Static Strain Aging in Low Carbon Ferrite-Pearlite Steel: Forward and Reverse Loading

by

Mojtaba Mansouri Arani

B.Sc., University of Tehran, 2012

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF
THE REQUIREMENTS FOR THE DEGREE OF

MASTER OF APPLIED SCIENCE

in

THE FACULTY OF GRADUATE AND POSTDOCTORAL STUDIES
(Materials Engineering)

The University of British Columbia
(Vancouver)

December 2015

© Mojtaba Mansouri Arani, 2015
ABSTRACT

The combination of static strain aging and plastic strain reversal is important to understand for both the forming of components and also analysis of in service performance, for example, in the case of fabrication of pipeline, motor shafts or structural components in buildings and ships.

Static strain aging phenomenon has been experimentally studied for the cases of forward and reverse re-straining after aging on a low carbon steel (0.16 wt% C) with a ferrite-pearlite microstructure. Torsion tests on hollow tubular samples were used for the mechanical tests. The shear strain on the surface of the sample was measured with the digital image correlation. The influence of the amount of pre-strain, aging time and temperature, and the strain path reversals on the stress-strain response after aging has been measured experimentally. A maximum increase of 46 MPa was obtained in the yield stress of the samples re-strained after full aging in the same direction as the initial straining. This maximum increase in yield stress as well as the rate of increment in yield strength during aging was almost independent of the amount of pre-strain and the increase in the flow stress occurred without a significant variation in the work hardening behavior. Further, it was shown that a yield point phenomenon was absent if the direction of re-straining after aging was reversed and the increase in the flow stress level after aging was proportional to the amount of pre-strain and increased with extended aging time. In this case, the absence of a sharp yield point after prolonged aging time led to the speculation that the activation of dislocations sources, rather than unpinning of locked dislocations in re-straining after aging was the controlling mechanisms although proof of this requires further investigation.
Abstract

Although it is difficult to unambiguously identify all of the underlying physical mechanisms, nevertheless, a comprehensive set of experimental results has been measured which can be used by the design engineer when considering cases where static strain ageing and strain path reversals are relevant for a ferrite-pearlite steel with 0.16 wt% carbon.
All the experimental work presented in this thesis has been carried out by the author in the Department of Materials Engineering at the University of British Columbia between September 2013 and May 2015. This included all the experimental design, material heat treatment and mechanical testing. Cited figures appearing in Chapter 2 are used with permission from applicable sources.
# Table of Contents

Abstract ................................................................................................................................. ii

Preface .................................................................................................................................... iv

Table of Contents ................................................................................................................ v

List of Tables ........................................................................................................................ viii

List of Figures ....................................................................................................................... ix

List of Symbols ...................................................................................................................... xv

List of Abbreviations ............................................................................................................ xviii

Acknowledgments .................................................................................................................. xix

Dedication .............................................................................................................................. xx

1. Introduction....................................................................................................................... 1

2. Literature Review .............................................................................................................. 3

   2.1 Interaction between interstitials and dislocations in iron .............................................. 3

   2.2 Diffusion of interstitials to dislocations (formation of Cottrell atmospheres) ........... 5

   2.3 The origin of the yield point ....................................................................................... 6

   2.4 Yield point elongation ............................................................................................... 9
# Table of Contents

2.5 Static strain aging after pre-strain ................................................................. 10

2.6 Experimental observations ................................................................................ 10

2.7 The Bauschinger effect ...................................................................................... 13

2.8 Directionality of strain aging .............................................................................. 15

2.8.1 Effect of grain size ......................................................................................... 18

2.9 Summary ............................................................................................................. 21

3. Scope and Objectives ............................................................................................ 22

4. Experimental Methodology .................................................................................... 24

4.1 Material ............................................................................................................. 24

4.2 Metallography .................................................................................................. 25

4.3 Torsion testing .................................................................................................. 27

4.3.1 Sample dimensions and mechanical testing .................................................. 27

4.3.2 Strain measurement ...................................................................................... 29

4.4 Aging procedure ............................................................................................... 35

5. Results .................................................................................................................. 36

5.1 Effect of sample geometry ................................................................................ 36

5.2 Basics of strain measurement using DIC ......................................................... 37

5.3 Yield stress measurement .................................................................................. 39

5.3.1 Yield stress for loading in forward direction .............................................. 40
Table of Contents

5.3.2 Yield stress in reloading in reverse direction .................................................. 43

5.4 Reproducibility of monotonic torsion test .......................................................... 44

5.4.1 Initial straining .................................................................................................. 44

5.4.2 Reloading in forward direction after strain aging ......................................... 48

5.4.3 Reverse loading after strain aging ................................................................. 49

5.5 Strain aging ......................................................................................................... 50

5.5.1 Yield behavior in forward straining after aging ............................................ 51

5.5.2 Yield behavior in strain reversal after aging ............................................... 54

6. Discussion ............................................................................................................. 60

6.1 Increase of the yield stress in forward re-straining after aging ......................... 60

6.2 Permanent strengthening in forward re-straining .............................................. 61

6.3 Interaction between the Bauschinger effect and strain aging ......................... 65

6.4 Effect of aging time and temperature on the flow stress after strain reversal .... 69

6.5 Summary ............................................................................................................ 73

7. Conclusions .......................................................................................................... 74

7.1 Future work ......................................................................................................... 75

Bibliography ............................................................................................................. 77
LIST OF TABLES

Table 4.1: Chemical composition of the 1020-JIS SN490 material (wt%). ........................................... 24

Table 5.1: Measured offset yield stresses of four samples reloaded in forward direction
        following 5% pre-strain and aging at 100°C for 10 hours...................................................... 48

Table 5.2: Measured offset yield stresses of five samples reloaded in reverse direction,
        following 5% pre-strain and aging at 100°C for 10 hours...................................................... 50
LIST OF FIGURES

Figure 2.1: Stress-strain curve for a low-carbon steel showing strain aging. Region A, original material strained through yield point. Region B, immediately retested after reaching point X. Region C, reappearance and increase in yield point after aging at low temperature. [9].................................................................................................................. 8

Figure 2.2: Diagrammatic representation of a row of solute atoms lying in the position of maximum binding at an edge dislocation. An applied shear stress will cause the dislocation to separate from the solute atoms by gliding in the slip plane to position x. [10].................................................................................................................. 8

Figure 2.3: Effect of grain size and aging time on changes in tensile properties due to strain-ageing in low carbon rimmed steel pre-strained 4% and held at 200°C; grain size (µm): (a) ~160; (b) ~80; (c) ~26. Strain-hardening coefficient n is given by σ = kε^n, where σ and ε are true stress and true strain, respectively. [17]............................................. 12

Figure 2.4: Shear stress–shear strain curves of two specimens of rimming steel. One curve with uninterrupted straining; the other was interrupted 4 times and continued in the same direction after aging. Yield points and the level of the curve after yielding rise with increasing strain. [8]................................................................. 12

Figure 2.5: Diagram showing the Bauschinger effect, in case of high carbon spheroidised steel. [21]................................................................................................................................. 13

Figure 2.6: Forward and reverse simple shear test after ~12% of equivalent pre-strain performed on IF steels with 3.5 and 12 µm grain sizes. [26]..................................................... 14

Figure 2.7: Directionality of yield point observed in two X100 steels taken directly from an as fabricated UOE pipe [44]. The tensile test performed parallel to the circumferential direction (Pipe A Circ and Pipe B Circ) exhibits yield points while samples tested parallel to the longitudinal direction exhibited continuous yielding. 16
Figure 2.8: Shear stress-shear strain curves of two specimens of rimming steel. One curve related to the sample strained to the point of buckling without interruption and the other was interrupted multiple times and continued in either the same direction as the original direction (full line) or the reverse direction (dashed line) after aging. [8] ........................................................................................................................................18

Figure 2.9: Effects of grain size after aging at 89°C on torsional stress-strain curves for a low carbon steel containing 0.11 wt% carbon tested in reverse restraining. (a) ~26 µm, and (b) ~98 µm. [46] ........................................................................................................................................19

Figure 2.10: Lightly strained area close to Lüders front in a test piece unloaded before aging, some dislocations appear to have moved back from the grain boundary wall. [46] . 20

Figure 4.1: Microstructure of the as-received steel plate..................................................................................25

Figure 4.2: Measured microhardness as a function of distance from the surface.......................26

Figure 4.3: Drawing of the tubular torsion sample with inner diameter of a) 7.1 mm, and b) 12.7 mm. ........................................................................................................................................28

Figure 4.4: Camera configuration for the DIC setup. The spread angle between the two cameras is maintained at 50 degrees in order to capture radial dilation and curvature of the sample.........................................................................................................................................................30

Figure 4.5: Method used by Davis8 software to calculate the strains from a speckle pattern, a) before deformation, and b) after deformation.................................................................31

Figure 4.6: Calculated shear strain over, a) interrogation surface of the sample, and b) a vertical line crossing the gauge section. .........................................................................................33

Figure 4.7: Elastic portion of a typical stress strain curve measured on steel grade JIS SN490. The strain was measured using the DIC set-up. The equation of the line was determined using a least squares fit in Microsoft excel.........................................................34
Figure 5.1: Comparison of the stress-strain curves related to samples with two different inner diameters, but the same wall thickness................................................................. 37

Figure 5.2: Shear strain distribution map in the gauge section of the samples strained initially to a) ~0.8%, b) 5%, and c) 10% equivalent strain. ................................................................. 38

Figure 5.3: Stress-strain curve of two samples reloaded in the same (green) and reverse (red) direction of the initial strain. Samples had a pre-strain of 5% and aged at 100°C... 40

Figure 5.4: An example of (a) lines with various offsets crossed a reloading curve, and (b) the measured yield stresses base on various offsets in three samples reloaded in forward direction after pre-strain of 5% and aging at 100ºC for 600 minutes. ........... 41

Figure 5.5: a) Loading curves of the as-received steel and 0.2% offset line. b) The magnified section shows the cross sections of the 0.2% offset line and the stress-strain curves in yield region................................................................. 42

Figure 5.6: (a) Defined $\Delta \sigma$ as the difference between stress-strain curve of a sample reloaded after aging in reverse direction and the base line, and (b) Plotted $\Delta \sigma$ as a function of strain in the same range of total equivalent strain. ............................................ 44

Figure 5.7: Stress-strain curves of sample pre-deformed to a, b) ~0.8%, c, d) 5%, and e, f) 10% equivalent strain. b, d, and f) are the magnified section of the stress-strain curves in the yield region........................................................................ 45

Figure 5.8: Histogram of the measured a) 0.2% offset yield stress, and the flow stress at b) ~0.8%, c) 5%, and d) 10% equivalent strain; as-received samples. ....................... 46

Figure 5.9: Stress-strain curves of four samples reloaded in forward direction, following 5% pre-strain and aging at 100ºC for 10 hours......................................................... 48

Figure 5.10: Stress-strain curve related to five samples reloaded in reverse direction, following 5% pre-strain and aging at 100ºC for 10 hours........................................... 49
Figure 5.11: Von Mises stress-strain curves of the samples pre-strained to a, b)~0.8%, c, d) 5%, and e, f) 10% equivalent strain, and re-strained in the same direction as the pre-strain after aging at 100°C for 15-600 minutes. The plotted magnified section (b, d, and f) of the yield region on the right, gives a close vision of this region. Open circles represent aging treatment upon unloading. ................................................................. 52

Figure 5.12: The effect of aging after various equivalent strains; tests were made in the same direction as the pre-strain. Aging times of a) 0-180 minutes, and b) 180-600 minutes. ................................................................................................................................................. 53

Figure 5.13: a) The effect of increasing aging temperature on the Von Mises stress-strain curves of the samples 5% pre-strained and re-strained in the forward direction after aging for 600 minutes. The plot of the yield region on the right (b), gives an expanded view of this region. The open circle represents aging treatment upon unloading. ................................................................................................................................................. 53

Figure 5.14: Von Mises stress-strain curves of samples pre-strained to a, b)~0.8%, c, d) 5%, and e, f) 10% equivalent strain, and re-strained in the reverse direction after aging at 100°C for 15-600 minutes. The plot of the yield region on the right (b, d, and f), gives an expanded view of this region. Open circles represent aging treatment upon unloading. ................................................................................................................................................. 55

Figure 5.15: Defined $\Delta YS_{Rev}$ as the difference between the offset yield stress of a sample reloaded in reverse direction after aging and that of the sample reloaded in the reverse direction without aging. ................................................................................................................................................. 56

Figure 5.16: The effect of aging and equivalent pre-strain on measured $\Delta YS_{Rev}$ based on using a) 0.05%, b) 0.1%, c) 0.2%, and d) 0.5% offset in samples reloaded in the reverse direction after ~0.8-10% equivalent pre-strain and aging at 100°C for up to 600 minutes ................................................................................................................................................. 57

Figure 5.17: a) The effect of increasing aging temperature on the Von Mises stress-strain curves of the samples 5% pre-strained and re-strained in the reverse direction after
aging for 600 minutes. b) The plotted magnified section of the yield region gives a closer vision. Open circle represents aging treatment upon unloading. .......................... 58

Figure 5.18: The effect of aging temperature on measured $\Delta Y_{SRev}$ based on various offsets in the samples reloaded in reverse direction after 5% equivalent pre-strain and aged for 600 minutes. ............................................................... 59

Figure 6.1: Plotted $\Delta \sigma_{forward}$ as a function of equivalent strain related to the samples a) $\sim$0.8%, b) 5%, and c) 10% pre-strained and re-strained in the same direction as the original pre-strain after aging at 100ºC for 15 and 600 minutes ....................... 62

Figure 6.2: Plotted $\Delta \sigma_{forward}$ as a function of equivalent strain related to the samples pre-strained up to 10% and re-strained in the same direction as the original pre-strain after aging at 100ºC for a) 15 and b) 600 minutes................................................................. 64

Figure 6.3: Plotted yield stresses of the samples re-strained immediately in the reverse direction after pre-strain of $\sim$0.8%, 5%, and 10% as a function of the applied offset. .................................................................................................................. 65

Figure 6.4: The plot of measured BEF in the samples re-strained immediately in the reverse direction after pre-strain of $\sim$0.8%, 5%, and 10% as a function of the applied offset. The extracted data from the study of Bouaziz et al. [26] on IF steel with grain size of 12 µm and 3.5 µm, after $\sim$12% equivalent pre-strain, and Li et al. [22] on AISI 1020 steel containing 0.21% carbon with grain size of 12 µm after 2% pre-strain are also plotted as a function of the applied offset....................... 66

Figure 6.5: The measured $\Delta Y_{SFwd}$ and $\Delta Y_{SRev}$ (0.2% offset) after 10% pre-strain and aging at 100ºC for up to 600 minutes................................................................. 68

Figure 6.6: The results of forward re-straining after 10% pre-strain and aging at 100ºC for up to 600 minutes. Plotted in accordance with Equation 2.5................................. 69
Figure 6.7: Plotted $\Delta \sigma_{\text{reverse}}$ as a function of equivalent strain related to the samples a) ~0.8%, b) 5%, and c) 10% pre-strained and re-strained in the reverse direction after aging at 100°C for 15 and 600 minutes. ........................................................................................................ 71

Figure 6.8: Plotted $\Delta \sigma_{\text{reverse}}$ as a function of equivalent strain. Samples restrained in the reverse direction after pre-strain of ~0.8%, 5%, and 10%. a) Without aging and b) aged at 100°C for 600 minutes before restraining. .............................................................................. 72

Figure 6.9: Plotted $\Delta \sigma_{\text{reverse}}$ as a function of equivalent strain for the samples restrained in the reverse direction after pre-strain of 5% and aging at 100°C, 150°C, and 200°C for 600 minutes before restraining. ........................................................................................................ 73
LIST OF SYMBOLS

$U$: the interaction energy between a dislocation and the solute atoms.

$r$: the distance from the core of the dislocation.

$r_o$: the minimum distance from the core of the dislocation where linear elasticity theory is applicable.

$\theta$: the polar coordinate for the positions of the solute atom relative to the core of the dislocation.

$A$: a parameter that depends on the elastic constants of the matrix and the shape change caused by the solute atom.

$c$: the equilibrium concentration of solute atoms in an “atmosphere” around each dislocation.

$c_o$: the bulk concentration of solute atoms.

$k$: Boltzmann’s constant.

$T$: the temperature in K.

$\lambda$: the atomic diameter of an iron atom.

$N_o$: the total number of carbon atoms per unit length of the dislocation required to form an atmosphere of one atom per atom plane.

$D$: the diffusion coefficient of carbon or nitrogen.

$n_0$: the initial concentration of carbon or nitrogen in solution.

$\rho_0$: the initial dislocation density.
\( \dot{\varepsilon} \): strain rate.

\( b \): the magnitude of Burgers vector.

\( \mu \): the shear modulus.

\( \rho_m \): the mobile dislocation density.

\( \bar{v} \): the average velocity of the dislocations.

\( \tau \): the applied shear stress resolved in the slip plane.

\( \tau_0 \): the shear stress for \( \bar{v} = 1 \text{m/s} \).

\( \sigma_f \): the maximum flow stress in the original pre-strain.

\( \sigma_r \): the measured offset stress in the reverse direction.

\( \beta_\sigma \): Bauschinger stress parameter.

\( \sigma_y \): the initial lower yield strength.

\( K_y \): a parameter that depends on local stress that must be developed at the yield front in order to nucleate mobile dislocation in adjacent unyielded grains.

\( d \): the average grain diameter.

\( \sigma_p \): the local stress required to unpin fully-locked dislocations.

\( l_p \): the distance from the tip of the slip band.

\( R \): the distance from the center of the tubular sample.

\( \Phi \): the angle of twist in torsion test.

\( \dot{\phi} \): the angular velocity in torsion test.
List of Symbols

\( l \): the gauge length of torsion sample.

\( \gamma \): shear strain.

\( M \): the measured torque in torsion test.

\( \sigma_{eq} \): the von Mises equivalent stress.

\( \varepsilon_{eq} \): the von Mises equivalent strain.

\( \Delta Y S_{\text{Fwd}} \): the increase of yield stress in forward re-straining after aging.

\( \Delta Y S_{\text{Rev}} \): the increase of yield stress in reverse straining after aging.

\( \Delta \sigma_{\text{forward}} \): the difference between the flow stresses of the aged sample and as-received sample at the same total equivalent strain.

\( \Delta \sigma_{\text{reverse}} \): the difference between the flow stresses in strain reversal after aging and monotonic loading at the same total equivalent strain.
LIST OF ABBREVIATIONS

BCC: Body Center Cubic.

YPE: Yield Point Elongation.

UTS: Ultimate Tensile Strength.

BEF: Bauschinger Effect Factor.

EQAD: Equivalent Average Diameter.

DIC: Digital Image Correlation.
ACKNOWLEDGMENTS

I wish to thank my supervisor Dr. Warren Poole for his time, encouragement, support, patience and enthusiasm during the course of my master over the past couple of years. I would further like to express my gratitude to Dr. Chad Sinclair and Dr. David Embury for the discussions about my project.

I can't leave this section without expressing my gratitude to Mr. Ross Mcleod, Mr. Carl Ng and Mr. Dave Torok of the machineshop for their wonderful job on fabricating my torsion samples. I also like to thank Mr. Wonsang Kim and Dr. Quentin Puydt for their precious help during early days of this project.

It is also a pleasure to acknowledge the TransCanada Pipeline Ltd. and Natural Science and Engineering Research Council of Canada (NSERC) for their generous financial supports.

My acknowledgments would not be complete without thanking all members of Microstructural Engineering Group, specially my officemates in AMPEL 349 and FF 201. I am also thankful to all my dear friends who made my stay in Vancouver pleasant. My sincere thanks to Pouria for the last minute proofread of this thesis.

Finally, I wish to express my deepest gratitude to my parents for their love and unconditional supports in my entire life.
TO MY PARENTS.
Chapter 1

INTRODUCTION

The phenomenon of strain aging in low carbon steels is of significant interest for industry due to its relevance to a wide range of applications for structural steels. The yield strength is an important factor for mechanical design and the assessment of material performance. For instance, the process of fabricating a ship hull, building structures and bent anchor bolts involves a set of thermomechanical steps including plastic deformation by bending as well as thermal processing by welding, and coating. A consequence of these combined processing steps is that the material’s properties vary significantly depending on the strain path and thermal history. This is important in particular when the in service loading is in a different direction than the deformation during manufacturing. Therefore, understanding the phenomena, which results in complex mechanical behavior, will help the designers better assess the final performance of the product.

The term “static strain aging” refers to the phenomenon where solute atoms, such as carbon and nitrogen atoms in iron, migrate to the dislocations and lock them, which results in the reappearance of the upper yield point and the Lüders plateau. Strain aging may result in significant increase in the yield strength when the material is reloaded in the same direction as the original deformation. However, changing the strain path after aging, i.e. first deformation in one direction and then the second deformation in the reverse direction (known as the Bauschinger effect), results in lower yield stress than the maximum flow stress in forward pre-strain. The effect of static strain aging on tension followed by holding and then reloading in tension has been widely studied in literature. On the other hand, the effect of the aging treatment
when strain path is altered has been far less studied. This anisotropy in the mechanical properties and dependency on the thermomechanical history of the material, provides designers with a challenge to predict the material behavior during and after the installation of the components.

In this work, the effect of the amount of pre-deformation, aging time and temperature, and strain path (fully reversed or reloaded in the same direction) on the yield strength and yielding behavior of a low carbon steel has been investigated. The studied material was JIS SN 490 steel, containing 0.16wt% carbon, with ferrite pearlite microstructure. The existence of the second phase (pearlite) allowed us to investigate the influence of the second phase while still avoiding microstructural complexities related to bainitic or martensitic dual phase steels, which are common in pipeline and automotive industries.

The torsion test was used in this study to evaluate the mechanical properties. The torsion test has an advantage compared to other mechanical property testing methods such as tension or compression since it is not limited by geometrical instabilities associated such as buckling in compression and necking in tension. For the above reasons it was decided to develop a procedure for conducting torsion tests on hollow tubular samples. In this case, tensile and compressive instabilities can be avoided, allowing the straining of the material to the point of fracture, and the unambiguous measurement of the Bauschinger and strain aging effect in strain reversal tests can be made.

In the following chapters, the current state of knowledge will be reviewed with the knowledge gaps identified. Based on this, the scope and objectives for the current study will be articulated. Then the methodology and results presented followed by a discussion of the current results and finally a short summary of the work and its implications will be presented as well as some suggestions for future work.
Chapter 2

LITERATURE REVIEW

This chapter reviews the basic knowledge relevant to the current study starting with the concept of static strain aging, its kinetics and current thoughts on the origin of the upper yield point in low carbon steels. Finally, strain path reversal (the Bauschinger effect) in combination with static strain aging will be reviewed.

2.1 Interaction between interstitials and dislocations in iron

The first scientific theory for static strain aging was proposed by Cottrell in 1949 [1]. He proposed that, it was the migration of solute atoms (interstitials in steels) to dislocations which pins them and results in a sharp yield point upon re-loading [2]. The attraction of smaller solute atoms to dislocations results from the compressive strain field around an interstitial solute atom (such as carbon and nitrogen) which can relieve some of the tensile strain field below the half plane which forms an edge dislocation. Moreover, in BCC iron, carbon and nitrogen interstitials occupy positions at the mid points of the edges of BCC cell (i.e. the octahedral sites). The resulting stress-strain field associated with these atoms has a shear component along with the hydrostatic field. Thus, these solute atoms are able to interact with the shear and hydrostatic stress field of dislocations, i.e. the interaction is almost equally strong with edge and screw dislocations [2].
If one considers that solute atoms are initially distributed at random positions around a dislocation at the nominal solute concentration, then the interaction energy between a dislocation and the solute atoms is given by [2]:

\[ U = \frac{A \sin \theta}{r} \]  \hspace{1cm} (2.1)

where \( r \) and \( \theta \) are the polar coordinates for the positions of the solute atom relative to the core of the dislocation, and \( A \) is a parameter that depends on the elastic constants of the matrix and the shape change caused by the solute atom (note: Equation 2.1 is not valid at the center of the core of the dislocation since linear elasticity theory is not applicable in this region). The energetically most favorable position for carbon and nitrogen atoms in BCC iron is expected to be at the position \( \theta = \frac{3\pi}{2} \) and \( r = r_0 \approx 2 \times 10^{-10} \), \( r_0 \) is the minimum distance from the core of the dislocation where linear elasticity theory is applicable. The maximum binding energy in this case is estimated to be \( \approx 1 \text{ eV} \) [2]. However, internal friction studies measure the binding energy to be \( \approx 0.45 \text{ eV} \) [2, 3]. This difference is reasonable since the binding energy obtained using linear elasticity is almost certainly too high.

The equilibrium concentration of solute atoms in an “atmosphere” around each dislocation is given by [2]:

\[ c = c_0 \exp\left(\frac{U}{kT}\right) \]  \hspace{1cm} (2.2)

where \( c_0 \) is the bulk concentration, \( k \) is Boltzmann’s constant, and \( T \) is the temperature in K. However, the interaction energy between carbon or nitrogen atoms and a dislocation is so strong that \( U_{\text{max}} \gg kT \) at room temperature, i.e. atmosphere becomes saturated. In this condition, the atmosphere condenses into a line of carbon and nitrogen lying parallel to the dislocation line and gets situated at the position of maximum binding [2].
2.2 Diffusion of interstitials to dislocations (formation of Cottrell atmospheres)

The migration of carbon or nitrogen atoms to the core of a dislocation can be considered as follows. According to Cottrell and Bilby [1], the number of solute atoms which arrive within time, $t$, in a unit volume at the core of the dislocation is:

$$N(t) = 2n_0 \left( \frac{\pi}{2} \right)^{\frac{1}{3}} \left( \frac{AD}{kT} \right)^{\frac{2}{3}} \int_0^t t^{-\frac{1}{3}} dt = 3n_0 \left( \frac{\pi}{2} \right)^{\frac{1}{3}} \left( \frac{ADt}{kT} \right)^{\frac{2}{3}}$$

(2.3)

therefore,

$$\frac{N(t)}{N_s} = 3n_0 \lambda \left( \frac{\pi}{2} \right)^{\frac{1}{3}} \left( \frac{ADt}{kT} \right)^{\frac{2}{3}}$$

(2.4)

where $\lambda$ is the atomic diameter of an iron atom, $N_s$ is the total number of carbon atoms per unit length of the dislocation required to form an atmosphere of one atom per atom plane (and this is equal to $1/\lambda$), $D$ is the diffusion coefficient of carbon or nitrogen, and $n_0$ is the initial concentration of carbon or nitrogen in solution. More recently, De Cooman et al. [4] considered the effect of saturation of atmospheres on the kinetics of strain aging by considering that carbon atoms which arrive at dislocation during the early stages of aging are more effective in pinning dislocations than those arriving later. Based on this consideration, Equation 2.5 was modified to give a better prediction of the static strain aging results, especially in the later stages of aging [4], i.e.

$$\frac{N(t)}{N_s} = \frac{1 - \exp \left( 3(\rho_0 - n_0\lambda) \left( \frac{\pi}{2} \right)^{\frac{1}{3}} \left( \frac{ADt}{kT} \right)^{\frac{2}{3}} \right)}{1 - \frac{N_s}{n_0} \exp \left( 3(\rho_0 - n_0\lambda) \left( \frac{\pi}{2} \right)^{\frac{1}{3}} \left( \frac{ADt}{kT} \right)^{\frac{2}{3}} \right)}$$

(2.5)

where $\rho_0$ is the initial dislocation density.
2.3 The origin of the yield point

Low carbon steels typically show an upper yield point, followed by an undulating stress-strain curve known as yield point elongation as shown in Figure 2.1 [5]. The origin of the upper yield point has two basic explanations: i) at the upper yield point, dislocations break free from their solute atmospheres and there is a drop in the stress or, ii) new mobile dislocations are generated (possibly at grain boundaries) and multiplication of dislocations occurs rapidly resulting in a drop of the stress. The first explanation was given by Cottrell and Bilby [1] and in this theory, it is the strong interaction between dislocations and their solute atmospheres that pins the dislocations. Therefore, a higher stress level is required to break the locked dislocations free from their atmospheres than that to keep them in motion once they have been unpinned. The interaction between dislocations and the solute atmospheres has two regimes. At small stresses, the dislocations cannot escape from these atmospheres and solute atoms migrate with dislocations during flow. However, at sufficiently high stress, the dislocations are collectively unpinned from these atmospheres. Unpinning causes a sharp increase in the mobile dislocation density and elastic relaxation of the rest of the sample result in smaller stress required for constant strain rate [2, 6]. This process results in sharp upper yield point and subsequently lower yield point during a tensile test. Figure 2.2 illustrates a row of solute atoms lying in the position of maximum binding along the core of a straight edge dislocation. The derivation of the interaction energy of a dislocation displaced by \( x \) from a line of solute atoms shows that the maximum force is required to displace the dislocation by \( x = r_0/\sqrt{3} \) [1]. Having this, the theoretical (break-away) tensile stress required to cause this force is estimated to be \( \approx 6 \) GPa in iron [2], however, it should be noted that this value is calculated based on the variation of the interaction energy during the course of unpinning which is not well known.

Due to the very strong interaction of dislocations with their atmospheres, the possibility of alternative mechanisms other than unpinning of dislocations upon yielding have also been
considered [7, 8]. Leslie [7] has shown that an abrupt multiplication of mobile dislocations can also cause a yield drop, i.e. even without dislocation unlocking. In this mechanism, the influence of mobile dislocation density on the stress required to obtain a constant strain rate is considered. The strain rate in a crystal can be related to the motion of dislocations using the Orowan equation:

\[ \dot{\varepsilon} = b\rho_m \bar{v} \]  \hspace{1cm} (2.6)

where \( b \) is the magnitude of Burgers vector, \( \rho_m \) is the mobile dislocation density, and \( \bar{v} \) is the average velocity of the dislocations. If the initial mobile dislocation density is very low due to pinning of the dislocations with Cottrell atmospheres, then the average velocity of dislocations has to be very high to meet the macroscopic strain rate. The average velocity of dislocations is dependent upon stress according to:

\[ \bar{v} \approx \left( \frac{\tau}{\tau_0} \right)^m \]  \hspace{1cm} (2.7)

where \( \tau \) is the applied shear stress resolved in the slip plane, \( \tau_0 \) is the shear stress for \( \bar{v} = 1 \text{m/s} \) and \( m \) is a constant which varies for different materials (about 35 for edge dislocations in steel according to Leslie [7]). Therefore, a high stress is required to provide the high velocity of dislocations at the beginning of yielding but as soon as the yielding occurs, multiplication of mobile dislocations results in stress drop. Therefore, large yield drops can be achieved without recourse to the unlocking of pinned dislocations [7].
Figure 2.1: Stress-strain curve for a low-carbon steel showing strain aging. Region A, original material strained through yield point. Region B, immediately retested after reaching point X. Region C, reappearance and increase in yield point after aging at low temperature. [9]

Figure 2.2: Diagrammatic representation of a row of solute atoms lying in the position of maximum binding at an edge dislocation. An applied shear stress will cause the dislocation to separate from the solute atoms by gliding in the slip plane to position x. [10]
2.4 Yield point elongation

In steels where carbon or nitrogen are present as interstitials, the initial plastic response typically shows an undulating stress-strain curve (there may or may not be a distinguished upper yield point) known as the yield point elongation (YPE). Macroscopic observations on the surface of a steel sample show the formation of surface markings known as Lüders bands. These bands often nucleate in regions of stress concentration such as the shoulders of the specimen [9] and these bands divide the specimen into the sections where plastic deformation is occurring and to those which are still unyielded. These bands propagate through the rest of the gauge section during the yield point elongation with complete yielding of the sample occurring at the end of the yield point elongation. Based on Cottrell’s theory, the nucleation of a Lüders band is due to break-away of a group of dislocations upon yielding in regions with substantial concentration of stress, which help the unpinning at smaller applied stress compared to the theoretical break-away stress. It was argued that the nucleation of a Lüders band did not have its origin from the break-away of single dislocation since making a light scratch on the surface of the sample, which should cause many dislocations to be unpinned, does not remove the upper yield point. Further, it was argued that during the propagation of the Lüders band(s), the stress concentration at the edge of the band helps release anchored dislocations in front of the Lüders band.

In the mobile dislocation theory of yielding, the nucleation of a Lüders band occurs when a sufficient stress is reached to activate a dislocation source (generally at the grain boundary) and then propagates the dislocations through the grain. Due to the local decrease in cross section, the Lüders band front acts as stress concentration, such that yielding is propagated over the gauge length of the specimen at a virtually constant stress, i.e. over the strain for the Lüders elongation.
Studies on Lüders elongation in low carbon steels show that it increases in magnitude with a decrease in ferrite grain size [11], temperature [12], and carbon concentration [11, 13], and with an increase in strain rate [12, 13].

2.5 Static strain aging after pre-strain

Figure 2.1 schematically describes the effect of static strain aging on stress-strain response of low carbon steels. In this figure, the sample is first plastically strained to point X (region A). The specimen is afterward unloaded and reloaded without any appreciable delay or any heat treatment (region B). In this case, the upper yield point is absent upon reloading since there was insufficient time for Cottrell atmospheres of carbon and nitrogen atoms to form. The specimen after reaching to the point Y is unloaded for a second time and an aging treatment is applied, by holding at low aging temperature (20-150°C) and the result is the return of the upper yield point, lower yield point and the Lüders plateau [9]. Holding the sample has resulted in the reappearance of the yield point due to the pinning of dislocations by formation of Cottrell atmospheres at dislocations present at the end of the the pre-strain.

2.6 Experimental observations

Figure 2.3 is a summary of typical results from static strain aging experiments, in this case extracted from the work of Wilson and Russell [14]. Their study considered the changes in the tensile properties of a commercial low carbon steel (0.038 wt% carbon), after tensile pre-straining, aging at 60°C and re-straining in the same direction. They propose four stages of strain aging as indicated in this figure. During stage I, the yield stress and the Lüders strain increase while other properties remain constant. At this stage, it is proposed that atmospheres are forming on dislocations and by the completion of this stage solute atoms in atmospheres are adequate in
number to occupy all the dislocation sites, i.e. the dislocation atmosphere density would be between one and two atoms per atom plane and all dislocations are fully locked, which results in the re-establishment of yield stress and Lüders strain. Further, it is proposed that during the Lüders strain, dislocations are unpinned from these atmospheres and the atmospheres are dispersed, which results in no further effect on other properties. In stage II, the yield stress continues to increase while the Lüders strain remains constant. The permanent hardening along with a constant work hardening coefficient is due to pinned arrays of dislocations or the fine precipitates on dislocations [15–17]. A similar trend is observed in stage III except that the strain hardening rate increases and results in subsequent increase in UTS. Here, it is proposed that all the dislocation sites are fully occupied and further segregation of solute atoms to dislocations results in the development of some form of solute cluster or precipitation at dislocations [14]. Overageing in stage IV leads to softening, however, the Lüders strain maintains or even rises in fine-grained material. The rise in Lüders strain in this stage is speculated to be due to the gradual re-solution of precipitates on dislocations and segregation of this solute to grain boundaries, thus increasing the stress required to unpin sources [15, 18]. Finally it was observed that increasing temperature up to just below the recovery temperature may accelerate the progress of strain aging stages but the magnitude of changes is not altered significantly [15, 19].

The effect of static strain aging was further investigated in a study on tubular torsion samples by Elliot et al. [8]. In this work, torsion experiments were conducted on a rimming steel containing 0.017 wt% carbon (rimmed steel is a type of steel which no deoxidizing agent is added to it during casting which causes carbon monoxide to evolve rapidly from the ingot). As illustrated in Figure 2.4, one torsion test was conducted up to torsional buckling and the other sample was unloaded four times and reloaded in the same direction following aging at 120 °C for 1 h. The interrupted curve shows an increase in both lower yield point and flow stress after each aging process. The permanent increase in the flow stress is contrary to the initial theory of dislocation locking, where it is assumed that all the dislocations are unlocked upon yielding, i.e.
this would result in return of stress to the uninterrupted curve when yield point elongation ends. Therefore, Elliot speculated that in aged samples, yielding occurs by multiplication of only few unlocked dislocation segments and the dislocation network remains mostly locked.

![Figure 2.3](image1.png)

Figure 2.3: Effect of grain size and aging time on changes in tensile properties due to strain-ageing in low carbon rimmed steel pre-strained 4% and held at 200°C; grain size (µm): (a) ~160; (b) ~80; (c) ~26. Strain-hardening coefficient \( n \) is given by \( \sigma = k \varepsilon^n \), where \( \sigma \) and \( \varepsilon \) are true stress and true strain, respectively. [14]

![Figure 2.4](image2.png)

Figure 2.4: Shear stress–shear strain curves of two specimens of rimming steel. One curve with uninterrupted straining; the other was interrupted 4 times and continued in the same direction after aging. Yield points and the level of the curve after yielding rise with increasing strain. [8]
2.7 The Bauschinger effect

In 1881, Bauschinger [20] discovered that if one reverses the direction of loading, the yield stress in the reverse direction is lower than that at the end of straining in forward direction. Figure 2.5 illustrates the stress-strain curve of a sample pre-strained in tension, unloaded and immediately re-strained in compression. In this figure, the compressive straining has been plotted as positive values of strain for convenience. As shown in this figure, when a specimen is reloaded in reverse direction after pre-strain, the yielding stress is generally lower than a sample re-strained in the same direction as pre-strain, i.e. the Bauschinger effect.

![Figure 2.5: Diagram showing the Bauschinger effect, in case of high carbon spheroidised steel.][21]

Different methods have been proposed to characterize the Bauschinger effect such as: I) Bauschinger strain as the difference in strain required to reach a specific stress in forward and revere [9], II) Bauschinger effect factor (BEF) as, \( BEF = \sigma_f / \sigma_r \), where \( \sigma_f \) is the maximum flow stress in the original pre-strain and \( \sigma_r \) is the measured offset stress in the reverse direction [22], and III) Bauschinger stress parameter: \( \beta_{\sigma} = (\sigma_f - \sigma_r) / \sigma_f \) [23].
Chapter 2. Literature Review

It is found that the Bauschinger effect increases with increasing the amount of pre-strain and saturates after a certain limit [24, 25]. The results of a study by Bouaziz et al. [26] on an interstitial free (IF) steel (i.e. a single phase ferritic steel) is shown in Figure 2.6 for two different grain sizes. The extracted results from this figure shows a BEF of 0.87 after ~12% equivalent pre-strain (as characterized by the 0.2% offset for yielding in the reverse direction) and 0.7 for samples with grain size of 12 and 3.5 µm, respectively. In contrast, a BEF of 0.55 was reported for AISI 1020 steel (0.21 wt% carbon) with grain size of 14 µm after 2% pre-strain [22]. The much larger Bauschinger effect in the 1020 steel is associated with the presence of the hard Fe₃C phase in the pearlite.

![Figure 2.6: Forward and reverse simple shear test after ~12% of equivalent pre-strain performed on IF steels with 3.5 and 12 µm grain sizes. [26]](image)

There have been various efforts to clarify the origin of the Bauschinger effect [27]. Two principal Bauschinger effect theories have been proposed: back stress and Orowan theory [21, 28, 29]. In the case of back stress theory, there are two main groups of back stresses [30]; i) the
long range internal stresses resulting from the interaction between moving dislocations and different obstacles (e.g., precipitates, grain boundaries, and other dislocations) during forward straining. This interaction generates back stress, which repels the dislocations from the obstacles in the reverse straining [21, 23, 28, 31–34] and ii) short range effects, such as annihilation of the dislocations in the course of reverse straining and directionality of mobile dislocations in their resistance to motion [30–32]. Internal back stress levels are shown to escalate rapidly with pre-strain at low pre-strain levels based on estimation of back stress levels measured through X-ray diffraction analysis [35] and neutron diffraction [36]. The back-stresses rapidly drop in the forward direction upon strain reversal and reform in the opposite direction [36].

In contrast, the proposal of Orowan [9, 29], suggests that dislocations pile up at barriers in tangles during plastic deformation, and eventually form cells. During unloading, the dislocation lines do not move significantly, since the structure is mechanically stable. However, upon reverse loading, they can move a significant distance at a low shear stress, since the barrier to the reverse motion of the dislocations are presumably weaker than those in the original motion and dislocations in the rear are not as closely spaced as in the front.

2.8 Directionality of strain aging

As discussed previously (Section 2.5), if a low carbon steel is deformed beyond the Lüders strain, then aged and strained further, the yield point reappears when straining is in the same direction. However, if restraining applies in direction significantly different from the pre-strain (e.g. fully reversed or orthogonal), a sharp yield point returns very slowly, if at all [37, 38]. While the strain aging of steels have been extensively studied and many aspects of it have been
Chapter 2. Literature Review

explored experimentally and theoretically (e.g. [6, 39–43]), the directionality of strain aging response has received very little attention [6, 8, 44–47].

Early investigations of Tipper [37] on small compression specimens machined from an extended hot rolled steel plates after aging in the direction of the preceding extension, showed that the yield phenomenon was completely absent. Figure 2.7 illustrates another example, where sharp yield point is visible in stress-strain curves of the tensile samples taken from the hoop direction and tested in the hoop direction of a pipe formed by UOE process [48]. However, continuous yielding was observed in the samples machined parallel to the longitudinal direction (i.e. at 90° to hoop direction) of the pipe. This directionality is due to the hoop expansion of the UOE process, which leads to plastic tensile deformation in the hoop direction before aging (e.g. during epoxy coating or during use [49]).

Figure 2.7: Directionality of yield point observed in two X100 steels taken directly from an as fabricated UOE pipe [44]. The tensile test performed parallel to the circumferential direction (Pipe A Circ and Pipe B Circ) exhibits yield points while samples tested parallel to the longitudinal direction exhibited continuous yielding.
The internal (residual) stresses in the material are proposed to be the reason for the anisotropic strain aging effect described above [8, 47, 50]. Williams [47] showed that the sharp yield point reappears in compression samples after tensile pre-straining and adequate aging. It was shown in his results that the yield stress increases by increasing the amount of pre-strain; however, the highest obtained yield stress in strain reversal (after 4% pre-strain) was 10% lower than that of the as-received steel. It was concluded that, although strain aging did not completely eliminate the portion of the Bauschinger effect, it was quite effective in reducing its magnitude. Polakowski [51] proposed that the absence of the yield phenomenon is due to the macroscopic residual stresses. As a result, the yielding starts at the very thin layer where the residual stress in reverse direction is highest, and it would propagate to layers of lower residual stress [27, 52]. The yield drop is not observable since at any time only a very small volume of sample is yielded. This led to the conclusion that yield point is absent even when all dislocations are strongly locked and that the directional internal stresses from pre-strain help easy dislocation unpinning during transverse straining [52]. This logic later was criticized because unpinning force required was larger than the internal stresses and also that the same argument could be applied to straining in the same direction. Additionally, the residual stresses can be responsible for the absence of the upper yield point only, but not the lower yield point and the yield itself [37, 46].

Further Elliot et al. [8] found similar results in torsion tests on a rimming steel (0.017 wt% carbon). Their results show that the upper yield point and the lower yield point re-appears if the strain before aging and after aging are of the same sign. However, in case of strain path reversal, yield point and yield point elongation were not observed. Figure 2.8 shows the results, where the solid lines indicate reloading in the same direction as the initial straining and dashed curves exhibit the reversal of strain path.
2.8.1 Effect of grain size

The effect of grain size on directionality of strain aging was studied by Wilson and Ogram [46] on a low carbon steel containing 0.11wt% carbon. Figure 2.9 illustrates that the sharp yield point does not return for coarse grained specimen for reverse straining after aging at 89 °C for several minutes. However, in fine grain steel the sharp yield point appears after 200 minutes. Wilson described the effect of grain size on the directionality of strain aging with Hall-Petch relationship [53–55] as follows:

\[ \sigma_y = \sigma_i + K_y d^{-1/2} \]  

(2.8)

where \( \sigma_y \) is the initial lower yield strength and \( K_y \) depends on local stress that must be developed at the yield front in order to nucleate mobile dislocation in adjacent unyielded grains and \( d \) represents average grain diameter. When the stress concentration is due to the blocking of a slip band by a grain boundary and nucleation occurs by unpinning a dislocation at a distance \( l_p \) from the tip of the slip band, then:

\[ K_y \approx 2\sigma_p l_p \]  

(2.9)

where \( \sigma_p \) is the local stress required to unpin fully-locked dislocations.
The contribution of the residual stresses from pre-straining and Bauschinger effect on \( \sigma_y \) with the variation of \( \sigma_i \) (friction stress term) was discussed in Section 2.7 as short range effects but the influence of pre-strain on the unpinning process and \( K_y \) is to be discussed as follows. The flow strength in forward direction at the end of pre-strain can be written as:

\[
\sigma_f' = \sigma_f' + K_f' d^{-1/2}
\]  

(2.10)

Measurements by Armstrong et al. [56] shows that \( k_f' \) is considerably lower than that in initial yield strength \( (k_f' \approx 0.5k_y) \).

![Figure 2.9](image)

Figure 2.9: Effects of grain size after aging at 89°C on torsional stress-strain curves for a low carbon steel containing 0.11 wt% carbon tested in reverse restraining. (a) ~26 µm, and (b) ~98 µm. [46]

The parameter \( K_y \) in Equation 2.8 is directional due to the polarity of dislocations in their resistance to motion near grain boundary barriers. Dense dislocation walls have been observed near grain boundaries in electron microscope images after pre-straining (Figure 2.10). These
dislocations are of a sign that permits them to move easily during reverse straining and run back to the grain body. Therefore, $l_p^*$ in Equation 2.9 for reverse straining is smaller than $l_p^*$ in forward straining [18, 46]. However, Wilson [46] suggests that this is not enough to fully account for directionality of $K_y$ and as such the inhomogeneity in solute segregation should be considered, which affect $\sigma_p$. Since the ratio of available solute atoms to dislocation density in regions near grain boundary is lower than that in the grain body, the dissolved solute available locally may not be enough to pin all the dislocations and then solute atoms would need to migrate to grain boundaries from the body of the grain. This implies that in the early stages of strain aging, dislocations near grain boundary that can operate in reverse strain have lower $\sigma_p$ than those in the grain body. Based on this theory, Wilson believes that increasing the grain size, results in longer diffusion distances from grain interiors to grain boundaries and therefore longer aging time for sources operating in reverse straining.

![Figure 2.10: Lightly strained area close to Lüders front in a test piece unloaded before aging, some dislocations appear to have moved back from the grain boundary wall. [46]](image)
2.9 Summary

This literature review has highlighted some key points on static strain aging. The elastic interaction between interstitials and dislocations in iron is discussed as the driving force for the diffusion of these atoms to dislocations and pinning them. The origin of the upper yield point and yield point elongation has been discussed based on the theories of dislocation unpinning and the mobile dislocation theory of yielding. It was shown that Bauschinger effect in strain reversal results in decrease of the yield stress. The experimental observations presented herein, showed a directionality in strain aging in the course of forward and reverse straining, however the source of this directionality is still not well understood.
Chapter 3

SCOPE AND OBJECTIVES

The previous chapter showed that despite the fact that phenomenon of static strain aging being known for more than 50 years, this phenomenon is still not completely understood or fully characterized. Therefore, the primary objective of this research project is to investigate the effect of static strain aging on the mechanical behavior of a low carbon steel, in particular for the case of a strain path reversal. An experimental study has been conducted to characterize static strain aging in a relatively simple steel. Torsion tests using tubular samples were conducted on a ferrite/pearlite steel (carbon content of 0.16 wt%) to examine static strain aging for cases of reloading in the same direction as the original strain or for fully reversed loading. All tests were conducted at room temperature with pre-strains between 0.8 and 10%. Static strain aging temperatures of 100-200°C were examined.

Ultimately in order to achieve the primary objective, this work aims to achieve the following:

- Develop a quantitative knowledge of the evolution of the yield stress by aging time in both forward and reverse re-straining.
- Investigate the effect of aging temperature on the magnitude of increase of the yield stress after aging.
Chapter 3. Scope and Objectives

- Study the influence of the amount of pre-strain on the yield stress of the material after static strain aging.
- Examine the effect of static strain aging on the stress-strain response of the material at relatively large strains in both forward and reverse direction.
Chapter 4

**EXPERIMENTAL METHODOLOGY**

This chapter describes the key experimental techniques used in this study. First, the material examined as a part of this thesis is presented. Next, sample design, mechanical testing and aging procedures are discussed.

4.1 Material

In this study, a hot rolled steel grade JIS SN490 (see chemical composition in Table 4.1) was used. This steel contains 1.53 wt% Mn and 0.26% wt% Si compared to 0.3-0.6 wt% Mn found in standard 1020 alloys. The JIS SN490 steel was received from JFE Steel Corporation in the form of plates with dimensions of 250x250x40mm.

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Al</th>
<th>Nb</th>
<th>Ti</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.16</td>
<td>0.26</td>
<td>1.53</td>
<td>0.016</td>
<td>0.002</td>
<td>0.025</td>
<td>0.009</td>
<td>0.001</td>
<td>0.0046</td>
</tr>
</tbody>
</table>

Table 4.1: Chemical composition of the 1020-JIS SN490 material (wt%).
4.2 Metallography

The microstructure of the as-received plate was examined using optical microscopy. The sample preparation included grinding with silicon carbide and polishing with 6 µm diamond particles suspended. It was then etched with a 2% nital solution for 10 seconds and examined with the Nikon EPIPHOT 300 series inverted optical microscope. Figure 4.1 shows the microstructure of this steel within the cross-section of the as-received plates. The figure shows that ferrite (blue) and pearlite (black) bands are formed in the rolling direction. To measure the average diameter of ferrite grains, micrograph was taken at a magnification that provides sufficient number of grains. Approximately 700 grains were examined. Grain boundaries were then highlighted manually and analyzed with ImageJ software version 1.45. The results of the measurements showed an equivalent average diameter (EQAD) of ≈30 µm for ferrite grains.

Figure 4.1: Microstructure of the as-received steel plate. RD is the rolling direction, TD is the transverse direction, and ND is the normal direction.
Chapter 4. Experimental Methodology

To check the homogeneity of hardness across the thickness of the as-received plates, Vickers microhardness measurements were made across their thickness. For sample preparation, a piece of plate was cut and polished to a mirror-like finish using silicon carbide grinding papers and 6 µm diamond particles suspended. A load of 500 g was applied for 10 s and measurements were taken at intervals of 0.5 mm. Figure 4.2 is a plot of microhardness as a function of distance from the surface. The average hardness along the thickness is 151 ± 7 HV.

Figure 4.2: Measured microhardness as a function of distance from the surface.
4.3 Torsion testing

4.3.1 Sample dimensions and mechanical testing

Torsion testing was done on two different hollow samples as shown in Figure 4.3. Since the imposed strain in a torsion experiment varies across the wall thickness [9], samples were designed with two different inner diameters, but the same wall thickness, to study the effect of the strain variation on the yield response of the material.

The starting geometry was taken from a previous PhD study [57], where the torsion samples dimensions were scaled from the specimens of Lindholm et al. [58] as shown in Figure 4.3a. In order to find the shear strain variation along the wall thickness in the elastic region, shear strain at distance $R$ of the center of the tubular sample can be measured using: $\gamma = \frac{R\Phi}{l}$ [59], where $\Phi$ is the angle of twist and $l$ is the gauge length.

Therefore, the shear strain ratio of the outer and inner surfaces can be calculated by: $\frac{\gamma_2}{\gamma_1} = \frac{R_2}{R_1}$, where $R_2$ and $R_1$ are the outer and inner diameters of the sample, respectively. Using this equation, it was found that the shear strain imposed on the outer surface is ~17% higher than the inner surface of this sample in elastic portion of deformation. However, by increasing the inner diameter of the sample while keeping the wall thickness constant, this variation lowers and reaches ~11% in the samples with inner diameter of 12.7 mm. Therefore, a few tests were conducted on larger samples with dimensions given in Figure 4.3b to examine this effect.
The samples were mechanically tested at room temperature in an Instron 8874 servohydraulic tension/torsion machine equipped with Wavematrix control software version 1.3. Grips with a collet system were used to enhance the alignment between the top and bottom grips. The alignment was checked with a dial gauge prior to testing. For this purpose, the sample was mounted on the upper grip (rotating grip) and during a rotation of 30 degrees of the top grip, no more than 0.05 mm of axial displacement was recorded. Considering the total length of the grip and sample as 240 mm, this amount of axial displacement gives no more than 0.07 degree axial deviation. The tests were run at an angular velocity of $0.05^\circ$/s. Using $\dot{\varepsilon}_{eq} = \frac{R_{out}}{\sqrt{3l}} \phi$, a theoretical equivalent strain rate of $1.3 \times 10^{-3}$ s$^{-1}$ and $2.2 \times 10^{-3}$ s$^{-1}$ for the outer surface of the samples having an outer
diameter of 8.3 mm and 13.9 mm can be calculated, respectively. In this equation, \( R_{\text{out}} \) is the outer diameter, and \( \Phi \) is the angular velocity. The test method was designed to keep the axial load as zero during the test in order to simulate the condition of a free end torsion test.

The torque was recorded during the test and shear stress was calculated [9]:

\[
\tau_m = \frac{MR}{\frac{\pi}{2}(R_o^4 - R_i^4)} \tag{4.1}
\]

where \( M \) is the measured torque and \( R_o \) and \( R_i \) are the outer and inner radii, respectively.

### 4.3.2 Strain measurement

The accurate measurement of the strain during the torsion test was a challenge. Due to the finite stiffness of the load frame, it was impossible to calculate the strain base on angular rotation. Sample geometry made further difficulty in measuring the strain since the gauge length was small compared to the fillet radius of the specimen, which resulted in inaccurate measurement of the gauge length of the samples.

Therefore, it was decided to use a non-contact measurement technique involving digital image correlation (DIC). The method by Badinier [57] was followed with slight modifications in the procedure. A LaVision DIC system was employed to measure strains on the surface of the samples. The LaVision Davis8 software was coupled to two synchronized Lavision Imager QE digital cameras as shown in Figure 4.4. The cameras were mounted horizontally on a single aluminum rail and brought close enough to the sample surface such that the specimen filled entirely the field of view of both cameras. The spread angle between cameras was maintained at 50 degrees, comfortably exceeding Lavision recommendation of not less than 30 degrees. Lighting was provided by two
LED lamps mounted on the same rail providing equal and uniform lightening. The DIC system was coupled to the torsion load frame by two BNC-BNC cables so as to allow for the synchronization of image acquisition with the imposed torque and angular torsion. In this case the image acquisition was triggered by the load frame and the level of torque at the point of image acquisition imprinted onto each image. An image acquisition frequency of 2 Hz was found to provide a good compromise between time for post processing and strain resolution. LaVision’s DaVis8 software package was used for both capturing as well as processing of the images.

![Camera configuration for the DIC setup. The spread angle between the two cameras is maintained at 50 degrees in order to capture radial dilation and curvature of the sample.](image)

The calculation of strain via DIC involves the quantitative comparison of two images taken before (reference image) and after deformation [60, 61]. These images need to have a quasi-random surface pattern with distinct features that can be easily identified. As shown in Figure 4.5a, a rectangle is defined around the desired area of the reference
image, which usually falls within the gauge section of the sample. Afterward, these images are divided into a regular grid on a pre-defined rectangle. The grid intersections are the locations where the displacement and strains are eventually evaluated. This subset of the reference image is then quantitatively compared to boxes of the same size in the deformed image to find the correlated boxes. This correlation is based on the unique surface pattern of each box. The displacement in each grid point is then calculated by measuring the displacement of the centroid of correlated boxes in the reference and deformed image leading to a spatial map of displacement over the sample surface. From this displacement map the strains can be calculated as the gradients of the displacement field [60].

![Figure 4.5: Method used by Davis8 software to calculate the strains from a speckle pattern, a) before deformation, and b) after deformation.](image)

Since the surface of the sample is curved, the binocular stereoimage technique has been used to obtain displacements on the surface of the sample in three-dimensional space [61]. In order to calibrate the cameras, first the cameras were manually focused on the surface of the sample and then the sample was removed and a calibration target was placed in the same position as the sample, and five pairs of images were captured by cameras while the calibration target was tilted and rotated slightly between each capture following the DaVis instruction. The calibration target was featuring a regular array of...
dots where the diameter of each dot was 0.25 mm and the distance between centers of
dots was 1 mm. It is important to note that even in three-dimensional stereo imaging DIC,
displacements and thus strains are measured only on the surface of the object.

Several parameters may influence the practical resolution of the DIC technique,
including but not limited to the sample surface quality, the initial calibration, lighting and
portion of the field of view filled with interrogation region [60]. Therefore, the practical
resolution is expected to vary from experiment to experiment although the resolution of
this technique may be sub-pixel theoretically. Bearing this in mind, one of the concerns
was to keep the variables of the experiments as constant as possible.

Sample preparation for DIC consisted of applying a matte white layer of enamel
spray paint to the sample and then applying a black speckle pattern using airbrush. This
provided a randomized fine speckle pattern for image analysis. In order to choose the grid
size for displacement measurements, first the displacement field was measured with a
relatively large grid size and, then it was repeated with a reduced grid size several times
until the displacement field within the gauge remained constant regardless of reduction in
grid size. The grid size was selected as ~10 to 20 grids across the gauge section as it was
found to give a good compromise in measurements and processing time [57].

Figure 4.6a illustrates the calculated shear strain over the interrogation surface. As
shown in this figure, the amount of shear strain in the middle of the sample (gauge
section) is the highest and the most uniform. Moving away from the center, as one comes
close to the shoulder of the sample where the thickness increases, the shear strain
decreases. In order to define the gauge length of the sample, the strains were measured
along a vertical line across the gauge section and plotted as a function of their position
during the experiment. As indicated in Figure 4.6b, the shear strain is most uniform in the central section of the sample during the test, which is defined as the gauge section and the distance where the strain is uniform would be the gauge length.

![Image of calculated shear strain](image)

**Figure 4.6:** Calculated shear strain over, a) interrogation surface of the sample, and b) a vertical line crossing the gauge section.
Finally the average shear strain was measured over the defined gauge length. The experimental shear stress-shear strain values were converted to von Mises equivalents using equations 4.2 and 4.3.

\[ \sigma_{eq} = \sqrt{3} \tau \]  

\[ \varepsilon_{eq} = \frac{\gamma}{\sqrt{3}} \]

where \( \sigma_{eq} \) and \( \varepsilon_{eq} \) are the von Mises equivalent stress and strain, respectively, and \( \tau \) and \( \gamma \) are the shear stress and engineering shear strain, respectively.

To check the accuracy of the DIC technique, the shear modulus in the elastic portion of stress strain curve was calculated. Figure 4.7 shows the resulting elastic curve of a sample where it was determined that the elastic response is linear (\( R^2 = 0.999 \)) having a slope of \( \mu = 79 \text{ GPa} \). This value was in the range of 74-81 GPa for the rest of experiments, which compares well against the expected shear modulus of iron, \( \mu = 78 \text{ GPa} \) [62].

![Figure 4.7: Elastic portion of a typical stress strain curve measured on steel grade JIS SN490. The strain was measured using the DIC set-up. The equation of the line was determined using a least squares fit in Microsoft Excel.](image)
4.4 Aging procedure

Pre-strained samples were subsequently aged at temperatures between 100°C to 200°C for 15-600 minutes. Aging was done in a stirred oil bath containing silicon oil, suitable for temperature range of 25 to 250°C. The temperature in the oil bath was controlled by a built in type K thermocouple. The samples were immersed in the oil bath 30 minutes after the bath reached the desired temperature. An extra thermocouple was used to check the temperature homogeneity. The variation of the temperature in the bath was no more than one degree Celsius compared to the set temperature. After completion of the aging, the samples were quenched in water at room temperature.
Chapter 5

RESULTS

This chapter summarizes the experimental results of this study. First, the results of the initial loading on samples having different dimensions are presented. Next, the strain maps calculated by DIC are presented. Subsequently, the method used to measure the yield stress upon loading is discussed and reproducibility of the measurements is reported. Finally, the results of aging at various conditions (aging time and temperature) and the influence of strain path are presented.

5.1 Effect of sample geometry

As described in Chapter 4, initially two sample geometries were examined for torsion tests. The stress-strain curves related to initial loading of both geometries are shown in Figure 5.1. As this figure shows, increasing the diameter does not make a detectable change in the yield respond of the stress-strain curve. Thus, in this thesis, experiments carried out only on samples with smaller radii ($r_{\text{inner}}=7.1\text{mm}$), which were previously designed by Badinier [57]. The lack of detectable change in yield behavior, and variation in yield stress even in samples having larger diameter seems to be due to the formed residual stresses during machining of samples. This effect will be further discussed in Section 5.3.1.
Chapter 5. Results

5.2 Basics of strain measurement using DIC

Figure 5.2 shows the strain map calculated by DIC on the surface of the samples after various amounts of pre-strain. As described in Chapter 4, the gauge section after each test is defined and the average of the shear strain is measured in it. The variation of the shear strain, $\varepsilon_{xy}$, in the gauge section of the samples is no more than $1 \times 10^{-3}$ in the sample strained to $8 \times 10^{-2}$ and reaches approximately $1 \times 10^{-2}$ in samples strained to 0.1.
Figure 5.2: Shear strain distribution map in the gauge section of the samples strained initially to a) ~0.8%, b) 5%, and c) 10% equivalent strain.
5.3 Yield stress measurement

Finding the yield stress in torsion tests was a challenge, especially for the samples loaded in the reverse direction after strain aging since the stress strain curve did not exhibit a sharp yield point. Figure 5.3 shows an example of the samples re-strained after aging, in either forward or reverse direction. As in all other graphs in this thesis, positive and negative stresses and strains are plotted in the same direction for easy comparison. This figure shows that the uncertainty in determining the yield stress of the samples strained in the same direction as that of the before aging is due to the absence of an upper yield point and slight increase of stress in the Lüders plateau. This effect could be understood by the variation of stress and strain distribution through the wall thickness of the sample [9]. Based on variation of stress through the thickness, the elements in the outer surface yield first and yielding continues to the inner elements [63]. While yielding progresses from the outer to the inner surface, the elements in the outer surface start to work harden. Therefore, the sharp upper yield point is absent in the stress-strain curve and a gradual increase in the Lüders plateau is visible.
5.3.1 Yield stress for loading in forward direction

In order to characterize the yield stress for reloading in forward direction, lines with the slope of the elastic portion of the stress-strain curve and offsets of 0.2-2%, were constructed and then the intersection of these lines and the stress-strain curve was measured (Figure 5.4a). The measured offset yield stresses were then plotted versus strain offsets as illustrated in Figure 5.4b. In this figure, the measured offset yield stresses of three samples reloaded in forward direction after pre-strain of 5% and aging at 100°C for 600 minutes are plotted. The offsets in these measurements were chosen to be relatively large due to the slight reduction of stress after yielding and the presence of stress plateau.
Figure 5.4: An example of (a) lines with various offsets crossed a reloading curve, and (b) the measured yield stresses base on various offsets in three samples reloaded in forward direction after pre-strain of 5% and aging at 100°C for 600 minutes.

As illustrated in Figure 5.4a, the line with 0.5% offset has passed the decline of stress after yielding and the curves in Figure 5.4b show a minimum in this offset. The same trend (minimum stress at 0.5% offset) was also observed in other samples reloaded
in forward direction. Thus, 0.5% offset was used as a general offset in this study to measure the yield stress of the samples reloaded in the forward direction.

As will be discussed in Section 5.3.1, a variation of the initial yield response was found for the as-received material. Therefore, in order to be consistent in the measurements, 0.2% offset was used as a generally accepted offset [9] for initial yield stress measurements as shown in Figure 5.5.

Figure 5.5: a) Loading curves of the as-received steel and 0.2% offset line. b) The magnified section shows the cross sections of the 0.2% offset line and the stress-strain curves in yield region.
5.3.2 Yield stress in reloading in reverse direction

The measurement of the yield stress in samples loaded in reverse direction is even more challenging due to a large Bauschinger effect and the lack of sharp yield point. The 0.2% offset is widely accepted to be used as a standard offset in this case [9]. However, there is no reason for this offset to give the best result. Using lower amounts of offset may be useful, although, higher uncertainty in the measurements will be the penalty.

Bearing this in mind, in order to find an appropriate offset to be used in reloading in reverse direction, the difference between the flow stresses of three samples reloaded in reverse direction after 5% equivalent pre-strain and aging, and the flow stress of an as-received sample loaded in the forward direction (Δσ) was plotted as a function of equivalent strain in Figure 5.6b.

The gradual decrease of the Δσ with increasing amounts of strain shows that it is insufficient to rely on a single offset value. Thus, in this thesis the yield stresses were measured with different offsets.
5.4 Reproducibility of monotonic torsion test

5.4.1 Initial straining

Figure 5.7 shows the von Mises stress strain curves of the samples initially deformed up to the strains of approximately 0.8, 5, and 10% equivalent pre-strains before aging.
Figure 5.7: Stress-strain curves of samples pre-deformed to a, b) ~0.8%, c, d) 5%, and e, f) 10% equivalent strain. b, d, and f) are the magnified section of the stress-strain curves in the yield region.
As shown in Figures 5.5 and 5.7, the measured yield stresses in the as-received samples indicate an average yield stress of 367 MPa and stress range of 30 MPa. The range of the flow stress at ~0.8% equivalent strain is 22 MPa and it decreases to 14 MPa in 10% equivalent strain. Figure 5.8a shows the histogram of the yield stresses for initial loading. Figure 5.8b, 5.8c, and 5.8d show the histograms for the results of the flow stresses measured at ~0.8%, 5%, and 10% equivalent strain, respectively.
The variation in the yield response is shown in Figure 5.7b, 5.7d, 5.7f, and 5.8a. The following factors may result in this variation:

1. Microstructural inhomogeneity.
2. Possible pre-loading of the sample during loading into the grips.
3. Possible deformation of the sample during machining.

Since no significant microstructural inhomogeneity was observed along the thickness of the as received plates (Figure 4.1) and microhardness measurements showed standard deviation of no more than 7 HV along the thickness of the as-received material (Figure 4.2), microstructural inhomogeneity seems to have a minor effect on the results.

In order to avoid pre-loading of the samples during mounting into the grips, the “specimen protect” was used, in which the machine compensated a maximum load of 0.2 KN, which corresponds to a stress of 13 MPa during mounting on the grips. Therefore, this factor also seems to have a negligible effect on the yield response of the material since this stress is less than 3% of the observed yield stress value (~360 MPa).

However, deformation of the sample during machining may have influenced the results since samples were not heat treated after machining to abolish the residual stresses formed during the process and these residual stresses may cause variation in the yield stress of the samples. As future work, the effect of residual stresses could be examined by conducting an annealing treatment of the material prior to and after machining.
5.4.2 Reloading in forward direction after strain aging

The reproducibility of the results in reloading in forward direction after strain aging was assessed by measuring the yield stress in reloading curves of four samples that were 5% pre-strained and aged at 100°C for 10 hours. The resulted stress-strain curves are shown in Figure 5.9. The extracted results, shown in Table 5.1, indicate a variation range of 6 MPa in the measured yield stresses.

![Figure 5.9: Stress-strain curves of four samples reloaded in forward direction, following 5% pre-strain and aging at 100°C for 10 hours.](image)

<table>
<thead>
<tr>
<th>Sample</th>
<th>YS 0.5% (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>589</td>
</tr>
<tr>
<td>2</td>
<td>585</td>
</tr>
<tr>
<td>3</td>
<td>587</td>
</tr>
<tr>
<td>4</td>
<td>583</td>
</tr>
<tr>
<td>Average</td>
<td>586</td>
</tr>
<tr>
<td>Range</td>
<td>±3</td>
</tr>
</tbody>
</table>

Table 5.1: Measured offset yield stresses of four samples reloaded in forward direction following 5% pre-strain and aging at 100°C for 10 hours.
5.4.3 Reverse loading after strain aging

In order to establish the uncertainty in the results of the samples reloaded in reverse direction after strain aging, five samples were reloaded after 5% pre-strain and aging at 100°C for 10 hours. Figure 5.10 shows the obtained von Mises stress-strain curves. The extracted results are shown in Table 5.2 based on various offsets. As expected, the variation range decreases from 46 MPa, measured in 0.05% offset, to 14 MPa measured in 0.5% offset.

![Stress-strain curve related to five samples reloaded in reverse direction, following 5% pre-strain and aging at 100°C for 10 hours.](image)

Figure 5.10: Stress-strain curve related to five samples reloaded in reverse direction, following 5% pre-strain and aging at 100°C for 10 hours.
Chapter 5. Results

Table 5.2: Measured offset yield stresses of five samples reloaded in reverse direction, following 5% pre-strain and aging at 100°C for 10 hours.

<table>
<thead>
<tr>
<th>Sample</th>
<th>YS 0.05% (MPa)</th>
<th>YS 0.1% (MPa)</th>
<th>YS 0.2% (MPa)</th>
<th>YS 0.5% (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>275</td>
<td>340</td>
<td>374</td>
<td>420</td>
</tr>
<tr>
<td>2</td>
<td>315</td>
<td>335</td>
<td>373</td>
<td>413</td>
</tr>
<tr>
<td>3</td>
<td>274</td>
<td>322</td>
<td>358</td>
<td>415</td>
</tr>
<tr>
<td>4</td>
<td>275</td>
<td>314</td>
<td>358</td>
<td>415</td>
</tr>
<tr>
<td>5</td>
<td>269</td>
<td>305</td>
<td>348</td>
<td>406</td>
</tr>
<tr>
<td>Average</td>
<td>282</td>
<td>323</td>
<td>362</td>
<td>413</td>
</tr>
<tr>
<td>Range</td>
<td>±23</td>
<td>±17</td>
<td>±13</td>
<td>±7</td>
</tr>
</tbody>
</table>

5.5 Strain aging

Having established the reproducibility of the measurements for loading in both the forward and reverse directions, three different amounts of equivalent pre-strains were selected. The minimum amount of pre-strain was selected as ~0.8% equivalent strain since with this amount of pre-strain, the yield point elongation was completed and the sample entered the region of uniform deformation. The largest pre-strain was selected as 10% equivalent strain, in which the sample undergoes a substantial level of work hardening and its dislocation density significantly increases. Finally, the pre-strain of 5% was selected as an intermediate level to complete the study. The samples, after pre-straining to the amounts described above, were unloaded and aged at 100°C for 15-600 minutes. Subsequently, reloading was carried out in both forward and reverse direction.

A small number of samples were aged at higher temperatures to evaluate the influence of aging temperature. In this case, samples were pre-strained to 5% equivalent strain prior to aging. Aging temperatures were chosen as 120, 150, and 200°C and the
aging time of 600 minutes was selected for all the above aging temperatures. Afterward, reloading was carried out in the reverse direction. In order to assess the influence of aging temperature on reloading in the forward direction, one sample was aged at 120°C for 600 minutes.

5.5.1 Yield behavior in forward straining after aging

Figure 5.11 shows the stress-strain curves of the samples reloaded in forward direction after pre-strain of 0.8, 5 and 10%, and aging at 100°C for 15-600 minutes. The notable features of these results are i) the increment of the yield stress and ii) the appearance of distinguishable yield point with increasing aging time. A comparison of the stress-strain curves shows that the reloading curves converge for different aging conditions in the case of 0.8% equivalent pre-strain, however, in the samples with 5% and 10% pre-strain, a permanent offset is observed in the stress-strain curves (at least over this range of equivalent strains).

Figure 5.12a shows $\Delta YS_{\text{Fwd}}$ (the increase of yield stress in forward re-straining) increases with aging time and reaches a maximum amount of $\sim 46$ MPa, after approximately 180 minutes. There appears to be essentially no change in $\Delta YS_{\text{Fwd}}$ for different pre-strain levels. Results of longer aging times, shown in Figure 5.12b, indicate that further aging times has minor effect (if any) on the yield stress.

The effect of aging temperature in forward re-straining after aging was evaluated by comparing the results of samples aged for 600 minutes at 100 and 120°C after 5% pre-strain. As shown in Figure 5.13, the yield stress was not influenced by increasing aging temperature.
Figure 5.11: Von Mises stress-strain curves of the samples pre-strained to a, b) -0.8%, c, d) 5%, and e, f) 10% equivalent strain, and re-strained in the same direction as the pre-strain after aging at 100°C for 15-600 minutes. The plotted magnified section (b, d, and f) of the yield region on the right, gives a close vision of this region. Open circles represent aging treatment upon unloading.
Figure 5.12: The effect of aging after various equivalent strains; tests were made in the same direction as the pre-strain. Aging times of a) 0-180 minutes, and b) 180-600 minutes.

Figure 5.13: a) The effect of increasing aging temperature on the Von Mises stress-strain curves of the samples 5% pre-strained and re-strained in the forward direction after aging for 600 minutes. The plot of the yield region on the right (b), gives an expanded view of this region. The open circle represents aging treatment upon unloading.
The measured $\Delta YS_{\text{Fwd}}$ for both aging temperature is 46 MPa. This result suggests that the increase of the yield stress upon reloading in forward direction after aging at 100°C for 600 minutes has reached its maximum and a further increase in aging temperature will not change the obtained value, at least in the range of 100-120°C. Although the case of aging at 120°C has been carried out once and further experiments are required to confirm this result.

5.5.2 Yield behavior in strain reversal after aging

The influence of aging time on the Bauschinger effect in strain reversal is illustrated in Figure 5.14 for samples pre-deformed to equivalent strains of 0.8%, 5%, and 10%, and aged at 100°C for 15-600 minutes. These graphs show a large Bauschinger effect in the samples reloaded immediately in the reverse direction after unloading. By increasing the aging time, the offset yield stress increases, however, this amount never reaches the flow stress values just before unloading. A permanent increase in the flow stress is observed although the slopes of the stress-strain curves are not equivalent at the end of these strain ranges and there is the possibility that the curves converge at larger strains.
Chapter 5. Results

Figure 5.14: Von Mises stress-strain curves of samples pre-strained to a, b)~0.8%, c, d) 5%, and e, f) 10% equivalent strain, and re-strained in the reverse direction after aging at 100°C for 15-600 minutes. The plot of the yield region on the right (b, d, and f), gives an expanded view of this region. Open circles represent aging treatment upon unloading.
To calculate the increment of the offset yield stress in reverse loading after strain aging, $\Delta YS_{Rev}$ was measured as the difference between the offset yield stress of the samples reloaded after aging and the sample reloaded without aging (Figure 5.15). Figure 5.16 shows the results of $\Delta YS_{Rev}$ with offsets of 0.5%, 0.1%, 0.2%, and 0.5% plotted as a function of aging time. Close inspection of these graphs shows a rapid increase in $\Delta YS_{Rev}$ during early stages of aging; however, the rate of change after the first 15 minutes starts to decrease until 180 minutes of aging, where the yield stress reaches to its maximum and almost does not change with increasing the aging time.

Figure 5.15: Defined $\Delta YS_{Rev}$ as the difference between the offset yield stress of a sample reloaded in reverse direction after aging and that of the sample reloaded in the reverse direction without aging.
In comparison to $\Delta YS_{Fwd}$, the maximum $\Delta YS_{Rev}$ has a value of $\sim$90-210 MPa depending on the offset, which is 2 to 4.5 times larger than the maximum $\Delta YS_{Fwd}$. Additionally, unlike the case of reloading in forward direction, the $\Delta YS_{Rev}$ in strain reversal after strain aging increases with the amount of pre-strain. Considering 0.05% offset yield stress, inspection of the results in Figure 5.16a shows that the increment in the yield stress of samples 10% pre-strained is 65 MPa higher than that of the samples $\sim$0.8% pre-strained. This amount reduces to 25 MPa when applying 0.5% offset.
The effect of aging temperature is shown in Figure 5.17, where samples with 5% equivalent pre-strain were aged at 100, 120, 150, and 200°C for 600 minutes. This figure shows that the yield stress and the flow stress increase by increasing aging temperature. The magnified view in Figure 5.17 shows evidence of a yield point for the sample aged at 200°C, however, this test was performed only once and further experiments are required to confirm this result. Figure 5.18 shows that the measured offset yield stresses increase with increasing aging temperature.

Figure 5.17: a) The effect of increasing aging temperature on the Von Mises stress-strain curves of the samples 5% pre-strained and re-strained in the reverse direction after aging for 600 minutes. b) The plotted magnified section of the yield region gives a closer vision. Open circle represents aging treatment upon unloading.
Figure 5.18: The effect of aging temperature on measured $\Delta YS_{Rev}$ based on various offsets in the samples reloaded in reverse direction after 5% equivalent pre-strain and aged for 600 minutes.
Chapter 6

DISCUSSION

This chapter presents a discussion from the current work on the effect of aging time, aging temperature, and the amount of pre-strain on the stress-strain response of the material for cases of deformation in both the forward and reverse directions. The role of aging on the increase of the yield stress, and permanent strengthening after aging are discussed based on possible mechanisms.

6.1 Increase of the yield stress in forward re-straining after aging

The results from the stress-strain curves of the samples reloaded in the same direction as the original pre-strain after aging (Figure 5.11) confirm that the steel underwent significant strain aging. An increase of 42MPa was observed in the samples aged for 180 minutes at 100°C and longer aging times did not result in a further significant increase (i.e. $\Delta YS_{Fwd}$ is equal to 46 MPa for the samples aged for 600 minutes). Reaching a plateau in $\Delta YS_{Fwd}$ after certain amount of aging time would be consistent with the completion of the Cottrell atmosphere formation [1, 46]. Considering that the average grain size in the studied steel was 30 µm, this increment in the yield stress compares well with literature, i.e. the increase of 48MPa has been reported by Wilson and Russell for a rimming steel containing 0.038% carbon with grain size of ~26 µm [17]. An important feature of the current results and those in the literature [64] is that the maximum increase of the yield stress, as well as the rate of increase during aging, is independent of the
amount of pre-strain. Therefore, it seems that to first order, the dislocation density has minimal influence on the maximum increase of the yield stress and the formation of the Cottrell atmospheres.

The results of forward re-straining as well as initial loading do not have an observable upper yield point. The absence of an upper yield point could have a number of origins including i) the low frequency of data acquisition (2Hz), ii) progressive yielding of the sample from the outer to the inner surface in the wall of the torsion sample, or iii) heterogeneity in the microstructure, i.e. the presence of pearlite colonies. A suggestion for further work would be to increase the data acquisition rate on the torque measurements to eliminate this possible explanation.

### 6.2 Permanent strengthening in forward re-straining

The effect of aging time on the question of permanent hardening during restraining after aging at 100 °C is shown in Figure 6.1 for the samples with ~0.8%, 5%, and 10% pre-strain. In these graphs, $\Delta \sigma_{\text{forward}}$ (the difference between the flow stresses of the aged sample and as-received sample at the same total equivalent strain), is plotted as a function of equivalent strain (see Section 5.2.2 for more details). With the exception to the case of ~0.8% pre-strain, where the difference between $\Delta \sigma_{\text{forward}}$ of the samples aged for 15 minutes and 600 minutes decreases as a function of the applied strain, the results in other cases remain constant with the difference of ~26 MPa and ~22 MPa for the 5% and 10% pre-strained samples, respectively. This implies that aging time has not significantly influenced the work hardening rate of these samples, at least in this range of strains, and only caused a shift in the flow stress.
Figure 6.1: Plotted $\Delta \sigma_{\text{forward}}$ as a function of equivalent strain related to the samples a) ~0.8%, b) 5%, and c) 10% pre-strained and re-strained in the same direction as the original pre-strain after aging at 100°C for 15 and 600 minutes.
In order to examine the effect of pre-strain on permanent strengthening, Figure 6.2 plots the results of samples with different pre-strains but the same aging time for the case of 15 and 600 minutes at 100°C. The important feature of this figure is that, regardless of the amount of pre-strain, samples aged for 15 minutes do not show significant permanent strengthening effect. However, the samples aged for 600 minutes (Figure 6.2b) appear to show a permanent strengthening. The increase in the flow stress after prolonged aging time cannot be rationalized with the original theory of Cottrell for strain aging, where dislocations are collectively unpinned upon yielding since in this case, the flow stress has to return to the monotonic loading curve once the yield point elongation is passed. Therefore, another mechanism is proposed, where dislocations remain locked during re-straining after sufficient aging times. In this respect, other sources of dislocation like grain boundaries activate [16, 17] or few unlocked dislocation segments start to multiply [8]. Figure 6.2b supports this hypothesis that increasing the amount of pre-strain, and therefore, dislocation density resulted in higher level of permanent strengthening. In this hypothesis, it is proposed that the locked dislocations provide stronger obstacles for mobile dislocations compared to unlocked forest dislocations. However, this mechanism may not be applicable for shorter aging times. For example, 15 minutes of aging did not result in meaningful increase in the flow stress. In this case, dislocations might not be fully locked and they are able to escape from their atmospheres upon yielding [16]. Finally, the observation that work hardening rate is similar for aged and as-received material, implies that either precipitation is not happening during this period of aging or the sizes of the formed precipitates are in the order that are penetrable, i.e. their existence did not change the work hardening behavior [9].
Figure 6.2: Plotted $\Delta\sigma_{\text{forward}}$ as a function of equivalent strain related to the samples pre-strained up to 10% and re-strained in the same direction as the original pre-strain after aging at 100ºC for a) 15 and b) 600 minutes.
6.3 Interaction between the Bauschinger effect and strain aging

The yield stress of the samples re-strained immediately in the opposite direction after pre-strains of up to 10% is plotted in Figure 6.3 as a function of the offset used to define yielding in the reverse direction. These data show that the yield stress measured based on 0.05% and 0.1% offset do not change with increasing the amount of pre-strain. However, the results of 0.2% and 0.5% offset yield stresses show an effect of pre-strain and this difference increases for offset strain of 0.5% compared to 0.2%.

![Figure 6.3](image)

**Figure 6.3:** Plotted yield stresses of the samples re-strained immediately in the reverse direction after pre-strain of ~0.8%, 5%, and 10% as a function of the applied offset.

As noted earlier in Chapter 2, different methods proposed to quantify the Bauschinger effect. In this study the Bauschinger effect factor (BEF) was selected to evaluate the Bauschinger effect in strain reversal where,

\[
BEF = \frac{\text{offset stress in reverse direction}}{\text{maximum flow stress in forward pre-strain}}
\]
Figure 6.4 shows the results of the measured BEF in the current study along with the extracted results of previous studies on an IF steel and AISI 1020 steel as a function of the applied offset. This figure shows that the BEF reduces with increasing the amount of pre-strain and reaches to 0.45 (taking 0.2% offset) for the sample 10% pre-strained. The results of 0.2% offset show good agreement with the work of Li et. al. [22], where the BEF of 0.55 was reported for the tensile sample prepared from AISI 1020 steel and compressed after 2% tensile strain. The work by Bouaziz et al. [26] on IF steel shows higher BEF in this steel than what was observed in the current study. The steel studied by Bouaziz differs in two important manners from the current steel, i) it is a single phase and ii) there is no interstitial C or N. The large Bauschinger effect observed in the current study might be due to the existence of a hard second phase, Fe₃C in the pearlite.

![Figure 6.4: The plot of measured BEF in the samples re-strained immediately in the reverse direction after pre-strain of ~0.8%, 5%, and 10% as a function of the applied offset. The extracted data from the study of Bouaziz et al. [26] on IF steel with grain size of 12 µm and 3.5 µm, after ~12% equivalent pre-strain, and Li et al. [22] on AISI 1020 steel containing 0.21% carbon with grain size of 12 µm after 2% pre-strain are also plotted as a function of the applied offset.](image-url)
Although the yielding was found to occur at very low stresses for the samples which were re-strained in the opposite direction (Figure 5.14 and 5.16), the yield stress increased when aging at 100°C. The 0.2% offset yield stress of the samples 10% and ~0.8% pre-strained and aged for 180 minutes reached 394 MPa and 323 MPa, respectively, which is close to the yield stress of the as-received material (367±15 MPa). Nonetheless, the yield stress of the aged samples did not reach the flow stress at the end of the pre-strain even after prolonged aging times. This observation is again inconsistent with Cottrell’s theory of strain aging since the stress required to unlock dislocations (~6 GPa) is anticipated to be larger than the directional internal stresses developed during pre-straining [46], therefore based on Cottrell’s theory, after sufficient aging time, where all dislocations are fully locked, it is expected that the Bauschinger effect is eliminated and the sharp yield point returns. Current work shows that strain aging was unsuccessful to completely eliminate the Bauschinger effect but it has reduced its magnitude. Therefore, mechanisms other than unlocking dislocations probably take place upon reloading after aging. In this case, the continuous yielding in strain reversal may be attributed to multiple nucleation of the yield front, and not by propagation of Lüders bands through the gauge section [46]. Activation of sources of dislocation in multiple grains containing stress concentrators and a higher level of internal stresses may be responsible for continuous yielding even when all dislocations are fully locked. The existence of the pearlite bands in the microstructure increases the local stress concentrators, which later possibly with the aid of internal stresses results in initiation of yielding at many locations in the sample.

The interesting feature of the results shown in Figure 5.16 is that, unlike $\Delta YS_{Fwd}$, the $\Delta YS_{Rev}$ is increasing with increasing the amount of pre-strain. Figure 6.5, compares the results of
the measured $\Delta YS_{Fwd}$ and $\Delta YS_{Rev}$ for the samples 10% pre-strained and after aging at 100°C. This figure shows that the maximum increase of the yield stress in strain reversal after aging is $\sim 2.5$ times larger than that in forward re-straining. Considering the aging time and temperature range, the effect of the dislocation rearrangement during aging on the extra increase of the yield stress is unlikely. At present, there is no simple explanation for these results, thus, further investigations need to be conducted to examine this effect.

![Figure 6.5: The measured $\Delta YS_{Fwd}$ and $\Delta YS_{Rev}$ (0.2% offset) after 10% pre-strain and aging at 100°C for up to 600 minutes.](image)

The results of forward re-straining after 10% pre-strain and aging are shown as $Ln (-Ln(1 - x))$ versus $Ln (t)$, where $x$ is the normalized value of $\Delta YS_{Fwd}$. This figure shows a slope of $2/3$, which is consistent with Equation 2.5 for the kinetics of static strain aging.
6.4 Effect of aging time and temperature on the flow stress after strain reversal

The effect of aging time on the measured $\Delta \sigma_{\text{reverse}}$ (the difference between the flow stresses in strain reversal after aging and monotonic loading at the same total equivalent strain) of the samples restrained in the reverse direction after aging at 100°C is presented in Figure 6.7. As shown in this figure, by increasing the aging time, the flow stress of these samples approaches the material which was loaded monotonically, i.e. the transient due to the strain path reversal was almost removed. Close inspection of the results shows that for samples with a pre-strain of ~0.8% (Figure 6.7a) after 600 minutes of aging at 100°C and 5% restraining, $\Delta \sigma_{\text{reverse}}$, reaches zero and remains at this level. In the case of 15 minutes aging at 100°C, there is no significant difference compared to the results for re-strain without aging (similar to forward re-straining after same aging condition). One can see a similar trend in Figure 6.7b and 6.7c for the samples 5% and 10% pre-strained. Although longer aging time has resulted in higher flow stress after

![Graph showing the relationship between aging time and flow stress](image-url)
yielding, the positive slope of the curves brings the possibility of convergence with the monotonic loading curve at larger strains.

In order to evaluate the effect of pre-strain on the flow stress of the samples re-strained in the reverse direction after aging at 100°C, the results for samples with different pre-strains, but the same aging time are plotted together in Figure 6.8. The strain in these graphs is measured from the beginning of the re-straining. As illustrated in this figure, increasing the amount of pre-strain from ~0.8% to 5% resulted in lower $\Delta \sigma_{\text{reverse}}$. However, such a decrease was not observed between the results of the samples 5% and 10% pre-strained. After 600 minutes of aging (Figure 6.8b), the flow stress of the sample ~0.8% pre-strained, reaches the flow stress of the as-received sample ($\Delta \sigma_{\text{reverse}} = 0$ MPa) at a strain of 0.05 but the samples 5% and 10% pre-strained even at re-strain of 0.25 are still 20 MPa below the flow stress of the as-received material at the same amount of total strain. Yet, the slope is positive and it is possible that $\Delta \sigma_{\text{reverse}}$ reaches zero at higher strains.
Figure 6.7: Plotted $\Delta \sigma_{\text{reverse}}$ as a function of equivalent strain related to the samples a) $\sim$0.8%, b) 5%, and c) 10% pre-strained and re-strained in the reverse direction after aging at 100°C for 15 and 600 minutes.
Figure 6.8: Plotted $\Delta \sigma^{\text{reverse}}$ as a function of equivalent strain. Samples restrained in the reverse direction after pre-strain of ~0.8%, 5%, and 10%. a) Without aging and b) aged at 100°C for 600 minutes before restraining.

The effect of aging temperature is shown in Figure 6.9, where the $\Delta \sigma^{\text{reverse}}$ of the samples 5% pre-strained and aged at 100°C, 150°C, and 200°C for 600 minutes is plotted as a function of equivalent strain. As illustrated in this figure, increasing the aging temperature, results in higher flow stress in strain reversal after aging, i.e. the $\Delta \sigma^{\text{reverse}}$ reaches -6 MPa after a total strain of 0.3 in the samples aged at 200°C. However, this amount is -20 MPa for the sample aged at 100°C.
Figure 6.9: Plotted $\Delta\sigma_{\text{reverse}}$ as a function of equivalent strain for the samples restrained in the reverse direction after pre-strain of 5% and aging at 100˚C, 150˚C, and 200˚C for 600 minutes before restraining.

6.5 Summary

A comprehensive set of experimental data on the yield stress and permanent strengthening after static strain aging with and without a strain path reversal has been reported. The results have been compared with proposed mechanisms in the literature. It is worth emphasizing that static strain aging is a very complex phenomenon and it is not always possible to draw definite conclusions on mechanisms, but the current results can be more easily rationalized in terms of permanent locking of dislocations and the operation of new sources of dislocations in restraining after aging. However, definitive proof of this hypothesis remains for future work.
Chapter 7

CONCLUSIONS

In this work, cases where static strain ageing was combined with strain path reversal were examined through a set of carefully designed experiments. The results showed that a significant increase in yield stress was found for cases where the plastic deformation was continued in the same direction after a thermal excursion. In contrast, the situation was much more complex when the strain path was reversed (i.e. Bauschinger tests). In this case, it was found that the static strain aging was unable to completely eliminate the Bauschinger effect and even prolonged aging time did not result in the return of the distinguished yield point and the samples yielded well below the maximum flow stress in forward pre-strain. Moreover, it was found that strain aging results in permanent strengthening in forward direction where a shift to higher stress levels without significant change in strain hardening was observed.

The key specific findings of this work are as follows:

- The amount and the rate of increase in the yield stress at forward re-straining after aging was independent of the amount of pre-strain.
- Re-straining in the reverse direction immediately after unloading caused a large Bauschinger effect and the samples yielded at significantly lower stresses than the as-received material. A comparison of the current results with the work of Bouaziz et al. [26] on IF steel shows that the Bauschinger effect observed in the current study is much larger than for the IF steel, probably due to the existence of a hard second phase, Fe₃C in the pearlite, in the current examined steel.
Chapter 7. Conclusion

- The absence of the distinguishable yield point in strain reversal implies that probably mechanisms other than unpinning (i.e. activation of sources of dislocation possibly at grain boundaries) are responsible for yielding upon re-straining after aging.
- The permanent strengthening in forward re-straining after aging reinforced the hypothesis of permanent dislocation locking during aging, where locked dislocations provide stronger obstacles for mobile dislocations compared to unlocked forest dislocations.
- In case of reverse straining after aging, an extended aging time resulted in increase of the flow stress and reduction of the strain hardening rate after yielding. Although, the flow stress after long duration aging was still lower than that of the as-received material, there is the possibility of convergence of the curves in larger strains due to the higher strain hardening rate in the aged samples.

In summary, based on this work, it is proposed that mechanisms other than dislocation unpinning are probably responsible for yielding after strain aging. A comprehensive set of experimental data were generated which can be used by designers working in this area, i.e. static strain aging, and strain path reversals in low carbon steels with ferrite-pearlite microstructure.

7.1 Future work

Based on the current study, the following is a list of suggestions for further investigation to give better explanation of the origin of static strain aging and its directionality:

- To examine the effect of grain size. This factor has been reported to influence the strain aging results. However there is a lack of knowledge about the source of this influence, i.e. whether it is due to the shorter distance which solute atoms should migrate at fine grains to reach dense dislocation walls at grain boundaries or due to the lower possibility of having stress concentrations at grains boundaries in this condition. Further investigation on this factor will help developing a better understanding for the source of directionality in strain aging systems.
To study the strain path dependency of the mechanical behavior. It seems necessary to examine strain paths other than fully reversed loading such as torsion-reverse torsion, e.g. conducting tension-internal pressure on tubular samples could be a good technique. This would provide further insight into the extent of directionality of strain aging.

Varying the carbon content in the steel to evaluate the influence of the volume fraction of pearlite and the interstitial carbon on the kinetics of strain aging and its magnitude.

Examine different steel microstructures such as bainite and martensite to provide insight into the detailed effects of local microstructure on yielding and strain ageing.

Finally, a model is needed to be developed to predict the mechanical behavior after strain aging in different conditions which can physically describe the experimental results from this study.
BIBLIOGRAPHY


49. C. Timms, D. Degeer, and M. McLamb, "Effects of a thermal coating process on X100


