon martensite.
2. The stress-assisted transformation product was more ductile than the thermally produced martensite (liquid N$_2$ quenched).
3. RA to martensite transformation generated a stable mean compressive residual stress in the component during the initial fatigue cycles (Figure 2.10 (a)).

Richman and Landgraf further hypothesized that an optimum amount of RA should be purposely designed in microstructures to ensure development of compressive residual stresses during fatigue cycling and thus longer lives in the elastic regime. RA also allows for possibilities of accommodating high strain excursions [2.16].

Jeddi and Lieurade investigated RA fractions and residual stress in a 14Ni-11Cr steel with different starting initial RA fractions during cyclic loading close to the endurance limit [2.17]. Similar to Richman and Landgraf [2.16], they found that a stable RA fraction and residual stress state are established in the initial loading cycles which govern the subsequent fatigue behavior. They also concluded that the transformation manifests as compressive residual stress that improves fatigue lives. The higher the amount of transformed martensite, the larger the stabilized compressive stress associated with the transformation induced volume expansion and subsequently, a greater improvement in fatigue performance [2.17].
Figure 2.10 (a) Maximum and minimum fatigue stress as a function of cycle number for several applied strain amplitudes for oil quenched 40126 steels, and (b) Strain based S-N curves for oil quenched and liquid N2 quenched 40126 steel [2.16].

2.4 Fatigue Crack Propagation in Steels

When a component containing a crack is subjected to constant cyclic loading, the crack length \( a \), increases with the number of fatigue cycles \( N \), for a constant loading amplitude \( \Delta P \), load ratio \( R \) (\( P_{\text{Min}}/P_{\text{Max}} \)) and test frequency. The fatigue crack growth rate \( da/dN \) increases with the number of cycles and can be represented as a function of \( \Delta P \) and \( a \) through the cyclic stress intensity parameter \( \Delta K \). This parameter represents stresses around a crack tip and a correlation exists between \( da/dN \) and \( \Delta K \) [2.18]. Metallic structural materials show three distinct regions in a \( da/dN \) versus \( \Delta K \) curve, as schematically shown in Figure 2.11. Region I exhibits a \( \Delta K_0 \), threshold value below which cracks do not propagate. Region II shows a linear slope, representing a stable crack growth regime. In contrast, region III depicts the unstable crack growth regime where fatigue crack growth (FCG) rates increase drastically as \( K_{\text{Max}} \) tends towards \( K_c \) [2.18].
The sigmoidal nature of the curve can be characterized by different modes of fracture [2.19]. In region II, transgranular fracture dominates and is largely insensitive to microstructure and mean stress. At high growth rates in region III, $K_{\text{Max}}$ tends towards $K_{\text{lc}}$; in this case, static modes of fracture like cleavage, micro-void coalescence and intergranular fracture may activate. In the ‘near threshold region’, there is a strong influence of microstructure, mean stresses and environmental factors [2.19].

2.4.1 Fatigue Crack Propagation in Embrittled Steels

Conventionally in metallic materials, in the ‘continuum controlled’ stage II stable fatigue crack growth regime [2.20], the crack propagates via ductile striations mechanism [2.21], and is largely insensitive to the microstructure. Ritchie and Knott [2.22] investigated the FCG behavior of high-strength medium-carbon steel (En30A, 0.35 wt pct carbon) susceptible to temper embrittlement. They studied the effect of mean stress on FCG rates in un-embrittled (tempered at 650 °C for 1 h) and temper embrittled steels (tempered at 540 °C for 8 h). Figure 2.12 (a) and (b) show the FCG rates versus $\Delta K$ plots for different values of load ratios for the un-embrittled and the embrittled steel respectively. A greater value of load ratio represents a higher value of mean stress during the fatigue cycling. Ritchie and Knott
observed that the FCG rates were insensitive to the value of mean stress for un-embrittled steels. However, for the temper embrittled steel, FCG rates were higher than un-embrittled steel and were strongly dependent on the load ratio and mean stress. The Paris law exponent $m$ also increased with increasing mean stress. The observed increase in FCG rates, dependence on mean stress and higher values of exponent $m$ were attributed to the occurrence of static modes of failure in the embrittled steels.

Figure 2.12 (c) and (d) show fracture surfaces of the un-embrittled and embrittled steels. The white arrows indicate the direction of crack propagation. In the un-embrittled steel, the fatigue crack propagated predominantly via ductile striations through the tempered martensite. In the embrittled steel, a significant amount of IG fracture was observed at a value of $K_{\text{max}}$ as low as half of $K_{Ic}$.

Irving and Kurzfield proposed two possible reasons for the occurrence of IG fracture during fatigue [2.23]. In high-strength steels, IG fracture was attributed to segregation of impurities to PAGBs resulting in high FCG rates. The other proposed mechanism was based on hydrogen derived from water vapor containing environments and did not affect the FCG rates. This mechanism was prevalent in the near threshold FCG behavior. Ritchie [2.24] suggested that both impurity segregation and presence of hydrogen reduce the cohesion at PAGBs and promote IG fracture during fatigue in temper-embrittled 300-M steel in the near-threshold FCG regime. Chapter 4 in this thesis discusses fatigue crack propagation in high-carbon plate-martensite austenite microstructures that are susceptible to quench embrittlement [2.25].
Figure 2.12 Fatigue crack propagation in low alloy steels. FCG rate versus $\Delta K$ for (a) un-embrittled and (b) embrittled steel. SEM images of fatigue fracture surfaces in (c) un-embrittled steel at $K_{\text{max}} = 47 \text{ MPa.m}^{1/2}$ and (d) embrittled steel at $K_{\text{max}} = 22 \text{ MPa.m}^{1/2}$[2.22].
2.4.2 Deformation Induced Martensite Transformation and Fatigue

Steels that contain metastable austenite are susceptible to deformation induced martensite transformation (DIMT) in the presence of stresses or strains. During fatigue crack growth, the crack tip stress/strain fields can induce localized phase transformation of RA to martensite in the vicinity of the crack tip, which can significantly alter the FCG resistance of the microstructure. In the literature, a range of mechanisms have been proposed to rationalize the influence of DIMT on FCG behavior. The volume expansion associated with the RA to martensite transformation imposes a compressive residual stress at the crack tip, which opposes the applied tensile stress, thereby resulting in reduced FCG rates [2.26]. Antolovich and Singh [2.27] suggested that the RA to martensite transformation absorbed energy during fracture, resulting in less energy available for crack extension. The transformation product, fresh martensite, is hard and brittle, which can lead to crack path deflection and reduction in crack propagation rates [2.28]. However, since the transformation product is brittle, the fresh martensite can lower the localized toughness, act as potential site for crack nucleation and deteriorate the FCG resistance of the microstructure [2.29, 2.30].

Mei and Morris [2.31] investigated the influence of mechanically induced martensite formation on fatigue resistance of 304 austenitic stainless steels (SS-304, metastable at low temperatures). Steels with two different compositions were subjected to fatigue loading (different R-ratios) at room temperature and liquid nitrogen temperature. Figure 2.13 (a) and (b) shows the comparison of FCG data and a representative crack tip in SS304L, highlighting martensitic transformation (using ferrofluid) around the crack tip. Low temperature, unstable austenite and greater $K_{\text{max}}$ promoted a larger amount of transformation in the vicinity of the crack tip. A 10 times reduction in FCG rate was observed in SS304LN (austenite stabilized using interstitial N) in the low $\Delta K$ regime as compared to SS304L at cryogenic temperatures (77 K) at $\Delta K = 10 \text{ MPa.m}^{1/2}$, resulting from larger amounts of mechanically-induced martensite in SS304L. $\Delta K_{\text{th}}$ increases with the increase in austenite to martensite transformation. The authors suggest mechanisms like perturbation of crack tip stress fields and crack deflection to explain
the improvement in resistance to fatigue crack propagation due to mechanically-induced martensite formation.

Wang et al. [2.33] investigated the role of DIMT on FCG in model medium Mn steels (0.2C-10Mn-3Al-1Si in wt pct). Using inter-critical annealing heat-treatment, they developed duplex ferrite-austenite microstructures with different austenite volume fraction and mechanical stability, and these conditions were fatigue tested. Figure 2.14 shows the FCG behavior of the two duplex microstructures with high and low amounts of DIMT. In the short-crack growth regime at low \( \Delta K \), the microstructure with higher amount of DIMT had better FCG resistance, and exhibited abnormal crack growth, such that upon encountering martensite, the crack was arrested. Once the crack-tip overcame the interface barrier, the crack grew rapidly inside the martensite grain. In the long crack growth regime, the microstructure with higher amount of DIMT had lower FCG resistance, and no crack-arrest was observed near martensite regions. Instead, secondary microcracks were observed near the fatigue crack, which lead to increased FCG rates via crack coalescence (transformation mediated crack coalescence, TMCC). The

Figure 2.13 (a) Fatigue crack growth rates in liquid N\(_2\), and (b) an optical micrograph highlighting martensite transformation in the vicinity of crack tip [2.31].
micro-cracks formed at the interface of austenite and brittle martensite due to the fatigue loading. A greater amount of DIMT improved FCG resistance in the stage I short crack regime but was detrimental to the FG resistance during stage II long crack regime. Chapter 4 and 5 in this thesis discuss the influence of DIMT on FCG behavior of high-carbon plate-martensite austenite microstructures.

![Graph showing the influence of DIMT on FCG behavior](image)

Figure 2.14 The influence of DIMT on FCG behavior in model medium Mn duplex ferrite austenite microstructures [2.32].

### 2.5 Summary

This chapter presented background information on the carburizing process, reheat treatment and the resulting microstructures. The carburizing process introduces gradients in carbon content, martensite morphology, hardness, RA content and residual stresses along the depth of the carburized components. The low-carbon case region contains lath-martensite while the microstructure in the high-carbon case regions consists of plate-martensite and RA (occasionally undissolved cementite). Further, the metastable RA in the case regions is susceptible to DIMT during service.

Microstructural factors affecting the bending fatigue behavior of carburized components were reviewed. Refinement in PAGS by reheating treatment results in improved bending fatigue strength since
finer PAGS have stronger martensite, lower susceptibility to quench cracking and fatigue crack nucleation (phosphorus dilution along PAGBs). Thermal processing and transformation induced compressive residual stress in the case regions lowers the mean stress experienced by the carburized component and results in greater fatigue lives. There is some ambiguity in the role of RA on fatigue behavior of carburized steels. During low-cycle fatigue, the presence of RA improves ductility, allows for plastic accommodation. The DIMT during service imposes compressive residual stress in the microstructure and improves fatigue performance. However, in high-cycle fatigue, presence of RA is detrimental to the fatigue strength, and the brittle martensite (DIMT product) acts as potential crack nucleation site.

A general overview of fatigue crack propagation in steels was presented. In un-embrittled steels, fatigue crack propagates via ductile striations mechanisms, while in the embrittled steels, static modes of failure such as IG fracture and cleavage are also present along with fatigue modes of failure. The influence of DIMT on fatigue crack propagation in metastable austenite containing steels is not well established. The volume expansion associated with DIMT imposes compressive stress at the crack tip, thereby reducing the effective ΔK and improving FCG resistance. However, DIMT product, fresh martensite lowers the local toughness and accelerates crack propagation.

This PhD thesis aims to better understand the role of RA on the FCG resistance of high-carbon plate-martensite austenite microstructures for potential applications in carburized and bearing steels. Microstructures with varying amounts and stability of RA were developed, fatigue tested and characterized to investigate the effect of DIMT on fatigue crack propagation.

2.6 References


2.4 G. Krauss, “Microstructures and Properties of Carburized Steels”, Heat Treating of Irons and


CHAPTER 3
MICROSTRUCTURE REFINEMENT STRATEGIES IN HIGH-CARBON PLATE-MARTENSITE AUSTENITE MICROSTRUCTURES

The following chapter discusses implementation of microstructure refinement strategies in high-carbon plate-martensite austenite microstructures because of their potential for improving the fatigue performance of case-carburized components and bearing steels. Further, stereological methods were developed to quantitatively characterize the refinement in PAGS, plate-martensite and RA constituents. Section 3.1 discusses relevant literature pertaining to microstructure development in high-carbon steels. Section 3.2 presents the thermal processing strategies and stereological characterization methods used in this study. Two different thermal strategies are presented: (i) thermal cycling, and (ii) step-quenching. The quantitative microstructural characterization results for the thermal cycling and step-quenching strategies are presented and discussed in Sections 3.3 and 3.4 respectively.

3.1 Background
Carburized steel components find applications in vehicle powertrains, machines, and power-generation equipment where high strength is required in combination with good fatigue resistance [3.1]. These desirable properties can be attributed to complex microstructures consisting of plate martensite and retained austenite (RA) in the carbon-rich case and lower-carbon microstructures in the core. There is a gradient of carbon concentration and hence RA content, microstructure, and hardness along the depth of the components [3.2]. Wise and Matlock [3.3] performed a statistical analysis to correlate microstructural variables with bending-fatigue endurance limits of carburized steels. Case grain size is the most critical parameter that influences endurance limits in carburized components [3.3]. Finer austenite grains result in improved fatigue lives, which is hypothesized to be due to refinement of martensite plates, phosphorus dilution along austenite grain boundaries, and lower susceptibility to quench micro-cracking [3.2]. Apple and Krauss [3.4] showed that refinement of prior austenite grains in the case regions using reheating cycles led to improvements in fatigue performance. During reheating to elevated temperatures, austenite
grains nucleate and grow at PAGBs and at martensite/austenite interfaces. Because of the multitude of grain nucleation sites, a finer PAGS can be achieved. Krauss [3.2] reported that triple reheating treatments led to a three times reduction in PAGS of carburized 4320 steel throughout the carburized case.

The mechanism of transformation of martensite from austenite is displacive and occurs at about the speed of sound in steel [3.5]. As a result, it is experimentally complicated to study, resolve, and interpret the nucleation and growth events. Two common martensite morphologies are found in steels: lath and plate, where the latter is common in high-carbon microstructures. Three different modes of kinetics are predominantly discussed in the literature: athermal, isothermal, and burst kinetics [3.6]. Extensive literature on isothermal and burst kinetics of martensite growth exists for Fe-Mn-Ni systems [3.7, 3.8]. Athermal martensite is usually observed in low-alloy carbon steel [3.5]. In most steels, transformation of austenite to martensite proceeds upon lowering the temperature below the martensite start temperature, $M_s$, and is not a function of time (because of rapid rate of transformation), hence the name athermal. The transformation extends to a temperature range between $M_s$ (martensite start) and a certain temperature $M_f$ (martensite finish) below which no transformation takes place even in the case of the presence of untransformed austenite. Under isothermal conditions at a temperature between $M_s$ and $M_f$, the transformation rate rapidly slows, but increases upon further lowering of temperature [3.6]. The kinetics of martensite transformation depend on the nucleation rate of martensite plates, as the plates grow rapidly thereafter. Therefore, the extent of transformation is dictated by the undercooling below the $M_s$ temperature (thermodynamic driving force for nucleation) and not on the time available for growth (athermal) [3.7]. Olson and Cohen [3.9,3.10] suggest that pre-existing defects in fcc (austenite) serve as favorable nucleation sites for martensite. Recent molecular dynamics simulations also discuss the thermodynamics and kinetics of several atomistic transformation mechanisms responsible for nucleation of martensite on defects in the austenite [3.11]. The structure of the prior austenite grain (size and density of defects) is thus, one of the most important factors that influences the athermal martensite transformation kinetics [3.12].
Previously, researchers have studied the effect of interrupted quenching on microstructure evolution in commercial steels. In low-carbon transformation induced plasticity (TRIP) steels containing alloying additions that suppress carbide precipitation, carbon can partition from supersaturated martensite into untransformed austenite upon aging during interrupted quenching [3.13]. The carbon-rich untransformed austenite can thus be chemically stabilized. In a Fe-0.8C high-carbon steel, Van Bohemen et al. [3.5] showed that partial bainite transformation during interrupted quenching at 325 °C for 3-5 mins stabilizes austenite. The volume change associated with bainite, and martensite transformation deforms and strengthens the surrounding austenite, thereby resulting in mechanical stabilization of RA. Interrupted quenching can thus chemically or mechanically stabilize the austenite and affect the subsequent martensite transformation.

In several published papers, stereological methods have been used to investigate and mathematically formalize the mechanisms and kinetics of martensite formation and to characterize the plate-martensite-and-RA microstructure [3.14–3.16]. Previous stereological studies also suggest that PAGBs strongly influence the nucleation of martensite. In the early stages of transformation, Chang et al. [3.14] observed that in coarse-grained microstructures, a greater fraction of martensite plates nucleate at PAGBs than in fine-grained samples. However, in the later stages of transformation, no differences were observed for the autocatalytic tendencies of the plates (i.e., nucleation of new plates on existing plates) at PAGBs and within austenite grains [3.14]. PAGS also influences the so-called “spread” of martensite (spatial distribution of martensite plates during transformation) [3.16]. The spread of transformation kinetics is different for large and small grains. Large grains promote uniform spread and fill-in of martensite transformation, whereas conditions with finer grain size contain partially transformed clusters of grains and in the later stages of the transformation, plate formation gradually spreads in all austenitic grains [3.17].

PAGS influences both the martensite transformation and the endurance limits in carburized components. However, the link between martensitic microstructure and fatigue performance is not well established. In this investigation, stereological methods are employed to provide a quantitative basis for
the evaluation of refinement in plate-martensite and RA constituents associated with thermal cycling and step quenching. Additionally, step-quenching is introduced as a potential heat treatment strategy to stabilize RA in high-carbon plate-martensite-and-austenite microstructures.

3.2 Experimental Methods

The following section presents the alloy composition, thermal processing strategies and microstructure characterization methods used in this work. Section 3.2.1 and Section 3.2.2 present the material and samples used in this study. Two microstructure refinement strategies: (i) thermal cycling, and (ii) step quenching were implemented as described in Subsection 3.2.3. Microstructures thus developed were quantitatively characterized using methods discussed in Subsection 3.2.4.

3.2.1 Material

In this study, commercial 52100 high-carbon steel is used to develop plate-martensite and RA containing microstructures. Studies on 52100 steel help better understand the evolution of microstructure in the case region of a case-hardened steel and bearing steel during thermal processing and fatigue application. The carbon content of 1 wt pct is within the range expected for carburized steels, and a similar microstructure develops during heat treatment.

Hot rolled and air-cooled 3.25 in diameter (82.5 mm) cylindrical bars of steel were received from TimkenSteel for this research. The as-received 52100 steel consisted of proeutectoid cementite along PAGBs and pearlite colonies with an initial colony size of 68.9 μm ± 3.9 μm. The composition of the steel is given in Table 3.1. 52100 steel, conventionally used in bearing applications, was selected due to its commercial availability and presence of existing literature on fatigue behavior. Additionally, using Andrew’s non-linear equation for $M_s$ temperatures [3.18],

$$M_s (^\circ C) = 512 - 453 C - 16.9 \text{Ni} + 15 \text{Cr} - 9.5 \text{Mo} + 212 C^2 - 71.5 C \text{Mn} - 67.6 C \text{Cr}$$  (3.1)

the estimated $M_s$ temperature of 52100 steel is 174 °C, which is comparable to $M_s$ temperatures for case region compositions of 8620 (180 °C) and 4320 (174 °C) steels assuming a carbon content = 0.9 wt pct (an average of 0.8 wt pct and 1 wt pct commonly observed in carburized components). Since the extent of
martensite transformation depends on undercooling below the $M_s$ temperature [3.7], similar amounts of RA fractions are expected at room temperatures for austenitized and quenched 52100 steel and shallow case regions of 8620 or 4320 steel carburized components.

Table 3.1 – Composition of 52100 Steel Provided by TimkenSteel (wt pct)

<table>
<thead>
<tr>
<th>Alloy</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>V</th>
<th>Al</th>
<th>N</th>
<th>S</th>
<th>P</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>52100</td>
<td>1.00</td>
<td>0.35</td>
<td>0.26</td>
<td>0.13</td>
<td>1.40</td>
<td>0.05</td>
<td>0.004</td>
<td>0.024</td>
<td>0.006</td>
<td>0.015</td>
<td>0.006</td>
<td>0.17</td>
</tr>
</tbody>
</table>

3.2.2 Sample Machining

As-received 52100 steel bars were machined into dilatometry specimens. Small cylindrical samples, 4 mm in diameter and 10 mm in length were obtained from the half radius position of the steel bar using wire electrical discharge machining (EDM). From a given cross-section of the steel bar, 16 equidistant small cylinders were machined from the half radius circle, as shown in Figure 3.1. A total of 96 dilatometry samples were obtained from a 6 cm long section of bar steel. The cylindrical axis of the sample was parallel to the rolling direction of the hot-rolled bar steel.

![Figure 3.1 Schematic drawing of dilatometry specimen and their location within the 52100 steel bar product (mid-radius circle).](image)

3.2.3 Thermal Processing

The cylindrical samples (diameter – 4 mm, length – 10 mm) were subjected to controlled heat-treatments in a TA Instruments Type 805L quenching dilatometer. In the dilatometer, the sample is heated within an induction coil and temperature is controlled using a thermocouple spot-welded onto the center
of the sample. The induction coil has spiral gas ducts which can be used to purge gas such as nitrogen
(N\textsubscript{2}), argon (Ar) or helium (He) for controlled cooling. Austenitizing and quenching heat treatments were
performed to develop plate martensite and RA containing microstructures. Two microstructure refinement
strategies, thermal cycling and step-quenching are described below.

**Thermal Cycling**

All samples were heated to 1000 °C at 5 °C·s\textsuperscript{-1} to ensure complete austenitization and carbide
dissolution (constant for all subsequent heat treatments), held isothermally for 100 s and quenched to
room temperature at 10 °C·s\textsuperscript{-1} to form a high-carbon plate-martensite austenite microstructure (hereby
called DQ, direct quenched). In the subsequent thermal cycling step, samples were reheated to three
different austenitizing temperatures of 900 °C, 1000 °C or 1100 °C, held isothermally for 100s and
quenched at 20 °C·s\textsuperscript{-1} to ensure martensitic formation (hereby called RH900, RH1000, and RH1100,
respectively). Further, to study the influence of time, a short reheating treatment was performed at 900 °C
for 1s. Up to three short reheating cycles (designated as RHS – n where n is number of cycles) at 900 °C
for 1s (with an interval of 60 s in between subsequent cycles) were also carried out to study the evolution
of PAGS during thermal cycling. Figure 3.2 (a) shows the summary of thermal cycles. The starting
microstructure for all the microstructures was plate-martensite and RA.

**Step Quenching**

A novel heat treatment, ‘Step-Quenching’ (referred to as SQ100) was implemented using the
dilatometer. During step quenching, the martensite transformation was interrupted by performing an
isothermal hold at a temperature, \( T_{SQ} \), below the \( M_s \) temperature, thereby resulting in primary and
secondary martensite transformation steps. It is hypothesized that during interrupted quenching,
martensite plate growth stops, and new martensite plates nucleate and grow upon subsequent quenching,
thereby resulting in finer martensite plates.

To perform step-quenching, samples were heated to 1000 °C at a heating rate of 5 °C·s\textsuperscript{-1} and
isothermally held for 100 s under vacuum. This time and temperature combination ensured a fully
austenitic microstructure with carbide dissolution. The samples were quenched to 100 °C ($M_s = 142$ °C) using argon gas with a cooling rate of 10 °C·s$^{-1}$, held isothermally for 1000 s and subsequently quenched to room temperature at 10 °C·s$^{-1}$ to form a plate-martensite and RA microstructure (Figure 3.2 (b)).

![Figure 3.2 Schematic diagram showing thermal history during (a) thermal cycling and (b) step quenching.](image)

3.2.4 Microstructure Characterization

The heat-treated samples were characterized using dilatometry data, light optical and scanning electron microscopy, and quantitative stereological analysis methods to understand the evolution of microstructure during the thermal cycling and step quenching. The details of the characterization methodology are presented below.

Dilatometry

The length of the sample and temperature data were recorded during controlled thermal cycling and step-quenching in the dilatometer. The trends in the relative change in length as a function of temperature were analyzed to study the microstructural response and the phase transformation behavior of the material during the heat-treatments.
Metallography

The heat-treated dilatometry samples were cross sectioned along their length using a TechCut 4™ (Allied High Tech Products Inc.) slow speed saw and hot mounted in Bakelite using a Leco® MX400 mounting press. Mounted samples were metallographically prepared by mechanical grinding, polishing, and chemical etching. Manual mechanical grinding was performed using a series of silicon carbide grit papers with grit numbers 180, 240, 320, 400 and 600 grit papers in that order (coarse to fine). Further, polishing steps were performed on a Leco® Spectrum System 2000 by using a diamond suspension solutions. The wheel speed was set to 150 rpm clockwise while the holder rotation speed was set to 75 rpm counterclockwise. The details of the auto-polishing procedure such as duration, polishing media and applied load are presented in Table 3.2. The polishing pads were alternatively sprayed with diamond suspension and alcohol based Leco® blue extender every 30 seconds.

Microscopy

Polished samples were etched with saturated picric acid (4 g picric acid, 100 mL water, 8 mL Teepol surfactant, and 4 drops HCl) to reveal prior austenite grain boundaries. To etch, the sample surface was heated for approximately 30 s using a heat gun, immersed in heated saturated picric acid solution (65 °C) for 60 s, and finally cleaned with a cotton swab and de-ionized water. Light optical micrographs were collected on the Leco® DSX 500 microscope and the Abram 3 circle method (ASTM E112) was used to determine the prior austenite grain size (PAGS) in ImageJ® software. More than 400 grains were sampled for determination of the PAGS.

Metallographically prepared sample surfaces were chemically etched with a 2 pct nital solution (1 mL nitric acid and 49 mL ethanol) for approximately 15 s to reveal the microstructural constituents. Etched samples were observed under a JEOL® 7000 field emission scanning electron microscope (FE-SEM) to identify phases present in the microstructures and perform quantitative stereological analysis. The FE-SEM was operated at an accelerating voltage of 20 keV and a working distance of 10 mm.
Table 3.2 – Auto-Polishing Steps used for Metallographic Sample Preparation

<table>
<thead>
<tr>
<th>Polishing Media</th>
<th>Polishing Pad</th>
<th>Load per Sample (lbf)</th>
<th>Duration (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>9 µm</td>
<td>Leco Ultra Silk</td>
<td>8</td>
<td>5</td>
</tr>
<tr>
<td>6 µm</td>
<td>Leco Ultra Silk</td>
<td>7</td>
<td>5</td>
</tr>
<tr>
<td>3 µm</td>
<td>Leco Ultra Silk</td>
<td>6</td>
<td>5</td>
</tr>
<tr>
<td>1 µm</td>
<td>Leco Imperial</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>0.5 µm</td>
<td>Leco Imperial</td>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td>0.05 µm</td>
<td>Leco Imperial</td>
<td>3</td>
<td>5</td>
</tr>
</tbody>
</table>

Stereological Analysis

The heat-treated, cross-sectioned, hot-mounted, and metallographically polished samples were etched with saturated picric acid to reveal the PAGBs and with 2 pct nital to quantify the martensite plates and RA sizes. The Abrams three-circle method was used to evaluate the PAGS of samples from picral-etched specimens according to ASTM E112. More than 400 prior austenite grains were measured. Polished cross-sections were also etched with 2 pct nital to reveal plate martensite and RA microstructures. Stereological measurements of the martensite micro-geometry were performed on scanning electron micrographs by standard stereological techniques such as point counting and lineal intercept counting [3.19].

Microstructures in metals and alloys contain 3-dimensional features (such as precipitates), 2-dimensional surfaces (such as grain boundaries), and 1-dimensional linear elements (such as dislocations). During metallographic analysis of 2-dimensional cross-sections, these microstructural features manifest as area sections, linear traces, and points in the plane of the observation, respectively [3.20]. Probability and statistics based stereological methods provide relationships between 3-dimensional features of the microstructure and the 2-dimensional observable properties of the micrographs [3.20]. Conventional notations used in stereology and fundamental relationships (Equations 3.2 – 3.4) are discussed below. The
derivations of these equations and a discussion of their accuracy is provided elsewhere [3.19,3.20].

The notation used here, and in the literature, consists of a geometrical element ‘E’ and a geometrical dimension ‘D’. The symbol $E_D$ represents the quantity of $E$ per unit dimension $D$; for example, $N_V$ represents the number of features per unit volume. Inaccessible three-dimensional features like $S_V$ or $L_V$, where $S$ is surface area, $V$ is volume and $L$ is length, can be expressed in terms of two-dimensional quantities using mathematical relationships [3.19, some of which are listed below.

\[
V_V = A_A = L_L = P_P
\]  

\[
S_V = \frac{4}{\pi} L_A = 2 P_L
\]  

\[
L_V = 2 P_A
\]

where $P$, $L$ and $A$ are point, length and area respectively.

Figure 3.3 schematically shows the evaluated stereological quantities: surface area per unit volume of martensite midplane ($S_V^{MID}$) and length per unit volume of midplane periphery ($L_V^{MID}$). Chen and Winchell proposed a correlation between shape-independent stereological features and the morphological aspects of plate martensite. In this study, two parameters were studied: $t_{avg}$ and $r_{avg}$ [3.14].

\[
t_{avg} = V_V / S_V^{MID}
\]  

\[
r_{avg} = 2S_V^{MID} / L_V^{MID}
\]

The $t_{avg}$ is the average thickness of martensite plates weighted by the midplane surface area ($S_V^{MID}$), $r_{avg}$ is the average distance between the center of the plate and its periphery along the midplane, and $V_V$ is the volume fraction of martensite (see Figure 3.3). Hereby, $r_{avg}$ will be referred to as the average martensite plate radius. Three-dimensional quantities in equations 3.5 and 3.6 can be expressed in terms of two-dimensional properties using equations 3.2 – 3.4:

\[
t_{avg} = P_P / 2P_L^{MID}
\]  

\[
r_{avg} = 2P_L^{MID} / P_A^{MID}
\]

The point fraction of martensite, $P_P$, was determined by overlaying a grid of points on a microstructure and calculating the fraction of points lying within martensitic regions. $P_L^{MID}$ was
determined by overlaying parallel lines on the microstructures and calculating the number of intersections between test lines and midribs per unit length of test line. $P_A^{\text{MID}}$ was determined by counting the number of martensite plate ends per unit area of the microstructure.

Figure 3.3 A schematic drawing showing various stereological quantities measured during the investigation.

To analyze the size distribution of RA constituents, image processing was performed using MIPAR™ software. Using thresholding, areas of austenitic regions were identified in 2-D planes and their circular equivalent diameters were determined. The size distribution of the RA constituents per unit area was determined, given by $(N_A)_i$, as the number of RA constituents in each class interval, $i$, with diameter $D_i$. The average RA constituent diameter, $D_{\text{avg}}$ is given by

$$D_{\text{avg}} = \frac{1}{N_V} \left( \sum (N_V)_i D_i \right)$$

where $(N_V)_i$ is the number of constituents per unit volume in each class interval. $N_V$ is not a measurable quantity in micrographs; however, it can be correlated to $N_A$ by considering the probability of the test plane sectioning the RA constituents. The Schwartz-Saltykov method was employed to determine the RA constituent size distribution $(N_V)_i$ [3.19]. The method was originally proposed for a poly-dispersed system of spheres, with inaccuracies resulting from non-spherical shapes. However, reasonable trends can be determined for equiaxed and spheroidal particles with this analysis assuming an isotropic and uniformly
scattered distribution of particles [3.19]. Takahashi and Suito [3.21] evaluated the accuracy of the Schwartz-Saltykov method and found that the histogram of size distribution of particles was independent of the number of intervals, even for a small number of intervals. Thus, in this study, the analysis of RA sizes will be based on the size distribution histograms.

**Neutron Diffraction**

To further understand the differences between direct and step-quenched microstructure, neutron diffraction scans were performed on the dilatometer samples at the Spectrometer for Materials Research using Temperature and Stress (SMARTS) at the Lujan Neutron Scattering Center, Los Alamos National Laboratory (LANL). The details of the instrument configuration and neutron diffraction data analysis methodology are included in Appendix A. In the field of materials science, neutron diffraction has been employed to study phase transformations, micromechanics, residual stresses, crystallographic texture, and kinetics of microstructural evolution [3.22]. Neutron diffraction is better suited for bulk samples due to higher penetration depths as opposed to surface analysis using x-ray diffraction.

SMARTS was used to study the differences in phase fractions and lattice parameters of the DQ (single thermal cycle at 1000 °C for 100 s) and SQ condition. Notably, for the neutron diffraction study, step quenching was performed at 110 °C for 1000 s (hereby referred to as SQ110 sample). The DQ and the SQ110 samples were mounted at an angle of 45° to the incident neutron beam (schematically shown in Figure 3.4), such that the diffraction vector along the radial direction was recorded in bank 1, while the axial diffraction pattern was recorded in bank 2. The cross-section of the incident beam was 2 mm x 2 mm, and radial collimators were used resulting in a probed volume of 8 mm³. The diffraction patterns were recorded from the center of the bulk heat-treated and compressed samples for 1.5 h each. Rietveld analysis was performed with GSAS to determine average lattice parameters, and phase fractions.
3.3 Results and Discussion – Thermal Cycling

Cylindrical samples of 52100 steel were subjected to controlled heat-treatments in a dilatometer with an aim to evaluate plate martensite-austenite microstructure refinement strategies. The two approaches, step-quenching and thermal cycling are evaluated. Section 3.3.1 presents and discusses the dilatometry data corresponding to the thermal cycling heat-treatment. Representative optical and electron micrographs of the thermal cycling are shown in Section 3.3.2. Quantitative stereological analysis of PAGS, plate-martensite microgeometry, and RA constituents are presented in Sections 3.3.3.

3.3.1 Dilatometry

Figure 3.5 (a) shows a typical quenching dilatometry curve (relative change in length, \( \text{rel. } \Delta l \) versus temperature) for high-carbon 52100 steel consisting of heating to 1000 °C, isothermal holding for 100 s, and quenching to room temperature, as highlighted. Selected regions of the curve shown in Figure 3.5 (b), (c) and (d) correspond to austenite reversion during heating, isothermal holding, and martensite formation during cooling, respectively. The arrow markers show the directionality of the \( \text{rel. } \Delta l \) representing the thermal history (heating and then cooling). Upon heating the as-received microstructure, the sample expands in length according to its coefficient of thermal expansion. Above the \( A_1 \) temperature, the austenite phase starts forming. Since austenite (FCC) is the high density close-packed
structure, this transformation is accompanied by a contraction in length. Upon further heating, the microstructure completely transforms to austenite at the $A_{CM}$ temperature followed by an increase in the sample length according to the coefficient of thermal expansion of austenite. The $A_f$ and $A_{cm}$ temperatures were 780 °C and 793 °C, respectively. The rel. $\Delta l$ of the sample as a function of temperature during austenite formation is shown in Figure 3.5(b). The reduction in rel. $\Delta l$ represents the inter-critical region of the phase diagram. The dilatometry data from isothermal holding is shown in Figure 3.5(c). The noise in temperature data is most likely an outcome of system noise due to the transition between heating and isothermal holding segments of the heat treatment. However, the key aspect of the data is an increase in the length of the sample by approximately 0.01 pct. During the isothermal holding, temperature homogenizes within the bulk of the sample and carbides dissolve. The compound effect of these phenomena results in the volume expansion thus observed. The martensitic transformation is shown in Figure 3.5(d). Insignificant changes in the slope of the cooling curve between 1000 °C and 200 °C temperature indicate that the cooling rate of 10 °C·s$^{-1}$ suppressed the formation of non-martensitic transformation products. The expansion in length below 142 °C ($M_s$ temperature, the onset of deviation from linearity in the rel. $\Delta l$ during cooling) can be attributed to the formation of plate-martensite.

3.3.2 Microscopy

Figure 3.6 (a) shows nital etched light optical micrographs of the as-received 52100 steel. The microstructure consisted of pearlite colonies with a mixture of alternate dark (cementite) and light (ferrite) lamellar constituents. Additionally, the PAGBs were decorated with hyper-eutectoid cementite. Figure 3.6 (b) shows a nital-etched optical micrograph of an austenitized and direct quenched sample. The microstructure consisted of plate-martensite (dark) and RA constituents (light) regions. Reheating to 1000 °C resulted in a ferrite to austenite phase transformation along with dissolution of cementite. Upon quenching to room temperature at 10 °C·s$^{-1}$, diffusionless transformation of austenite to plate-martensite was observed, and non-martensitic transformation products (NMTP) such as bainite and ferrite were avoided. Since austenite to martensite transformation was incomplete, metastable RA constituents were
also observed. A small fraction of large carbides remained undissolved along PAGBs for all conditions. However, small carbides dissolved during reheating, and no grain pinning effect is expected from the remaining coarse carbides.

Figure 3.5 A typical dilatometry curve for austenitized and quenched 52100 steel (a) An overview, (b) austenitization during heating, (c) isothermal holding and (d) martensite formation.
After the initial austenitization and quenching step, reheating thermal cycles were performed at 900 °C, 1000 °C, and 1100 °C as described in Section 3.2.3. Figure 3.7 (a)-(c) show optical micrographs of the RH900, RH1000, and RH1100 samples respectively, etched with saturated picric acid. Etching with picric acid reveals the prior austenite grain boundaries. Due to over-etching, the plate-martensite and RA substructure is also visible. Notably the scale bar in Figure 3.7 (c) is different from Figure 3.7 (a) and (b). A greater PAGS was observed for the sample with higher reheating temperature. High-carbon steels are susceptible to quenching induced micro-cracking, and the susceptibility increases with increasing PAGS [3.4]. These microcracks were observed in the optical micrographs (indicated by red arrows), particularly in the RH1100 microstructure with the largest PAGS (Figure 3.7 (c)).
Figure 3.7 Light optical micrographs of 52100 steel dilatometry samples etched with saturated picric acid to reveal PAGBs. (a) RH900, (b) RH1000, and (c) RH1100 samples.

Figure 3.8 (a)-(d) shows light optical micrographs of RH900 sample, and short time single, double and triple reheating cycle samples (performed at 900 °C for 1 s, RHS – 1, 2 and 3). While the RH900 sample was held isothermally at 900 °C for 100 s, the other RHS samples were held isothermally for only 1 s. The scale bar for RH900 (Figure 3.8 (a)) is different from the RHS samples, suggesting that a longer isothermal hold resulted in an approximately 2 times larger PAGS. Subsequent short time reheating cycles, i.e. double and triple RH cycles, do not alter the grain size significantly. Quantitative
stereological trends in PAGS, plate-martensite microgeometry and size distribution of RA constituents are discussed in the next section.

Figure 3.8 Light optical micrographs of 52100 steel dilatometry samples etched with saturated picric acid to reveal PAGBs. (a) RH900, (b) RHS-1, (c) RHS-2, and (d) RHS-3 samples.

3.3.3 Quantitative Stereology

Stereological analysis was performed on microstructures developed using thermal cycling (described in Section 3.2.4). The results of trends in PAGS, plate-martensite and RA sizes as a function of thermal cycling parameters are presented below.
Prior Austenite Grain Size

Optical micrographs etched with saturated picric acid were used to determine the PAGS for different thermal cycling microstructures according to Abrams three circle method ASTM E112. The thermally cycled samples showed significant microstructural banding of grain sizes possibly due to alloy segregation and compositional banding during prior processing. The narrow bands consisting of significantly smaller PAGS were avoided for stereological measurements.

Figure 3.9 shows the prior austenite grain sizes as a function of reheating temperatures. Error bars represent the standard deviation in PAGS determined from different micrographs. Reheating to higher temperatures leads to coarser PAGS. During reheat treatments, austenite reversion takes place, where austenite grains nucleate and grow on PAGBs and at martensite/austenite interfaces. They subsequently impinge against one another resulting in fine austenitic grains. However, during isothermal holding at elevated temperatures, grain growth subsequently occurs. Larger grains grow at the expense of smaller grains to reduce the grain boundary area of the microstructures.

Figure 3.9 Prior austenite grain size as a function of the reheating temperature during thermal cycling (isothermal holding of 100 s). More than 400 prior austenite grains were sampled to determine the PAGS for each condition.
Stereological analysis was performed to quantify the size distribution of PAGs resulting from multiple short time re-heating treatments. Figure 3.10 shows the cumulative distribution function (CDF) of grain boundary intercept length for microstructures produced by multiple re-heating cycles. For an austenite reversion cycle of 1 s at 900 °C, a significantly smaller final PAGS of 5.89 ± 0.48 μm was obtained, less than half the value for a 100s hold at 900 °C (RH900). It was observed that subsequent thermal cycling at 900 °C did not alter the average grain sizes significantly; however, for multiple re-heating cycles, the grain-size distribution became narrower. The triple re-heated sample has a steeper CDF curve as compared to the single reheated sample, indicating a lower standard deviation in grain size in the triple reheated specimen. Roughly 20 pct of the lineal intercepts in RHS-1 are greater than 10 μm in length, while in RHS-3 less than 10 pct of the intercepts are greater than 10 μm. Lineal intercepts as large as 24.8 μm were observed for the RHS-1 sample. Similar trends were observed for multiple reheating cycles at 1000 °C for 100 s. Picric acid etched optical micrographs and trends in PAGS for multiple reheating cycles (1000 °C for 100 s) are included in Appendix B.

Crack nucleation during fatigue in carburized components can occur via intergranular fracture along PAGBs, and larger grains can serve as larger, more detrimental crack nucleation sites and may act as the weak link controlling fatigue performance. The difference in CDF between re-heat conditions indicates that there are fewer large grains after subsequent thermal cycles. It is hypothesized that improved fatigue lives result from this narrower grain size distribution.
Figure 3.10 Cumulative distribution function plots for grain boundary linear intercepts of microstructures produced by thermal cycling (900 °C and 1 s) subjected to multiple austenite reversion cycles.

**Plate-Martensite Micro-Geometry**

The refinement in average plate martensite size, resulting from the refinement in PAGS was quantitatively characterized. Figure 3.11 shows a representative electron micrograph from the RH1000 microstructure etched with 2 pct nital. The dark regions correspond to martensite, whereas the light regions are RA. The high-carbon martensite has a plate morphology with varying sizes of martensite plates and RA encapsulated in between. Different micro-constituents, namely plate martensite (α’) and RA (γ) microconstituents, are labeled. Midribs of some martensitic plates are indicated using white lines. The stereological features $S_{mid}^V$ and $L_{mid}^V$ (measured in terms of midplane periphery points per unit area, indicated by black circles) are also schematically highlighted.
Figure 3.11 Scanning electron micrograph of RH1000 microstructure etched with 2 pct nital. Constituents and stereological quantities are schematically labeled in the micrograph.

Figure 3.12 (a) and (b) show the trends in average martensite plate thickness and radius as a function of PAGS where the error bars represent the standard deviation in values obtained from different micrographs. Fisher et al. [3.23] formulated an austenite partitioning model for martensite growth kinetics where martensite units grow and divide austenite grains. New martensite units form in finer austenite constituents and as a result, the mean martensite plate volume decreases with increasing transformation. All three conditions contain approximately 70 pct martensite by volume, and thus a similar degree of austenite partitioning can be assumed. Therefore, it is reasonable to quantitatively compare the micro-geometry of plate martensite for different re-heating treatments. The trends in Fig. 3.12 (a) and (b) show that the average plate size decreases with a decrease in PAGS. In a given microstructure, a wide range of sizes of martensite plates can be observed. Martensite transformation in the “fill-in” mode initiates with uniform formation of martensite plates in all austenite grains, while the subsequent smaller plates form in the smaller austenite volumes partitioned by previously formed plates, resulting in relatively large values of the reported standard deviations in plate sizes [3.15]. The white arrow in the Figure 3.11 points towards fine martensite plates that potentially partitioned a large RA constituent during the fill-in stage of the transformation. The stereological results are summarized in Table 3.3.
Figure 3.12 Quantitative trends in morphology of plate martensite as a function of prior austenite grain size of reheated specimen: (a) average plate thickness and (b) average plate radius.

Guimarães et al. [3.16] suggest that martensite formation proceeds in two steps: elastic spread of the martensite midrib followed by thickening of plates. PAGBs and martensite-austenite interfaces are obstacles to the elastic propagation of midribs. The average plate radius is geometrically limited by impingement against physical barriers [3.16]. Furthermore, as the midrib lengthens, the deformation of austenite in the vicinity of the propagating front impedes its growth, limiting the length of plates [3.16].

The average plate thickness is greater at higher values of PAGS. As the martensite plate thickens, it deforms the surrounding austenite plastically. The surrounding austenite imposes a local mechanical constraint over the thickness of plates [3.16]. The complex interplay of mechanical and geometrical constraints during martensite growth via thickening can potentially explain the refinement in plate thickness observed in Figure 3.12 (a). Previous studies on martensitic transformations in high-carbon steels have shown that $t_{avg}$ is composition dependent [3.24]. Further, Chang et al. [3.14] reported that the values of $t_{avg}$ are more sensitive to austenite grain size in O1 tool steel than in a Fe-1.4C-0.02P alloy. Overall, the growth of martensite and its morphology is limited by both characteristics of the austenite and the grain/phase boundary interfaces.
Table 3.3 – Summary of Plate Martensite Stereological Measurements

<table>
<thead>
<tr>
<th></th>
<th>Experimental Quantities</th>
<th>Stereological Quantities</th>
<th>Morphological Quantities</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reheating Temp (℃)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>900</td>
<td>12.4±2.2</td>
<td>0.72±0.01</td>
<td>0.59±0.06</td>
</tr>
<tr>
<td>1000</td>
<td>31.6±3.6</td>
<td>0.68±0.03</td>
<td>0.79±0.07</td>
</tr>
<tr>
<td>1100</td>
<td>69.8±5.8</td>
<td>0.72±0.03</td>
<td>1.01±0.07</td>
</tr>
<tr>
<td>PAGS (µm)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Vv</td>
<td>1.21±0.06</td>
<td>1.1±0.1</td>
<td>2.2±0.24</td>
</tr>
<tr>
<td>Sv\textsuperscript{MID} (µm\textsuperscript{-1})</td>
<td>0.86±0.06</td>
<td>0.59±0.05</td>
<td>2.92±0.32</td>
</tr>
<tr>
<td>Lv\textsuperscript{MID} (µm\textsuperscript{2})</td>
<td>0.71±0.04</td>
<td>0.41±0.02</td>
<td>3.46±0.25</td>
</tr>
<tr>
<td>t\textsubscript{avg} (µm)</td>
<td>0.59±0.06</td>
<td>0.79±0.07</td>
<td>1.01±0.07</td>
</tr>
<tr>
<td>r\textsubscript{avg} (µm)</td>
<td>2.2±0.24</td>
<td>2.92±0.32</td>
<td>3.46±0.25</td>
</tr>
</tbody>
</table>

Retained Austenite Size Distribution

Austenite grains are partitioned as the transformation progresses and a wide range of constituent sizes are obtained. Austenite regions were identified in 2-D micrographs and their circular equivalent diameters were determined. The Schwartz-Saltykov method was employed to calculate the volumetric RA constituent size distribution ($N_{Vv}$) from the 2-D RA area sections [3.19]. Figure 3.13 shows the trend in average RA constituent diameter as a function of PAGS. Figures 3.14 (a), (b) and (c) show the frequency distribution for RA constituent sizes in RH900, RH1000 and RH1100 microstructures, respectively.

The average RA constituent diameter increases with an increase in prior austenite grain size of the microstructure. Since all the conditions have approximately the same volume fraction of RA (determined stereologically), the influence of PAGS can be isolated in these conditions. The linear trend for average RA diameter can thus be primarily attributed to geometrically constrained spread of martensite in different PAGS. RA constituents share interfaces with plate martensite. As discussed in the
previous section, finer PAGS results in finer martensite plates, and thus a greater surface area to volume ratio ($S_v^{\text{MID}}$). Therefore, finer RA constituents are found in fine PAGS microstructures.

![Figure 3.13 Average equivalent diameter of the RA constituents as a function of prior austenite grain size (PAGS) of reheated specimen.](image)

The range of the sizes and the size of largest austenite constituent increases with increase in PAGS. In the existing literature pertaining to RA-containing advanced-high-strength-sheet (AHSS) steels, the stability of RA has been attributed to factors such as composition, size, shape, and strain partitioning in surrounding microstructures [3.26–3.28]. The RA volume contribution for each size class interval was determined by multiplying the number fraction, $(N_V)_i$, with the specific spherical volume (determined using the average diameter for the size class). The size distribution data suggests that constituents with diameter greater than 2.5 $\mu$m contribute to approximately 75 pct RA by volume in RH1100 and approximately 25 pct RA by volume in RH1000, while all the RA constituents in RH900 are finer than 2.5 $\mu$m. In RH900, the sub-micron constituents form the greatest fraction of RA, and they are potentially mechanically stable compared to the larger RA. It is hypothesized that microstructural refinement from thermal cycling resulting in a fine distribution of RA constituents can consequently affect the mechanical behavior, e.g. fatigue.
3.4 Result and Discussion – Step Quenching

Interrupted quenching below the $M_s$ temperature (step-quenching) was implemented on high-carbon 52100 steel using a dilatometer. A comparison between direct quenched RH1000 sample and the step quenched (SQ100 and SQ110) samples is presented in this section. The $rel. \Delta l$ versus time and
temperature data is analyzed in Section 3.4.1. Quantitative trends in the martensite micro-geometry for the two conditions are presented in Section 3.4.2. Finally, results of the neutron diffraction studies on direct and step-quenched samples are presented in Section 3.4.3.

3.4.1 Dilatometry

Due to similar reheating and isothermal holding parameters between the direct-quenched RH1000 sample and the step quenched (SQ100) sample, the trends in rel. Δl versus temperature were the same (discussed in greater detail in Section 3.3.1). The thermal history of the RH1000 and SQ100 samples were significantly different in the quenching step. Consequently, the rel. Δl versus temperature data for the martensite transformation segment were remarkably different. Figure 3.15 (a) and (b) show the rel. Δl versus temperature data for RH1000 and SQ100 samples, respectively. The trend in Figure 3.15 (a) follows the typical athermal martensite transformation kinetics. In Figure 3.15 (b), form 142 °C to 100 °C (primary transformation step), the rel. Δl versus temperature curve followed a similar trajectory to that presented in Figure 3.15 (a). During the isothermal holding at 100 °C for 1000 s, a small change in length of the SQ sample was observed (discussed later in this subsection). Upon further cooling of the SQ100 sample, an initial drop in rel. Δl is observed for the secondary step of quenching up until approximately 85 °C after which, the sample starts expanding again due to the transformation of RA to martensite. An evident deviation from K-M behavior was observed.

Figure 3.15 (c) shows the rel. Δl versus temperature curve for SQ100 sample in the vicinity of the \( T_{SQ} \) (a zoomed-in section of Figure 3.15 (b)). The segments of the curve corresponding to the primary quenching step, isothermal holding, and secondary quenching step are highlighted in the plot. Further, three distinct regions were observed in the rel. Δl versus temperature curve in the secondary quenching step: (i) a reduction in the rel. Δl between 100 °C and 85 °C, (ii) a rapid increase in the rel. Δl between 85 °C and 80 °C, and (iii) a moderate increase in the rel. Δl below 80 °C. An increase in the rel. Δl represents martensite formation while the decrease in the rel. Δl represents thermal contraction. Thus, the dilatometer data during secondary step quench suggest that after the isothermal holding step, martensite
formation was initially suppressed, or in other words, the austenite was stabilized. However, on further cooling, a secondary $M_s$ temperature (85 °C in this case) was observed corresponding to the secondary martensite formation. Entwisle [3.7] has previously reported a cooling rate dependent martensite transformation behavior below the $M_s$ temperature due to a stabilizing phenomenon. He suggests that any interruption or slowing down in cooling retards the transformation. Studies on isothermally held or partially transformed microstructures showed that the stabilizing treatments caused a delay in the recommencement of martensite transformation (similar to the secondary $M_s$ temperature in this study).

Seo et al. [3.30] also reported a shift in $M_s$ temperature in dilatometry data during the formation of secondary martensite in quenching-and-partitioning (Q&P) processed advanced high-strength steels, which contain lath-martensite and RA microstructures. This shift in $M_s$ temperature was attributed to carbon enrichment in austenite during the partitioning step of the Q&P process (chemical stabilization). In another study, Van Bohemen et al. [3.5] studied the influence of partial bainitic transformation on the subsequent martensitic transformation in high-carbon 1080 steel. They observed a drop in $M_s$ temperature due to prior bainite transformation, which was attributed to mechanical stabilization, wherein the shape change associated with the displacive bainite transformation plastically deforms and strengthens the austenite. The increased strength of austenite leads to a need for higher driving force for subsequent martensite formation and thus a lower $M_s$ temperature for secondary martensite. However, the dislocations formed due to the deformation of austenite during primary transformation can assist the subsequent nucleation of secondary martensite, which is referred to as autocatalytic nucleation [3.5].

A systematic step-quenching study was performed to investigate the influence of $T_{SQ}$ and isothermal holding time on the subsequent shift in secondary $M_s$ temperature after isothermal holding (extent of austenite stabilization). The results of the study are included in Appendix C. Figure 3.16 (a) shows the trends in ‘secondary $M_s$ temperature’ as a function of $T_{SQ}$ for two different isothermal holding times, 20 s and 1000 s. The dashed line represents $T_{SQ}$ data or the ‘x = y’ line. In Figure 3.16 (b), the same data are plotted as the shift in secondary $M_s$ ($T_{SQ} –$ secondary $M_s$, in °C)’ as a function of $T_{SQ}$ for the two different isothermal holding times; the relative shift in secondary $M_s$ reflects the degree of austenite
stabilization due to the step quench process. It was observed that the extent of stabilization increased with increasing hold times and decreasing $T_{SQ}$. The observations were consistent with previous studies in literature [3.31]. With an increasing amount of primary martensite (lower $T_{SQ}$), a greater shift in secondary $M_s$ was observed; this trend suggests that the compressive stresses induced by the primary martensite transformation mechanically stabilized the austenite.

In quenching and partitioning steels, silicon (greater than 1 wt pct) is added to suppress precipitation of carbides in martensite and promote partitioning of carbon from martensite to austenite (chemical stabilization of austenite). In lean-silicon steels such as 52100 steel, partitioning of carbon from martensite to austenite is not expected at temperatures as low as 100 °C. However, the time dependent shift in secondary $M_s$ possibly points toward a chemical nature of austenite stabilization.

Kinsman and Schyne [3.31] studied recommencement of martensite transformation in partially transformed and aged Fe-Ni-C steels and proposed a kinetic model for the thermal stabilization phenomenon. The authors assumed that austenite-martensite embryos exist which can grow into martensite below the $M_s$ temperature. During the aging treatment (isothermal holding during step-quenching), they propose that carbon segregates to glissile interfaces of the martensite nucleus and pin it. Thus, the observed shift in secondary $M_s$ represents the undercooling required to provide the chemical free energy driving force sufficient to overcome the pinning effect of interstitial carbon. In agreement with their model, the authors reported that the thermal stabilization increased with increasing aging temperature and time, while it did not depend on prior martensite content.
Figure 3.15 Martensite transformation segment of the rel. $\Delta l$ versus temperature curve for (a) RH1000 sample and (b) SQ100 sample. (c) rel. $\Delta l$ versus temperature curve in the vicinity of isothermal holding step during the step-quenching heat treatment (SQ100 sample).

The thermal stabilization mechanism can be used to explain the time dependent shift in secondary $M_s$ temperature observed during the step-quenching study. However, unlike Fe-Ni-C steels, $T_{SQ}$ (aging temperature) and prior martensite content were interconnected parameters (K-M equation) in the 52100 steel. Hence, it is speculated that a combination of mechanical stabilization and thermal stabilization
mechanisms might be at work in this step-quenching heat-treatment. The influence of step-quenching on microstructure development is discussed in the next sections.

![Graphs showing the relationship between secondary Mₜ temperature and step quench temperature](image)

Figure 3.16 Martensite transformation segment of the rel. Δl versus temperature curve for (a) RH1000 sample and (b) SQ100 sample. (c) rel. Δl versus temperature curve in the vicinity of isothermal holding step during the step-quenching heat treatment (SQ100 sample).

### 3.4.2 Stereological Analysis

Stereological analysis was performed on SEM micrographs of the SQ100 sample to quantify the volume fraction of RA and martensite micro-geometry. Figure 3.17 (a) and (b) show quantitative stereological results. The step-quenched condition SQ100, has 38.1 pct RA while the direct quenched sample RH1000 contained 32 pct RA. Both the RH1000 and SQ conditions had the same austenitizing cycle, and thus the PAGS is similar. It is to be noted that SQ underwent one reheating cycle (PAGS = 32.1±1.1 µm), while RH1000 experienced two reheating cycles (PAGS = 31.1± 3.6 µm). A significant reduction in average plate radius and average plate thickness in the SQ condition (as shown in Figure 3.17 (b)) suggests a refinement in martensite plate sizes. During quenching below the $M_s$ temperature, the formation of martensite deforms and introduces dislocations in the surrounding austenite due to the approximately 2 pct volume expansion and shear strains, which makes the plastic
accommodation of subsequent martensite more difficult. Chatterjee and Bhadeshia [3.29] hypothesized that the formation of dislocations in austenite interferes with glissile movement of martensite/austenite interfaces, and a plate stops growing when a strain is reached where the driving force for martensite formation (undercooling) equals the resistance to interface motion. During the second step, new plates of fresh martensite nucleate and grow in the deformed austenite. The deformed austenite has high density of defects, which serve as nucleation sites for martensite. The presence of defects also increases the resistance to glissile motion of martensite-austenite interface during martensite growth. Hence, the subsequent transformation of the deformed-and-strengthened austenite (mechanically stabilized) results in finer martensite plates during the secondary quenching step. Additionally, the greater amount of RA for the SQ100 condition is in agreement with the stabilization of austenite observed in the dilatometry data and in previous literature [3.7, 3.31].

![Figure 3.17](image)

**Figure 3.17** Comparison of stereological parameters of the direct quenched and step-quenched microstructures: (a) RA volume fraction and (b) plate martensite size.
3.4.3 Neutron Diffraction

Neutron diffraction scans were performed on the DQ sample and the SQ 110 (interrupted quenching at 110 °C for 1000 s) samples using SMARTS in LANL. Due to high-carbon content of the alloy, a body centered tetragonal (BCT) structure was assumed for martensite phase during analysis.

Figure 3.18 (a) shows the neutron diffraction pattern (red) for the DQ sample along the axial direction (bank 2) and the corresponding Rietveld refinement fit (green) that was calculated using GSAS software. The x-axis shows d-spacing in Å, while the y-axis shows normalized peak intensities in counts·µs⁻¹. The red and black markers below the diffraction patterns represent the calculated peak positions for martensite and austenite, respectively. The pink curve below the peak markers shows the difference between the measured pattern and the calculated fit. In order for the least squares calculations to converge, martensite was assumed to be body centered cubic (BCT) and a fiber texture was assumed for both phases (since the two detector panels provide limited coverage of the pole figure). The magenta colored (lower) difference curve suggests that a good fit was obtained using Rietveld refinement. Figure 3.18 (b) shows the high intensity austenite and martensite peaks from the same neutron diffraction pattern. Evident martensite peak splitting was observed due to its high tetragonality. Further, peaks corresponding to transition carbides were not detected in the diffraction patterns.

Average lattice parameters and weight fractions of austenite and martensite were determined from the Rietveld refinement fits. Figure 3.19 (a) shows the average austenite lattice parameters measured along the radial and axial directions of the cylindrical sample. By radial and axial direction, it is meant that lattice parameter is calculated from the grains that diffract the incident neutron beam towards the bank 1 and bank 2 directions respectively. The high-carbon martensite has a BCT structure which is characterized by two lattice parameters ‘a’ and ‘c’. Figure 3.19 (b) and (c) show the average martensite lattice parameters ‘a’ and ‘c’, respectively. Phase weight fractions were determined from Rietveld refinement analysis of neutron diffraction patterns. Using the density values (calculated from lattice parameters), the phase volume fractions were calculated (shown in Figure 3.19 (d)).
Martensite formation is accompanied by volume expansion, which in turn imposes compressive residual strains on the surrounding austenite [3.29]. The heat-treated alloys RH100 and SQ contained approximately 67 pct martensite, and thus, the RA was in a compressive stress state. Since the strain free lattice parameters were unknown, the absolute values of elastic macrostrains could not be determined. However, a comparison of austenite lattice parameters suggested that step-quenching heat treatment resulted in larger compressive elastic strains in the austenite phase. As the martensite plate grows within a RA constituent, it plastically deforms and strengthens the surrounding RA [3.4]. While neutron diffraction measures elastic strains, and not the plastic strains, the ability to withstand greater compressive elastic strains is indicative of a greater strength of RA in the SQ microstructure.

Since there was no external applied load, the presence of compressive elastic strain in austenite would mean that the martensite phase had a tensile strain. The martensite lattice parameter ‘\(a\)’ was indeed larger for the SQ 110 sample than the DQ sample indicating a presence of tensile residual strains. However, an opposite trend was observed for the martensite lattice parameter ‘\(c\)’. The tetragonality of martensite stems from the presence of interstitial carbon atoms within the supersaturated iron lattice. In the literature pertaining to low temperature tempering of martensite, redistribution of carbon atoms to lattice defects and carbon atom clustering are two proposed phenomena [3.32]. For the SQ110 sample, the lowering of tetragonality (\(c/a\) ratio) can thus be attributed to the redistribution of carbon atoms to lattice defects during the isothermal holding step. This observation provides evidence for short-range diffusion of carbon atoms in support of the Kinsman and Schyne’s kinetic model for thermal stabilization of austenite during the SQ process [3.31].

Neutron diffraction data suggested that SQ110 sample had a marginally lower amount of RA than the DQ sample (Figure 3.19 (d)). This observation conflicted with the existing literature and stereological analysis previously performed in this study. Neutron diffraction is a bulk phase fraction measurement technique which likely has a higher accuracy as compared to stereological analysis of SEM images. Additionally, the differences in etching response of primary and secondary martensite in the SQ microstructures may be a source of error in the phase volume fraction measurement. Further
characterization and data analysis such as in-situ heat treatment and neutron diffraction would help better understand the phenomenon of austenite stabilization during interrupted quenching in carbon steels.

Figure 3.18 Measured and calculated neutron diffraction pattern of the DQ sample recorded on bank 2 of SMARTS diffractometer at LANL. (a) full pattern, and (b) high intensity austenite and martensite peaks.
Figure 3.19 Rietveld refinement analysis of direct and step quenched samples. (a) austenite lattice parameters, (b) martensite lattice parameters ‘a’, (c) martensite lattice parameters ‘c’, and (d) retained austenite volume fraction.
3.5 References


CHAPTER 4
FATIGUE BEHAVIOR OF HIGH-CARBON PLATE-MARTENSITE AUSTENITE MICROSTRUCTURES

This work aims to elucidate the role of RA on fatigue behavior of high-carbon plate-martensite austenite microstructures. The following chapter discusses development, characterization and fatigue testing of microstructures with varying RA content.

4.1 Background
Carburized components are used in vehicle powertrains and power generation equipment where high strength is required in addition to good fatigue resistance [4.1]. The high-carbon case region provides strength and wear resistance, whereas the low carbon core provides toughness to the carburized components. The case regions contain plate-martensite, retained austenite (RA) and occasionally undissolved cementite whereas the core regions contain lath martensite, ferrite or bainite [4.2]. Along with the carbon composition gradient, the carburized components also have a gradient in hardness, RA content, and residual stresses along the depth of the component [4.3]. The differences in austenite to martensite transformation kinetics between the case and core during quenching introduces compressive residual stresses at the surface [4.4]. The microstructural complexity further increases owing to the possibility of deformation induced phase transformation of RA to martensite during service [4.2].

During cyclic loading in service, parts such as driveshafts or a root of a gear tooth are susceptible to bending fatigue failure. Fatigue cracks typically initiate intergranularly along prior austenite grain boundaries (PAGBs) and propagate transgranularly in the stable crack growth regime. Upon reaching a critical crack length, the component fails via intergranular (IG) unstable crack growth in the high-carbon region [4.2]. The occurrence of IG fracture in high-carbon microstructures has been attributed to phosphorus segregation and cementite formation at PAGBs [4.5]. The role of RA and microstructural gradients on bending fatigue behavior of carburized components is not well established. Krauss suggested that the presence of high amounts of RA (soft and ductile phase) reduces the yield strength and fatigue
strength. Stress/strain assisted RA to martensite transformation introduces compressive residual stress in the microstructure and improves fatigue strength [4.6]. Alternatively, Hu et al. [4.7] showed that brittle martensite formed during fatigue cycling increased crack propagation rates and reduced crack growth resistance. Often, the influence of RA fraction, carbon content, hardness and residual stress are coupled in the observed results. Further, conducting stable fatigue crack growth (FCG) experiments in carburized components is challenging owing to the strong microstructural gradients in the case region, as the FCG rates and failure mechanisms vary as a function of carbon content and hardness along the depth of the case [4.8].

The goal of this study was to investigate the role of RA on fatigue crack propagation in a microstructure containing plate-martensite and RA (similar to the case region of a carburized steel). Using commercial high-carbon 52100 steel was used to develop high-carbon plate-martensite austenite microstructures with varying amounts and stability of RA using thermal processing strategies. The mode I fatigue crack growth behavior of the different microstructures was evaluated, with an emphasis on understanding the interaction between the fatigue crack and the surrounding high-carbon plate-martensite austenite microstructures. Quantitative stereological analysis was performed on fatigue fractographs to determine the differences in failure mechanism between microstructures with varying RA content.

### 4.2 Experimental Methods

The following section presents experimental methods used to develop, characterize, and conduct fatigue testing on high-carbon plate-martensite austenite microstructures with varying RA content. Sections 4.2.1 and 4.2.2 describe the material and samples used in this study. Heat treatments for the fatigue samples were performed with a salt pot (Section 4.2.3) to develop microstructures with variations in RA (amount and stability). Microstructure characterization methods are discussed in Section 4.2.4. The various stages of fatigue testing are described in Section 4.2.5. Finally, methods used to characterize the interaction between the fatigue crack and the surrounding microstructures are presented in Section 4.2.6.
4.2.1 Materials

In this study, commercial 52100 high-carbon steel was used as to develop microstructures containing plate-martensite and RA. The alloy composition, rationality behind alloy selection, and the details of the as-received material were presented in Section 3.2.1.

4.2.2 Sample Machining

The as-received 52100 steel bars were machined into sub-sized compact tension (CT) fatigue samples for fatigue crack growth studies. Rectangular blanks of length 76.2 mm, height 32 mm, and width 6.35 mm were machined from the radial position of the 52100 steel bar. From each 76.2 mm (3 inch) long section, 8 such rectangular blanks were machined as shown in Figure 4.1. Two sub-sized compact tension (CT) samples were wire-EDM machined from the rectangular blanks (Figure 4.2 (a)) for fatigue crack growth testing. The dimensions of the sub-sized CT samples are shown in Figure 4.2 (b). In addition to the CT samples, three microstructural characterization coupons were also obtained from each rectangular blank (indicated in Figure 4.2 (a)). The crack growth direction was aligned along the transverse direction of rectangular blank and thus along the radial direction of the as-received steel bar.

![Diagram of radial rectangular blanks](image)

Figure 4.1 A schematic diagram of the orientation of rectangular steel blanks that were machined from the as-received 52100 steel bar.
Figure 4.2 Schematic diagram of fatigue samples. (a) Orientation of CT sample and microstructure characterization coupons within the radial blanks, (b) dimensions of the sub-size CT samples used in the study, and (c) notch geometry of the CT sample.

4.2.3 Heat Treatment

Austenitization heat treatments on the relatively larger fatigue samples were carried out on as-received pearlite/pro-eutectoid cementite specimens in salt pots, which contained a molten salt mixture of sodium and potassium nitrate (supplied by – Park Thermal, ISO-THERM 220). The dilatometry samples (Chapter 3) reheated to 1000 °C were prone to micro-cracking during quenching. Thus, a lower austenitization temperature was selected for fatigue samples to provide a fine PAGS and suppress micro-cracking. The sub-size CT sample and a steel coupon were submerged in salt pot below the $A_{cm}$ at 870 ± 5
°C, held for 30 min, and subsequently quenched to room temperature in an agitated oil bath. The quenched sample was then tempered at 175 °C for 6 h in a Carbolite Gero CWF 1300 box furnace prior to fatigue testing. The room temperature oil quenched and low temperature tempered condition will be referred to as the RTDQ (room temperature direct quench) condition throughout this chapter.

Microstructures with a reduced amount of RA were developed using two routes: (i) cryogenic quenching to transform RA to martensite and (ii) tempering to decompose existing RA. Cryo-quenching results in an increase in microhardness since martensite is the hard phase while RA is the soft phase. Alternatively, tempering the microstructure results in a reduction in the bulk microhardness. For the cryo-quenched condition, the room temperature oil quenched sample was quenched in liquid nitrogen at -196 °C and subsequently tempered at 175 °C for 6 h in the box furnace prior to fatigue testing. The cryo-quenched microstructure will be referred to as the LN2 condition throughout this thesis. A reduction in RA content and an increase in microhardness value was anticipated for this condition.

To develop a microstructure with lower RA content but similar microhardness, a higher tempering temperature condition was identified. The room temperature oil quenched sample was first cryo-quenched in liquid nitrogen to transform RA to martensite. Further, the sample was tempered at 240 °C for 1.5 h in box furnace. The higher temperature condition will be referred to as the LN2T condition in this thesis. Notably, the liquid N₂ quenched samples (LN2 and LN2T) were first quenched to room temperature oil bath and then quenched into a liquid N₂ containing dewar prior to tempering treatment. Dilatometry data pertaining to low temperature tempering is included in Appendix D.

Finally, the ‘step quenching’ heat treatment (described in Section 3.2.3) was implemented using salt pot and oil bath. The sub-size CT sample and a steel coupon were introduced in a salt pot at 870 ± 5 °C for 30 min and subsequently quenched to 100 °C in an agitated hot oil bath. The sample and the coupon were held at 100 °C for 1 h in the oil bath and subsequently air-cooled to room temperature. A low temperature tempering treatment was performed at 175 °C for 6 h in the box furnace. The step-quench condition will be referred to as the SQ condition in this thesis. Figure 4.3 schematically shows, and Table 4.2 summarizes the heat treatments used to develop the 4 microstructures studied in this thesis.
Figure 4.3 A schematic diagram showing austenitization, quenching and tempering cycles for the four microstructures developed in this study (a) RTDQ and SQ, and (b) LN2 and LN2T.

Table 4.1 – Summary of Heat Treatments

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Austenitization (Salt Pot)</th>
<th>Quenching Steps</th>
<th>Tempering (Box Furnace)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Temperature</td>
<td>Time</td>
<td>Temperature</td>
</tr>
<tr>
<td>DQ</td>
<td>875 °C</td>
<td>30 min</td>
<td>20 °C</td>
</tr>
<tr>
<td>LN2</td>
<td>875 °C</td>
<td>30 min</td>
<td>20 °C and -196 °C</td>
</tr>
<tr>
<td>LN2T</td>
<td>875 °C</td>
<td>30 min</td>
<td>20 °C and -196 °C</td>
</tr>
<tr>
<td>SQ</td>
<td>875 °C</td>
<td>30 min</td>
<td>100 °C and 20 °C</td>
</tr>
</tbody>
</table>
4.2.4 Microstructure Characterization

The characterization methods used to quantify and compare microstructures are presented in this section. Light optical microscopy (LOM) and scanning electron microscopy (SEM) were used to identify the phases present in the microstructures. X ray diffraction was used to quantify the fraction of RA, and Vickers hardness testing was performed to measure the microhardness values of the different conditions.

Microscopy

The austenitized, quenched, and tempered steel coupons were cross-sectioned perpendicular to their length using an MSX saw, and hot mounted in Bakelite using a Leco® 450X mounting press. Mounted samples were metallographically prepared by mechanical grinding, polishing, and chemical etching. Manual mechanical grinding was performed using a series of silicon carbide grit papers, and polishing steps were performed on Leco® spectrum system 2000 by using diamond suspension solutions. The details of the metallographic procedure have previously been provided in Section 3.2.4.

Metallographically prepared sample surfaces were chemically etched with a 2 pct nital solution (1 mL nitric acid and 49 mL ethanol) for approximately 15 s to reveal the microstructural constituents. Etched samples were observed under a Leco® DSX 500 optical microscope and JEOL 7000 field emission scanning electron microscope (FE-SEM) to identify phases present in the microstructures. The FE-SEM was operated at an accelerating voltage of 20 keV and a working distance of 10 mm.

Polished samples were etched with saturated picric acid (4 g picric acid, 100 mL water, 8 mL Teepol surfactant, and 4 drops HCl) to reveal prior austenite grain boundaries. To etch, the sample surface was heated for approximately 30 s using a heat gun, and then immersed in heated saturated picric acid solution (65 °C) for 60 s. The etched sample was cleaned with a cotton swab and de-ionized water. Optical micrographs were collected on a Leco® DSX 500 microscope, and the Abram 3 circle method (ASTM E112 – 96) was used to determine the prior austenite grain size (PAGS) in ImageJ® software. More than 1000 grains were sampled to determine PAGS.
X-Ray Diffraction

X-ray diffraction (XRD) was used to determine the RA volume fraction for different microstructures developed in this study. To prepare samples for XRD, four approximately 5 mm long segments of steel coupons were cut, stacked together, and mounted in Bakelite. Figure 4.4 schematically shows cuts in the steel coupon and the resulting Bakelite mount. Standard metallographic procedures were used to polish the samples down to 1 µm diamond suspension (described in Section 3.2.4). By stacking four cross-sections together, the sample surface area was maximized for improved intensity counts in the XRD patterns.

Figure 4.4 A schematic diagram showing sample preparation for X-ray diffraction measurement.

XRD patterns were recorded on a Siemens Kristalloflex 810 instrument using a ‘Siemens Type KFL CU 2 K’ copper source and a scintillation detector, with operating voltage and current of 30 kV and 25 mA, respectively. A graphite monochromator was installed on the detector side of the beam to remove the K-beta signals. There were four slits in the system, two on the incident side of the sample, 1° each, and two on the diffracted side, 1° and 0.15°. The samples were scanned over a 2θ range of 40° and 105°, with a step size of 0.02° and a dwell time of 5 s per step.

Peak intensity data was used to determine the RA fraction from XRD patterns. Peak fitting was performed on the PANalytical High Score Plus® software. Using the software, the peak positions and background intensity were first determined. Further, using the ‘Profile Fitting’ tool in the High Score software, a pseudo-Voigt profile (a convolution of Gaussian and Lorentzian functions) was fit to the peak
shape to determine peak heights and full width at half maximum (FWHM). The fraction of RA was calculated using the SAE [4.9] method. The RA fraction is given by

\[
V_\gamma = \frac{\left(\frac{1}{n_\gamma}\right) \left(\sum_0^n \left(\frac{I^{hkl}_\gamma}{R^{hkl}_\gamma}\right)\right)}{\left(\frac{1}{n_\gamma}\right) \left(\sum_0^n \left(\frac{I^{hkl}_\gamma}{R^{hkl}_\gamma}\right)\right) + \left(\frac{1}{n_\alpha}\right) \left(\sum_0^n \left(\frac{I^{hkl}_\alpha}{R^{hkl}_\alpha}\right)\right)}
\]

(4.1)

where \(V_\gamma\) is the volume fraction of the RA, \(n_\gamma\) and \(n_\alpha\) are the number of peaks of austenite and martensite respectively, \(I^{hkl}_\gamma\) and \(I^{hkl}_\alpha\) are the \(hkl\) austenite and martensite peak intensities, and \(R^{hkl}_\gamma\) and \(R^{hkl}_\alpha\) are the theoretical intensities for the \(hkl\) peaks. The \(I^{hkl}\) values were calculated as the product of peak height and peak FWHM values from the peak profile fits. Four austenite peaks (\{111\}, \{200\}, \{220\}, \{311\}) and four martensite peaks (\{110\}, \{200\}, \{211\}, \{220\}) were used for the analysis.

**Vickers Microhardness**

Microhardness measurements were performed on the austenitized, quenched and tempered conditions using an Instron Wilson Tukon® series 200 manual hardness tester. Indentation was performed under 1000 g-f load and a dwell time of 10 s using a pyramidal diamond indenter. Sixteen hardness measurements were taken for each microstructure condition (four indentations from each segment of the Bakelite mounted steel coupon previously used for XRD measurements (Figure 4.3)). Each indentation was at least 1.5 mm away from the edge of the sample and from each other.

**4.2.5 Fatigue Testing**

Fatigue crack growth tests were performed on the heat-treated sub-size CT samples. First the details of the mechanical testing frame and sample preparation are presented. Then, the pre-cracking procedure and fatigue crack growth rate testing procedure are described. Finally, the fatigue data analysis methodology is discussed.
Mechanical Testing Frame and Fixturing

Fatigue experiments were performed on an MTS® Model 204.63 servo-hydraulic load frame. The hydraulic frame was controlled using an MTS® TestStar™ IIs controller and Station Manager version3.5c software. Custom fixturing grips were used for sub-size CT samples. The grips were 12.7 mm (0.5 in) wide to account for samples with varying thicknesses. The samples used in this work were 6.35 mm thick (0.25 in). To ensure good alignment, 1.6 mm (1/16 in) thick ultra-low-friction Rulon®J (polymeric material like Teflon™) spacers (McMaster product – 7488K11) were used between the grips and the sample. High-hardness carbon steel shafts, 6.35 mm (0.25 in) diameter and 76.2 mm (3 in) length (McMaster product – 6061K411) were used as loading pins.

Sample Preparation

The heat-treated samples were metallographically polished prior to fatigue testing. Both the faces of the sub-size CT samples were ground on Leco® PX 300 grinding station using a sequence of 120, 180, 240, 320, 400 and 600 grit silicon carbide (SiC) grinding pads. Using a sequence of diamond polishing suspensions with sizes 9 µm, 6 µm, 3 µm, and 1 µm, the sample surfaces were polished to a mirror finish on a manual Leco® VP-160 polishing wheel. During polishing, Ultra-Silk lapping cloth was used for the 9 µm, 6 µm, and 3 µm step, while the Imperial polishing pad was used for the 1 µm step.

Prior to pre-cracking, a chevron notch was introduced in the polished CT samples using a diamond wafering blade on Allied Tech® TechCut 4 slow speed saw (recommended for brittle and low toughness materials). Symmetry in the notch was ensured by measuring the length of the groves on sample surface using optical imaging on a Leco DSX500 microscope. Approximately 2 mm long grooves were cut on both sides of the sample resulting in a 120 º symmetrical notch at the center.

Fatigue Pre-Cracking

As per the fatigue crack growth testing standard, ASTM E647, a sharp fatigue crack must be induced in the sample before testing in order to avoid the effects of the machined notch. The process of introducing a sharp crack in the sample is referred to as pre-cracking. CT samples have an increasing
K-gradient, which means that for a constant load amplitude, as the crack grows, the stress intensity at the crack tip increases. Pre-cracking is challenging in low toughness materials since brittle materials have a narrow $\Delta K$ range where stable crack growth can occur, and a crack that initiates can rapidly propagate to failure [4.10]. By introducing a chevron notch prior to fatigue testing, a decreasing K-gradient is geometrically generated at the start of pre-cracking. Further, it is recommended that loading be performed in displacement control so that as the crack grows, the applied load and the $\Delta K$ decreases [4.10].

In this study, fatigue pre-cracking was performed on a chevron notched sub-size CT sample in displacement control. Fatigue cycling was performed at a frequency of 5 Hz and a displacement amplitude of 0.14 mm, which nominally corresponded to a peak load value between 1.8 kN and 2.2 kN and an $R$-value of approximately 0.2. The region near the notch was observed in real time using a high magnification USB camera (USB2.0 HD UVC WebCam, Plugable Digital Viewer software) for the onset of pre-crack initiation at the notch. If no pre-crack was observed for 40,000 cycles (approximately 2 h), the displacement amplitude of fatigue cycling was increased by 0.01 mm for another set of 40,000 cycles. Once a crack of appreciable length (approximately 500 µm) was observed at the notch edge, the fatigue pre-cracking procedure was stopped. The sample was removed from the load frame, both surfaces were imaged using a Leco DSX 500 optical microscope, and the lengths of the pre-crack were recorded.

**Fatigue Crack Growth Testing**

Once the subsize CT sample had a sharp pre-crack, fatigue crack growth experiments were performed under constant load amplitude (increasing $\Delta K$) mode as per the ASTM E647 standard. Interrupted fatigue cycling was performed at a frequency of 5 Hz. A sinusoidal load function with a peak load ($P_{\text{max}}$) of 1.5 kN and a minimum load ($P_{\text{min}}$) of 0.15 kN ($R$- ratio = $P_{\text{min}}/P_{\text{max}}$ = 0.1) was used for all the tests. High magnification stitched images were recorded using a Leco® DSX 500 optical microscope on both the surfaces of the sample between each set of interrupted fatigue cycles. The peak load during pre-cracking was greater than the peak load during FCG testing. To eliminate the influence of load history and plastic zone associated with higher pre-cracking loads on the FCG testing results, the crack length
data between initial pre-crack length (approximately 500 µm) and 1 mm was disregarded during the
calculation of FCG rates. An approximately 0.25 mm crack extension was ensured between each crack
length measurement. As a result, variable fatigue cycling intervals were used. For crack lengths ranging
from 1 mm to 3 mm, the crack length was measured every 10,000 cycles. Similarly, for the crack lengths
ranging from 4 mm to 6 mm, the crack length was measured every 2,000 cycles. If the crack extension on
one side of the sample was significantly larger than the other side (due to misalignment of the load
frame), the sample was rotated for the next set of fatigue cycling to ensure a planar crack front and a valid
test.

For each microstructure condition, two samples were tested. For the first sample, the FCG
experiment was stopped at a crack length of approximately 6 mm to study the interaction of the arrested
crack with the surrounding microstructure. The second sample was tested to failure and fractography was
performed on the fracture surfaces. The trends in crack length as a function of number of cycles were
recorded for the samples. The next section presents data processing methods used to analyze FCG rates.

Data Analysis

Crack length versus number of cycles data were collected for the four different microstructure
conditions DQ, LN2, LN2T, and SQ. Conventionally, the results of the FCG tests are presented as fatigue
Crack growth rate ($\frac{da}{dN}$) versus the stress intensity range ($\Delta K$) plots on a log scale. Both $\frac{da}{dN}$ and $\Delta K$
values can be determined from the crack length versus number of cycles data.

For CT samples, the stress intensity range at the crack tip, a function of applied load and
gy, is given by the expression

$$\Delta K = \frac{(P_{\text{Max}} - P_{\text{Min}})}{B\sqrt{W}} \left( \frac{(2 + \alpha)}{(1 - \alpha)^2} \right) (0.886 + 4.46\alpha - 13.32\alpha^2 + 14.72\alpha^3 - 5.6\alpha^4) \quad (4.2)$$

where $\alpha$ is $a/W$, $a$ is the crack length measured from the line connecting the bearing points of load
application, $W$ is the width of the sample (25.4 mm), and $B$ is the thickness (6.35 mm).
The FCG rates \((da/dN)\) were calculated from the average crack length versus the number of cycles data using the incremental polynomial methods as described in Appendix X1 of ASTM E647. A parabola (second degree polynomial) was fit to a set of five data points on the crack length versus number of cycles data using the least square method in MATLAB 2021a. The rate of crack growth \((da/dN)\) was then determined by taking the derivative of the fitted parabola at the central data point.

4.2.6 Fatigue Crack Characterization

For the sample with the arrested crack, the interaction between the fatigue crack and the surrounding microstructure was characterized using LOM and SEM. The distribution of phases in the vicinity of the fatigue crack was determined using electron backscatter diffraction (EBSD). For the sample that was tested to failure, fractography was performed on the fracture surface to identify and quantify the fatigue failure mechanisms.

Microscopy

The CT samples with arrested fatigue cracks were metallographically ground and polished as described in Section 3.2.4. Sample surfaces were etched with saturated picric acid solution at 65 °C for 60 s to reveal PAGBs. A Leco® DSX500 optical microscope was used to acquire high resolution stitched images of the region in the vicinity of the fatigue crack near the crack tip. The interaction between the fatigue crack and surrounding PAG microstructure and the martensitic sub-structure was also observed using FE-SEM, and high magnification micrographs were recorded. The FE-SEM was operated at an accelerating voltage of 20 keV and a working distance of 10 mm during imaging.

Electron Backscatter Diffraction

To determine the distribution of phases near the fatigue crack, particularly the distribution of RA, EBSD mapping was performed near the fatigue crack for the RTDQ and SQ samples with the arrested crack. The edges of the CT sample were sectioned and removed using an MSX saw to isolate the region of interest containing the notch, the fatigue crack, and the material around it. In Figure 4.6(a), the dashed lines schematically represent the cuts made, gray regions represent the removed edges, and the white
region represents the samples used for EBSD characterization. The sectioned samples were metallographically polished as described in Section 3.5.1. An additional polishing step was performed using a 0.03 µm colloidal silica solution on the Beuhler VibroMet™ (vibratory polisher) for 72 h to remove the grinding and polishing induced deformation layer prior to EBSD mapping. With a step-size of 100 nm, 60 µm by 30 µm sized, EBSD scans were performed at approximately 4 mm (corresponding to a $\Delta K$ value of 5.2 MPa·m$^{0.5}$ at the crack tip) and 6 mm ($\Delta K = 5.7$ MPa·m$^{0.5}$) form the notch edge on a FEI Helios SEM. Figure 3.6(b) schematically shows scanned areas. An operating voltage of 25 keV and a beam current of 11 nA were used. TEAMSTM EBSD analysis software was used for data collection. Ferrite (BCC) and Austenite (FCC) phases were selected while Kikuchi pattern indexing. Once the maps were collected using TEAMSTM software, the EBSD data were analyzed using TSL OIM software. Data points with a confidence index lower than 0.1 were removed from the data set. Phase distribution and inverse pole figure (IPF) maps were generated for the EBSD scans to study the microstructure in the vicinity of a fatigue crack.

![EBSD Sample](image)

(a)

![EBSD Scans](image)

(b)

Figure 4.5 A schematic diagram of the (a) EBSD sample and (b) EBSD scan methodology.
Fractography

To identify the failure mechanisms present in different microstructure conditions, fractography was performed on the fracture surfaces of the CT samples that were fatigue tested to failure. SEM micrographs of the fracture surfaces were recorded using an FE-SEM and FEI Helios SEM. The operating voltage was 20 keV and the working distance was approximately 15 mm. All the fatigue fracture surfaces exhibited a mixed transgranular (TG) and intergranular (IG) fracture morphology. The overload region of the fracture surface also exhibited mixed TG and IG fracture. To quantify the trends in percentage IG character of the fracture surfaces, stereological analysis was performed on fractographs. For all the sample conditions, a set of images was recorded from different regions along the center (width direction) of the sample as a function of distance from the notch edge (2 mm, 4 mm, and 6 mm). Figure 4.5(a) schematically shows the imaged regions as a function of distance from the notch edge. Additionally, fractographs were also recorded from the overload region of the CT sample, at a distance of 3 mm from the edge of the sample at the center of the specimen.

Figure 4.6 (a) An overview of the fractograph acquisition methodology, and (b) a representative fractograph of RTDQ condition taken at 2 mm crack length, showing stereological measurement of percentage IG character.
Using ImageJ, an 8 x 10 grid of points was overlaid on the fractographs, and grid points lying on
the faceted intergranular regions were indexed for each fractograph. The percentage IG character was
calculated as the ratio of indexed points and the total number of points (80 in this case). For each region,
the percentage IG character value was averaged over 6-8 such fractographs. More than 2000 prior
austenite grains within a region were sampled for each data point. Figure 4.5 (b) schematically shows the
indexing methodology used for stereological analysis where the magenta grid points highlighted in yellow
correspond to IG fracture. The fatigue fracture surface shown in Figure 4.5 was collected from the RTDQ
sample at a distance of 2 mm from the notch edge. 7 out of 20 grid points lie on IG fracture features, thus
the percentage IG character was calculated to be 35 pct for this image.

4.3 Results

Sub-size CT samples were machined from 52100 steel bars, heat treated to develop varying high-
carbon plate-martensite austenite microstructures (RTDQ, LN2, LN2T, and SQ), and their fatigue
behavior was investigated. Section 4.3.1 presents a comparison of the various microstructures developed
in this study. The trends in fatigue crack growth behavior of the microstructures are presented in Section
4.3.2. Finally, a quantitative and qualitative analyses of the interaction between the fatigue crack and
high-carbon plate-martensite austenite microstructures are discussed in Section 4.3.3.

4.3.1 Microstructure Characterization

The heat-treated steel coupons were metallographically polished and etched for LOM and SEM
characterization. Further, XRD patterns were collected from the polished cross-sections to determine the
RA fraction for all four microstructures. Finally, microhardness measurements were performed on the
heat-treated coupons.

Microscopy

Figure 4.7 (a) shows a low magnification optical micrograph of the RTDQ steel coupon, etched
with saturated picric acid solution. The vertical white arrow on the micrograph shows the rolling direction
(RD) and the axis of the as-received cylindrical bar. Alternate dark and light microstructural bands were
observed along with inclusions within the dark bands. Microstructural banding in steels originates from the dendritic nature of solidification, where impurities and microalloying elements segregate to interdendritic regions. In commercial hypereutectoid steels such as 52100 steel, the microstructure banding results from variation in carbides size and density [4.11]. Figure 4.7 (b) shows a high magnification optical micrograph with a light microstructural band between two dark bands. Etching with saturated picric acid revealed PAGBs. Additionally, a fine dispersion of carbides and the plate-martensite and RA substructure were also visible within the PAGs.

Figure 4.7 (c) and (d) show SEM micrographs from the light and dark etching regions of the microstructure. The dark regions showed significantly greater density of carbides. The dark regions had a greater number of inclusions, suggesting that the dark bands had greater microsegregation of alloying elements such as Cr, C, S and others. Upon heating a pearlitic microstructure above $A_{cm}$ temperature, the carbides spherodize and dissolve. The presence of higher levels of Cr suppressed the carbide dissolution locally, thus resulting in a higher density of carbides in the Cr rich band [4.11].

Figure 4.8 (a) shows a SEM micrograph of the nital-etched RTDQ microstructure revealing the sub-structure within the PAGs. Tempered plate-martensite, RA and cementite were identified within the micrograph as labeled. At an austenitizing temperature of 875 °C within the austenite and cementite phase field, cementite dissolution and coarsening occur simultaneously. Two distinct carbide morphologies are observed: large, approximately 1 µm size carbides along the PAGBs and fine sub-micron carbides within the PAGs. A bimodal distribution of carbides in RTDQ 52100 steel has previously been reported in literature [4.12]. Larger cementite potentially forms during the coarsening of hyper-eutectoid grain boundary cementite, while the finer carbides result from dissolution and coarsening of fine cementite lamella present in the pearlitic colonies of the prior microstructure.

Figure 4.8 (b) shows a nital etched SEM micrograph of the LN2 condition. Cementite particles and tempered martensite are evident, similar to the RTDQ condition. As compared to the RTDQ condition, fewer and smaller RA constituents (indicated by white arrows) are present in the LN2 condition. Cryo-quenching resulted in considerable RA to martensite transformation. Figure 4.8 (c) shows
a nital etched micrograph of the LN2T condition. Similar to the RTDQ and LN2 conditions, undissolved cementite was observed in the micrograph. RA constituents were not evident since tempering at 240 °C would have resulted in at least partial decomposition of RA into ferrite and cementite. Figure 4.8 (d) shows a nital etched SEM micrograph of the SQ sample. Similar to the RTDQ microstructure, tempered plate martensite, RA constituents, and cementite particles were observed.

![Micrographs of the RTDQ condition etched with saturated picric acid to reveal PAGBs.](image)

Figure 4.7 Micrographs of the RTDQ condition etched with saturated picric acid to reveal PAGBs. (a) Low magnification optical micrograph showing microstructural banding, (b) high magnification optical micrograph showing the microstructure in the light and dark bands. SEM micrographs from (c) the light region and (d) the dark region.
Figure 4.8 SEM micrographs of heat treated 52100 steel etched using nital. (a) RTDQ microstructure showing plate-martensite, RA and cementite, (b) LN2 micrograph, (c) LN2T SEM micrograph, and (d) SQ micrograph.

X-Ray Diffraction

XRD patterns were recorded for each of the four microstructure conditions RTDQ, LN2, LN2T and SQ. Using the integrated intensities of austenite and martensite peaks, RA volume fractions were calculated as described in Section 4.2.4. Figure 4.9 (a) shows XRD patterns for the four different
microstructures, where the x-axis represents the diffraction angle two theta in degrees and the y axis represents the normalized intensity (counts/counts). Both austenite and martensite peaks were observed for all four conditions; however, variations in intensities of austenite peaks were evident. The RTDQ and SQ conditions had significantly higher austenite peak intensities while the LN2 and LN2T had much smaller austenite peaks.

Using the SAE method [4.9], the RA fraction was determined to be 17.6 vol pct for RTDQ, 19.3 pct for SQ, 9.3 vol pct for LN2, and 7.1 vol pct for the LN2T conditions. Figure 4.9 (b) shows the quantitative results for the RA fraction values. For the LN2 condition, quenching to -196 °C provided the thermodynamic driving force for further transformation of RA. Upon tempering at 240 °C, some of the remaining RA further decomposed to ferrite and cementite. The decomposition of austenite was incomplete over 1.5 h of tempering since approximately 7.1 vol pct RA remained in the LN2T condition. For the SQ condition, interrupted quenching below the Ms temperature potentially stabilized the austenite, as discussed in detail in Section 3.4.1.

Microhardness

Vickers microhardness measurements were performed on the metallographically polished steel coupons for the four conditions. The volume fraction of RA and degree of martensite tempering played a critical role in the observed Vickers microhardness values. For the RTDQ, SQ, and LN2 conditions, which underwent the same tempering treatment (175 °C for 6 h), microhardness values decreased with increasing RA volume fraction, since RA is the softer phase. Despite a small amount of RA in the LN2T condition, tempering the martensite at a higher temperature led to a relatively softer martensite phase. The RA content and microhardness values are summarized in Table 4.2.
Figure 4.9 X-ray diffraction patterns of the heat-treated 52100 steel. (a) Full scan with a 2θ range of 40° to 105°, and (b) quantitative results for RA volume fraction for each microstructure condition, calculated using the SAE method. (see electronic copy for color image)

Table 4.2 – Summary of Microstructural Parameters

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>RA Content (vol pct)</th>
<th>Microhardness (HV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RTDQ</td>
<td>17.6</td>
<td>820</td>
</tr>
<tr>
<td>LN2</td>
<td>9.4</td>
<td>905</td>
</tr>
<tr>
<td>LN2T</td>
<td>7.1</td>
<td>831</td>
</tr>
<tr>
<td>SQ</td>
<td>19.3</td>
<td>812</td>
</tr>
</tbody>
</table>

4.3.2 Fatigue Crack Growth Behavior

The fatigue behavior of the four different microstructure conditions was investigated to understand and deconvolute the influence of microstructural parameters such as microhardness, RA volume fraction and RA stability. Table 4.3 summarizes similarities and differences between the different combinations of microstructure conditions.
Table 4.3 – A Summary of Similarities and Differences between Different Microstructures

<table>
<thead>
<tr>
<th>Sample IDs</th>
<th>Similarities</th>
<th>Differences</th>
</tr>
</thead>
<tbody>
<tr>
<td>RTDQ and LN2</td>
<td>Tempering Treatment</td>
<td>RA content and Micro-Hardness</td>
</tr>
<tr>
<td>RTDQ and LN2T</td>
<td>Micro-Hardness</td>
<td>RA content and Tempering Treatment</td>
</tr>
<tr>
<td>RTDQ and SQ</td>
<td>RA content, Microhardness and</td>
<td>Stability of RA</td>
</tr>
<tr>
<td></td>
<td>Tempering Treatment</td>
<td></td>
</tr>
<tr>
<td>RTDQ, SQ and LN2T</td>
<td>Micro-Hardness</td>
<td>Amount and Stability of RA</td>
</tr>
<tr>
<td></td>
<td>Tempering Treatment</td>
<td></td>
</tr>
</tbody>
</table>

Fatigue crack growth experiments were performed on the CT samples (details are presented in Section 4.2.5) and the trends in crack length versus the number of fatigue cycles were recorded. Figure 4.10 (a) shows a representative crack length versus number of cycles data for the RTDQ microstructure. The average crack length presented is the average of the crack lengths on both surfaces of the CT sample. As the crack length increases, the rate of crack growth increases. This behavior is characteristic of constant load amplitude fatigue cycling, i.e., increasing $\Delta K$ testing conditions.

The crack length versus number of cycles data were converted to FCG rate versus $\Delta K$ using the incremental polynomial method as described in Section 4.2.5. Figure 4.10 (b) shows the results for FCG rate, $da/dN$, as a function of the stress intensity range at the crack tip, $\Delta K$, for the RTDQ condition. The data are plotted on a log-log scale. The error bars represent the standard deviation in the FCG rates between three different RTDQ samples. Since the test was stopped at a crack length of 6 mm for two of the samples, the FCG rates at $\Delta K$ greater than 5.5 MPa-m$^{0.5}$ represent data from only one sample that was tested to failure.
Figure 4.10 Fatigue crack growth behavior of the RTDQ microstructure. (a) A representative average crack length versus number of fatigue cycles plot, and (b) FCG rate versus ΔK plot on a log-log scale.

For the ΔK range from 4.8 MPa·m$^{0.5}$ to 6 MPa·m$^{0.5}$, the FCG rates ranged from approximately $10^{-8}$ m/cycle to $10^{-6}$ m/cycle (two orders of magnitude). Bilinear behavior was observed in the FCG data for the RTDQ. A steeper slope of FCG data was observed for values of ΔK greater than approximately 5.5 MPa·m$^{0.5}$, as compared to the FCG data for ΔK values less than 5.5 MPa·m$^{0.5}$. Similar FCG behavior was observed by Rescalvo and Averbach [4.13], which they ascribed to the transition from ΔK controlled FCG to $K_{\text{max}}$ controlled FCG behavior. At values of ΔK greater than 6 MPa·m$^{0.5}$, a deviation from the linear trend was observed. At this point, the CT sample failed by overload failure in less than 200 cycles, suggesting the onset of unstable fatigue crack propagation at ΔK greater than 6 MPa·m$^{0.5}$.

Figure 4.11 (a) shows representative crack length versus number of fatigue cycles data for the RTDQ, LN2 and LN2T microstructures. For all three conditions, as the crack length increased, the rate of crack growth also increased due to the increase in applied stress intensity for a constant load amplitude test. To quantify the differences between crack growth behavior of different microstructures, the number of cycles for similar crack extension were compared.
Figure 4.11 (b) shows the FCG rates (\(\frac{da}{dN}\) data) as a function of \(\Delta K\) at the crack tip for the three conditions, where the x and y-axis are on a log scale. At \(\Delta K\) values below 4.8 MPa·m\(^{0.5}\), a plateau in the FCG rates was observed for the LN2 and RTDQ condition. Low \(\Delta K\) values might represent microstructure sensitive near-threshold short crack propagation behavior [4.14]. Within the \(\Delta K\) range from 4.8 MPa·m\(^{0.5}\) to 5.6 MPa·m\(^{0.5}\), the FCG rate increased almost linearly, suggesting that the crack growth followed the stable stage II fatigue crack growth behavior. The FCG rates are highest for the LN2 condition, lowest for the LN2T condition, and intermediate for the RTDQ condition.

The RTDQ microstructure has greater RA fraction than the LN2 condition and the same degree of martensite tempering. These results suggest that higher fraction of RA improved the fatigue crack growth resistance by 1.4 times (calculated as the ratio of number of cycles for the same crack extension, 2 mm to 6 mm). Between the LN2 and LN2T conditions, the RA fraction was slightly higher in the LN2 condition, but the LN2T condition had a greater degree of martensite tempering. The FCG resistance improved by 1.6 times for LN2T as compared to the LN2 condition due to the greater degree of martensite tempering. Between the LN2T and RTDQ condition, the RTDQ condition had greater fraction RA but the LN2T
condition had a greater degree of martensite tempering. The FCG resistance of the LN2T microstructure is marginally better (approximately 1.1 times) than the RTDQ microstructure. The results suggest that martensite tempering may have a more prominent effect on the FCG rates.

Figure 4.12 (a) shows representative average crack length versus number of fatigue cycles data for the RTDQ and SQ microstructures. For a 4 mm crack extension from a crack length of 2 mm to 6 mm, the RTDQ and SQ samples underwent 78k and 86k cycles, respectively. Interestingly, at smaller crack length values (2 mm to 4 mm), the \( a \) versus \( N \) curves were similar for the RTDQ and SQ microstructures. However, at greater crack lengths (4 mm to 6 mm), the crack grew slower in the SQ microstructures.

Figure 4.12 (b) shows the FCG rates versus \( \Delta K \) plot for the RTDQ and SQ conditions on a log-log plot. Two different regimes of FCG behavior were observed, characterized by the different slopes of the \( da/dN \) versus \( \Delta K \) data for low and high \( \Delta K \) regions. For values of \( \Delta K \) greater than 5.6 MPa·m\(^{0.5}\), the FCG rates increased more rapidly. At low \( \Delta K \) range of 4.6 MPa·m\(^{0.5}\) to 5 MPa·m\(^{0.5}\), both the RTDQ and SQ microstructures had similar FCG rates. However, for values of \( \Delta K \) greater than 5 MPa·m\(^{0.5}\), the FCG rates for the SQ microstructures were consistently lower than those in the RTDQ microstructure. Further, for the SQ microstructure, the onset of stage III unstable crack growth at a larger \( \Delta K \) (6.4 MPa·m\(^{0.5}\)), as compared to the RTDQ microstructure (6.0 MPa·m\(^{0.5}\)).

The comparative results suggest that greater RA volume fraction and potentially greater stability of RA for the SQ condition resulted in improved fatigue crack growth resistance at high \( \Delta K \) values. Further, the results also indicated that the onset of unstable fatigue crack growth occurred at a greater \( \Delta K \) value for the SQ condition, as compared to the RTDQ condition. However, more tests are needed to verify the observation.

Conventionally, in metallic materials, the FCG rates \((da/dN)\) depend on the alternating stress intensity at the crack tip \((\Delta K)\) and are typically expressed by the Paris-Erdogan law:

\[
\frac{da}{dN} = C \Delta K^m
\]  

(4.3)
where \( \frac{da}{dN} \) is the FCG rate, \( \Delta K \) is the stress intensity range at the crack tip, and \( C \) and \( m \) are experimental parameters that depend on factors such as material and test conditions. The parameter \( m \) is the slope of the FCG rate versus \( \Delta K \) plot (log scale). The value of exponent \( m \) was calculated for the four conditions in this study by performing linear regression on the FCG rates versus \( \Delta K \) data for the \( \Delta K \) range of 4.6 MPa·m\(^{0.5}\) to 5.6 MPa·m\(^{0.5}\). The values of \( m \) were found to be 11.9 for the RTDQ condition, 14.7 for the LN2 condition, 14.5 for the LN2T condition, and 9.6 for the SQ condition. For the RTDQ and SQ microstructures, at \( \Delta K \) greater than 5.6 MPa·m\(^{0.5}\), the values of \( m \) were 24.7 and 23.2 respectively. Such high values of \( m \) exponent suggest that static modes of failure such as IG fracture or cleavage might be present in the microstructures [4.15]. The relevance of high values of \( m \) exponent is discussed in Section 4.4.1. The fatigue cracks were characterized further to better understand the interaction between the fatigue crack and high-carbon plate-martensite austenite microstructures.

![Fatigue crack growth behavior](image)

Figure 4.12 Fatigue crack growth behavior of RTDQ and SQ microstructures. (a) Average crack length versus number of fatigue cycles plot, and (b) an FCG rate versus \( \Delta K \) plot on a log-log scale.

4.3.3 Fatigue Crack Characterization

Sub-size CT samples with RTDQ, LN2, LN2T, and SQ microstructures were fatigue tested. For the samples where the fatigue crack was arrested, polished and etched surfaces were observed using
optical and electron microscope to study the interaction between the fatigue crack and the microstructure. Further, for the RTDQ and SQ samples with substantial amount of RA volume fraction (approximately 18 pct), EBSD mapping was performed in the vicinity of the fatigue crack to determine the distribution of RA constituents. Finally, for the samples that were tested to failure, fracture surfaces were studied using FE-SEM and FEI Helios to identify and stereologically quantify failure mechanisms within different samples.

Microscopy – Crack Path

CT samples with arrested cracks were etched with saturated picric acid to reveal PAGBs in the vicinity of the fatigue cracks. Figure 4.13 (a) shows the RTDQ microstructure in the vicinity of the crack tip showing the crack path with respect to the PAGBs. The direction of crack growth is from left to right in the micrograph. The crack path exhibited both intergranular (IG) and transgranular (TG) sections (as indicated by white arrows) along the crack. The microstructure consists of various features such as PAGBs, cementite, (plate) martensite – martensite interfaces, and plate-martensite RA interfaces which may all interact with the fatigue crack to result in a jagged appearance. Figure 4.13 (b) shows a higher magnification optical micrograph of the arrested fatigue crack in an SQ sample. The crack followed a mixed IG and TG path as labelled on the figure. Two regions of interest along the crack path (Region I and Region II) were observed using FE-SEM.

Figure 4.13 (c) shows a high magnification SEM micrograph of Region I as seen on Figure 4.13 (b). The direction of crack growth is from left to right. In Figure 4.13 (c), the crack that was travelling along the PAGB of ‘Grain A’, entered the ‘Grain A’, changed direction, and then continued along PAGB of a ‘Grain B’. Figure 4.13 (d) shows a high-magnification SEM micrograph of Region II as seen on Figure 4.13 (b). A dense dispersion of carbides was observed in the PAG, and the crack travelled along carbide particles. The region potentially belonged to the dark etching band with high carbide density. Two different transgranular crack paths, Figure 4.13 (c) and (d) had a jagged appearance. A similarly mixed TG and IG crack path was also observed for the LN2 and LN2T conditions.
Figure 4.13 LOM micrograph of saturated picric acid etched (a) RTDQ microstructure and (b) SQ microstructure, (c) and (d) High magnification FE-SEM micrographs showing mixed IG and TG crack paths in the SQ microstructure.
EBSD – Distribution of RA

The RTDQ and SQ microstructures have significantly higher RA content when compared to the LN2 and LN2T conditions. The RA constituents can undergo mechanically induced austenite to martensite transformation [4.1]. An EBSD scan was performed on the metallographically and VibroMet™ polished RTDQ sample and SQ sample with the arrested crack to study the distribution of phases in the vicinity of the fatigue crack as described in Section 4.2.6. The scan area was located at a distance of 4 mm from the notch tip. Figure 4.14 (a), (b), and (c) show the SEM detector (SEI) image, phase map (raw data) and cleaned phase map (confidence index >= 0.1) of the RTDQ sample. The red regions in Figure 4.14 (b) and (c) show the regions indexed as body centered cubic (BCC) phase, which are indicative of plate-martensite, while the green regions show the face centered cubic phase, which are RA constituents. Owing to a fine microstructure with high dislocation density (plate martensite), a low confidence index was observed for the scan. Approximately 40 pct of the pixels were eliminated from the map upon data clean up (Figure 4.14 (c)). A gradient in RA content was observed as a function of distance from the crack. At approximately 20 µm away from the crack, many RA constituents were observed, whereas in the narrow 10 µm region near the fatigue crack almost no RA constituents were observed, indicating RA to martensite phase transformation in the vicinity of the crack tip. The stress/strain fields ahead of the crack tip can result in deformation induced phase transformation of RA to martensite. The size of the plastic zone, \( r_p \), can be estimated by the expression

\[
 r_p = \frac{1}{2\pi} \left( \frac{K_I}{\sigma_y} \right)^2
\]

(4.4)

where \( K_I \) is the applied stress intensity at the crack tip and \( \sigma_y \) is the yield strength [4.16]. Using the value of \( K_I = 6.7 \text{ MPa} \cdot \text{m}^{0.5} \) (experimentally determined \( K_{max} \) from Figure 4.12 (b)) and \( \sigma_y = 1697 \text{ MPa} \) [4.17], the value of plastic zone size was calculated to be approximately 2.5 µm, which is smaller than the transformation zone (approximately 9 µm wide). The transformation of RA to martensite outside the
plastic zone indicated that the stress-assisted austenite to martensite transformation mechanism was responsible for the phase transformation in the vicinity of the fatigue crack [4.18].

Figure 4.14 EBSD maps of a region near the fatigue crack in the RTDQ sample at a distance of 4 mm from the notch tip. (a) SEM detector image, (b) raw phase map and (c) phase map showing data with confidence index greater than 0.1. The red regions correspond to plate- martensite, while green regions represent RA constituents. (See electronic copy for color image)

Figure 4.15 (a), (b), and (c) show an SEM detector image, phase map (raw data), and cleaned phase map (confidence index > 0.1) for the SQ sample. The scan area was located at a distance of 4 mm from the notch tip. The red regions in Figure 4.15 (c) are indicative of plate martensite and the green regions show RA constituents. Similar to the RTDQ sample, a gradient of RA content as a function of distance from the crack tip was observed for the SQ sample. At approximately 20 µm away from the crack, many RA constituents were observed, while in the narrow 10 µm wide zone near the crack, fewer and finer RA constituents were observed, indicating that stress-assisted RA to martensite transformation occurred near the crack tip. However, as compared to the RTDQ sample where a negligible amount of RA
was found in the transformation zone, few fine RA constituents were observed in the SQ sample (indicated by the white rectangle in Figure 4.15 (c)). These results indicate that the extent of RA to martensite transformation was less in the SQ microstructure, suggesting that the RA constituents in the SQ microstructure are mechanically more stable. Notably, only a small 30 µm wide region (5 PAG diameters) along the crack was scanned for this analysis, and the results may not be statistically significant. However, fine RA constituents were consistently observed near the fatigue crack in SQ sample but not in the RTDQ samples, for all the EBSD scans (multiple scans at crack lengths 4 mm and 6 mm) performed along the fatigue crack in the two microstructures.

![EBSD maps](image)

Figure 4.15 EBSD maps of a region near the fatigue crack in the SQ sample at a distance of 4 mm from the notch tip. (a) SEM detector image, (b) raw phase map and (c) phase map showing data with confidence index greater than 0.1. The red regions correspond to plate- martensite, while green regions represent RA constituents. *(See electronic copy for color image)*

Fractographic Analysis

Fracture surfaces of the CT samples for all four microstructure conditions were imaged using the FE-SEM. Stereological analysis was performed to quantify the variations in the failure modes in fatigue
and overload regions of the fracture surfaces as described in Section 4.2.6. Figure 4.16 shows high-magnification SEM fractographs of the overload regions of the RTDQ, LN2, LN2T, and SQ samples. The fractographs were taken from the center of the sample along the width, at a distance of 3 mm from the back edge of the sample.

Figure 4.16 SEM fractographs from the overload regions of the (a) RTDQ, (b) LN2, (c) LN2T, and (d) SQ samples. The fracture surfaces show mixed IG and MVC fracture modes.

In each of the microstructure conditions in the overload region, two distinct fracture morphologies were observed: intergranular fracture along PAGBs (IG) and ductile micro-void coalescence (MVC) fracture. PAGBs in the high-carbon 52100 steel are susceptible to quench
embrittlement [4.5]. In the existing literature, the observed IG fracture has been attributed to the presence of undissolved cementite along the PAGBs (austenitization below the $A_{cm}$ temperature) and phosphorus segregation during the austenitizing step, which in turn promotes cementite formation at PAGBs during quenching. The MVC fracture behavior was attributed to regions containing fine undissolved cementite particles (less than 200 nm in diameter) within the tempered martensite matrix. The cementite particles acted as void nucleation sites and the material failed via fine micro-void coalescence. Figure 4.17 (a) shows a fracture surface from the overload region of the RTDQ sample. The center of the fractograph shows an MVC region and the surrounding regions failed via IG failure. In the surrounding IG failure region, the dashed circles on the PAG facets showed the presence of carbide particles and pits (presumably the sites where the carbide particles de-bonded). The size of the pro-eutectoid cementite in Figure 4.8 (a) was consistent with the particles observed on PAG facets in Figure 4.17 (a). Figure 4.17 (b) shows a higher-magnification image of the MVC region. The white arrows show micro-voids with cementite particles in them. The observed cementite particle size matched the cementite observed in Figure 4.13 (d).

Figure 4.17 SEM fractographs from the overload region of RTDQ microstructure showing (a) IG and MVC fracture morphologies; dashed circles show cementite particles at the PAGB interfaces and (b) high magnification image of MVC region showing fine carbides at the core of micro-voids.
High resolution fractographs were recorded using a FEI Helios SEM to perform stereological analysis on the fracture surfaces for all four conditions. Fracture surfaces spanning over 1000 PAGs were analyzed to quantify the percent IG character of the overload regions. Figure 4.18 shows the percent IG character of the four microstructure conditions. For the three microstructure conditions that were tempered at 175 °C, RTDQ, LN2 and SQ, the overload fracture surface had approximately 75 pct IG character. For the LN2T condition which was tempered at 240 °C, the overload failure region had a significantly lower percent IG character of approximately 50 pct. Yaney and Krauss [4.19] previously investigated the influence of tempering temperature and phosphorus content on the overload fracture behavior of 52100 steel. They observed that at increasing tempering temperatures, greater transgranular fracture (MVC) was observed for 52100 steel (austenitizing temperature: 850 °C, P content: 0.023 wt pct). They suggested that with increasing tempering temperatures, the matrix softens, and carbides within decomposed RA act as crack nucleation sites. The decomposition of RA to ferrite and carbide in this study might have contributed to the increase in the MVC character observed for the LN2T condition.

Figure 4.18 Quantitative stereological trends in percent IG behavior of overload fracture for the different microstructure conditions. The error bars represent the standard deviation in the results from the different fractographs.
Fatigue fracture regions were also analyzed for the four microstructures to identify and quantify the various modes of fatigue failure. Figure 4.19 (a) shows fractographs from the RTDQ sample, taken at 4 mm crack length from the center region. The white arrow above the scale bar shows the direction of crack propagation (right to left). Similar to the overload region, IG fracture along PAGBs was observed. Additionally, two different fracture morphologies, mixed ductile-brittle (MDB) and transgranular (TG) fracture were also observed in the fractographs. All three different fracture features: MDB, IG and TG are labelled. Figure 4.19 (b), (c) and (d) show high magnification SEM images of the MBD, IG and TG regions shown in Figure 4.19 (a). The fatigue fracture region in Figure 4.19 (b) shows fine ductile dimples connected via relatively flat facets (potentially cleavage) and is thus labelled MDB. It is important to note that the MDB regions in the fatigue fracture surfaces were different from the MVC regions in the overload fracture surfaces (Figure 4.16 (a)). Pro-eutectoid carbide particles were observed on the IG fracture surface of the fatigue region (Figure 4.19 (c)). In Figure 4.19 (d), fine sub-micron facets, ridges and cementite particles were observed on the TG fatigue fracture surface. The microstructures contain different constituents such as pro-eutectoid carbides, fine dispersed carbides, tempered plate-martensite, RA, fresh martensite (RTDQ and SQ), and RA decomposition products (LN2T). Hence, a fatigue crack can interact with one or more of the above-mentioned constituents resulting in various TG morphologies such as ductile striations, quasi-cleavage, and cleavage. Fractographs showing different TG failures regions are included in Appendix E.

Figure 4.20 shows representative fracture surfaces from the four microstructures, taken at a crack length of 4 mm from the center of the sample in the width direction. The crack length of 4 mm corresponded to a stress intensity range of 5.2 MPa·m^{0.5} at the crack tip. The direction of crack growth was from right to left in the fractographs. While some grains failed via one of the failure modes, the crack path also transitioned from IG to TG mode within some grains. Alternatively, large transgranular patches spanning several grains were also observed in the fatigue regions of the samples (not shown here).
Figure 4.19 SEM fractographs from the fatigue regions of the RTDQ sample at 4 mm crack length (corresponding to $\Delta K = 5.2$ MPa m$^{0.5}$) showing (a) an overview, (b) MDB, (c) IG, and (d) TG regions.
Figure 4.20 SEM fractographs from the fatigue regions (4 mm crack length corresponding to $\Delta K = 5.2 \text{ MPa}\cdot\text{m}^{0.5}$) of the (a) RTDQ, (b) LN2, (c) LN2T, and (d) SQ samples. The fracture surfaces showed mixed IG, TG and MDB fracture modes.

Quantitative stereological analysis was performed on the fracture surfaces to determine the trends in percent, IG, TG and MDB fracture in the fatigue region of different CT samples. SEM images were recorded at different crack lengths to study the fatigue mechanisms operating at low (4.8 MPa-$\text{m}^{0.5}$), intermediate (5.2 MPa-$\text{m}^{0.5}$), and high (5.75 MPa-$\text{m}^{0.5}$) $\Delta K$ values in the stable fatigue crack growth regime. Figure 4.21 (a), (b) and (c) show the trends in percent fracture features for the different microstructures. The initial RA vol pct is included in parenthesis with the sample labels on the x-axis.
Figure 4.21 Percent fracture features for the four different microstructures at $\Delta K = (a) 4.8 \text{ MPa}\cdot\text{m}^{0.5}$ (b) 5.2 MPa$\cdot$m$^{0.5}$, and (c) 5.75 MPa$\cdot$m$^{0.5}$. The initial RA content is included in parentheses in x-axis.
Figure 4.21 Continued

Fracture surfaces of all the samples showed mixed IG, TG and MDB failure at low, intermediate, and high values of $\Delta K$. However, the relative fractions for each fracture feature varied between different microstructures for the same value of $\Delta K$. Also, for a given microstructure, the relative fraction of fracture features varied at different $\Delta K$ values. Different TG modes (such as ductile striations, quasi-cleavage, and cleavage) were not categorized and quantified separately due to the complexity and challenges in their identification. Overload or fatigue features showing ductile dimples were collectively categorized as MDB during the stereological analysis. Meaningful trends in percent fracture features were extracted from the data presented in Figure 4.21 to study the effect of microstructure and $\Delta K$ on failure modes with the aim to rationalize the observed FCG rates.

Figure 4.22 (a) shows the trends in percent IG factor as a function of $\Delta K$ for all four microstructures. The initial RA content of each microstructure is included in parentheses in the figure legend. The pct IG fracture increased with increasing $\Delta K$ for all the microstructures. IG fracture is
associated with brittle failure and rapid crack propagation [4.20]. The increase in the slope of the FCG data (Figure 4.12 (b)) at high values of $\Delta K$ can be attributed to the increase in percent IG fracture.

Additionally, in Figure 4.22 (a), it was observed that both the RTDQ and SQ conditions had lower percent IG character than the LN2 and LN2T conditions in the fatigue fracture regions at all values of $\Delta K$. The difference in IG character was greater at low and intermediate $\Delta K$ values. The LN2T condition had lower IG character than the LN2 condition, but the difference was insignificant at low and intermediate $\Delta K$. Figure 4.22 (b) shows the percent TG fraction as a function of the initial RA content of microstructures for different $\Delta K$ values. With increasing $\Delta K$, the TG character expectedly reduced since the IG character increased. Further, for microstructures with higher initial RA content, TG character was greater. Thus, it was observed that presence of RA promoted TG fracture and suppressed the IG character during fatigue crack growth.

![Figure 4.22 Quantitative trends in percent fracture character.](image)

(a) Percent IG character versus $\Delta K$, and (b) percent TG character versus RA content.

The relative fractions of fracture features provided insights into the observed FCG rates. The FCG rate followed the trend: LN2 > RTDQ > LN2T (Figure 4.11 (b)). Figure 4.21 (c) showed the percent fracture features for LN2, LN2T, and RTDQ conditions at $\Delta K = 5.75$ MPa·m$^{0.5}$. The LN2 microstructure
had the highest IG character and also the highest FCG rate amongst all the sample conditions, since IG fracture is a brittle mode of failure. For the LN2T condition, the greatest fraction of MDB character was observed. Hence, despite a greater IG character in the LN2T condition (than RTDQ), lower FCG rates can be attributed to the ductile character of the MDB failure mode.

At low $\Delta K = 4.8 \text{ MPa} \cdot \text{m}^{0.5}$, the FCG rates and the percent fracture features (Figure 4.21 (a)) were similar for RTDQ and SQ conditions. FCG rates for the SQ microstructure were lower than the RTDQ microstructure at intermediate and high $\Delta K$ values. The SQ condition had marginally lower IG character and greater MDB character as compared to the RTDQ condition at $\Delta K = 5.75 \text{ MPa} \cdot \text{m}^{0.5}$ (Figure 4.21 (c)). The quantitative fractography results suggested that the FCG resistance was highest for MDB fracture, followed by TG fracture and the least for the IG fracture mode. The microstructures exhibiting greater fractions of MDB and TG fracture character had lower FCG rates.

4.4 Discussion

The FCG rate and fatigue crack characterization results are discussed in the context of existing literature pertaining to fatigue behavior of steels, quench embrittlement in 52100 steel, and the effects of deformation-induced martensite transformation. The IG failure during fatigue, the effect of RA to martensite transformation, and the stability of RA are related to the interaction between fatigue crack and high-carbon plate-martensite austenite microstructures. Finally, the implications of the results of this study on fatigue performance of carburized components are discussed.

4.4.1 Fatigue Crack Growth Rates and IG Fracture in 52100 Steel

Most continuum-based fatigue striation models predict the value of exponent $m$ in the Paris-Erdogan law (Equation 4.2) to be between 2 and 4 [4.21]. The stable FCG rates in ductile microstructures are independent of strength levels and microstructurally insensitive [4.22]. However, Ritchie and Knott [4.15] observed that the values of $m$ could be higher (up to 10) in temper embrittled steels. Values of $m$ as high as 15 – 50 have been reported for brittle ceramics where FCG rates are highly sensitive to $\Delta K$ [4.23]. In this study, $m$ values of approximately 12 to 15 were observed for the different microstructures. Such
large values of $m$ have been attributed to the presence of ‘static’ or ‘monotonic’ modes or failure in steels such as IG fracture, cleavage and MVC [4.15]. Extensive IG fracture was found in the fatigue regions of all the samples, and thus high values of $m$ were observed for the FCG data. The RTDQ and SQ microstructures with a higher initial RA content had lower $m$ values than the LN2 and LN2T microstructures in the range of $\Delta K$ values between 4.6 MPa·m$^{0.5}$ and 5.6 MPa·m$^{0.5}$.

Sadananda and Vasudevan have proposed a Unified Approach where the FCG rates depend on two crack tip driving parameters, $\Delta K$ and $K_{\text{max}}$ [4.20]. They propose that static fracture modes during fatigue crack propagation are $K_{\text{max}}$ dependent. At low $K_{\text{max}}$, the crack growth process is fatigue dominated whereas at high $K_{\text{max}}$, the crack growth is fracture dominated [4.24]. The extent of IG fracture (static failure) increased with increasing $\Delta K$ (increasing $K_{\text{max}}$) for all the microstructure conditions (Figure 4.20 (a)). Alternatively, the percent TG character (fatigue mode of failure) was highest at low $\Delta K$ and low $K_{\text{max}}$ values for all the microstructures. To better understand the observed trends in FCG behavior, fracture modes in 52100 steels are discussed next.

#### 4.4.2 Fatigue and Overload Failure Mechanisms in 52100 Steel

Extensive IG fracture was observed for all the microstructures at low, medium and high $\Delta K$ (Figure 4.20 (a)). The occurrence of IG failure during fatigue in embrittled high strength steels has been attributed to segregation of impurities to the PAGBs and was responsible for high FCG rates [4.25]. High-carbon steels containing greater than 0.5 pct carbon content are susceptible to IG failure along PAGBs after austenitizing and low temperature tempering due to quench embrittlement [4.26]. The phenomenon of quench embrittlement and fracture has been widely investigated for 52100 steels [4.5, 4.27, 4.28]. Ando and Krauss [4.27] proposed that IG cracking in high-carbon steels was caused by cementite formation along PAGBS during quenching. They further suggested that phosphorus segregates to PAGBs and promotes the formation of cementite along PAGBs. Kar et al. [4.17] alternatively suggested that large transformation stresses associated with plate-martensite formation produce microcracks within PAGs which are weak due to the presence of undissolved pro-eutectoid cementite, thereby resulting in IG
fracture. The presence of significant amounts of P (0.023 wt pct) in the 52100 alloy used in this study, further aggravated the susceptibility of the 52100 steel used in this study towards IG fracture.

Nazakawa and Krauss [4.5] investigated the effect of austenitizing treatments on the overload fracture behavior of 52100 steel; microstructures with fine spherical carbides (200 nm to 500 nm in diameter) failed via transgranular micro-void coalescence as the network of cementite particles act as stress-risers, and potential crack nucleation sites [4.29]. Brittle IG fracture of 52100 steel was attributed to the formation of fine cementite particles along PAGBs [4.30]. In this study, the microstructures consisted of undissolved cementite after the austenitization step. A bi-modal cementite particle distribution with coarse pro-eutectoid cementite along PAGBs and a fine dispersion of spherical cementite (less than 200 nm in diameter) within the PAG was observed. Furthermore, microstructural banding due to differences in number density of carbides was observed (Figure 4.7). Thus, for all conditions, mixed MVC (resulting from fine carbide dispersion) and IG fracture was observed (resulting from cementite along PAGB). For the LN2, RTDQ and SQ condition, due to the similar carbide distribution, similar overload fracture behavior was observed (approximately 75 pct IG and 25 pct MVC character). However, for the LN2T condition tempered at 240 °C, a greater amount of MVC fracture was observed (Figure 4.16 (b)). Yaney and Krauss [4.19] investigated the effect of tempering temperature on overload fracture behavior of 52100 steel and observed that with increasing tempering temperature, IG character reduced. They suggest that temper carbides and regions of RA that decomposed to carbides became crack initiation sites within the matrix resulting in a greater amount of MVC fracture.

In addition to the static modes of failure, i.e. IG and MVC observed in the overload regions of all the samples, the fatigue regions also had varying amounts of TG fracture. Nazakawa and Krauss [4.5] also reported significant amounts of TG fracture in the fatigue pre-crack regions of their overload fracture samples. Various fracture features such as facets, ridges and cementite particles were observed along the TG regions of the fatigue samples in this study. Striations were not observed and there was very little evidence of plastic flow, which is similar to the observations by Rescalvo and Averbach [4.13]. Previously, transgranular fracture modes such as cleavage, quasi-cleavage, mixed ductile-brittle, inter-
martensite and intra-martensite have been reported for embrittled steels [4.31]. Some of the above-mentioned fracture features such as cleavage, quasi-cleavage and mixed ductile-brittle fracture were present in the TG regions of the different microstructures in this study (Figure 4.18 (d) and Appendix E); however, identification and quantitative analysis of the different TG fracture modes is beyond the scope of this work.

4.4.3 Effects of Retained Austenite

The RTDQ and the SQ microstructures had significant initial RA volume percent of approximately 17.6 and 19.3 vol pct respectively. The two cryo-quenched microstructures, LN2 and LN2T, had much lower initial RA volume fraction of 9.3 and 7.1 vol pct, respectively. Fractographic analysis showed that the microstructures with higher initial RA content had lower percent IG character for the fatigue fracture surface at low, medium and high $\Delta K$ (Figure 4.20 (a)). The difference in percent IG character was prominent at low $\Delta K$ values. Conversely, for any given $\Delta K$, the percent TG character was greater for the microstructures with higher initial RA content (RTDQ and SQ). Thus, it was inferred that the presence of RA suppressed IG character on the fatigue fracture surface and promoted TG failure.

Additionally, deformation induced martensite transformation (DIMT) was observed in the vicinity of the fatigue crack for the RTDQ and SQ microstructures. For the RTDQ microstructure, the size of the transformation zone at $\Delta K = 5.2$ MPa·m$^{0.5}$ was approximately 10 µm, while the estimated size of the plastic zone was approximately 3 µm. Since DIMT was observed outside the estimated plastic zone, it is suggested that stress-assisted RA to martensite transformation occurred in the RTDQ sample [4.18]. The stress and strain fields at the crack tip provided the driving force for the transformation of meta-stable RA to fresh untempered plate-martensite. DIMT near the fatigue crack potentially influenced the crack path, failure modes and FCG rates.

In the literature, many complementary or contradictory mechanisms have been proposed to explain the role of DIMT on FCG behavior in the steels. The mechanisms that predict an improvement in FCG resistance due to DIMT are listed below:
• Transformation induced crack closure – DIMT introduces tensile deformation in the wake of the crack, causes pre-mature contact between the crack faces, and reduces the effective crack driving force. [4.32]

• Transformation Toughening – The DIMT imposes compressive residual stress at the crack tip which offset the applied stress intensity and retards FCG rates. [4.33]

• Transformation induced energy absorption – The strain energy absorbed during DIMT reduces the energy available for crack extension. [4.34]

• Transformation induced crack deflection – The DIMT transformation product acts as an obstacle in the crack propagation and increases tortuosity of the crack path. [4.33, 4.35]

• Transformation mediated crack arrest – The hard and brittle martensite acts as a barrier for crack propagation. [4.36]

Conversely, the brittle martensite (the transformation product) was found to reduce the fracture toughness and FCG resistance via the following mechanisms:

• Martensite induced crack nucleation – The fresh martensite lowers the local toughness and acts as potential crack nucleation sites in TRIP steels [4.37].

• Transformation mediated crack coalescence – DIMT is accompanied by micro-cracking near the crack tip. The microcracks coalesce with the fatigue crack and accelerate the FCG rates [4.36].

Two possible mechanisms for the role of RA in promoting transgranular failure in fatigue regions of the RTDQ and SQ microstructures in this study are proposed below:

Transformation Toughening Mechanism

The RTDQ and SQ microstructures have approximately 9 vol pct more RA than the LN2 condition that may undergo DIMT. The DIMT is accompanied by a 4 pct volume expansion as RA has a face centered cubic (FCC) crystal structure, whereas the martensite phase has a body centered tetragonal (BCT) crystal structure. The volume expansion imposes a compressive residual stress at the crack tip \( K_{\text{Trans}} \). The \( K_{\text{Trans}} \) effectively reduces the applied \( K_{\text{max}} \) at the crack tip to \( K_{\text{eff max}} = K_{\text{max}} - K_{\text{Trans}} \).
provides the driving force for FCG via static failure mode, in this case IG fracture. Thus, the reduction in $K_{\text{mat}}$ due to $K_{\text{Trans}}$ suppressed the static IG failure and promoted TG failure in the RTDQ and SQ microstructures. The proposed mechanism is consistent with the transformation toughening model developed by Mei and Morris [4.33] for stainless steel and the mechanism of transformation toughening in brittle materials proposed by McMeeking and Evans [4.38].

**Martensite Influenced Crack Propagation**

This proposed mechanism is associated with the transformation product, fresh martensite, and is based on the transformation mediated crack coalescence (TMCC) model proposed by Wang *et al.* [4.36]. The fresh untempered martensite (the transformation product) is hard and brittle. Due to the stress fields associated with the fatigue loading, microcracks develop at the fresh plate martensite interfaces and at cementite particles in the transformation zone ahead of the fatigue crack tip. These microcracks coalesce with the primary fatigue crack resulting in a transgranular crack path. However, since the martensitic features are finer than the PAGS, martensite induced crack deflection lowers the FCG rates as the transgranular crack follows a tortuous path through the martensitic substructure. Figure 4.23 shows evidence of crack branching, crack coalescence, microcracking, and a transgranular zig-zag crack path in the RTDQ sample. The direction of crack growth is left to right in the micrograph. Recently, Kowathanakul *et al.* observed martensite induce crack deflection in SUJ2 bearing steels [4.39]. Previously, Mei and Morris have also proposed that the transformed region produces a zig-zag crack path [4.33].
Figure 4.23 SEM micrograph of fatigue crack in the RTDQ microstructure showing primary crack, secondary cracks, and evidence of microcracking, crack coalescence and crack deflection. The direction of crack growth is from left to right.

4.4.4 Implications on Fatigue Behavior of Carburized Microstructures

The carburizing process introduces a gradient of carbon composition along the depth of a steel component [4.40]. Thermal and transformation gradients during the quenching step result in compressive residual stresses in the carburized component [4.2]. Figure 4.24 (a) shows a bending fatigue fracture surface of a gas carburized 8620 Brugger specimen. The crack initiated at the surface intergranularly along PAGBs. The stable fatigue crack growth through the tempered martensite and RA microstructure was largely transgranular. Figure 4.24 (b) shows the gradients in carbon content and compressive residual stress as a function of depth from the surface in carburized 52100 steel disks [4.41]. As the carbon content decreased along the depth of the sample, the susceptibility to IG fracture due to quench embrittlement also reduced. Furthermore, the thermal processing induced compressive stress effectively lowers the maximum stress intensity at the crack tip, which is the driving force for static IG failure. Thus, a
combination of composition gradient and compressive residual stress resulted in transgranular fatigue fracture in the case region of the carburized component.

In this study, the fatigue crack propagation was investigated in high-carbon plate-martensite austenite microstructures in 52100 steel to evaluate the role of RA on fatigue behavior in the absence of macro residual stresses and compositional gradients. Undissolved carbides were also present along with plate martensite and RA. Extensive IG fracture was observed in the fatigue regions of the high-carbon microstructures due to quench embrittlement (Figure 4.20). The network of cementite played a key role in the failure mechanisms. It was observed that a greater amount of RA suppressed IG fracture and promoted TG fracture (SQ and RTDQ conditions).

Based on the results of FCG behavior in quenched and tempered 52100 steel, it is hypothesized that during low cycle fatigue (LCF) of carburized components, the presence of RA and its subsequent transformation to martensite would impose a compressive residual stress (transformation toughening mechanism) at the crack tip, thereby further lowering the processing induced compressive residual stress [4.43]. As a result, a greater applied tensile stress would be required to activate the static modes of
failure and propagate fatigue cracks in the case regions of carburized components. Thus, the LCF fatigue strength of the microstructure would improve due to the presence of RA.

As opposed to LCF (high values of \( K_{\text{max}} \)), during high cycle fatigue (HCF) at low value of \( K_{\text{max}} \), the static modes of failure would not be active. It is hypothesized that during HCF, the hard and brittle fresh martensite in the transformation zone would be susceptible to micro-cracking, and fatigue crack propagation rates would increase via the TMCC mechanism (Wang et al.). Thus, it is speculated that due to the presence of mechanically unstable RA, accelerated FCG would be observed for carburized microstructures in the HCF regime. However, the presence of mechanically stable RA (SQ microstructure) would improve local toughness, potentially reduce microcracking susceptibility and increase FCG resistance of the carburized microstructures in the HCF regime.

4.5 References


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CHAPTER 5
TRANSFORMATION TOUGHENING IN HIGH-CARBON PLATE-MARTENSITE MICROSTRUCTURES

The following chapter discusses implementation of a transformation toughening model for high-carbon plate-martensite austenite microstructures. Existing transformation toughening models, the McMeeking and Evans [5.1] model for brittle materials and the Mei and Morris model [5.2] for stainless steels were modified to predict the trends in fatigue crack growth (FCG) rates for the high-carbon plate-martensite austenite microstructures. Experimentally determined parameters were used to inform the model and the role of RA on FCG rates was rationalized. Section 5.1 presents the relevant transformation toughening literature. Two of the existing transformation toughening models are described in Section 5.2. The methodology for the implementation of the Mei and Morris model for high-carbon plate-martensite austenite microstructures is presented in Section 5.3. Finally, the model predictions of the FCG rates for high-carbon plate-martensite austenite microstructures are discussed in Section 5.4.

5.1 Background
Transformation toughening refers to an improvement in fracture toughness of the material resulting from localized phase transformation in the vicinity of a crack tip. For transformation toughening to occur, it is important that the material has a meta-stable phase, that is capable of transformation due to crack tip stress fields [5.3]. Additionally, the transformation should be instantaneous (not requiring time-dependent long-range diffusion) and result in a shape change or volume expansion. Transformation toughening results from the dissipative work required for the phase transformation, as less energy is available for crack extension [5.4]. Alternatively, it has been proposed that the transformation strains produce local compressive stresses, thereby lowering the stress intensity at the crack tip, i.e., the driving force for crack propagation [5.1].

Displacive martensitic transformations satisfy the necessary conditions of transformation toughening [5.3]. In zirconia (ZrO₂) ceramics, the tetragonal to monoclinic transformation of ZrO₂ has
been shown to improve the toughness [5.5, 5.6]. In metastable austenitic stainless steels, austenite to martensite transformation alters the work hardening rate, delays the tensile instability to larger strains and can also enhance fracture toughness by suppressing micro-void nucleation [5.7, 5.8]. However, in the case of high-strength transformation induced plasticity (TRIP) steels, conflicting results have been reported for the effect of RA to martensite transformation on fracture toughness of microstructures. The conflict stems from the fact that the transformation product, fresh martensite, is brittle. Thus, the improvement in toughness due to the transformation may be negated by the brittle nature of the transformation product [5.3]. In medium carbon high strength steels (4340 and 300 M), the presence of a fine dispersion of stable RA was shown to improve the fracture toughness [5.9]. Jacques et al. [5.10], on the other hand, showed that in TRIP steels, fracture originated in regions containing only martensite and attributed the low fracture toughness to pre-mature cracking in martensitic regions.

In ceramic systems, transformation toughening has been quantitatively rationalized via a reduction in stress intensity at the crack tip. McMeeking and Evans [5.1] developed a transformation toughening model that calculated the effect of phase transformation induced volume expansion on the local stress intensity factor at the crack tip. Mei and Morris [5.2] implemented the McMeeking and Evans model on austenitic stainless steels to rationalize the influence of deformation induced austenite to martensite transformation on fatigue crack growth behavior in SS304. In this chapter, the Mei and Morris model is implemented on high-carbon plate-martensite austenite microstructures with the aim to better understand the influence of RA on fatigue crack growth resistance in carburized and bearing steels.

5.2 Transformation Toughening Models

The following section describes two existing transformation toughening models. Both models use linear elastic fracture mechanics approaches as opposed to the transformation-energy based models. The McMeeking and Evans model for brittle materials is discussed in Section 5.2.1. Next, the Mei and Morris transformation toughening model for stainless steel is discussed in Section 5.2.2.
5.2.1 McMeeking and Evans Model

McMeeking and Evans modeled the reduction in stress intensity at the crack tip due to transformation-induced stresses in brittle materials. There are two parts to the model: (i) determination of the transformation zone (size and shape) and (ii) calculation of the reduction in stress intensity at the crack tip due to transformation.

For the determination of the transformation zone, it was assumed that the transformation in the vicinity of the crack tip occurred when a critical value of local stress \( (p_{ij})_c \) is reached. Thus, for a given applied stress, a transformation zone of size \( r^* \) would develop at the crack tip, given by

\[
 r^* = \left[ \frac{K^2 D(\theta, \nu)}{2\pi (p_{ij}^c p_{ij}^c)} \right] \tag{5.1}
\]

where \( D \) is a function of orientation, \( \theta \), the angle from the crack plane, Poisson’s ratio \( \nu \), and \( K_c \) is the critical stress intensity at the crack tip. Since the size of the transformation zone was variable and depended on parameters such as particle size, temperature, chemical composition, and matrix stiffness, they used an experimentally determined transformation zone half-width parameter, \( w \), instead. They further assumed that the transformation zone followed a constant hydrostatic stress contour, which meant that the transformation was activated by a critical value of hydrostatic stress at the crack tip stress field. The transformation zone size was given by –

\[
 r = \left( \frac{8}{3\sqrt{3}} \right) w \cos^2 \frac{\theta}{2} \tag{5.2}
\]

where \( w \) is the experimentally determined half-width of the transformation zone.

The toughening due to a transforming particle resulted from the transformation strain, which imposed stress at the crack tip. The deviatoric component of the transformation strain was considered to be negligible, and only the influence of the dilatant component of the transformation strain on the stress intensity at the crack tip was analyzed. The model was thus relevant for crack-tip transformations which induce an equal number of variants per particle that effectively nullify the deviatoric component of the transformation strain. McMeeking and Evans used the Eshelby technique [5.11] to determine the
transformation stress and the weight function method [5.12] to calculate the change in stress intensity at the crack tip. The change in stress intensity factor due to the transformation zone was given by the general expression

$$\Delta K_I = (E V_f \epsilon^T \sqrt{w}) \left[ \kappa \left( \frac{\Delta a}{w}, \nu \right) \right]$$  \hspace{1cm} (5.3)

where $E$ is the Young’s modulus, $V_f$ is the fraction of transformation, $\epsilon^T$ is the dilatant transformation strain, and $\kappa$ is a transformation zone dependent function of crack extension ($\Delta a/w$) and Poisson’s ratio, $\nu$.

For the transformation zone shape described by Equation 5.2, the calculated values of $\kappa$ function taken from the McMeeking and Evans article are tabulated in Table 5.1.

<table>
<thead>
<tr>
<th>$\Delta a/w$</th>
<th>$\kappa = \Delta K_I (1-\nu) / E \epsilon^T V_f \sqrt{w}$</th>
<th>$\Delta a/w$</th>
<th>$\kappa = \Delta K_I (1-\nu) / E \epsilon^T V_f \sqrt{w}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>5.3</td>
<td>-0.209</td>
</tr>
<tr>
<td>0.8</td>
<td>-0.148</td>
<td>7.9</td>
<td>-0.211</td>
</tr>
<tr>
<td>1.6</td>
<td>-0.184</td>
<td>12.5</td>
<td>-0.213</td>
</tr>
<tr>
<td>2.3</td>
<td>-0.196</td>
<td>24.4</td>
<td>-0.214</td>
</tr>
<tr>
<td>3.9</td>
<td>-0.206</td>
<td>$\infty$</td>
<td>-0.215</td>
</tr>
</tbody>
</table>

The initial value of $\kappa$ was zero due to the initial transformation zone shape, which meant that there was no toughening. However, as the crack entered the transformation zone ($\Delta a/w$ increased), the value of $\kappa$ decreased (transformation toughening), suggesting that the transformation in wake of the crack tip contributed to the reduction in $\Delta K_I$ at the crack tip. Figure 5.1 (a) schematically shows a crack entering the transformation zone and the resulting transformation toughening. The gray arrows indicate the tractions on the transformation zone boundary and crack surfaces. The value of $\kappa$ saturated asymptotically at approximately -0.22. Thus, for crack extension values greater than approximately $5w$, the change in stress intensity due to transformation toughening was given by
\[
\Delta K_I = -\frac{0.22(EVf\varepsilon^T\sqrt{w})}{(1 - \nu)}
\]

(5.4)

Further, they also proposed an expression for toughening due to an arbitrarily located cylindrical through-thickness particle near the crack tip. The change in stress-intensity due to a transformed area, \( A_p \), was given by

\[
\Delta K_I = -\frac{(EVf\varepsilon^T\sqrt{w})}{6\sqrt{2}\pi(1 - \nu)} \int_{A_p} r^2 \cos \left( \frac{3\theta}{2} \right) dA
\]

(5.5)

where \( r \) is the distance of the transformed particle from the crack tip, \( \theta \) is the angle between crack and the particle location, and \( A_p \) is the area of the transformed particle. Equation 5.5 can be integrated over any arbitrary transformation zone shape to calculate the reduction in stress-intensity due to transformation toughening.

5.2.2 Mei and Morris Model

Mei and Morris [5.2] modified the McMeeking and Evans model [5.1] to analyze the effect of volume expansion associated with the austenite to martensite transformation on fatigue crack growth rates in SS304 alloys. They too assumed that martensite variant selection during the transformation effectively nullified the shear component of the transformation strain, and thus modelled the transformation toughening purely due to the \( 2 \) pct volume expansion. Contrary to McMeeking and Evans’ approximation of transformation being driven by hydrostatic stress, Mei and Morris assumed that the austenite to martensite transformation was driven by local shear stress. Thus, the transformation zone shape was defined by a constant equivalent shear stress contour

\[
r = wc(\nu) \cos^2 \left( \frac{\theta}{2} \right) \left[ 3 \sin^2 \left( \frac{\theta}{2} \right) + (1 - 2\nu)^2 \right]
\]

(5.6)

Where the \( c(\nu) \) factor was used to fix the half-width, \( w \), of the contour. Figure 5.1 (b) schematically shows the two different transformation zone shapes. Using the constant equivalent shear stress contour, the asymptotic value of the change in stress intensity at the crack tip (similar to Equation 5.4) was given by
\[
\Delta K_i = K_{\text{Trans}} = -\frac{0.33(EV_f e^T \sqrt{w})}{(1 - \nu)}
\]  

(5.7)

The transformation toughening due to the shear stress contour was 50 pct greater than the hydrostatic contour used by McMeeking and Evans. The transformation occurring ahead of the crack tip in the wedge-shaped region between \( \theta = \pm 60^\circ \) (dashed gray lines in Figure 5.1 (b)) increases the value of \( K_{\text{Trans}} \) (makes it less compressive), due to the \( \cos (3\theta/2) \) factor in Equation 5.5. The martensite transformation ahead of the \( \pm 60^\circ \) wedge increases the stress intensity at the crack tip, while the transformation behind the \( \pm 60^\circ \) wedge and in the wake of the fatigue crack reduces the stress intensity at the crack tip (transformation toughening). Since in the case of constant hydrostatic stress contour, a greater amount of transformation takes place ahead of the crack tip, lower magnitude of transformation toughening is observed for the McMeeking and Evans model as compared to the Mei and Morris model.

To implement the transformation toughening model on their FCG testing results, Mei and Morris first determined \( w \) as a function of applied \( \Delta K \) using optical microscopy. Assuming a linear transformation profile with 50 pct transformation at the crack surface and 10 pct transformation at the zone boundary, they then calculated the values of \( K_{\text{Trans}} \) as a function of the applied \( \Delta K \). The resulting transformation toughening adjusted effective \( \Delta K \) at the crack tip was given by the expression

\[
\Delta K_{\text{eff}} = K_{\text{max}} - \max \{K_{\text{min}}, |K_{\text{Trans}}|\}
\]

(5.8)

where \( K_{\text{max}} \) and \( K_{\text{min}} \) are the maximum and minimum applied stress intensities at the crack tip during fatigue cycling. Upon plotting the FCG rates \( (da/dN) \) as a function of \( \Delta K_{\text{eff}} \), they observed agreement in the FCG \( da/dN \) versus \( \Delta K \) behavior for SS304 samples exhibiting varying degrees of transformation toughening. The next section discusses the implementation of the Mei and Morris model for high-carbon plate-martensite austenite microstructures.
5.3 Model Implementation Methodology

The Mei and Morris model was implemented on the high-carbon plate-martensite austenite microstructure RTDQ to quantify the influence of transformation toughening due to the RA-to-martensite transformation on the observed FCG rates, previously presented in Chapter 4. Section 5.3.1 describes the assumptions associated with implementation of the model. The value of $K_{\text{Trans}}$ was calculated using Equation 5.7. The values of Young’s modulus ‘$E$’ = 203 GPa [5.13] and Poisson’s ratio ‘$\nu$’ = 0.277 [5.14] were taken from literature. Three of the parameters in Equation 5.7, the volume fraction of transformed martensite, $V_f$, the transformation strain, $\varepsilon_T$, and the transformation zone half-width, $w$, were experimentally determined (described in Section 5.3.2 – Section 5.3.4).

5.3.1 Assumptions

The Mei and Morris model used the stress intensity at the crack tip approach (as opposed to energy-based approach) in the analysis of transformation toughening. Thus, to implement their model for high-carbon plate-martensite austenite microstructures, firstly, it was assumed that linear elastic fracture
mechanics (LEFM) applied to the system, including small scale yielding. The estimated plastic zone size, $r_p$, is given by the expression

$$r_p = \frac{1}{2\pi} \left( \frac{K_I}{\sigma_y} \right)^2$$

(5.9)

where $K_I$ is the applied stress intensity at the crack tip and $\sigma_y$ is the yield strength [5.15]. For the RTDQ sample, the onset of unstable crack growth was observed at $K_{\text{max}} = 6.7$ MPa·m$^{0.5}$ (crack length = 7 mm).

High-carbon 52100 steel is susceptible to quench embrittlement, and conditions with these martensitic microstructures (untempered or low temperature tempered conditions) fail before yielding during tensile tests. Thus, the yield strength of martensitic microstructures in 52100 steel (hardness approximately 64 HRC) cannot be experimentally determined. Further, hardness to yield strength conversion relationships were not available for microstructures with such high hardness values (RTDQ – 825 HV) [5.16]. However, to conservatively estimate the yield strength of RTDQ microstructure, a literature value for a bainitic microstructure in 52100 steel (hardness - 59 HRC), $\sigma_y = 1697$ MPa [5.17], was assumed as an under-estimate. Based on the experimentally determined $K_I$ value and conservatively estimated $\sigma_y$ value from the literature, the maximum size of the plastic zone for the RTDQ microstructure was calculated to be 2.5 µm, which was much smaller as compared to the crack lengths of the order of millimeters. Thus, the small-scale yielding assumption was valid.

To determine the transformation zone, it was assumed that the transformation of RA to plate-martensite near the crack tip was activated when the value of the local equivalent shear stress reached a critical value. Thus, the shape of the transformation zone was given by the constant equivalent shear stress contour, Equation 5.6. To calculate the reduction in stress intensity at the crack tip ($K_{\text{Trans}}$) due to transformation strain ($\varepsilon_T$), only the volume expansion component of the strain was considered. It was assumed that due to variant selection during RA to martensite transformation, the effective shear strain due to the transformation was nullified. This assumption was adopted to simplify the analysis since the influence of shear strain depends on size, orientation and location of transformed martensite [5.18]. The
assumptions made during experimental determination of $V_f$, $\varepsilon^T$, and $w$ are described in detail in the following subsections.

### 5.3.2 Volume Fraction of Transformed Martensite

A comparative analysis between the LN2 and the RTDQ conditions (Chapter 4) was performed in this study. Negligible transformation in the vicinity of the fatigue crack was assumed for the LN2 condition (baseline). Thus, the difference in the initial RA content between RTDQ condition and the LN2 condition was considered to be the fraction of RA available to transform to martensite in the RTDQ condition. Using the RA volume fraction values from Table 4.3, the value of $V_f$ was calculated to be

$$V_f = RA_{RTDQ} - RA_{LN2} = 17.6 \text{ vol pct} - 9.4 \text{ vol pct} = 8.2 \text{ vol pct}$$

(5.10)

Electron backscatter diffraction (EBSD) mapping was used to determine the distribution of RA to martensite transformation near the fatigue crack. Two different models, the uniform transformation and linear transformation models were developed [5.19]. The uniform transformation model (UTM) assumed that the amount of martensite transformation was 8.2 vol pct within the entire transformation zone. The linear transformation model (LTM) assumed that the amount of martensite transformation varied linearly between 8.2 vol pct at the crack surface and 0 vol pct at the edge of the transformation zone, as a function of distance from the crack tip.

### 5.3.3 Transformation Strain due to Volume Expansion

The transformation strain ($\Delta V/V$) associated with the RA to martensite transformation was calculated using the lattice parameters determined from the neutron diffraction data of the DQ condition. The neutron diffraction results were presented in Section 3.4.3 (Figure 3.19). The transformation strain is given by

$$\varepsilon^T = \frac{\Delta V}{V} = \frac{(V^y - V'^a)}{(V^y)}$$

(5.10)

where $V^y$ is the volume of an atom in austenite (Å$^3$/atom) and $V'^a$ is the volume of an atom in the martensite (Å$^3$/atom). Using the values of austenite lattice parameter to be 3.603 Å and the martensite lattice parameters to be 2.857 Å and 2.972 Å for $a$ and $c$ respectively, the volumetric $\varepsilon^T$ was calculated to
be 3.72 pct. In the existing literature, approximately 4 pct volume expansion has been reported for the austenite to martensite transformation in steels [5.20, 5.21].

Notably, the DQ condition was austenitized at 1000 °C for 100s and had a negligible amount of undissolved cementite, while the RTDQ condition was austenitized at 875 °C for 30 mins and had a greater amount of undissolved cementite. The associated differences in the austenite and martensite carbon content between the two different austenitization conditions may be a source of error in the transformation strain calculation.

5.3.4 Transformation Zone Half-Width

EBSD scans were performed along the crack in the RTDQ sample to determine the distribution of RA in the vicinity of the fatigue crack. Three different regions at crack lengths of 2 mm, 4 mm, and 6 mm corresponding to $\Delta K$ values of 4.8 MPa·m$^{0.5}$, 5.2 MPa·m$^{0.5}$, 5.75 MPa·m$^{0.5}$ were scanned as described in Section 4.2.6. A MATLAB code was developed to identify the location of the crack using the SEM image (Figure 4.14 (a)) and then quantify the fraction of RA, martensite, and unindexed points (confidence index < 0.1) using the phase map (Figure 4.14 (c)) as a function of distance from the crack in microns.

To determine the value of $w$ using the RA phase fraction versus distance from the crack data, the transformed zone was assumed to be the region near the crack where RA fraction was less than 2 pct (arbitrarily chosen threshold). The value of 2 vol pct, corresponded to regions of the EBSD map where relatively large RA constituents were present. Additionally, the fine martensite and RA regions with high dislocation density had low pattern quality (more than 50 pct pixels were unindexed). It was possible that there was additional RA to martensite transformation beyond the estimated half-width, $w$; however, RA constituents were the minority phase, and due to the spatial limitations of the EBSD mapping, the distribution of the martensite transformation in the narrow transformation zone was not recovered. The sensitivity of the transformation toughening model on variations in $w$ was also examined.

Three values of $w$ corresponding to $\Delta K$ values of 4.8 MPa·m$^{0.5}$, 5.2 MPa·m$^{0.5}$, 5.75 MPa·m$^{0.5}$ were extracted from the EBSD scans. The value of $K_{\text{Trans}}$ can be calculated using $w$. Assuming that the
transformation is shear activated, and that the value of $K_{\text{max}}$ governs the extent of martensite transformation, $w$ can be described as a function of $K_{\text{max}}$. In LEFM, the stress field near the crack tip is proportional to $K/\sqrt{r}$, where $r$ is the distance from the crack tip [5.15]. Thus, for a given critical shear stress for transformation, $w$ is proportional to $K_{\text{max}}^2$. The general expression for $w$ was given by the expression

$$w = A(\Delta K_{\text{max}})^2$$

(5.10)

where $A$ is an experimentally determined fitting parameter. Using the expression for $w$, the values of $K_{\text{Trans}}$ and subsequently $K_{\text{eff}}$ were calculated for the RTDQ microstructure to help interpret the influence of DIMT on FCG behavior.

### 5.4 Results and Discussion

The results of transformation zone size and reduction in stress intensity due to DIMT in the vicinity of the crack tip are presented in Section 5.4.1 and Section 5.4.2 respectively. Two different transformation toughening models were developed with variations in martensite distribution within the transformation zone: (i) UTM, and (ii) LTM. The models were implemented on the FCG data for the LN2 and RTDQ conditions (Chapter 4). Section 5.4.3 discusses the UTM and LTM predictions, and Section 5.4.4 presents an analysis of the sensitivity of the two models to experimental parameters such as amount of martensite transformation and transformation zone half width.

#### 5.4.1 Transformation Zone Half-Width

EBSD scans were performed at varying crack lengths to determine the distribution of RA near the crack tip. Figure 5.2 (a) shows the SEM detector image from the EBSD scan taken at 4 mm crack length, corresponding to the $\Delta K$ values of 5.2 MPa·m$^{0.5}$ for the RTDQ sample. The black region at the bottom represented the crack, and the direction of crack growth is from left to right in the SEM detector map. Approximately 35 µm x 45 µm area on one side of the fatigue crack was scanned. Figure 5.2 (b) shows the corresponding phase distribution map where red pixels represented plate-martensite and the green pixels represented RA constituents. To better visualize the distribution of RA near the crack tip, only the
austenite pixels with confidence index lower than 0.1 were omitted from the map. It was observed that the RA constituents (green pixels) were fewer and finer near the fatigue crack as compared to the regions approximately 20 µm away from the fatigue crack. Figure 5.2 (c) shows the phase map where all the pixels with confidence index less than 0.1 were omitted (unindexed points). More than 50 pct of the pixels had confidence index values below 0.1. Fine and deformed sub-micron martensite and RA constituents presumably resulted in low quality Kikuchi patterns.

To determine the values of $w$ from the EBSD maps, trends in fraction of phases (pixels) were plotted as a function of distance from the crack tip. Figure 5.3 shows the relative fraction of unindexed, martensite and RA pixels as a function of the distance from the crack tip. The white arrow in Figure 5.2 (c) indicates the direction of positive x-axis in Figure 5.3. More than 50 pct of the pixels were unindexed. In the 3 µm narrow region near the crack, the number of unindexed pixels rapidly increased. The fatigue crack was approximately 5 µm wide and the sample was tilted at 70º during EBSD mapping. It is possible that the electrons were not able to backscatter from the crack region, and thus poor pattern quality was observed. Alternatively, due to significant plastic deformation near the crack (estimated plastic zone size $= 4.5$ µm), the pattern quality was poor. The green trend shows the fraction of RA pixels. Up to approximately 9 µm distance from the fatigue crack, a negligible fraction of RA constituents was observed. At a distance greater than 9 µm, significant amounts of RA fraction (large RA constituents) were found. Thus, using the threshold value of 2 vol pct RA, the transformation zone half-width $w$ was determined to be 8.8 µm for $\Delta K = 5.2$ MPa·m$^{0.5}$ ($K_{max} = 5.8$ MPa·m$^{0.5}$).
Figure 5.2. EBSD maps of a region near the fatigue crack in RTDQ sample at a distance of 4 mm from the notch tip. (a) SEM detector image, (b) phase map where austenite pixels with CI less than 0.1 are omitted, and (c) phase map showing data with CI greater than 0.1. The red regions correspond to plate-martensite while green regions represent RA constituents. (See electronic copy for color image)

The green trend in Figure 5.3 corresponding to RA fraction captures the amount of transformation in the vicinity of the crack. Negligible RA fraction was observed until 9 µm distance from the crack. For the distance range between 9 µm and 20 µm, a small increase in RA content (significant noise) was observed as a function of distance from the crack surface. The EBSD technique generally underestimates the RA phase fractions [5.22]. Thus, it was possible that some fine RA constituent within the 9 µm zone went undetected, which would mean that the UTM potentially over-predicts the extent of transformation.

Similarly, it was possible that RA to martensite transformation outside the 9 µm zone was not captured due to the noise in the data, which meant that the LTM potentially under-predicts the extent of transformation.
Figure 5.3 Fraction of phases as a function of the distance from fatigue crack in the RTDQ sample at $\Delta K = 5.2$ MPa·m$^{0.5}$ ($K_{\text{max}} = 5.8$ MPa·m$^{0.5}$). Data was extracted from Figure 5.2 (c). (See electronic copy for color image)

Similarly, transformation zone half-widths for crack lengths of 2 mm and 6 mm were also determined using respective EBSD maps. Three such scans were analyzed for each value of $\Delta K$. Figure 5.4 (a) shows the trend in transformation zone half-width as a function of $\Delta K$ at the crack tip. Error bars represent the standard deviation in the $w$ values determined from three different EBSD scans from the same region. The average values of $w$ varied between 9 and 11 µm for all the conditions. There was a small increase in $w$ with increasing values of $\Delta K$.

Figure 5.4 (b) shows the $w$ versus $K_{\text{max}}$ data. A general expression for the $w$ as a function of $K_{\text{max}}$ was derived by fitting Equation 5.10 through the data presented in Figure 5.4 (b) (dotted line). The data did not follow the expected functionality and a poor fit was obtained. The dotted line shows the fitted equation, given by the following

$$w = 0.2876(\Delta K_{\text{max}})^2$$  \hspace{1cm} (5.11)
Figure 5.4 Determination of transformation zone half-width, w. (a) w versus $\Delta K$ data, and (b) w versus $K_{\text{max}}$ data. The dotted line represents Equation 5.11.

5.4.2 Stress Intensity due to Transformation

The Mei and Morris model (Equation 5.7) was implemented on high-carbon plate-martensite austenite microstructures. Table 5.2 presents a summary of the parameters used in the model.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Notation</th>
<th>Methodology</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Young’s Modulus</td>
<td>$E$</td>
<td>Literature [5.13]</td>
<td>203 GPa</td>
</tr>
<tr>
<td>Poisson’s Ratio</td>
<td>$\nu$</td>
<td>Literature [5.14]</td>
<td>0.277</td>
</tr>
<tr>
<td>Volume Fraction of Transformed Martensite</td>
<td>$V_f$</td>
<td>XRD [5.23]</td>
<td>8.2 pct</td>
</tr>
<tr>
<td>Volume Expansion Strain</td>
<td>$\varepsilon^T$</td>
<td>XRD Lattice Parameters</td>
<td>0.0372</td>
</tr>
<tr>
<td>Transformation Zone Half-Width</td>
<td>$w$</td>
<td>EBSD/Image Processing</td>
<td>$w = 0.2876 K_{\text{max}}^2$</td>
</tr>
</tbody>
</table>
For the UTM, it was assumed that 8.2 vol pct RA transformed to martensite in the transformation zone. The reduction in stress intensity at the crack tip due to uniform transformation model ($K_{U Trans}^{\text{Trans}}$) was given by the expression

$$K_{U Trans}^{\text{Trans}} = \frac{0.33(EV_f e^T \sqrt{w})}{(1 - \nu)}$$  \hspace{1cm} (5.12)

For the LTM, it was assumed that the RTDQ sample had a linear gradient of amount of transformed RA from 8.2 vol pct at the crack surface to 0 vol pct at the edge of the transformation zone, across the half-width of the transformation zone. The reduction in stress intensity at the crack tip due to linear transformation model ($K_{L Trans}^{\text{Trans}}$) was given by the expression [5.19]

$$K_{L Trans}^{\text{Trans}} = \frac{0.22(EV_f e^T \sqrt{w})}{(1 - \nu)}$$  \hspace{1cm} (5.13)

Using the values of model parameters from Table 5.2 in Equations 5.12 and 5.13, transformation toughening due to UTM and LTM was calculated for a range of applied $\Delta K$ values from 4.5 MPa·m$^{0.5}$ to 6 MPa·m$^{0.5}$. Figure 5.5 (a) shows the values of $K_{\text{max}}$ (black open circle), $K_{\text{min}}$ (black closed circles), $K_{U Trans}^{\text{Trans}}$ (blue squares), and $K_{L Trans}^{\text{Trans}}$ (red squares) as a function of applied $\Delta K$. The values of $K_{L Trans}^{\text{Trans}}$ are approximately 1 pct greater than the values of $K_{\text{min}}$ and hence, the data practically overlap. Thus, for the proposed values of transformation zone half-width (Equation 5.11), negligible toughening was observed for the linear transformation model. The linear transformation model will be further revisited in the sensitivity analysis section (Section 5.4.4).

The values of $K_{U Trans}^{\text{Trans}}$ were approximately 50 pct greater than $K_{\text{min}}$, which meant that according to the UTM, the crack tip would experience a reduction in alternating stress intensity due to transformation toughening. The effective $\Delta K_{eff}$, calculated using Equation 5.8 is plotted against the applied $\Delta K_{app}$ in Figure 5.5 (b). The FCG results for the RTDQ and LN2 samples are rationalized in the context of the transformation toughening model in the next section.
Figure 5.5 (a) Applied maximum and minimum stress intensity factors, along with the magnitude of UTM and LTM predicted reduction in stress intensity at the crack tip (transformation toughening, $K_{\text{Trans}}$) (b) a comparison between the applied $\Delta K$ and effective $\Delta K$ accounting for the reduction in stress intensity at the crack tip predicted via UTM.

### 5.4.3 Uniform and Linear Transformation Models

To rationalize the influence of RA on FCG rates between the LN2 (9.4 vol pct RA) and RTDQ sample (17.6 vol pct RA) conditions (Figure 4.11 (b)), the UTM (Equation 5.12) and LTM (Equation 5.13) models were implemented on the experimental $\Delta K$ data. It was assumed that for the LN2 condition, negligible RA to martensite transformation occurred near the fatigue crack, and thus the effective $\Delta K_{\text{eff}}$ was the same as the applied $\Delta K_{\text{app}}$. However, for the RTDQ condition, the excess RA content of approximately 8.2 vol pct (as compared to the LN2 condition), was available to transform to martensite within the transformation zone. Thus, for the RTDQ condition, $\Delta K_{\text{eff}}$ was equal to the difference between the applied $\Delta K_{\text{app}}$ and the model calculated transformation induced stress intensity factor $K_{\text{Trans}}^{U}$ (UTM) or $K_{\text{Trans}}^{L}$ (LTM).

Figure 5.6 (a) shows the experimental FCG rates ($da/dN$) versus the applied $\Delta K_{\text{app}}$ data for the LN2 and RTDQ conditions (same as Figure 4.11 (b)). FCG rates were approximately 1.5 times greater for
the LN2 condition. The improvement in FCG resistance of the RTDQ condition was attributed to the RA to martensite transformation induced compressive residual stress at the crack tip (Section 4.4.3).

Figure 5.6 (b) shows the FCG rates as a function of the effective $\Delta K_{\text{eff}}$ (adjusted for transformation toughening in the RTDQ) for the LN2 condition, and the UTM and LTM model predictions. Additionally, the RTDQ experimental FCG data as a function of $\Delta K_{\text{app}}$ is also included. The experimental RTDQ FCG data practically overlaps with the LTM model predictions. The transformation toughening models shifted the experimental RTDQ FCG rates data to the left (reduced the value of $\Delta K_{\text{eff}}$) by a magnitude equal to $K_{\text{U,Trans}}^{U}$ in the case of the UTM prediction and $K_{\text{L,Trans}}^{L}$ in the case of LTM prediction. The LTM model predicted negligible toughening for the model parameters used. In Figure 5.6 (b) it is observed that the UTM predicted FCG rates were greater than the experimentally observed FCG rates (LN2 condition), while the LTM predicted FCG rates are lower than the experimental data. Predicted FCG behavior meant that UTM over-predicted while LTM under-predicted the reduction in stress intensity at the crack tip due to transformation toughening.

Both UTM and LTM are based on simplistic approximations of the martensite transformation distribution near the crack tip. According to LEFM, the local stress field near the crack tip decreases as a function of the distance from crack tip by a $r^{-1/2}$ dependence. In reality, based on the relationship between the applied local stress and the extent of martensite transformation a more complex martensite distribution may be present within the transformation zone (also indicated by Mei and Morris [5.2]). Furthermore, the magnitude of transformation toughening has an $r^{-3/2}$ dependence, which means that martensite transformation near the crack has a stronger influence on toughening than martensite transformation at farther away from the crack. Improved characterization of transformation gradients within the transformation zone can help with better predictions of FCG behavior in high-carbon plate-martensite austenite microstructures. EBSD mapping, which was used in this study, has spatial limitations in terms of scan area sizes within reasonable amounts of time.
Figure 5.6 (a) Experimentally determined FCG rates for the LN2 and RTDQ condition (Chapter 4), (b) FCG rates, predicted using UTM and LTM (modified RTDQ data), along with the experimental LN2 and RTDQ results. Negligible transformation toughening was assumed for thus, experimental and model predicted LN2 FCG data was the same.

5.4.4 Model Sensitivity Analysis

The sensitivity of the UTM and LTM to model parameters $V_f$ (volume of transformed martensite) and $w$ (transformation zone half-width) was analyzed. Since UTM over-predicted the extent of transformation toughening, the response of UTM for lower values of $V_f$ and $w$ was calculated. Conversely, since, LTM under-predicted the extent of transformation toughening, LTM predictions for greater values of $V_f$ and $w$ were calculated.

Figure 5.7 (a) show the FCG results of UTM with three different values of $V_f$, 8.2 vol pct, 7 vol pct, and 6 vol pct. A good agreement was observed between experimental FCG (LN2 condition) and UTM results for $V_f$ value of 0.07, while UTM under-predicted the extent of transformation toughening for $V_f$ value of 0.06. Upon changing the amount of transformed martensite by 2 vol pct, from 8.2 vol pct to 6 pct, the UTM predicted FCG rates increased by up to a factor of 2.

Figure 5.7 (b) shows the predicted FCG rates of LTM for three values of $V_f$, 8.2 vol pct, 10 vol pct, and 12 vol pct. A good agreement between experimental FCG rates (LN2) and LTM predictions was
observed for $V_f$ value of 10 vol pct, and the LTM over-predicted the toughening for $V_f$ value of 12 vol pct. By changing the amount of transformed martensite by approximately 4 vol pct, the LTM predicted FCG rates increased by up to 2 times. As compared to LTM, UTM was more sensitive to the changes in amount of martensite transformation, $V_f$.

The LN2 microstructure was developed by quenching the RTDQ microstructure in liquid N$_2$. The RA content of 9.1 vol pct in the LN2 condition was thermally stable at -196 °C (temperature of liquid N$_2$). However, the mechanical stability of RA in the crack tip stress field of the RTDQ microstructure might be different from the thermal stability at liquid N2 temperature. Thus, it is possible that the amount of transformation due to the fatigue crack was less than the difference between initial RA content of the RTDQ and LN2 microstructures, potentially supporting the better UTM prediction with a small volume fraction of martensite transformation. On the other hand, the best fit value of $V_f$ for the LTM was 10 vol pct, which is unrealistic since it is greater than the initial difference between the RA content in the RTDQ and LN2 condition.

The sensitivity of UTM and LTM to transformation zone width was also analyzed. The value of $w$ was varied by changing the fitting parameter $A$ in Equation 5.10. Three different values of $A$, 0.2876, 0.25 and 0.2, resulted in the range of values for $w$ to be 9.1 µm – 10.9 µm, 6.7 µm – 9.5 µm, and 5.3 µm – 7.6 µm, respectively. For this sensitivity analysis, the value of $V_f$ was fixed at 8.2 vol pct. Figure 5.8 (a) shows the FCG results of UTM with varying values of $w$. The legend in the plot includes the average value of the calculated $w$. The model with an average transformation zone half-width of 6.4 µm showed the best agreement between the experimental results and the model predictions.
Figure 5.7 Sensitivity of UTM and LTM to changes in amount of transformed martensite Vf. (a) UTM model predictions and (b) LTM model predictions for varying values of Vf.

Figure 5.8 (b) shows LTM model predictions for different average values of $w$ – 9.9 µm, 11.3 µm, and 14.5 µm. The LTM prediction for an average transformation zone width of 14.5 µm resulted in the best agreement between experimental and model calculated FCG rates. The difference between the sensitivity of UTM and LTM to values of $w$ was insignificant.

The best fitting value of $w$ of 6.4 µm for UTM was unrealistic since the RA to martensite transformation was experimentally observed at greater distances from the crack. It is however possible that small amounts of RA might have transformed to martensite outside the estimated transformation zone size, which was not captured due to the spatial limitations of the EBSD technique. The trends in RA phase fraction in Figure 5.3 show a slight increase in RA content between 10 µm and 20 µm. Thus, the best fitting value of average $w$ for the LTM seems realistic.
Figure 5.8 Sensitivity of UTM and LTM to changes in amount of transformation zone half-width, w. (a) UTM model predictions and (b) LTM model predictions for varying values of w.

The value of the Paris-law exponent \(m\) for the FCG results was approximately between 12 and 15 (Section 4.4.3), much higher than ductile metallic materials (approximately between 2 and 4) due to static modes of failure during fatigue like intergranular fracture [5.24]. High values of \(m\) meant that the FCG rates strongly depended on the value of \(\Delta K\), such that a small difference in the value \(K_{U_{\text{Trans}}}\) or \(K_{L_{\text{Trans}}}\) would dramatically influence the model predicted FCG behavior. Transformation toughening resulting from DIMT can thus be an important mechanism for improving FCG resistance of these high-carbon plate-martensite austenite microstructures. The UTM and LTM were less sensitive to changes in \(w\) as compared to the changes in \(V_f\), potentially due to the linear dependence of \(K_{U_{\text{Trans}}}\) on \(V_f\), as opposed to a square root dependence on \(w\). Overall, LTM was less sensitive to model parameters than UTM. Improved characterization of RA content in the narrow region near the fatigue cracks is necessary for better predictions using the transformation toughening models presented in this study.

The improvement in FCG resistance for the RTDQ microstructure was attributed to the reduction in stress intensity at the crack tip due to the RA to martensite transformation near the fatigue crack. The model predictions support the proposed hypothesis (Section 4.4.3) that the presence of RA suppressed IG
fracture by lowering the $K_{\text{max}}$ at the crack tip (driving force for IG fracture). However, it was observed that the SQ microstructure with a lesser amount of RA to martensite transformation in the transformation content showed greater improvements in FCG resistance. Thus, it is possible that a mechanism such as micro-crack coalescence or the brittle nature of transformed martensite (Section 4.4.3) may lower the net improvement in FCG resistance for the RTDQ microstructure as compared to SQ microstructures. The LN2T microstructure with a lower initial RA content than the RTDQ condition had a greater FCG resistance, suggesting that other mechanisms pertaining to plate-martensite and undissolved cementite might play a role in the observed FCG behavior of high-carbon plate-martensite austenite microstructures.

5.5 References


CHAPTER 6

SUMMARY AND CONCLUSIONS

The goal of this PhD work was to study the influence of microstructural features on the fatigue crack growth (FCG) resistance of high-carbon plate martensite austenite microstructures for potential application in carburized and bearing steels. Using commercial high-carbon 52100 steel (1 wt pct carbon), microstructures consisting of plate martensite, RA and undissolved cementite were developed, fatigue tested and characterized. The following research questions were answered:

- How do thermal processing strategies, specifically, thermal cycling and step-quenching, influence the refinement of prior austenite grains (PAG), plate martensite and RA?
- How do fatigue cracks interact with the surrounding high-carbon plate martensite austenite microstructure?
- How does the transformation of RA to martensite in the vicinity of fatigue crack affect FCG rates in high-carbon plate martensite austenite?

The three research questions were individually addressed in Chapters 3, 4, and 5 and the key findings are summarized in sections 6.1, 6.2, and 6.3, respectively.

6.1 Microstructure Refinement Strategies for High-Carbon Plate Martensite Austenite Microstructures

Microstructure development during thermal cycling and step-quenching of commercial high-carbon 52100 steel was investigated with the following highlights and conclusions:

- A thermal cycling heat treatment is an effective approach for prior austenite grain size (PAGS) control. During reheating above the $A_{cm}$ temperature, low temperatures and short isothermal holdings resulted in the greatest grain size refinement.
- Multiple reheating cycles did not substantially reduce the cementite size distribution and PAGS further. However, a narrower PAGS distribution was obtained, potentially resulting in a reduced number of large intergranular crack nucleation sites in fatigue.
• The average martensite plate radius and plate thickness increase with an increase in PAGS. The growth of martensite and its morphology are limited by physical obstacles such as grain/phase boundary interfaces.

• Stereological measurements on RA constituents showed that finer PAGS leads to finer RA constituents. Since the mechanical stability of austenite depends on its size, grain refinement can potentially lead to a more stable RA in carburized and bearing steels.

• Interrupted quenching below the $M_s$ temperature results in austenite stabilization (mechanical and/or thermal). Based on literature, it is suggested that martensite formation in the primary quench step plastically deforms and strengthens the remaining austenite, thereby impeding plastic accommodation of subsequent transformation. During isothermal holding below $M_s$, carbon atoms migrate to glissile martensite interfaces, thereby pinning them and increasing the chemical free energy driving force for subsequent phase transformation.

• Stereological analysis showed that step-quenching led to refinement in martensite plate sizes. Neutron diffraction data provided evidence of greater compressive residual strains in step-quenched austenite and lower tetragonality in step-quenched martensite, suggesting carbon redistribution during the SQ process.

6.2 Interaction between High-Carbon Plate Martensite Austenite Microstructures and Fatigue Cracks

52100 steel sub-size CT samples were austenitized, quenched and tempered to develop high-carbon plate martensite austenite microstructures with varying amounts and stability of RA. Four different microstructure conditions were generated: RTDQ, LN2, LN2T and SQ with RA contents of 17.6, 9.4, 7.1, and 19.3 vol pct respectively, and hardness values of 820, 903, 831, and 812 HV, respectively. A bimodal distribution of undissolved cementite, pro-eutectoid cementite along PAG boundaries, and fine spheroidal cementite within the grains was present. The FCG behavior and the interaction between the fatigue crack and microstructures were investigated with the following highlights and conclusions:
• RTDQ and SQ samples showed a bilinear behavior in the FCG rates versus $\Delta K$ data, potentially associated with the transition from $\Delta K$ – controlled to $K_{\text{max}}$ – controlled FCG. FCG rates were highest for the LN2 condition, intermediate for the RTDQ condition, and lowest for the LN2T and SQ conditions. Higher initial amounts of RA, mechanical stability of RA, or greater degree of martensite tempering improved FCG resistance.

• High values of the Paris law exponent, $m$, in the range of 12 to 24 were observed for the FCG data due to static modes of failure such as IG fracture during fatigue owing to the high susceptibility of 52100 steel to quench embrittlement.

• The crack path exhibited a mixed intergranular (IG) and transgranular (TG) morphology. Stress-assisted RA to martensite transformation was observed in the vicinity of the fatigue cracks in the RTDQ and SQ microstructures.

• The overload regions of the fracture surfaces exhibited mixed IG fracture and micro-void coalescence (MVC) for all samples. The fatigue fracture regions exhibited a combination of IG fracture, mixed-ductile brittle (MDB) and transgranular (TG – cleavage, quasi-cleavage and ductile striations) fracture.

• The IG fracture mode had the least FCG resistance while the MDB failure mode had greatest FCG resistance. The FCG resistance depended on the relative fractions of the different fracture modes. Greater amounts of TG (RTDQ and SQ) and MDB fracture (LN2T and SQ) resulted in improved FCG resistance.

• The presence of higher initial RA content (RTDQ and SQ) suppressed brittle IG fracture and promoted TG fracture. Two mechanisms were proposed to rationalize the role of RA in promoting TG fracture in the fatigue regions of the RTDQ and SQ samples:
  - Transformation Toughening Mechanism: The RA to martensite transformation in the vicinity of the fatigue crack imposes compressive residual stress at the crack tip, thereby
lowering the $K_{max}$ at the crack tip, which is interpreted to be the driving force for IG fracture.

- Martensite Induced Crack Propagation: Due to crack tip stress fields, micro-cracks form at the interfaces of fresh untempered martensite. These micro-cracks coalesce with the primary fatigue crack resulting in TG fracture during fatigue.

### 6.3 Fatigue Life Prediction Model for High-Carbon Plate Martensite Austenite Microstructures

The Mei and Morris transformation toughening model was implemented on high-carbon plate martensite austenite microstructures to predict the influence of RA to martensite transformation on FCG behavior. The highlights and conclusions are presented below:

- The transformation toughening model calculated the reduction in stress intensity at the crack tip resulting from the volume expansion associated with the stress-assisted RA to martensite transformation in the vicinity of the fatigue crack tip.

- Experimentally determined parameters including the transformation strain, $\varepsilon^T$ (using neutron diffraction), amount of martensite transformation, $V_f$ (XRD), and width of transformation zone, $w$ (EBSD) were used to inform the model prediction.

- Two different models were considered: a uniform transformation model (UTM) and a linear transformation model (LTM) assuming different martensite transformation distributions. The UTM over-predicted while the LTM under-predicted the influence of transformation on FCG rates, suggesting that a more complex martensite transformation distribution might be present in the transformation zone near the fatigue crack.

- The UTM and LTM were more sensitive to variations in $V_f$ than to variations in $w$. The UTM was more sensitive to experimentally determined parameters than the LTM.

- The FCG rates predicted by the models were highly sensitive to small differences in $\Delta K$ at the crack tip, which were induced by the variations in austenite volume fraction and stability. Thus,
transformation toughening may be an important microstructure design strategy for improved FCG resistance in high-carbon plate martensite austenite microstructures.
CHAPTER 7
FUTURE WORK

This chapter presents recommendations for future research directions that can help better understand some of the observations in this thesis, validate proposed mechanisms and improve fatigue crack growth (FCG) resistance of high-carbon plate martensite austenite microstructures.

Interrupted quenching below the $M_s$ temperature (step quenching) resulted in the refinement of plate-martensite and more-stable RA constituents. However, the nature of austenite stabilization (mechanical or thermal) was not clear. Using in-situ neutron diffraction or high-energy x-ray diffraction (HEXRD) techniques, the evolution of austenite and martensite lattice parameters and phase fractions during and after the isothermal holding can be investigated to quantitatively study the time and temperature dependence of microstructural phenomena like carbon re-distribution, development of residual stresses and the differences in martensite transformation kinetics. Further, the SQ microstructure exhibited better FCG resistance than RTDQ, which was attributed to the local improvement in toughness of the microstructure due to the presence of stable RA. Interrupted or in-situ measurement of the evolution of RA volume fraction during monotonic and cyclic deformation can help quantify the differences in martensite transformation behavior and the resulting mechanical performance of the RTDQ and SQ microstructures.

High-carbon plate martensite austenite microstructures developed from the high-carbon 52100 steel were susceptible to quench embrittlement and exhibited extensive intergranular (IG) fracture along prior austenite grain boundaries (PAGBs) during fatigue. A network of undissolved pro-eutectoid cementite particles along the PAGBs strongly affected the fracture modes during mode I fatigue testing. The case regions of carburized components often contain less than 1 wt pct carbon. By altering the starting microstructure, lowering the carbon content of the alloy, or varying the austenitization temperature, the distribution of carbides can be modified and FCG resistance can be improved. A
systematic study of variations in carbide distribution, amount of RA and stability of RA is needed to optimize the fatigue performance of high-carbon plate martensite austenite microstructures.

Presence of higher initial RA content (RTDQ and SQ) suppressed IG fracture and promoted transgranular (TG) fracture during fatigue crack propagation in high-carbon plate martensite austenite microstructures. It was proposed that the deformation induced martensite transformation (DIMT) in the vicinity of the crack imposed compressive residual stress at the crack tip, thereby lowering the driving force for static failure and fatigue crack propagation. HEXRD scans along the length of the fatigue cracks can quantify the local micro-residual stresses, the amount of DIMT near the fatigue crack and provide the evidence of transformation induced compressive residual stresses at the crack tip. The differences between DIMT behavior of RTDQ and SQ samples owing to the differences in mechanical stability can be evaluated using the HEXRD scans.

Transformation toughening models were developed to predict fatigue lives of high-carbon plate martensite austenite microstructures. The model-predicted FCG rates were highly sensitive to the amount and distribution of austenite to martensite transformation near the fatigue crack. An improved characterization of martensite transformation gradients, via TEM or HEXRD will help increase the accuracy of the transformation toughening models. Further, by modifying the composition of RA, the transformation strain (volume expansion) and amount of martensite transformation can be tailored to maximize DIMT and the reduction in stress intensity at the crack tip, and thereby improve the FCG resistance of high-carbon plate martensite austenite microstructures.

The LN2T microstructure with lower amounts of RA showed better FCG resistance as compared to the RTDQ microstructure. Other competing mechanisms affecting the FCG resistance of high-carbon plate martensite austenite microstructures such as micro-crack coalescence, crack path deflection, and transformation-induced energy absorption were not explored in detail in this thesis and require further investigation.
APPENDIX A

NEUTRON DIFFRACTION MEASUREMENTS AND ANALYSIS

Neutron diffraction is a powerful technique that is used to determine atomic and magnetic structures of materials. In the field of materials science, neutron diffraction has been employed to study phase transformations, micromechanics, residual stresses, crystallographic texture, and kinetics of microstructural evolution. Neutron diffraction is better suited for bulk samples due to higher penetration depths as opposed to surface analysis using x-ray diffraction.

The instruments at the Lujan Neutron Scattering Center at the Los Alamos National Lab (LANL) receive a pulsed neutron beam from a tungsten spallation source. The neutron beam is moderated using cold water (H\textsubscript{2}O) to ensure the wavelength is in the range of atomic distances in engineering materials. Time-of-flight neutron diffraction is used to study stress and strains in bulk material in a thermo-mechanical environment using instruments such as Spectrometer for Materials Research at Temperature and Stress (SMARTS).

Figure A.1 shows the schematic diagram of SMARTS instrument. The instrument consists of a parallel beam of pulsed neutrons, the cross-section of which can be adjusted using incident collimator slits (smallest cross-section used was 1 mm x 2 mm). The sample is placed at an angle of 45° with respect to the incident beam while the detectors are located at an angle of ±90° to the neutron beam. The two detectors are located at a distance of 1.5 m from the sample; each consist of 196 He\textsuperscript{3} (isotope of Helium) tubes that subtend an angle of 20° both vertically and horizontally. The sample is mounted on a stage that allows independent rotation and translation with a precision of 1 µm thereby allowing one to perform neutron diffraction scans. The translation and rotation stage can be equipped with a tensile load frame (capacity - 250 kN) and a furnace that can reach 1500 °C to perform in-situ neutron diffraction measurements. The two different detectors provide information about stress/strains in the axial/radial, normal/transverse, or in-plane/parallel directions of the sample based on the mounting configuration.
To investigate the RH100 and SQ110 microstructures, neutron diffraction measurements were recorded using the SMARTS instrument. Figure A.2 (a) shows a schematic diagram of the top view of the SMARTS setup. The cylindrical sample was mounted at an angle of 45° to the incident neutron beam. The diffraction pattern was recorded in two stationary detector banks, located at an angle of ± 90° with respect to the incident beam. Furthermore, the incident beam was collimated using incident slits, resulting in a 2 mm x 2 mm cross-section of the beam, while radial collimators were placed between the sample and the detector to collimate the diffracted beam, resulting in a 8 mm³ probed volume. The diffraction patterns were recorded from the center of the heat-treated samples for 1.5 hr each. Data analysis of the diffraction patterns was performed using GSAS (general structure analysis system) software. Average values of interatomic spacing and phase weight fractions were determined using Rietveld refinement in GSAS.
Figure A. 2 A schematic diagram of SMARTS neutron diffraction measurement setup.
APPENDIX B

EVOLUTION OF PAGS DURING MULTIPLE REHEATING CYCLES

52100 steel samples were inductively heated to an elevated temperature, isothermally held in vacuum ($10^{-5}$ bar pressure) and subsequently quenched using argon gas in a TA instruments Type 805L quenching dilatometer. Thermal cycling was also employed to produce microstructural refinement. Samples were heated to 1000 °C at a heating rate of 5 °C·s$^{-1}$ and isothermally held for 100 s under vacuum to dissolve all pre-existing carbides. The samples were argon gas quenched to 40 °C with a cooling rate of 20 °C·s$^{-1}$ to ensure plate-martensite and RA microstructure, similar to the case of carburized components. This thermal cycle was repeated up to 3 times to obtain single, double and triple reheated specimens. Table B.1 summarizes the PAGS for Direct Quench, Single Reheat, Double Reheat and Triple Reheat conditions, and Figure B.1 shows the optical micrographs of the four conditions etched with saturated picric acid to reveal the prior austenite grain boundaries.

Table B.1 – Evolution of PAGS during Multiple Reheating Cycles

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Prior Austenite Grain Size (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Direct Quenched</td>
<td>38.8</td>
</tr>
<tr>
<td>Single Reheat</td>
<td>31.1</td>
</tr>
<tr>
<td>Double Reheat</td>
<td>29.9</td>
</tr>
<tr>
<td>Triple Reheat</td>
<td>28.4</td>
</tr>
</tbody>
</table>

A single reheating cycle resulted in approximately 20 pct reduction in the PAGS. Multiple thermal cycles did not significantly refine the PAGS further. Figure B.1 (b) for the single-reheating microstructure shows some very large and some very small PAG, while in the triple reheat microstructure (Figure B.1 (d)), the large PAG were eliminated. A comparison between the size distribution of PAGs suggested that multiple reheating cycles lead to a narrower PAGS distribution in the microstructure.
Figure B. Light optical micrographs of transverse sections of 52100 steel dilatometry cycles etched with picric acid. (a) Austenitized at 1000 °C for 100 s and direct quenched, (b) single reheated, (c) double reheated, and (d) triple reheated with the same thermal cycle.
APPENDIX C

INFLUENCE OF HOLD TEMPERATURE AND TIME ON MICROSTRUCTURE DEVELOPMENT DURING STEP QUENCHING

Cylindrical samples of 52100 steel samples were heated to 1000 °C at a heating rate of 5 °C·s⁻¹ and isothermally held for 100 s under vacuum and then argon gas quenched to 200 °C with a cooling rate of 20 °C·s⁻¹. Below 200 °C in the martensite formation regime, the sample was allowed to cool without active heating or gas cooling. For step-quenched microstructures, the sample was isothermally held at 100 °C for 10 s, 100 s, or 1000 s and then subsequently allowed to cool to room temperature in the ambient dilatometer atmosphere. Figure C.1 (a) shows the martensite transformation segment data for the change in length versus temperature for the isothermal holding at 100 °C for 100 s. After the isothermal hold, a drop in length of the sample was observed for approximately 8 °C cooling, suggesting that the austenite did not transform (stabilization of austenite). Figure C.1 (b) shows the change in length versus temperature data for three different isothermal hold time, 10 s, 100 s, and 1000 s. A greater extent of austenite stabilization (shift in secondary $M_s$) was observed for longer isothermal holds.

![Figure C.1](image)

(a) Change in length v/s temperature dilatometry data for 100 °C and 100 s step quench. (b) Effect of isothermal holding time on austenite stabilization during step quenching.
Heat treatments with varying step-quench temperatures and the same isothermal hold time of 1000 s were performed on cylindrical samples. The extent of stabilization was quantitatively determined as the difference between isothermal holding temperature and the secondary $M_s$ temperature.

Figure C.2 (a) shows the trends in extent of austenite stabilization as a function of step-quench temperature for isothermal hold time of 1000 s. Greater stabilization of austenite was observed for lower step quench temperatures (greater amounts of primary martensite). Figure C.2 (b) shows the change in length of the sample during the isothermal hold at various temperatures. For all the conditions, the length of the sample first increases and then decreases. Besides temperature homogenization within the sample, the increase in length of the sample can be attributed to decomposition of austenite while the decrease in length results from precipitation of carbides or carbon clustering within martensite. Variations in the amount of increase and the subsequent decrease in lengths of the samples were observed for different step-quenching temperatures.

Figure C. 2 (a) Trends in the extent of austenite stabilization as a function of step-quench temperature (constant isothermal hold 1000 s). (b) Change in length of the sample during isothermal holding at different step-quench temperatures.
APPENDIX D

LOW TEMPERATURE TEMPERING OF AUTENITIZED AND QUENCHED 52100 STEEL

Cylindrical samples of 52100 steel samples were subjected to controlled heat treatments in a TA Instruments 805L quenching dilatometer. The samples were heated to 875 °C at a heating rate of 5 °C·s⁻¹ and isothermally held for 30 min under vacuum and then argon gas quenched to 200 °C with a cooling rate of 20 °C·s⁻¹. Below 200 °C in the martensite formation regime, the sample was quenched to 40°C with a cooling rate of 2 °C·s⁻¹. The austenitized and quenched samples were then subjected to tempering treatments at 100 °C, 175 °C, and 240 °C for 1 h each. Figure D.1 (a) schematically shows the thermal history of low temperature tempered sample. Figure D.1 (b) shows the change in length versus time data for the three tempering conditions. During the isothermal hold, the length of sample increased for the 240 °C tempering condition, owing to the partial decomposition of austenite. For the other two samples tempered at 100 °C and 175 °C, the length of the sample decreased due to precipitation of carbides and carbon clustering. Decomposition of austenite was not observed at 100 °C and 175 °C.

Figure D. 1 (a) A schematic plot of low temperature tempering heat-treatments. (b) Change in length of the sample during isothermal holding at different tempering temperatures.
APPENDIX E

TRANSGRANULAR FATIGUE FRACTURES IN HIGH-CARBON PLATE-MARTENSITE AUSTENITE MICROSTRUCTURES

The fatigue fracture surfaces in all the four microstructures, RTDQ, LN2, LN2T, and SQ (Chapter 4) exhibited intergranular (IG) fracture, transgranular (TG) fracture and mixed ductile-brittle (MDB) fracture features. The transgranular features potentially showed presence of cleavage, quasi-cleavage, and ductile striations fracture morphologies.

Figures E.1 (a-b), (c-d), and (e-f) show high-magnification SEM fractographs from the transgranular fatigue fracture regions in the RTDQ, LN2, and LN2T microstructures respectively. All the fractographs were recorded at 4 mm crack length, corresponding to a $\Delta K$ value of 5.2 MPa·m$^{0.5}$. 
Figure E.1 Transgranular fatigue fracture features in the (a) and (b) RTDQ sample, (c) and (d) LN2 sample, and (e) and (f) LN2T sample. All images taken at crack length of 4 mm from the notch edge, corresponding to $\Delta K = 5.2 \text{ MPa}\cdot\text{m}^{0.5}$. 
APPENDIX F
SUPPLEMENTAL FILES

The Chapter 3 in the thesis document is adapted from the journal article - M. Agnani, O. L. DeNonno, K. O. Findley, and S. W. Thompson, “Quantitative Analysis of Microstructural Refinement in Simulated Carburized Microstructures,” *Journal of Materials Engineering and Performance*, vol. 29, no. 6, pp. 3551–3559, 2020.” The permission to use the content of the article from the publisher Springer Nature and the co-author not on the thesis committee may be found in the supplemental file Ch3 JMEP Self Co Author.pdf.

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